

**DEVELOPMENT, MATERIAL AND STRUCTURAL
PERFORMANCE OF TENSION HARDENING
FIBER REINFORCED GEOPOLYMER
CONCRETE (THFRGC)**

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Abstract

On account of growing environmental and economic concerns, decarbonization of the concrete industry has become a priority with the development of environmentally friendly building materials to attract both research community and industry. A class of advanced eco-friendly building materials is geopolymer concretes. Their production incorporates industrial by-products in lieu of cement which has a double benefit in terms of sustainability: recycling industrial wastes instead of harmful disposal and reducing carbon footprint by eliminating cement. Meanwhile, Tension-Hardening Fiber Reinforced Concrete (THFRC) shows great potential as a structural material for modern infrastructure due its enhanced tensile strength and ductility. Although THFRC is considered a sustainable solution thanks to the tension-hardening delaying the need for retrofits, the high amount of Ordinary Portland Cement (OPC) used as a binder raises concerns regarding the sustainability of the material hindering the widespread application on account of the high cost and carbon footprint. This dissertation aims to advance the knowledge on sustainable and high-performance building materials by developing and characterizing a Tension-Hardening Fiber Reinforced Geopolymer Concrete (THFRGC) in terms of its material and structural behaviour. After a thorough review of the related literature, the experimental stage comprises the characterization of various mineral powders, mix design based on chemical and physical optimization, material identity characterization according to North American Standards prescribed for conventional THFRCs, determination of bond-slip law of reinforcing bar in THFRGC and performance under biaxial stress states. To promote the widespread use of the material, rheology, and fiber orientation in THFRCs are also explored using destructive and non-destructive techniques. Furthermore, this project aims to tackle the conundrum of tensile characterization of THFRC by developing a novel indirect tension technique that combines the simplicity of splitting test with the ability to obtain the whole response in pure tension. The proposed test is numerically validated using advanced Non-Linear Finite Element Analysis. The latter was also employed to investigate the validity of the tensile response obtained using the Inverse Analysis proposed by CSA, S6, Annex 8.1. Finally, self-sensing performance of a multifunctional nanoengineered THFRC is explored to pave the way of smart THFRCs in Structural Health Monitoring (SHM) applications.

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List of Abbreviations

AAFA	Alkali Activated Fly Ash
AAM	Alkali Activated Materials
AAR	Alkali Aggregate Reaction
AASHTO	American Association of State Highway and Transportation Officials
AC	Alternate Current
AC-IS	Alternate Current Impedance Spectroscopy
ACI	American Concrete Institute
ADA	Air-Detraining Admixture
AFNOR	Association Française de Normalisation
AS	Alkaline Solution
ASR	Alkali Silica Reaction
ASTM	American Society for Testing Materials
BFS	Blast Furnace Slag
CB	Carbon Black
CC	Conventional Concrete
CDP	Concrete Damage Plasticity
CEPRA	Connectionless Electrical Pulse Response Analysis
CF	Carbon Fibers
CFD	Computational Fluid Dynamics
CFL	Courant-Friedrich-Levy number
CFRP	Carbon Fiber Reinforced Polymer
CHBDC	Canadian Highway Bridge Design Code
CNF	Carbon Nanofiber

CNT	Carbon Nanotube
CSA	Canadian Standards Association
CSH	Calcium Silicate Hydrate
CT	Computed Tomography
CTE	Coefficient of Thermal Expansion
DB	Dry Binder
DC	Direct Current
DEWST	Double Edge Wedge Splitting Test
DIC	Digital Image Correlation
DPT	Double Punch Test
DSF	Densified Silica Fume
DSP	Densified Systems containing homogeneously arranged ultra-fine Particles
DT	Displacement Transducer
DTPT	Direct Tension Pullout Test
DTT	Direct Tension Test
ECC	Engineering Cementitious Composites
EDS	Energy-Dispersive X-ray Spectroscopy
FA-C	Fly Ash Type C
FA-F	Fly Ash Type F
FCR	Fractional Change of Resistance
FHWA	Federal Highway Administration
fib	International Federation for Structural Concrete
FP	Feldspar Powder
FRC	Fiber Reinforced Concrete
FS	Ferronickel Slag

FVM	Finite Volume Method
GGBFS	Ground Granulated Blast Furnace Slag
GNF	Graphene Nanofibers
GNP	Graphene Nanoplatelets
GPC	Geopolymer Concrete
GS	Granite Sand
GWP	Global Warming Potential
HPC	High Performance Concrete
HRWRA	High Range Water Reducing Admixture
ISSC	Intrinsic Self Sensing Concrete
JSCE	Japanese Society of Civil Engineers
LAC	Low Alkali Concrete
LBM	Lattice Boltzmann Method
LCA	Life Cycle Assessment
LOI	Loss on Ignition
LVDT	Linear Variable Differential Transformer
MCFT	Modified Compression Field Theory
MG	Metagabbro
MK-750	Metakaolin calcined at 750 °C
MR	Molar Ratio
MST	Modified Splitting Test
MTO	Ministry of Transportation Ontario
MWCNT	Multiwall Carbon Nanotubes
NLFEA	Non-Linear Finite Element Analysis
OPC	Ordinary Portland Cement

PCM	Phase Changing Materials
PCNT	Pristine Carbon Nanotubes
PDF	Probability Density Function
PFA	Pulverized Fly Ash
PM	Pumice
PP	Polypropylene
ppb	parts per billion
ppm	parts per million
PSD	Particle Size Distribution
PSS	Potassium Silicate Solution
PVA	Polyvinyl Alcohol
QEMSCAN	Quantitative Evaluation of Materials by Scanning Electron Microscopy
RA	Retarding Admixture
RCC	Roller Compacted Concrete
RCPT	Rapid Chloride Ion Penetration Test
RH	Relative Humidity
RHA	Rice Husk Ash
RHT	Riedel Hiermaier Thoma model
RILEM	International Union of Laboratories and Experts in Construction Materials, Systems and Structures
RM	Red Mud
RPC	Reactive Powder Concrete
rpm	rotations per minute
RSS	Residual Sum of Squares
RVE	Representative Volume Element

SCC	Self-Compacting Concrete
SCM	Supplementary Cementitious Materials
SD	Standard Deviation
SEM	Scanning Electron Microscopy
SHM	Structural Health Monitoring
SIA	Swiss Society of Engineers and Architects
SIFCON	Slurry Infiltrated Fibered Concrete
SPH	Smoothed Particle Hydrodynamics
SPT	Standard Pull-out Test
SSS	Sodium Silicate Solution
ST	Splitting Test
SWCNT	Single-wall Carbon Nanotubes
TA	Tartaric Acid
TEA	Triethanolamine
TEM	Transmission Electron Microscopy
THFRC	Tension Hardening Fiber Reinforced Concrete
THFRGC	Tension Hardening Fiber Reinforced Geopolymer Concrete
TRD	Trap Rock Dust
TSFRC	Tension Softening Fiber Reinforced Concrete
UHPC	Ultra-High-Performance Concrete
UHPFRC	Ultra-High-Performance Fiber Reinforced Concrete
USF	Undensified Silica Fume
UTM	Universal Testing Machine
VE	Virtual Extensometer
VEM	Variable Engagement Model

VMA	Viscosity Modifying Admixture
WB	Wet Binder
WR	Weight Ratio
WRA	Workability Retaining Admixture
WST	Wedge Splitting Test
XRD	X Ray Diffraction
XRF	X Ray Fluorescence

List of Symbols

$\mathbf{A}$	Orientation Tensor
A_b	Cross sectional area of reinforcing bar
A_f	Cross sectional area of fiber
b	Width
B	Constant from curve fitting -RHT model
C	Cover thickness
c	Cohesion
C_{ii}	Material stiffness
D_1	Shape parameter for damage factor -RHT Model
D_2	Shape parameter for damage factor -RHT Model
D_b	Diameter of reinforcing bar
$D_{dynamic}$	Diameter of dynamic spread
d_f	Diameter of fiber
d_{fo}	Nominal diameter of fiber
D_{max}	Maximum aggregate size
D_{min}	Minimum aggregate size
D_{static}	Diameter of static spread
E_c	Modulus of elasticity of concrete
E_s	Modulus of elasticity of steel
f_1	First peak strength per ASTM 1609
f	Stability timestep factor
f_b	Bond strength
F_b	Pullout force
$f_{b,ave}$	Average bond strength
$f_{b,res}$	Residual bond strength
f_c	Compressive strength
f_c^{ef}	Effective compressive strength
F_{cap}	Parabolic cap function
f_{cr}	Tensile cracking strength

f_{150}^D	Residual strength at displacement L/150 per ASTM 1609
f_{600}^D	Residual strength at displacement L/600 per ASTM 1609
$F_{elastic}$	Scaling factor
f_p	Peak strength per ASTM 1609
f_r	Modulus of rupture
$F_{rate}(\dot{\epsilon})$	Dynamic increase factor
f_t^{ef}	Effective tensile strength
f_{tu}	Ultimate tensile strength
$f_{y,s}$	Yield stress of steel
G	Shear modulus
$G_{elastic}$	Elastic shear modulus
GF	Gauge factor
G_f	Fracture energy
GL	Gauge length
$G_{residual}$	Residual shear modulus
h	Height
I	Current
I_1	First invariant of stress
J_2	Second invariant of stress
K	Consistency factor
k	Stiffness of average bond-slip law
L	Length
L_b	Embedment length
L_{base}	Length of base shrinkage
L_{ch}	Characteristic length
L_{comp}	Comparator length
l_f	Fiber length
l_{fo}	Nominal fiber length
$l_{p,max}$	Maximum plastified bond length
m	Mass
M_1	Constant from curve fitting - RHT model

n	Flow index
$n_{r,f}$	Real number of fibers crossing a plane
$n_{th,f}$	Theoretical number of fibers crossing a plane
P	Load
$P_{1,ASTM}$	First peak load per ASTM 1609
P_{actual}	PSD curve
P^D_{150}	Residual load at displacement L/150 per ASTM 1609
P^D_{600}	Residual load at displacement L/600 per ASTM 1609
P_p	Peak load per ASTM 1609
p_{spall}	Spalling pressure
P_{target}	Target PSD curve
q	Distribution modulus
q_{lat}	Lateral confining pressure
R	Resistance
r_c	Compressive strength correction factor
R_o	Material resistance
R_{sl}	Sliding plane length
R^T_{150}	Equivalent flexural strength ratio
S	Sensitivity index
s_1	Slip at the end of the linear elastic range of the bond-slip law
s_2	Slip at the end of the plateau of the bond-slip law
s_3	Slip at residual bond of the bond-slip law
T^D_{150}	Toughness (area under the curve from 0 to L/150) per ASTM1609
V	Voltage
V_e	Element volume
V_f	Volume of fibers
w	Crack width
w_l	Longitudinal crack width
$Y_{elastic}$	Elastic limit surface – RHT model
$Y_{failure}$	Failure surface - RHT model
$Y_{pre-peak}$	Pre-peak loading surface - RHT model

$Y_{residual}$	Residual surface - RHT model
Y_{TXC}	Compression meridian - RHT model
α	Orientation factor
$\dot{\gamma}$	Shear strain rate
δ_1	Displacement at first peak load per ASTM 1609
ΔL_{bar}	Elongation of bar
δ_p	Displacement at peak load per ASTM 1609
$\dot{\epsilon}$	Strain rate
ϵ_c	Axial compressive strain
ϵ_{cfmin}	Minimum compressive failure strain
ϵ_{co}	Strain corresponding to compressive strength
ϵ_{cr}	Cracking tensile strain
ϵ_{lat}	Lateral strain
ϵ_{pl}	Plastic strain before ultimate strength
ϵ_s	Reinforcing steel strain
ϵ_t	Axial tensile strain
ϵ_{tfmin}	Minimum tensile failure strain
ϵ_{tu}	Strain corresponding to ultimate tensile strength
κ	Sliding plane angle
μ_d	Dynamic friction coefficient
μ_s	Static friction coefficient
μ_o	Plastic viscosity
ρ	Resistivity
ρ_c	Density of concrete
ρ_s	Density of structural steel
σ_n	Normal stress
τ	Shear stress
τ_{oct}	Octahedral shear stress
τ_o	Yield stress (rheology)
φ	Internal friction angle

Chapter 1: Introduction

1.1. Problem Statement

1.1.1. Geopolymer Concrete

The accelerated global population growth that inevitably leads to the increase of urbanization rates intensifies the demand for construction materials. Conventional concrete is the foundation of modern infrastructure and is the second most commonly used material on this planet after water. It is known that the main component of conventional concrete is Ordinary Portland Cement (OPC), and its estimated yearly production will almost reach 5 billion tons by 2030 ((Statista Research Department, 2020) (Figure 1.1)).

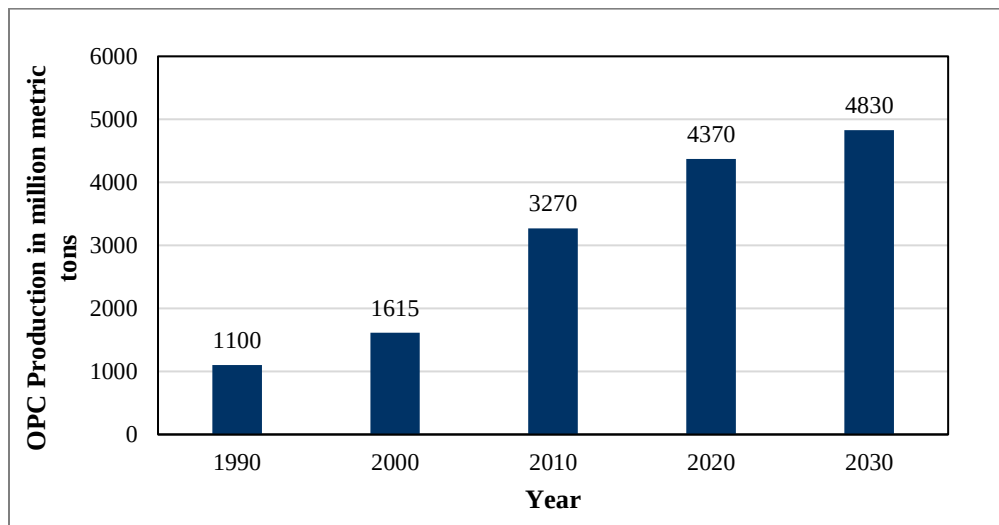


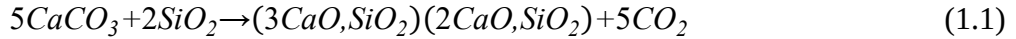
Figure 1.1: Global cement production from 1990 to 2030 in million metric tons (modified from Statista Research Department, (2020))

Undeniably, global warming is a threat for the planet with the greenhouse gases, especially carbon dioxide (CO_2) emitted from human activities, being one of the main contributors. Table 1.1 (Blasing, 2016) shows the concentration in the troposphere and the lifetime of the most common greenhouse gasses. The period of time that these gases stay in the atmosphere ranges from a few to thousand years. To reflect the duration of time and the ability to absorb energy, a parameter known as Global Warming Potential (GWP) has been used. The higher the value of GWP, the higher the contribution of the gas to global warming.

Table 1.1: The highest tropospheric concentration, GWP and lifetime of greenhouse gases

Gas	Pre-1750 tropospheric concentration	Recent tropospheric concentration	GWP (100-year time horizon)	Atmospheric lifetime (years)
Concentrations in parts per million (ppm)				
Carbon Dioxide (CO ₂)	~280	399.5	1	100-300
Concentrations in parts per billion (ppb)				
Methane (CH ₄)	722	1834	28	12.4
Nitrous Oxide (N ₂ O)	270	328	265	121
Tropospheric Ozone (O ₃)	237	337	N/A	hours-days

The Cement industry is the third largest source of CO₂ emission with about 7% of the total footprint. The impact of the production of OPC on global warming is clear once the chemistry mechanisms are explained. Cement is a product of calcination of limestone (calcium carbonate, CaCO₃) and silico-aluminous material according to the following reaction (Equation 1.1):



For every 1 ton of produced cement, 0.55 tons of chemical-CO₂ are emitted, and an additional 0.4 tons of CO₂ are required for the combustion of carbon-fuel (Benhelal et al., 2013). This roughly means that the production of 1 ton of cement generates 1 ton of carbon dioxide.

Due to growing environmental and economic concerns associated with conventional cementitious building materials, research interest following a mitigation path gravitates towards the development of novel environmentally friendly materials. The mitigation path is based on human interventions to decrease the greenhouse gases footprint. Although there have been admirable attempts for reducing the carbon footprint, the ominous projections about cement industry indicate an increase of 260% from 1990 to 2050 (Müller & Harnisch, 2008).

The rapid energy demand due to the accelerated population and urbanization growth already results in depletion of natural resources, with cement industry also being affected by this phenomenon. According to Figure 1.2, cement and lime industry has prevailed as the most energy-intensive industry. The depletion of fuel resources combined with the green taxes imposed in case of not limiting the emissions will skyrocket the cement price.

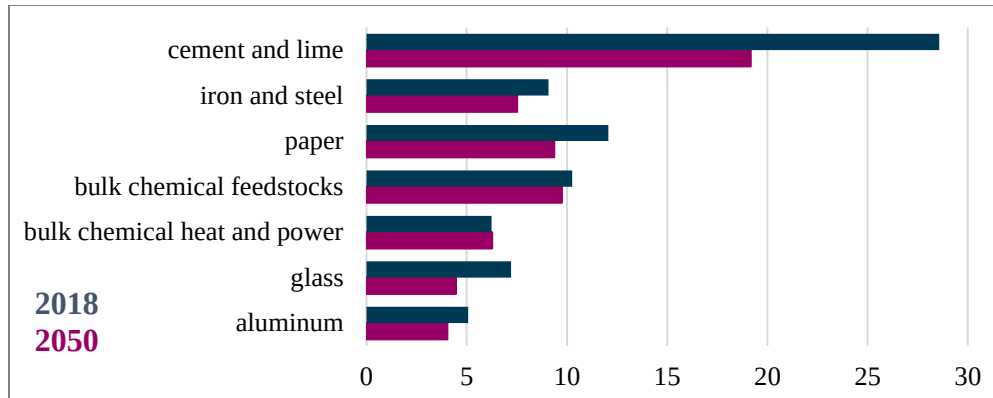


Figure 1.2: Energy-intensive manufacturing by industry (trillion British thermal units per billion 2012 dollar of shipments, modified from (U.S. EIA, 2022))

The environmental and economic concerns emerging from the energy-intensive industry of building materials impose a need for immediate action regarding the mitigation of CO₂ emissions. The basic strategies recently followed for achieving this goal are: (i) combustion of alternative fuels such as biomass and waste-derived fuels (ii) carbon capture and storage (iii) use of Supplementary Cementitious Materials (SCMs) such as fly ash, blast furnace slag and silica fume. There are several examples of cement industries using refuse-derived fuel, liquid and solid hazardous waste, biomass, and waste oil (The Pembina Institute, 2005) and research studies about CO₂ injections in concrete production (Monkman et al., 2016). The most promising strategy of mitigation is the utilization of SCMs. So far, several studies have been conducted on SCMs as partial replacement of cement even at percentages of 70% (Eshghi, 2018). In the past years, SCMs have been used as mineral admixtures in conjunction with OPC to reduce the cost of the mix design, and to enhance the properties of fresh or hardened concrete, such as workability, early strength, and durability.

Only recently, driven by climate change, there have been systematic efforts in developing a cement-free concrete completely made of industrial by-products. The two basic methods used for the development of green concrete is alkali activated materials and geopolymers, with the latter being of the utmost interest in the present thesis. Unfortunately, in current literature, there is a lot of misconception about these methods, as many researchers treat them as the same. Thus, the greatest challenge in developing a durable green concrete is to eliminate material-specific findings that cannot be extrapolated from one type of development to the other because the technologies emanating from these efforts are based on totally different mechanisms. The emerging, alkali-

activated and geopolymer material technologies should not be treated as similar to each other, or even as conventional concrete technologies and a more in-depth understanding is needed to successfully manipulate them for further progress in the respective fields.

1.1.2. Tension Hardening Fiber Reinforced Concretes (THFRCs)

There is no doubt that conventional concrete, owing to its inexpensive constituents, easy on-site preparation and favorable mechanical strength has been considered throughout the 20th century the cornerstone of infrastructure. It has been used mainly for reconstruction, and urban development which consists of residential and high-rise buildings, bridges, and highways. However, there is ample evidence that the combination of chemically aggressive environments, extreme climate conditions and aging, lead to a progressive deterioration of the material either in the form of carbonation in the presence of CO₂, reinforcement corrosion or disintegration due to freeze-thaw, or other forms of ageing. Especially concrete infrastructure in cold climates, where exposure to freezing and use of de-icing salts is inevitable, or marine environments, as shown in Figure 1.3 (Pantazopoulou et al., 2019), is prone to such extent of degradation that its service life may be dramatically decreased to almost half of the designed value (Mehta & Burrows, 2001). Nevertheless, maintenance and rehabilitation of the degraded concrete infrastructure could be more expensive than construction of new buildings, as it may exceed annually \$1 and \$20 billion for bridges and buildings, respectively (Yıldırım et al., 2018). In the last 20 years, Tension Hardening Fiber Reinforced Concrete (THFRC) which emerged as a technological evolution of Fiber Reinforced Concrete (FRC) has been successfully used as a resilient and sustainable material to overcome the aforementioned challenges. The low water to cement ratio, the dense microstructure and small particle size of its components contribute to high compressive strength and durability, while the addition of steel or synthetic fibers provide superior tensile strength, crack control and ductility. An identifying attribute of THFRCs is its tensile resilience: contrary to conventional FRC, where cracking strength almost coincides with the ultimate and upon exceedance of the latter, no significant post-cracking strength and strains may be supported, THFRC materials exhibit a hardening behaviour prior to localization with substantial post-localization strains and residual strength. This behaviour imparts characteristics such as increased ductility and fracture toughness to structural components constructed with THFRC whereas the crack control imparted by the fibers limits the penetration of aggressive agents markedly

improving durability, service life and eventually effecting a long terms reduction of the CO₂ footprint of such structures on account of the enhanced performance and reduced need for maintenance.

Nonetheless, accurate determination of the dependable tensile strength and ductility remains a challenge in the field of THFRCs. Whereas there are various direct and indirect procedures available in the literature for tensile characterization of THFRC, they are mostly difficult to conduct and fraught with uncertainty or reduced reliability. In the meantime, the composite nature of the material adds extra complications in the understanding and determination of tensile behaviour of THFRCs. Fiber orientation is a catalytic factor in the mechanical behaviour of the material that is often responsible for differentiating mechanical properties, especially in tension. Therefore, as no consensus has been reached in the research and standards community regarding a standard methodology to derive the constitutive law, further research is needed for development of a testing method that will alleviate the challenges associated with the available procedures.



Figure 1.3: 20-year-old prestressed box-section concrete bridge over sea (Pantazopoulou et al., 2019)

Although THFRC is considered a sustainable solution compared to other cementitious composites, the high amount of OPC that is used as a binder (around 700 kg /m³ of THFRC) raises concerns about the sustainability of the material. Currently, there is a gap in literature regarding the development of green THFRC.

With geopolymer concrete being the latest advancement in building materials, most of the available studies in the literature are focused on the development and material characterization of geopolymer materials that would replace conventional concrete. A few studies (Al-Majidi et al., 2017) have been conducted on development of advanced geopolymer mixtures as a substitute of Engineering Cementitious Composites (ECC), but even in this case, studies are restricted only in investigating the material behaviour rather than the structural response. The next frontier that hasn't been explored yet is the development and the structural characterization of a Tension

Hardening Fiber Reinforced Geopolymer Concrete (THFRGC). The fabrication of THFRGC will combine the advantages of sustainability of geopolymer concrete with the beneficial performance of THFRC in terms of strength, resilience, tensile ductility, and durability.

1.1.3. Structural Health Monitoring Through the use of Carbon Nanomaterials

Throughout the years urban development relies on civil infrastructure such as bridges, buildings, highways, and dams. Proper maintenance and management are key to the continuing, reliable, safe, and economical operation of these structures. Despite the initial design, deterioration due to aging, fatigue (cyclic traffic loads), environmental factors (carbonation and corrosion) and unexpected extreme phenomena (earthquakes, fires, hurricanes, and floods) diminishes the infrastructure's structural capacity and serviceability (Chen & Ni, 2018). Currently, condition assessment of the existing infrastructure is being conducted via visual inspection, a very subjective technique that sometimes is not possible if the site is not accessible. The concerns raised from conventional assessment strategies led to the development of several novel monitoring technologies known collectively as Structural Health Monitoring (SHM) methods. With SHM, infrastructure is continuously monitored by advanced sensors which transmit data via networks to hardware and software facilities. The collected data are used for evaluating the current health and predicting the future performance of the infrastructure. SHM is a growing research field showing great potential as a reliable, economic, and safe strategy of maintenance and management of infrastructure. However, due to its multidisciplinary nature, the technology is either too expensive or relatively primitive in certain aspects relating to practical implementation. In this regard, many advancements in fields associated with mechanical, computer and civil engineering are required for maturity of SHM. So far, monitoring systems proposed and used for SHM are based on external sensors embedded or attached in the structure transmitting signals through wired technology. The sensors are discrete, therefore finite and placed in strategic locations. The next frontier to be explored is wireless and continuous self-sensing systems.

Latest advances in the field of Nanotechnology have directed to the development of novel multifunctional smart materials. Notably, cementitious composites reinforced with Carbon Nanotubes (CNT) is a novel promising contactless application of SHM (Ubertini et al., 2014). Carbon nanotubes are the most resilient materials known in nature due to the exceptional

mechanical, electrical and chemical properties they demonstrate. One interesting observation in this type of material is that when they are subjected to stress/strain, their electrical properties are altered, expressing a linear and reversible piezoresistive response. These properties enable a new class of nanoscale mechanical sensors and make them ideal candidates as a reinforcement in cementitious materials with self-sensing properties.

1.2. Research Significance

On account of growing environmental and economic issues, the decarbonization of concrete industry has become a priority with the development of sustainable building materials to attract both research and industry community. Sustainability can be achieved either through mitigating CO₂ emissions in concrete's production or by advancing its performance and thus reduce the need for maintenance. The enhanced performance is mainly governed by microstructure and tensile hardening properties of concrete. An ideal building material should combine both aspects of sustainability, namely in terms of its constituents and performance. However, the development of such material should be followed with material and structural characterization with focus on the tensile response in order to promote its use in the industry. Meanwhile, the presence of fibers opens new prospects for self-sensing abilities and SHM applications.

1.3. Objective

The title of the thesis effectively summarizes the scope of the research, which is focused on the development and characterization of sustainable tension hardening and high performance multifunctional structural materials. with an emphasis to achieving (a) reduced carbon footprint through exploitation of geopolymer technology; (b) improving the tensile behaviour characterization procedures, and (c) endowing the material with sensing capabilities that would facilitate SHM procedures in the field. Through this objective the use and structural application of geopolymer THFRC is promoted, while contributing to practical knowledge for future application of THFRCs in general, such as the development of a novel tensile testing setup that would be easily accessible to the material certification industry, methods for fiber orientation assessment and exploitation of inherited self-sensing abilities in case of nanoengineered THFRCs.

1.4. Research Methodology

To achieve the above objectives, the work for this dissertation was spun along three mutually complementary directions, namely: Assessment of the state of the art in the fields of the study, followed by an experimental and analytical/computational investigation. The state-of-the-art review provides a basis to identify research gaps on the issues discussed above. The experimental part includes development and complete characterization of a novel geopolymer tension-hardening concrete as well as the raw materials, mix design, physico-mechanical properties, durability, structural behavior, electrical properties, electromechanical response, and experimental destructive/non-destructive fiber orientation technique measurements. With regards to the analytical investigation, the work comprises of FEM simulations of the proposed tensile test and four-point bending tests used for derivation of tensile properties through inverse analysis, but also FE- based RVE modeling for fiber distribution and orientation assessment.

The state-of-the-art review covers not only the area of geopolymer concrete, tensile characterization of THFRC and SHM through nanoengineered cementitious composites but also the tools needed towards achievement of the aforementioned objectives. Such tools include mix optimization techniques, methods for determination of fiber orientation and influencing factors and constitutive THFRC models for FEA simulations. The review is based on a search of scientific journals and proceedings of related conferences, as well as North American and European Standards.

For the experimental campaign, with regards to the geopolymer THFRC, the characterization of reactive powders was performed in terms of physical, chemical, and mineral analysis. Through trial and error and mix optimization techniques, 60 different mix designs were tried based on performance parameters such as fresh (workability and setting time) and hardened state (microstructure and mechanical strength) properties. Then, the developed geopolymer THFRC is characterized based on tests that are currently included in the material identity characterization prescribed for THFRCs by North American Standards. Along with these tests, the structural performance was evaluated using Direct Tension Pullout and Push-off tests, for determination of bond-slip law of reinforcing bars in geopolymer THFRC matrix and interface shear, respectively. For the development of the novel tensile tests, different proprietary THFRC mixes were cast and tested, while different geometries and boundary conditions of the test were also investigated. With

regards to nanoengineered THFRC, the self-sensing capabilities were investigated through conventional and a wireless electromechanical testing. The results of all mechanical tests were further analyzed using Digital Image Correlation. Finally, for the assessment of fiber distribution in THFRC, cuts on tested specimens with manual fiber counting were performed and a transparent fluid model THFRC was used as a visualization technique.

The computational part included FE modeling and analysis of the proposed tensile test for numerical validation, while two different FE softwares and plasticity models in static and quasi-static analysis were used to assess the tensile law of THFRC obtained through inverse analysis and four-point bending.

1.5. Structure of the Thesis

This dissertation presents a research study completed by the author from 2017 to 2022. Figure 1.4 is a schematic illustration of the thesis outline, while the following paragraphs provide a brief description of each chapter.

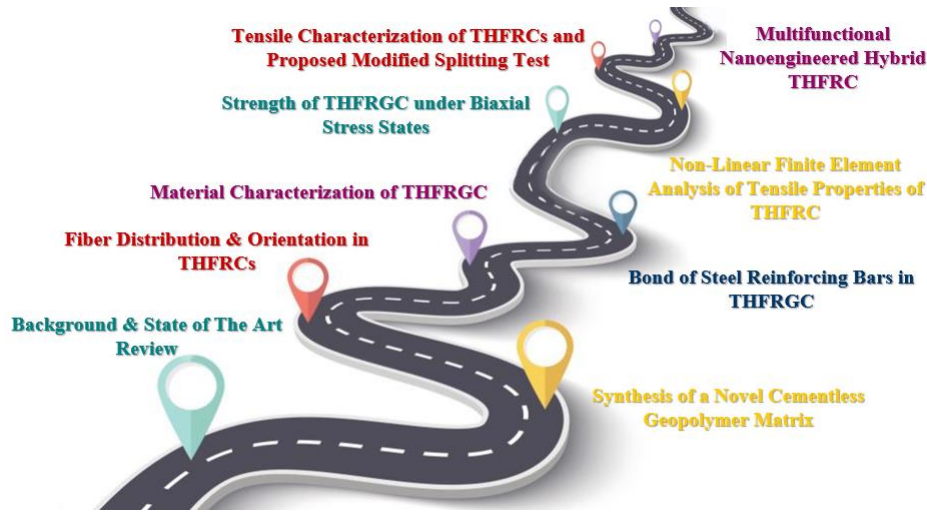


Figure 1.4: Thesis outline: A roadmap towards sustainability and resilience

Chapter 2- Background and State of The Art Review lays the background and presents an exhaustive literature review on geopolymer concrete, tensile characterization of THFRC materials, and intrinsic self-sensing nanoengineered concrete.

Chapter 3- Synthesis of a Novel, Cementless Geopolymer Concrete Matrix presents the process of development of an innovative and novel cement-less concrete which was based on the

geopolymerization technology. The chapter details the characterization of raw materials and concrete microstructure, optimization techniques based on particle packing and chemistry and various mix designs. Moreover, it describes the effect of different raw materials and chemical admixtures on fresh and hardened state properties.

Chapter 4- Fiber Distribution and Orientation in THFRC explores the correlation of different orientation factors proposed in the literature with the actual fiber distribution obtained by experimental manual counting on a variety of different THFRC surfaces. Then using the above results, a fiber orientation factor that is depended on fiber geometry, casting method and formwork geometry is proposed. Finally, the fiber distribution and influencing factors are studied through the use of a translucent fluid model of self-consolidating THFRC.

Chapter 5- Material Characterization of the Developed THFRGC includes the experimental campaign for the determination of mechanical, physical and durability properties of the developed geopolymer THFRC.

Chapter 6- Bond Behaviour of Reinforcing Bar in THFRGC describes the interfacial bond-slip law between deformed steel bars and geopolymer THFRC developed in Chapter 3 and characterized in Chapter 5. The investigation parameters include two different embedment lengths and clear covers. The results are further analyzed with DIC analysis and used to obtain a constitutive bond-slip law of reinforcing bars in geopolymer THFRC.

Chapter 7- Strength of THFRGC Under Biaxial Stress States presents push-off tests of the developed geopolymer THFRC without reinforcement and with varying shear plane angle. The results are further used to obtain the constitutive behavior in terms of a 2-D Mohr-Coulomb envelope and the 3-D Bresler-Pister Failure Criterion for this material so as to facilitate its adaptation in finite element software using nonlinear plasticity models.

Chapter 8- Non-Linear Finite Element Analysis of Tensile Properties of THFRC describes a comparative evaluation of numerical simulations of four-point bending tests conducted on THFRC used for inverse analysis and extraction of the tension stress-strain properties of these materials by the available standards (CSA, S6, CHBDC-Annex 8). Two different concrete damage plasticity models with tensile constitutive law based on the results of inverse analysis

proposed by CSA S6 Annex 8.1 are used. The chapter is developed based on a comparative investigation published by the researcher (Ralli et al., 2021).

Chapter 9- Tensile Characterization of THFRCs and Proposed Modified Splitting Test presents the design, numerical and experimental validation of a novel indirect tension test for recording the tensile stress-strain response of THFRCs. The experimental results of a total of 34 specimens from different THFRC mixes, with varying geometry and support conditions are presented and further analyzed using DIC in order to document the performance of the new testing method.

Chapter 10- Multifunctional Nanoengineered THFRC describes a series of mechanical, electrical, and self-sensing experiments performed on a proprietary nanoengineered THFRC. The main focus of the chapter is the assessment of self-sensing capabilities through a wireless approach which could be used in the future for contactless monitoring of structural elements endowed with carbon nanomaterials.

Chapter 11- Conclusions and Future Research summarizes the work and findings of the present dissertation while provides an outline of proposed areas for future research.

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Chapter 2: Background and State of The Art Review

Latest and increased technological demands of structural materials combined with environmental concerns have generated the need for the development of sustainable high performance engineered cementitious composites, while endowing them with remote sensing and condition assessment capabilities would be a desirable advancement towards structural health monitoring of structures containing these materials. In this chapter a thorough review of geopolymer concrete, tension hardening behaviour and self-sensing properties of nanoengineered cementitious composites is given, in order to form a strong conceptual and technical basis regarding sustainable and smart high performance building materials on which to build the present research.

2.1. *Geopolymer Concrete*¹

This section comprises an exhaustive critical review of the currently available literature. The purpose of this review is the deep understanding of the underlying physicochemical mechanisms that govern geopolymerization and how geopolymer concrete performs in terms of durability, mechanical and structural properties.

2.1.1. Historical Background of Geopolymer Science

Geopolymer materials represent the latest advancement in alumino-silicate chemistry, which was first investigated in the 1930's (Olsen, 1934). By that time and for the next two decades, research focused on development of zeolites and alkali activated materials by reacting the aluminosilicates with alkali hydroxides (Glukhovsky, 1965; Howell, 1963; Olsen, 1934). Later on, in the 1970s, Davidovits defined the geopolymer chemistry with his first work based on kaolinite which paved the way for the establishment of geopolymer chemistry. In the effort to develop non-flammable and non-combustible polymeric stone-like materials, principles of geochemistry were used to produce mineral polymers, which have since been referred to as geopolymers. The invention of the first mineral fire-resistant resin consisting of metakaolin and soluble alkali silicate was patented in 1975, whereas the first geopolymer cement was developed and patented by Davidovits and Sawyer in 1984 in the US as "Early High-Strength Mineral Polymer (J. Davidovits & Sawyer,

¹ Part of this chapter has been used in the Author's publication with her supervisor, titled: "State of the art on Geopolymer Concrete" *International Journal of Structural Integrity*. Vol. 12 No.4, pp.511-533

1985). Since then, geopolymer building materials have gained significant ground in the field of sustainable infrastructure.

So far geopolymer concrete has been extensively used both in precast production and on site in the field. Besides structural applications, geopolymer mortars have been used as repair mortars in public facilities, namely in 2019, in Ohio, to restore a sewer lining (Royer, 2019). Numerous projects around the world have been implemented using structural geopolymer concrete with Australia to pioneer in the sector by constructing the first public building, Global Change Institute made entirely from geopolymer concrete in 2013 (Bligh & Glasby, 2013), followed by West Wellcamp Airport in Brisbane in 2014 (Glasby et al., 2015) and more recently, in 2019, Sydney launched the first trial of public road construction using entirely geopolymer concrete (City of Sydney, 2019). Besides Australia other known projects implementing geopolymer concrete are found in Russia where a pedestrian bridge in Skolkovo and the foundations of Gazprom Neft storage facility were built entirely from this novel material (Dudnikova et al., 2018). Finally, the next frontier already being explored is in the field of additive manufacturing (3-D printing) with the 1st geopolymer 3-D printed house in 2018, in Siberia (Dudnikova et al., 2018). However, no two geopolymer products are the same. Because they combine mostly wastes with chemistry carefully adapted to the source material characteristics, development of a geopolymer material that is entirely cementless, strong as concrete and at least as durable, is a challenging endeavor that requires extensive experimentation and testing.

Note that although geopolymer technology emerged as an advancement of alumino-silicate chemistry, it does not mean that zeolites, alkali activated materials and geopolymers belong in the same category generally referred to as alkali activated materials (AAMs). The underlying mechanisms of AAMs and Geopolymers are completely different, and the differences pervade every aspect of material behavior. The differences will be discussed in the following sections.

Apart from the field of construction materials where the development of geopolymer building materials and especially cement and concrete are driven by the need to reduce CO₂ emissions, the relevant technologies find a wide range of multidisciplinary application in the fields of ceramics, decoration, coatings, high-tech resins for fiber composites in aircrafts, environmentally

friendly cements, waste encapsulation, fire-resistant materials, infrastructure retrofit and strengthening composites and bio-technologies.

It is notable that early evidence of geopolymer synthesis has been found in ancient monuments around the world (J. Davidovits, 2020; J. Davidovits et al., 2019). For example, it has been claimed that manufactured stones with a microstructure very similar to that of geopolymer systems could be identified in the Pyramids of Cheops and Chephren built in 2700 B.C. High similarity with modern geopolymer building materials is also found in Roman Concrete (300 B.C.) and south American monuments (600 A.D.).

2.1.2. Definition and Terminology

Geopolymers are mineral chemical compounds or mixtures thereof, comprising repeated chemical units, for example silico-oxide (-Si-O-Si-O-), silico-aluminate (-Si-O-Al-O-), ferro-silico-aluminate (-Fe-O-Si-O-Al-O-) or alumino-phosphate (-Al-O-P-O-), created through a polymerization process. In geopolymerization, many small molecules, also referred to here on as oligomers, are combined to form a covalently bonded three-dimensional macromolecular network. The geo-chemical syntheses resulting from the polymerization occur either in alkaline media (where alkali ions such as Na^+ , K^+ , Li^+ , Ca^{++} and Cs^+ are present) or in acidic media (in the presence of phosphoric and humic acids). The most common route for synthesis of geopolymer concrete is in an alkaline medium, especially in the presence of sodium or potassium. The existence of soluble isolated oligomers was proven by North and Swaddle, (2000). They are the initiating units of potassium-based alumino-silicate geopolymers; they are also called sialates based on geological terminology, where the term sialic is encountered and is used to characterize a material containing high amounts of aluminum and silica. The different forms of the elementary units found in geopolymers are classified according to their Si: Al atomic ratio, which essentially specifies the type of application the geopolymer can be used in.

2.1.3. Geopolymerization Mechanism

Geopolymerization takes place when an alumino-silicate source material (such as calcined clays or fly ash) reacts with a soluble alkali silicate (such as sodium or potassium silicate solution) under conditions to be discussed in the forthcoming sections. There are six steps to the process: (i) alkalination, (ii) depolymerization of silicates, (iii) formation of oligo-sialates, (iv)

polycondensation, (v) reticulation, networking and finally, (v) geopolymer solidification. An example geopolymer product obtained from the reaction of metakaolin 750 with a sodium silicate is used to illustrate the process and kinetics of geopolymerization in this section (J. Davidovits, 2020).

Based on the molar ratio of $\text{SiO}_2/\text{Na}_2\text{O}$ in the silicate solution, specific oligomer structures form with molecular structure being determined in room temperature. However, when the silicate solution is mixed with the metakaolin (a process known as alkalination), these oligomers depolymerize owing to the exothermic reactions that occur and the consequent rise of temperature. The oligomer species consist of silicon compounds also known as Q species and have been identified via ^{29}Si NMR Spectroscopy by researchers in the past (Felmy et al., 2001; Harris et al., 1980). The identification is Q_n where n is number of bridging oxygens (O_B); thus, Q_0 has no bridging oxygen, Q_1 has one, and so on. In this case, the sodium silicate solution at room temperature consists of Q_0 , Q_1 , Q_2 and Q_3 species (siloxonates), but after the exothermic reaction, the major siloxonates are Q_0 and Q_1 with some Q_2 in linear or cyclic structural form as a result of depolymerization (Davidovits, 2020). During alkalination, the particles of metakaolin are not dissolved but only react on the surface with the alkali (Figure 2.1). The kinetics of the chemical mechanism could be distinguished into two phases. In Phase 1, the surface and the edges of the particles react with NaOH and the siloxonates. Phase 2 initiates as a result of Phase 1. The reaction of Phase 1 causes the interlayers of MK-750 to swell enough so as to allow only the smaller ions of Na^+ and OH^- to penetrate the layer and react. The rest of the molecules remain outside of the layers and react superficially.

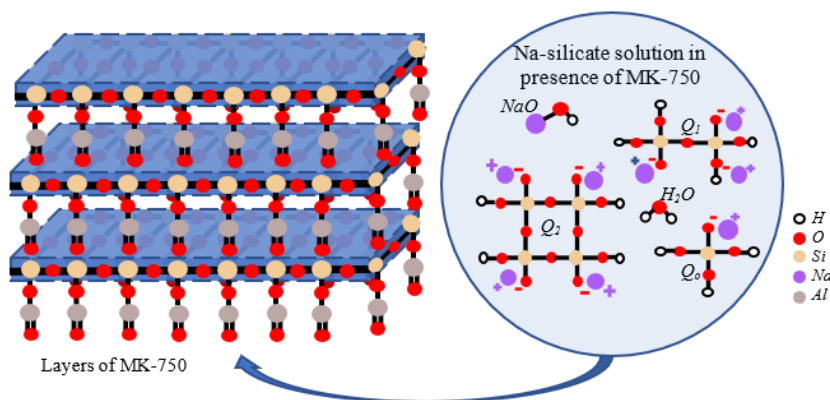


Figure 2.1: Schematic representation of the chemical attack of Na-silicate on alumoxyl groups ($-\text{Al}=\text{O}$) of Metakaolin-750 layers

More analytically, Phase 1 starts with alkalination of metakaolin particles. The Al atom becomes tetravalent in the side group sialate $O_3-Si-O-Al-(OH)_3Na^+$ (Figure 2.2a). It is known that the Al atom has an amphoteric character and depending on the medium can be trivalent (acidic medium) or tetravalent (alkaline medium). Subsequently, the cation OH^- attaches to the silicon atom forming a pentavalent Si and thus initiating the alkali dissolution on the edges of the particles (Figure 2.2b). Next, the silicon atom becomes tetravalent again as cleavage of the siloxane oxygen occurs due to transfer of an electron from the Si atom to O atom. This results in the formation of silanol (Si-OH) and basic siloxo (Si-O-) as illustrated in Figure 2.2c. The process goes on with further formation of silanol groups and isolation of the ortho-sialate molecule which is the fundamental unit of geopolymerization (Figure 2.2d). The basic siloxo (Si-O-) reacts with the Na^+ and forms the (Si-ONa) terminal bond (Figure 2.2e). So far, these steps occur in both phases. At this point, the Q_1 di-siloxonate of the soluble silicate and formed oligo-sialate condense and create an ortho-sialate-disiloxo cyclic structure, where NaOH is liberated to further react again in Phase 2 (Figure 2.2f). The polycondensation continues into forming the typical geopolymer chain, Na-poly(sialate-disiloxo) (Figure 2.2g). The free NaOH will be later fully consumed during Phase 2 leading to the formation of the 3-D network. The formed chains interact with each other through water (hydrogen bonds) to form the three-dimensional geopolymer network. The dehydroxylation causes the evaporation of water which is followed by solidification leading to formation of the final stable network (Figure 2.2h).

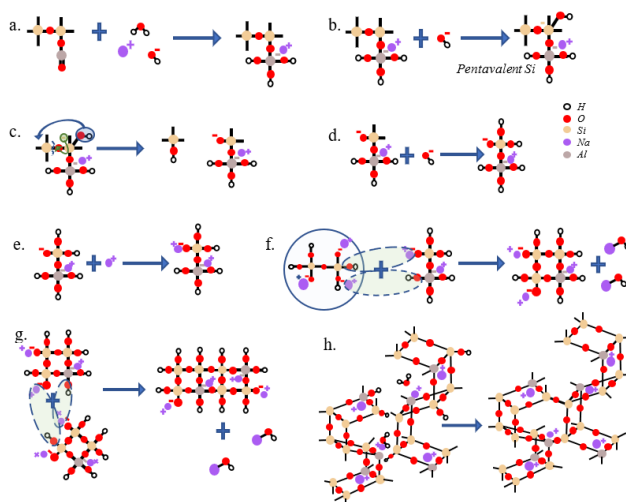


Figure 2.2: Steps of geopolymerization mechanism: a. Alkalination of sialate group, b. Initiation of superficial alkali dissolution, c. Formation of intermediate silanol Si-OH and Basic Si-O-, d. Formation of primary unit of geopolymerization, e. Formation of ortho-sialate molecule, f. Condensation of the ortho-sialates, g. Polycondensation into Na-poly(sialate-disiloxo), h. Formation of 3D geopolymeric network with Si:Al=2

2.1.4. Applications

Geopolymer technology as mentioned earlier, has a vast field of applications, with geopolymer concrete being one of them. Other than geopolymer concrete, depending on the atomic ratio Si: Al present in the geopolymer's molecular structure, applications have ranged from ceramics to high-tech heat resistant composites used in aircraft or automobile applications. Geopolymer concrete is characterized by a 3-D network of Si: Al=3-4 units.

Composition of a geopolymer material requires a combination of material sources from the following groups, which are subsequently analyzed in detail:

- Geopolymer precursor, which is the source material that will react with the soluble silicate.
- Hardener – in the form of Soluble Silicates. In the remainder, the term geopolymer *binder* encompasses the solid part of the soluble silicate, together with the precursor.
- Aggregates
- Additives

In the following sections, the all-important curing conditions and durability performance of the materials are reviewed. The differences between Geopolymers and Alkali-Activated Materials are also analyzed, aiming to contribute to the clear distinction between these two methodologies.

2.1.5. Alumino-Silicate Source Materials Used as Precursors

Selection of pertinent alumino-silicate source materials is key in developing a geopolymer concrete. Setting target performance criteria such as high performance and sustainability require thorough understanding of the physicochemical properties of the material. A suitable source material for geopolymer concrete should be: (i) highly amorphous, (ii) possess the ability to release aluminum easily and (iii) contain sufficient reactive glassy content with low water demand. Several studies have been conducted on different types of binders and on how these may affect the properties and the characteristics of the resulting geopolymer synthesis. Various aluminosilicate raw materials such as metakaolin or industrial by-products such as fly ash, and ground granulated blast furnace slag have been used alone or in combination as binders for

geopolymer concrete. A detailed review follows, regarding the alumino-silicate source materials that have been used or have a potential as a part of the geopolymeric binder, with reference to cost, availability, suitability, and performance.

- *Metakaolin*

Metakaolin (MK) is a pozzolan resulting from calcination (dehydroxylation of the water contained, through heating to 600-800 °C) of kaolinite, a common clay mineral found in kaolin. At 550 °C, kaolinite which is previously extracted from kaolin becomes anhydrous and loses its crystalline order. The resulting Metakaolin ($\text{Al}_2\text{Si}_2\text{O}_7(\text{OH})_{2(1-\kappa)}$ where κ is the dehydroxylation ratio that depends on the calcination temperature) is amorphous (Soleil, 2019). The average particle size can be as low as 1.5µm. The material becomes again crystalline if calcined to 975 °C, but with a different structure forming Al-Si Spinel which is commercially known as calcined kaolin ($\text{Al}_2\text{Si}_2\text{O}_7$). Further calcination up to 1150 °C results to the formation of chamotte which is mullite ($3\text{Al}_2\text{O}_3:2\text{SiO}_2$) and an excess of silica. The prefix “meta” is used to imply the effected change, i.e., the dehydroxylation of the initial mineral that occurs due to elevated temperature. Quality of the product is controlled during manufacturing and is less variable than industrial pozzolans.

Usually, MK is used as a SCM in conjunction with cement in concrete as it yields high performance and early strengths (Siddique, 2008). MK-750 (i.e., product of kaolinite calcination at 750 °C) is the most common type of MK used in geopolymer concrete. The dehydroxylation of kaolinite into MK-750 yields Al atoms in IV-, V- and VI coordination; this formation can only be explained with the covalent bond concept. The number of coordination shows how many oxygens surround one Al atom. The surrounding oxygens may not all be bonded to Al, as Al can be trivalent or tetravalent depending on the pH of the medium (acidic/basic, respectively). These species characterize MK and are used as a reactivity index. They react differently, the most reactive being Al(V). On account of the amorphous state of the material, the concentration of the phases (Al(IV), Al(V) and Al(VI)) depends on the manufacturing process and may be characterized by NMR Spectroscopy.

The manufacture of MK starts with mining and preparation of kaolin. At this point, quality control is performed based on factors such as mineralogy, chemistry and specific surface. Subsequently,

kaolin is calcined in continuous furnaces such as rotary and Herreshoff multilevel kiln or flash kilns. The key performance factors here are time, temperature and redox conditions. The duration of calcination for rotary and Herreshoff kiln is 4 and 6 to 12 hours respectively, while the flash kiln needs only a few seconds. Different kilns yield MK with different properties. Table 2.1 shows how the kiln type affects the amount of Al species and thus the reactivity of the MK-750 that comes from the same geological source, kaolin. The last step of manufacture is the milling which is performed in a ball, hammer or roll-press mill, with particle size to be the quality control parameter.

Table 2.1: Concentration of Al species according to the manufacture processing (R. Davidovits et al., 2019)

Kiln Type	Al-OH Al(VI) %	Al=O Al(V) %	Al-O-Al Al(IV) %	Al(V) +Al(IV) reactivity %
Rotary	35	50	15	65
Flash	25	55	20	75
Herreshoff	24	49	27	76

Although MK is a manufactured alumino-silicate source material, there are not yet standardized methods for determining the suitability of the product for geopolymer applications. Instead, companies provide information about tests related to the use of MK in conjunction with OPC. A common test performed by the manufacturing industry is the one that determines the pozzolanic activity (modified Chapelle test in the French norm, (NF (Normes Françaises) P18-513, 2010)).

As mentioned in the preceding example of MK-based geopolymer, MK exhibits a strong exothermic behavior when it reacts with alkalis. To assess the real conditions during geopolymerization, R Davidovits et al., (2019) prepared a geopolymer slurry by mixing commercially available MK-750 with potassium silicate and heat-curing for one hour at 80 °C. The exothermicity was measured with thermocouples. The test illustrated that different kiln types affect the reactivity of the final product. Despite the reactivity of MK due to high alumina content, a moderate-to-low pH in the range of 5-6 is beneficial in terms of hardening, as it prolongs the mixing time thereby avoiding flash setting which is the biggest threat to the success of geopolymerization.

Being a manufactured pozzolan, MK-750 undergoes quality control and standardization, rendering it an excellent candidate for geopolymers. The high reactivity and fine size contribute to high strength and durability, whereas when used in conjunction with industrial by-products as

geopolymeric binder it prolongs the mixing and setting time. A drawback is the high cost. For this reason, it is used in small amounts to enhance the properties of industrial by-product based geopolymers.

- *Fly Ash*

Fly Ash (FA) also referred as Pulverized Fuel Ash (PFA) is a precipitation ash product from exhaust gases of coal-based power plants. It is the most common artificial pozzolan due to the high content of amorphous silica and sometimes it is considered as a lower quality natural metakaolin. Due to its ability to improve rheology and reduce alkali aggregate reaction it was initially utilized as SCM in ordinary Portland cement (Mindess & Young J.F., 1981).

Bhatt *et al.* (2019) predict an increase of global energy consumption by 30% by 2035 with a commensurate increase in annual coal consumption from 3840 million tons in 2015 to 4032 in 2035. The abundance of FA combined with the fact that this industrial waste does not require any pre-treatment renders its use an attractive option from a sustainability perspective whereas disposal of FA in open landfills would cause massive environmental problems such as ground water contamination, spills, and ground pollution by heavy metals, with many possible effects on public health. Therefore, valorization of the FA is essential for sustainability of the coal industry, particularly now with the resumption of intensive coal burning in response to the exacerbated energy crisis.

Regarding production of FA, finely grounded coal is combusted by injection with a stream of hot air at high speed into the furnace of electricity-generating power plants. The coal is burnt when the temperature of the boiler reaches 1500 °C. The remaining material which consists of shale and clays melts and via rapid cooling solidifies into spherical particles of high fineness, comprising amorphous and crystalline phases with the majority being mullite, magnetite, quartz, haematite, and unburned carbon (Neville, 2012). Fineness also depends on the degree of pulverization and combustion environment, whereas minerals contained in coal and the burning conditions are the basic factors affecting chemical composition. As with MK-750, different methods of FA extraction yield different material qualities.

ASTM C 618 (ASTM C618, 2022) classifies FA into Class F and Class C types based on the CaO content; the former is the commonest type, deriving from anthracite and bituminous coal burning.

FA-F is mainly siliceous (over 50%), has dark color, constant fineness, carbon content and a low CaO content (<10%). This type of FA has higher alumina content as compared to Class C. FA-C is a light grey high-lime ash product of sub-bituminous coal and lignite that contains low amounts of carbon and high amount of MgO and CaO (>10%). The chemical composition of typical fly ash types from different coals and the requirements according to ASTM C 618 (2022) are listed on Table 2.2 (Chou, 2012). CSA A3001 (2004) identifies three types of FA based on the lime content, namely, F, CI, and CH. (In Canada only type F and CI are produced, where the limit of CaO is 8%, and 8-20%, respectively). Type CH has a higher content, around 20%, and is usually imported from the USA). FA-F is preferred as a source material for geopolymers because of the lower CaO content, high Al₂O₃ content and stable minerals.

Table 2.2: Chemical composition of different types of Fly Ash and ASTM specifications (Chou, 2012)

Compound	Bituminous	Subbituminous	Lignite	ASTM C 618 specifications	
Silica (SiO ₂)	57.0	34.6	28.9		
Alumina (Al ₂ O ₃)	25.3	17.8	11.1		
Ferric Oxide (Fe ₂ O ₃)	5.3	6.1	11.1		
SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃	87.6	58.5	54.1	70.0 min	50.0 min
Lime (CaO)	2.0	27.2	18.9		
Magnesia (MgO)	1.2	5.8	6.0		
Sodium Oxide (Na ₂ O)	0.9	2.3	8.0	1.5 max	1.5 max
Potassium Oxide (K ₂ O)	3.1	0.5	0.9		
Sulfur Trioxide (SO ₃)	0.2	2.8	6.8	5.0 max	5.0 max
Loss of Ignition (LOI)	3.1	0.9	0.4	6.0 max	6.0 max
Moisture Content (%)	N/A	N/A	N/A	3.0 max	3.0 max

The reactivity of FA depends on the mineral composition and the vitreous phase content which on its end, depends on the temperature of combustion (the glass content increases with firing temperature). The majority of power-plants operate in temperatures between 800 °C to 1600 °C, and thus the formed phases are not distinct, as would be the case, for example, at a temperature of 1200 °C; it is possible to have remains of quartz but also some cristobalite formed. For this reason, the reactivity of the fly ash may be determined from Electron Microscopy.

Apart from the determining effect of combustion temperature on the morphology of FA particles, other important factors are, the source of coal and the oxygen supply in the furnace. Depending on these parameters the particles could be coarse or fine and have a range of possible surface texture characteristics (angularity/smoothness and porosity). Generally, a high temperature of combustion (around 1500 °C) yields a fine FA consisting mostly of hollow particles (cenospheres), amorphous in nature and spherical in shape, that are particularly suitable for

geopolymerization. Grain size also depends on the processing parameters, with particle sizes ranging from 5 μm to 300 μm . In fact, due to the highly fine nature of the material, particles tend to agglomerate and mechanical or sonic disintegration is required for the determination of the true grain size distribution (Kumar et al., 2005).

According to a characterization study of 17 European FA's (J. Davidovits et al., 2014) no cenospheres are formed at combustion temperatures lower than 1250 °C and only remains of minerals such as illite $((\text{K},\text{H}_3\text{O})\text{Al}_2\text{Si}_3\text{AlO}_{10}(\text{OH})_2)$ occur; at lower temperatures biomass remains occur that disturb the polymerization process. Perfect vitreous spheres are formed at temperatures around 1500 °C, but at even higher temperatures (e.g., 1800 °C) used in gasification technology the occurrence of spherical particles is reduced. Regarding the effect of mineralogy (determined from XRD analysis) on polymerization: some minerals tend to be favorable whereas others hinder the process. Major mineral phases in the samples include quartz (SiO_2), mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$), hematite (Fe_2O_3), magnetite (Fe_3O_4), anorthite-albite $((\text{Ca},\text{Na})(\text{Al},\text{Si})_4\text{O}_8)$, illite $((\text{K},\text{H}_3\text{O})\text{Al}_2\text{Si}_3\text{AlO}_{10}(\text{OH})_2)$, calcite (CaCO_3), lime (CaO), microcline (KAlSi_3O_8) and gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$). Minerals containing Mg generally do not participate in the geopolymer process.

All samples of FA considered were mixed with a soluble potassium silicate solution, water and a small amount of blast furnace slag and cured at room temperature. Because of the complex chemical reactions and the effect of many parameters on their outcome, it is difficult to fully identify the role of each of alternative FA sample on final material performance. To this end, compressive strength was used for determining indirectly the reactivity of the source materials and the suitability of FA. It was generally observed that the high glass content, well-graded fine or intermediate grain size distribution are favourable characteristics for geopolymerization whereas high amounts of magnetite, and unburned carbon and presence of sulfides hinder the process. It was also reported that FA-F with higher amounts of Ca in the form of free lime have an increased risk of flash-setting (Nugteren et al., 2009), whereas FA-C cannot be used as a geopolymer precursor due to the lack of suitable minerals. It was recommended that FA-C be avoided because the presence of calcium in high amounts may interfere with the polymerization process and may alter the microstructure (Gourley, 2003).

- *Blast Furnace Slag*

This industrial waste of the steel production industry has been used as a SCM in concrete for more than 80 years as it yields high early strength. For every ton of manufactured metal, 0.2-0.4 tons of slag is produced (Yuksel, 2018). With the global steel production on the rise, according to the United States Geological Survey, it is estimated that 16 Mt of ferrous slag was produced in 2018, alone (van Oss, 2019). Today, it is considered a proper aluminosilicate source material for geopolymers.

Blast Furnace Slag (BFS) is a non-metallic by-product of the ore smelting process comprising silicates and aluminosilicates of calcium and other alkalis. It is characterized by moderate-to-high alkalinity (pH in the range of 8-12). Despite the presence of sulfur in its composition in small amounts, there is no corrosion risk for the embedded reinforcing steel in concrete consisting of slag due to the relatively high pH environment effected in the material. The methods of cooling the molten slag categorize this by-product in different types. Air-cooled blast furnace slag, expanded slag, pelletized slag and granulated blast furnace slag are some of these types. The last one is non-crystalline and formed when molten BFS is rapidly chilled through immersion in water (Mehta & Monteiro, 2014). Further modification of this type by grinding to the fineness of cement leads to Ground Granulated Blast Furnace Slag (GGBFS), which is suitable as a geopolymer binder. As with FA, the factors that control the reactivity of slag are, chemical, mineral composition, glassy phases content and fineness.

Generally, only highly amorphous slags could be used as a geopolymer precursor. The small particle size in the range of 5 to 25 μm increases the specific surface area and thus the reactivity. GGBFS grains are angular and irregular resembling fractured glass particles possessing a high CaO content (bound in minerals and not in free lime form), with silica and alumina and magnesia to follow. Chemical composition in this form is the most common information slag manufacturers provide, but it is not enough to determine whether the slag is appropriate for geopolymers.

Mineral composition is critical in determining the suitability of a BFS as a geopolymer precursor. The BFS should be gehlenite based (i.e., the glassy content should consist of the mineral gehlenite, $2\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$). Gehlenite and akermanite ($2\text{CaO}\cdot\text{MgO}\cdot 2\text{SiO}_2$) form a solid mixture which is known as the mineral melilite. Another mineral usually found in slag is merwinite

($3\text{CaO}\cdot\text{MgO}\cdot 2\text{SiO}_2$). Amongst gehlenite, akermanite and merwinite, the first is the most reactive due to the fact that Al is more favorable for geopolymerization compared to Mg. Figure 2.3 shows the molecular structure of melilite and merwinite and it is clear that Ca^{++} is captured in the molecular structure of the mineral and not free to react.

Despite the high reactivity of BFS, this alumino-silicate source material does not have the right mineralogy to be used as the sole or as the main precursor in a geopolymer binder. The absence of stable molecular structures found in MK mainly, but also in FA, which provide stability, hinders the formation of the 3-D geopolymer network and thus the final product is incomplete as it consists of chains and not a stable geopolymer block. The final product falls under the classification of alkali activated material (AAM) and its chemistry resembles the chemistry of OPC. More specifically during the first step of geopolymerization, i.e., alkalination, the two basic minerals of melilite, gehlenite, akermanite and merwinite depolymerize into hydrates and precipitations of $\text{Al}(\text{OH})_3$, $\text{Mg}(\text{OH})_2$, $\text{Ca}(\text{OH})_2$ respectively, as shown in Figure 2.4a (J. Davidovits, 2020). These precipitations react with the CO_2 in the atmosphere yielding carbonates that accelerate the hardening process (Astutiningsih & Liu, 2005). Subsequently, those two products inter-react and form the first unit of alkalination, precipitations of alkali and some hydrate remains as shown in Figure 2.4b (J. Davidovits, 2020). The yielding product is alkali activated slag and despite the high strength that it provides, there is a high risk of efflorescence and reduced durability (eventual break down and disintegration). This is because the process of geopolymerization is incomplete and the alkali cation Na^+ is not chemically attached in the structure. Thus, when in contact with water, this cation may migrate and form free alkalinity. Geopolymerization will be facilitated and will continue if MK-750 or FA are added in order to provide stable molecular structures of Q species leading to the formation of the 3-D geopolymer network (J. Davidovits, 2020).

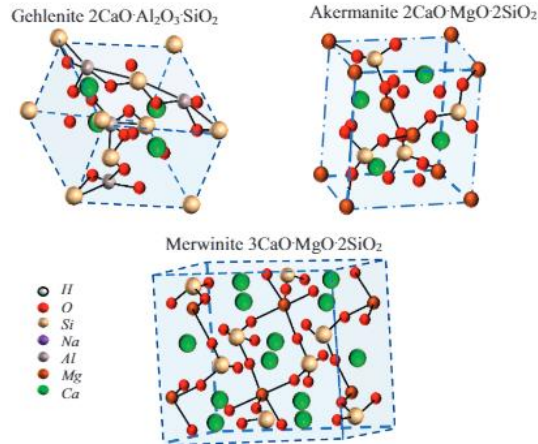


Figure 2.3: Molecular structure of gehlenite, akermanite and merwinite

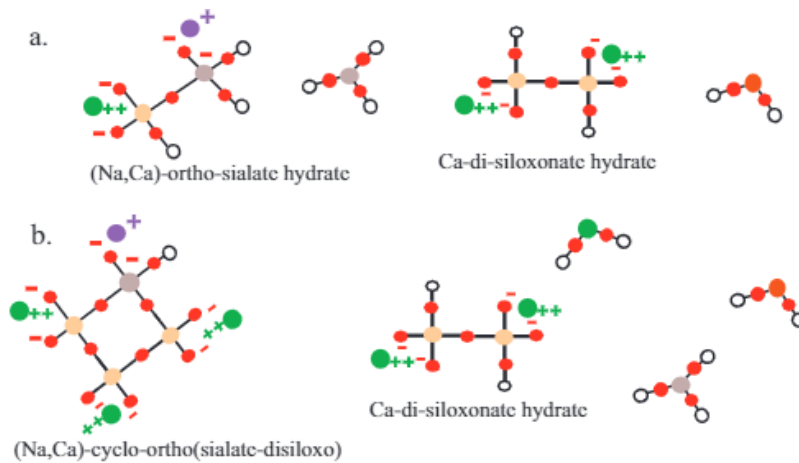


Figure 2.4: Alkalination of gehlenite, akermanite, and merwinite. (a) Depolymerization of melilite (b) formation of oligomer, CSH and alkali hydroxides precipitations

- *Other aluminosilicate precursors*

With the emerging emphasis on the use of alternative energy sources for sustainability, it is expected that coal burning will be eliminated eventually, and so the production of FA. On the other hand, despite of its abundance, BFS does not have the right mineralogy to serve as the main geopolymer precursor, so it cannot fully substitute fly ash. For this reason, various alternative aluminosilicate source materials have been explored in the literature. Ferronickel slag (FS) is an industrial by-product of melting nickel ore and bituminous coal in electric arc furnaces. The amount of produced slag in a ferronickel plant is 80-90% of the original source material (Komnitsas et al., 2007). FS is very fine with a mean particle size of 6.59 microns and is mainly X-ray amorphous (Maragkos et al., 2009); its main difference from iron BFS is the very low content of CaO and the high content of FeO. It has been shown that FS could be used as a

geopolymer precursor, yielding a high compressive strength (70-118 MPa) and low water absorption (0.7-0.8%) (Komnitsas et al., 2007). However, the high iron oxide content indicates that the geopolymerization mechanism is quite different from the alumino-silicate source materials described so far.

Another available geopolymer precursor is Red Mud (RM), the major industrial by-product resulting from the Bayer process, used to extract alumina from bauxite ores. In fact, the amount of this residue is greater than the amount of produced alumina. Currently RM is disposed in a slurry form into on-site waste lakes for further dewatering, consolidation, and storage, which is a costly option due to mandatory environmental monitoring and the need for long-term maintenance. This source material contains up to 60% of its mass in oxidized iron (responsible for the red colour of RM). The minerals containing iron are maghenite, haematite and goethite. In addition to iron, RM is mainly composed of silica, unleached residual aluminum, and titanium oxide. The resulting geopolymer matrix consists of ferro-sialate links and this concrete belongs to the category of ferro-sialate geopolymer concrete.

Calcined kaolinitic shale residues have been also studied as a geopolymer alumino-silicate source materials as their chemical and mineral composition is found to be very similar to the feeding material (Bortnovsky et al., 2005). When mixed with potassium silicate (molar ratio ranging from 1.2 to 1.66) and sand as fine aggregate, high compressive strengths may be attained. Another possible precursor is Rice Husk Ash (RHA), a silica rich agricultural by-product containing 90-95% by wt. amorphous silica. The high reactivity and the substantial pozzolanic properties of RHA, makes it suitable precursor geopolymer concrete. So far, the most common method of RHA disposal is in landfills with many environmental implications. As the annual amount of RHA is excessive, valorization of this by-product in geopolymers is an opportunity; however, the potential inhalation hazard of the crystalline silica content presents health risks (Prabu et al., 2014). Apart from the prevailing use of RHA in products of soluble silicates, Rattanasak et al., (2011) also used RHA in conjunction with $\text{Al}(\text{OH})_3$ powder as a geopolymeric binder.

Some natural rock minerals may also be used as geopolymer precursors such as volcanic ashes and weathered rocks. Volcanic ashes (pumice and scoria) are remains of pulverized rocks, minerals and volcanic glass created during volcanic eruption (Djobo et al., 2016). Partial

replacement of MK, FA or BFS with selected volcanic ashes (further calcined or not) produces geopolymer cement with enhanced properties and lower CO₂ emissions than the simple FA-BFS based geopolymer cement. On account of the lower reactivity of rock-based cements, they have not been tested in full replacement of one of the main geopolymer precursors. Reactivity depends on the loss of mass on ignition and the amount of clay minerals in volcanic ashes.

Recent studies by J. Davidovits (2020) illustrated that weathered and finely-ground rocks (either calcined or weathered) could also be used as geopolymeric precursors particularly if containing high amounts of iron oxides (such as granite, gneiss, basalt, and gabbro). Two types of weathered rocks were investigated both being residues of basalt. The samples were calcined at 750 °C for 3 hours and then ground down to a mean particle size of 10-25 microns, followed by mixing with potassium silicate to yield geopolymer pastes that reached a compressive strength of 80 MPa. The mineralogy of the weathered rocks is listed on Table 2.3.

Table 2.3: Mineralogical composition of lithomarge and saprolite samples (%) (J. Davidovits, 2020)

Type	Quartz	Kaolinite	Hematite	Goethite	Plagioclase	Pyroxene	Gibbsite	Montmorillonite	Other*
Lith.	12	45	30	3	-	-	-	-	10
Sapr.	3	35	18	3	15	10	6	5	5

*: Anatase, ilmenite and olivine

Most of the aforementioned materials are by-products, an attribute that is beneficial from a sustainability perspective. A major drawback is the unknown mineralogy, physical and chemical properties which vary around the world. For this reason, the suitability of any non-standard precursor should be evaluated through a series of tests, with particular interest in the determination of microstructure, chemical composition along with mineral phases and pH value. The last is very important as high pH values indicate increased risk of flash setting (J. Davidovits, 2020).

2.1.6. Soluble Silicates

Soluble alkali silicates are also known as hardeners in geopolymer chemistry, as they serve as the trigger of the geopolymerization mechanism. The key physico-chemical properties of these essential materials are presented in this section, to highlight their role in the process.

Silicates are the largest, most interesting, and complicated class of minerals. With oxygen and silicon being the two most abundant elements in this planet, the fact that 90% of the Earth's crust is made up of those is no real surprise. Due to the wide range of applications, silicates could have a liquid, powder, or granular form. The combination of their wide availability, low cost and user-friendly nature makes them suitable for the geopolymerization of various aluminosilicate source materials.

Nowadays, silicates are produced mainly through either the furnace route or the hydrothermal process. In the first method, waterglass is made by melting primary sand and sodium (Na_2CO_3) or potassium carbonate (K_2CO_3) at temperature between 1350-1450 °C. The product is dissolved in water to form a solution. The hydrothermal process requires an autoclave, by means of which direct dissolution of siliceous materials like sand occurs in caustic soda (NaOH) or in potassium hydroxide (KOH). Another less common method is the dissolution of silica fume in alkaline solutions (J. Davidovits, 2020). The method is not common for it is relatively expensive and limited for mass production of geopolymers.

Regarding chemical composition, silicates are basically combinations of alkali metal oxide M_2O , silica SiO_2 and water. The alkali metal (M) could be either Sodium (Na), Potassium (K), Lithium (Li), Magnesium (Mg), Calcium (Ca) or Cesium (Cs), the first two being the most common. The general formula is $t\text{SiO}_2:\text{M}_2\text{O}:u\text{H}_2\text{O}$, where t is the degree of polymerization and u the number of water molecules (Kriven, 2017). These two parameters are very important for choosing the right soluble silicate for the manufacture of geopolymer concrete.

Commercially available silicates are defined by similar parameters, namely, the weight ratio, WR, and the molar ratio, MR. A common mistake is to identify those ratios as the same. WR (also called Modulus) refers to the weight parts of SiO_2 to weight parts of M_2O , whereas MR refers to moles of SiO_2 to moles of M_2O contained in the silicate. Commercially available silicates have a MR ranging from 0.4 to 4.0, with silicates having values under 1.45 being very corrosive and unstable due to the high causticity and the lack of three-dimensional structure. On the other hand, high MR values such as 3 and 4 are less reactive and could be lowered by dissolving sodium or potassium hydroxide pellets into the silicates. It should be noted here that this process should be done in an autoclave due to the high instability of the solution and is generally not recommended

(J. Davidovits, 2020). Other properties that are crucial for choosing a soluble silicate are viscosity, causticity, pH value, solubility, and dissolution speed. All of these are influenced mostly by MR, density, and temperature. In fact, increasing the ambient temperature leads to an increase of reactivity and a reduction of viscosity. However, MR seems to play a critical role in the physico-chemical properties of the silicate. For high MR, viscosity, cold sensitivity, chemical resistance, and loss of water are higher, while at the same time alkalinity, pH buffer, solubility and reactivity are lower.

Due to the limited availability of commercial silicates made for geopolymer applications, different alkaline reagents have been used in the literature. The most common is blending a standard commercially available soluble silicate of low reactivity, with a hydroxide solution 24 hours prior to mixing. The advantage in this method is the flexible adjustment of MR, but the final product is unstable and not ideal for controlling repeatability of the product when used in the geopolymer. Another alternative is a solution of silica sol and hydroxide. Generally, a commercially available soluble silicate with MR around 1.5-1.7 is preferred as it is user friendly, reproducible, and stable (J. Davidovits, 2020).

As mentioned before, MR plays a significant role in the selection of the right soluble silicate as it affects almost all the properties of the alkaline reagent. The higher the MR the more stable the silicate is. This is related to the depolymerization degree of the oligomers in solution. The higher the MR, the higher the concentration of high order Q species, the strongest the structure of the soluble silicate and thus the lower the reactivity. However, very low MR indicates the presence of low order species which are more reactive but also very unstable due to their simple structure.

The most common silicates found in the literature, due to their wide availability, are Potassium (K) and Sodium (Na) -based silicates. Na-silicate has prevailed due to its low cost and the ability to yield higher compressive strengths as compared to K-silicates. However, the lower atomic mass of Na (22) compared to K (40) is not beneficial, as Na ions migrate more easily and might cause durability issues such as efflorescence and leachates. Another problem of Na-silicate is that the viscosity is highly dependent on temperature as compared to K-based silicates (Figure 2.5). This might result in the use of higher amounts of water to increase fluidity, thereby increasing the w:b ratio, and therefore the porosity of the resulting solid structure.

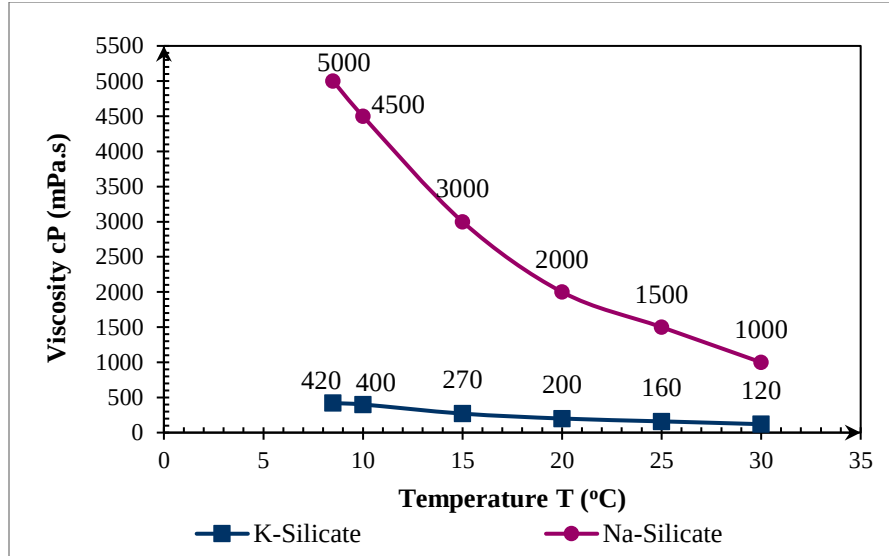


Figure 2.5: Viscosity of Na-silicate (MR=2) and K-Silicate (MR=2.2) solutions, same concentration, as a function of temperature (modified from J. Davidovits, 2020)

2.1.7. Aggregates and Fillers

Aggregates and fillers are a crucial component of conventional concrete as they provide strength, volume stability, workability, lower cost (60-70% of the total volume) better packing and prevent drying shrinkage. There is a range of options, from well graded coarse and fine with angular or rounded shapes that are inert in terms of reactivity, to lower the risk of Alkali Aggregate Reaction (AAR). Inert aggregates are conventional gravel, river, and silica sand. They can also be used in geopolymer concrete, at the same volume (i.e., up to 75% by volume as in conventional concrete) and they have the same role in the resulting solid. However, since the geopolymerization is a completely different mechanism, there is no risk of AAR, and thus the use of partially reactive aggregates has been encouraged. In fact, aggregates with reactive surface and inert core, such as waste sands, enhance the geopolymer matrix, as they form a better bond with their reactive surface. However, the research community is still suspicious of the use of partially reactive aggregates due to AAR risk and most of the available work in the literature has been conducted using conventional, inert aggregates. A less common inert filler used in geopolymer matrix is quartz sand. Incorporation of quartz sand as an aggregate, results in better mechanical strength and more stable microstructure as compared to river sand (Wan et al., 2017).

Feldspar rock, granite, basalt, and zircon waste are some partially reactive aggregates that have been used at the industrial level and have shown better performance results compared to standard

concrete or silica or quartz sand (Reggiani, 2019). Although in conventional concrete granulometry and optimal packing are the key factors, in geopolymer concrete except from those, mineralogy also plays an important role. The partially reactive aggregates mentioned have a high concentration of the mineral feldspar which is rich in K or Na.

2.1.8. Additives

As in conventional concrete, additives in geopolymer concrete are used to enhance the performance of the composite in terms of strength and ductility or to impart to it new properties such as antimicrobial action, self-sensing, self-cleaning and self-healing abilities. Additives that enhance geopolymer systems with various properties other than enhanced durability and performance are presented only for reference to the current state-of-the-art on geopolymer concretes. Besides Silica Fume, fibers, chemical admixtures, nanomaterials, and healing agents have been studied. For example, carbon nanomaterials have been used to enhance the mechanical properties (Abbasi et al., 2016; Yan et al., 2015) or to impart new properties such as self-sensing (Bi et al., 2017). TiO_2 is another nanomaterial that has been explored for its photocatalytic properties (Falah & MacKenzie, 2015). Nanoclay has also been incorporated in geopolymer matrices and results showed enhanced matrix density and prevention of fiber disintegration as it lowers the alkalinity of the matrix (Assaedi et al., 2017). Finally, another interesting nanomaterial that has been tested in geopolymer mortars is silver nanosilica which exhibited strong antimicrobial action (Adak et al., 2017). Bacteria based healing agents have been tested to impart self-healing abilities for the geopolymer mortars (de Koster et al., 2015; Jadhav et al., 2018). Of the various additives, considered, the most prominently used in geopolymer concrete technology are discussed in greater detail in the following section.

- *Silica Fume/ Microsilica*

Silica Fume (SF), also known as microsilica, volatilized silica or condensed silica fume, is a by-product of the manufacture of silicon and ferrosilicon alloys - such as ferromagnesium, ferromanganese, ferrochromium, and calcium silicon - from high-purity quartz and coal in a submerged-arc electric furnace (Neville, 2012). The fine spherical particles of non-crystalline silica (SiO_2) is the reason why silica fume is highly reactive and improves the packing of the binder. It can be used as part of the geopolymeric binder in either a slurry, densified or

undensified form. Studies have shown that the addition of silica fume enhances the compressive strength of geopolymer mortar and concrete (M. Al-Majidi et al., 2016). Especially for mortars, undensified silica yielded higher strength, whereas silica fume in slurry form demonstrated lower strength gain due to agglomeration of the particles. Both types reportedly reduced the workability of the mix (M. Al-Majidi et al., 2016). Besides the above applications, microsilica has also been used in conjunction with alkali oxides (Na_2O or K_2O) to prepare in laboratory conditions soluble silicates (Trindade et al., 2020).

- *Fibers*

Geopolymer concrete is brittle in tension and can only be used in reinforced structural members. Fiber reinforced geopolymer concrete has been developed in recent years. The role of fibers is the same as in fiber-reinforced concrete; by virtue of bridging the cracks, fibers increase ductility, tensile strength, and strain capacity. A variety of fiber materials have been tried, such as wood, flax and hemp fibers, Polyvinyl Alcohol (PVA) (Al-Mashhadani et al., 2018) steel (M. H. Al-Majidi et al., 2017a) and basalt fibers (Dias & Thaumaturgo, 2005).

- *Chemical Admixtures*

Plasticizers that have been used in geopolymers are usually carboxylate-ether based and lignosulphonate or naphthalene sulfonate based. The first are very effective with advanced OPC concrete such as UHPC. However, the organic polymers of such a plasticizer may hinder geopolymerization and thus strength development. On the other hand, naphthalene sulfonate-based plasticizers generate small bubbles that increase workability without interfering with geopolymerization. Plasticizers may be avoided by using extra drops of water at the end of the mixing. As with conventional concrete the optimal dosage is in the range of 1 to 1.5% wt of the geopolymeric binder (J. Davidovits, 2020). Except from workability, another issue of geopolymer concrete is the quick setting time due to the high alkalinity of the binder. Whereas in conventional concrete, retarders are used to prolong the hardening, they are not effective in geopolymer concrete and might even cause erratic behaviour because they are based on hydration mechanism. Some salts have been used in the literature with the dipotassium phosphate (K_2HPO_4) to be efficient in terms of delaying the hardening but significantly reduce the compressive strength due to the weakness of the apatite mineral formed (Nugteren *et al.*, 2009); using ice water or freezing

the binders before mixing has also been suggested and tried by the author with notable success (Reggiani, 2019).

2.1.9. Curing Conditions

Geopolymer composites develop their strength via curing. It begins by covering in plastic sheets the specimens right after casting to prevent shrinkage due to water evaporation, for 24 hours. Although the chemical mechanism of geopolymer concrete consolidation is not hydration, it is crucial for the water in the fresh mix to remain and participate in the formation of the three-dimensional geopolymer network. If left uncovered, the specimen will start slowly to lose its strength. After the first 24 hours, the specimens could be air-cured at ambient temperature, water-cured or oven-cured for 2 hours at 70 °C. The last yields the highest possible strength, but it is not practical for large scale or on-site casting. Water and air curing yield almost the same results with water-curing showing sometimes higher strength, due to remaining unreacted pozzolan.

2.1.10. Fresh Properties

Geopolymer concrete has problematic workability. However, the desired workability of fly-ash based geopolymer concrete could be achieved by modifying slightly the water content in the mixture by adding a few extra drops of water in the amount of 1-2% of the binder (Hardjito et al., 2004). The addition of extra water should be handled with care as excessive amount in the geopolymer mixture can reduce the compressive strength as it promotes high porosity. A factor that affects significantly the workability is the addition of Silica Fume as an additive. This is a very fine material (i.e., it has very specific high surface area), and despite the superior properties that it provides, it also increases dramatically the mix's viscosity and should be handled with care (Al-Majidi et al., 2016).

The setting time of fresh geopolymer could be determined once the pH value is known. Thus, it highly depends on the chemical composition and especially on the amount of alkalis. The addition of a small amount of MK-750 in a FA-based geopolymer with medium to high alkalinity, prolongs the setting time. On the other hand, geopolymers made of by-products rich in CaO, such as GGBFS, exhibit even shorter setting time ranging between 15 to 45 minutes at 60 °C (Cheng & Chiu, 2003). But when using fly ash with low alkalinity as source material, the geopolymer

concrete did not show any sign of setting after 120 minutes at room temperature (Hardjito & Rangan, 2005).

2.1.11. Hardened Mechanical Properties

Material characterization through determination of the mechanical properties of a material is the first step of evaluating its performance. The very first published study on geopolymer concrete with determined mechanical properties was developed by Rangan et al., (2005). The heat-cured fly ash based geopolymer concrete comprises fly ash, sodium silicate, water, some superplasticizer, fine sand, and granite coarse aggregates. The highest compressive strength 60 MPa was achieved for water/geopolymer solids ratio of 0.16 but it was very stiff mix, while the lowest of 30 MPa was achieved with water/geopolymer solids ratio of 0.24 and high workability. Regarding modulus of elasticity, it was found to be around 27000 MPa for a 60 MPa compressive strength while Poisson's ratio is lower than that of conventional concrete, around 0.17. Later on, Wallah & Rangan, (2006) also studied the creep and drying shrinkage on the same mix design. The results showed that the heat-cured fly ash-based geopolymer concrete undergoes very little shrinkage in the range of 100 microstrains after a year, which is significantly smaller than 500-80 microstrains observed in OPC concrete. Finally, the creep coefficient for a geopolymer concrete with a compressive strength of 67 MPa, the creep values were between 0.4-0.5. For lower strengths (40 MPa) the coefficient was 0.6-0.7.

Table 2.4 lists a few of past experimental studies on Fiber Reinforced Geopolymer Concretes. The majority focuses on evaluation of compressive and flexural strength with some of them investigating modulus of elasticity and tensile strength. Yet, so far there has been no complete material characterization study on fiber reinforced geopolymer concretes and especially in THFRGC, as such a development has not been achieved yet.

Table 2.4: Studies on mechanical properties of Fiber Reinforced Geopolymer Concrete/Mortars

Reference	Details/ Parameters	Compressive Strength f_c (28d)	Tensile Strength f_t	Modulus of Rupture f_r	Young's Modulus E_c	Observations
(Al-mashhadani et al., 2018)	PVA, PP and Steel Fibers	60-63	-	7-11	-	Improved Flexural strength due to the addition of fibers
	Different fiber content					PPs and PVAs reduce shrinkage

	FA/BFS					Fibers improved abrasion performance
	Heat curing at 80°C for 24hr					
(Guo & Pan, 2018)	PP, Basalt and Steel Fibers	30-40	-	6-8	-	Fibers improves overall performance
	Different fiber content					Steel fibers performed better than Basalt and PP
	FA/Steel Slag					
(M. H. Al-Majidi et al., 2017a)	Steel fibers	20-45 plain 20-65 reinf.	1.7-3.3	-	2000 - 15000	Enhanced mechanical properties with increase of slag content
	FA/BFS/SF					Densified SF had a negative effect on strength development
	Different forms of SF					Post cracking behaviour and energy absorption capacity improved due to inclusion of fine SF
	Ambient curing					
(Korniejenko et al., 2018)	Flax and Hemp Fibers (1% wt)	30.72-42.70	-	4.67-5.09	-	Hemp fibers outperformed flax fibers
	Different forms of Fibers					Decorticated hemp fibers and microfibers yielded the highest mechanical properties
	FA					
	Heat curing at 75°C for 24 hr					
(M. H. Al-Majidi et al., 2017b)	Strain Hardening	42-69	1.6-3.8	2.5-7 (1 st crack) 3-12 (2 nd peak strength)	9855 - 24885	Addition of fibers increased compressive strength by 15-20 MPa
	PVA, Steel and Glass Fibers					Steel fibers performed better in terms of mechanical strength and post-cracking behaviour
	Various Volume Fractions and Aspect Ratios					3% of 13mm straight long steel fibers yielded compressive strength of 70MPa
	FA/BFS/SF					Hybrid reinforcement enhance mechanical performance
	Ambient curing					
(Nematollahi et al., 2017)	Strain Hardening Composite	8.8-54.6	0.25-0.75 (1 st crack) 1.7-4.7 (ft _u)	-	1800 - 11300	0.34-4.3% tensile strain capacity
	Oil-coated PVA fibers					Average residual crack width:24-83mm
	FA					Na-based alkaline solutions and extra water improved strength

	Heat curing at 60°C for 24h					High chemical bonding of uncoated PVA with geopolymer matrix leading to delamination
	Na-based, K-based, and lime- based alkaline reagents					Proposed oil coating yields lower chemical bonding and protects the fiber leading to pull-out failure

2.1.12. Differences Between Geopolymer Concrete, AAM and OPC

Alkali Activated Materials and zeolites are often referred to in current literature as synonyms to geopolymers. However, these are completely different technologies, with different hardening mechanisms, raw materials, mixing procedures and resulting products. In this section a review of the fundamental differences between these technologies is presented to mitigate some of the common ambiguities regarding the relationship between these technologies.

Geopolymer chemistry is motivated by the natural geosynthesis processes. Its objective is the creation of polymeric alumino-silicate chains that are connected so as to form three dimensional polymeric networks similar to rocks. This is achieved by partial dissolution of alumino-silicate source material particles in a user friendly alkaline or acidic medium. On the other hand, the scope of zeolite and AAM chemistry is the creation of hydrates similar to those found in OPC via total dissolution of the particles of the precursor in highly alkaline and caustic medium. The user hostile alkaline medium which could be an alkali metasilicate or any alkali silicate with MR lower than 1.4 might yield a high compressive strength. However, the system has free alkalinity, a fact that creates stability and durability problems.

The mechanism of AAM resembles the hydration mechanism of OPC where, instead of Calcium Silicate Hydrates (CSH) in form of gel, there are potassium or sodium aluminosilicate hydrates (K-A-S-H or N-A-S-H). Those products are completely different and chemically less stable than geopolymer block units. Cement hydration technology yields more stable hydrates since Ca is insoluble in water, whereas alkali-activation hydrates with K or Na instead of Ca are not stable, due to the high solubility of the alkali cations in water. Another difference lies on the selection of the aluminosilicate source material. In the case of geopolymers, careful investigation of the material is needed in terms of mineralogy, reactivity, and stability, whereas AAM could be

practically made of any industrial waste. This carries a risk in terms of predictability and repeatability of material behavior. A critical difference lies in the mixing process.

AAM concrete is made following the same mixing procedure as OPC. The process usually starts with a dry mix of alumino-silicate materials and aggregates, followed by the addition of soluble silicates along with the water and any admixtures, with total mixing time almost same with conventional OPC concrete mixing. Geopolymer concrete mixing process is completely different. Aggregates should not be mixed in conjunction with the geopolymer precursors as they function as barriers in the reaction between the aluminosilicate powders and the soluble silicate. The soluble silicate should be mixed with the extra water prior to mixing, preferably at least 24 hr before, in order to achieve highest stability. The process should start with the most important part of mixing the precursors with the silicate for at least 5 minutes depending on the mixer and its speed. Once the geopolymeric slurry is ready, the aggregates, preferably partially reactive are added and mixed for 2-3 minutes. Before adding any fibers in the mix, a few drops of water could be used in an amount not greater than 1 to 3% wt. of the binder if necessary, so as to ensure better workability. Finally, the effect of curing is an important indicator that the aforementioned technologies are different. Air and water curing could be used in geopolymer concrete technology yielding almost the same results in terms of strength. However, AAM are cured either by air or are oven-cured at temperatures of 60 °C not to accelerate the alleged geopolymerization but to harden by fixing the saturated alkali ions of the highly caustic alkaline solution. Concrete made of alkali activated materials does not possess mineral polymeric nature whereas the free alkalinity in the structure leads sometimes to efflorescence when cured in water.

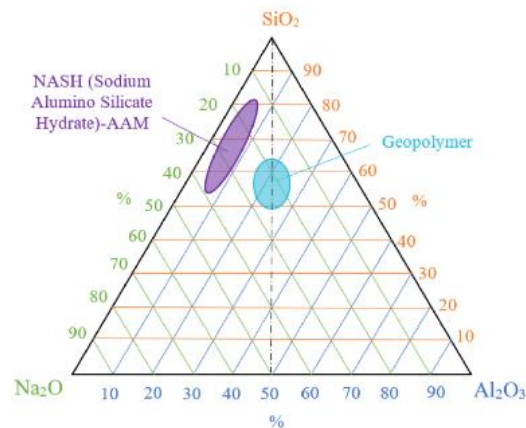


Figure 2.6: Ternary diagram of $\text{Na}_2\text{O}:\text{SiO}_2:\text{Al}_2\text{O}_3$ for comparison of AAM and Geopolymer

The differences between the two material classes are also apparent in terms of the durability properties of each system. Since the AAM target the dissolution of aluminosilicate source materials using highly caustic medium, their resulting structure is similar to OPC composites, as potassium or sodium aluminosilicate hydrates are formed. Figure 2.6 (Kriven, 2017) shows the difference between geopolymer and AAM in a $\text{Na}_2\text{O}:\text{Al}_2\text{O}_3:\text{SiO}_2$ phase diagram. Geopolymers always show the ratio 1:1:4 in EDS characterization technique which is not related to hydration products stating that geopolymers do not belong in the same category with AAM.

Leaching properties could be used as a durability index, as they show how chemically stable is the microstructure of a material. It has been found that AAM exhibit high mobility of oxyanionic species in a range of 5-50 times higher than geopolymers (J. Davidovits et al., 2014; Izquierdo et al., 2009). Alkali Activated Fly Ash demonstrates the highest leachates in terms of elements and contents. Geopolymer leachate elements are Zn, Ti and Rb with a maximum content of 10 mg/kg while the leaching elements for AAFA are Cd, Sb, Ba, Se, Cr, W, Li, Mo, As, V, B and Ca with highest leachate content of 100 mg/kg.

2.1.13. Durability

Historical geopolymer monuments are prime evidence that in terms of durability, geopolymer composites outperform conventional concrete and AAMs. In this section a review on fundamental durability aspects is given along with comparison to AAM and conventional OPC.

As mentioned before in the definition of geopolymers, geosynthesis can take place in an acidic medium. Thus, in the presence of acid, destruction of geopolymer chain is limited as compared to Portland cement and Ca-rich aluminosilicates such as slag. The % weight loss of geopolymer in the presence of strong acids such as HCl and H_2SO_4 is less than 20% while Portland cement is extremely susceptible to both acids with the loss of strength ranging between 80-100%.

Wallah et al., (2005) investigated the acid resistance of geopolymer concrete in sulfuric acid. The investigation comprises immersion of the samples in sulfuric acid solution of different concentrations for 24 weeks. Their results have shown that after 24 weeks, 0.5% concentration had almost no effect on compressive strength whereas specimens submerged in 1 and 2% sulfuric acid lost around 35% and 60% of their compressive strength, respectively.

- *Sulphate Resistance*

Another important durability index is the sulfate resistance. OPC is highly susceptible to sulfate attack due to formation of expansive gypsum and ettringite, which subsequently leads to expansion and cracking (Neville, 2014). However, the chemical mechanism of geopolymers is completely different from OPC as no gypsum or ettringite is formed. Some slight risk exists in case of Ca-rich binders (Rangan, 2009).

A part of the GEOCISTEM project (J. Davidovits et al., 1997) where geopolymer concretes were produced and compared to mortar samples from Roman concrete monuments, was intended to assess sulfate resistance. The test was performed on geopolymer cement and CEM-I 42.5 R according to ASTM C1012 (2018). The results showed that geopolymer cement underwent no expansion, but instead, it shrunk slightly whereas the dimensional change for the OPC binder was up to 0.02%.

Sulphate resistance of geopolymer concrete was also investigated by Rangan (2009). The heat-cured low calcium fly-ash based concrete was immersed in a 5% sodium sulfate solution for different exposure periods. Change in mass, length and compressive strength were used for assessing the sulfate resistance. The results showed that geopolymer concrete has superior resistance to sulfate attack as even after a year of immersion in the solution, no signs of erosion, cracking or spalling were detected on the specimens. In fact, it is very interesting that the immersion caused a slight increase in compressive strength.

- *Alkali-Silica Reaction (ASR)*

Owing to the high content of alkalis used in mixing geopolymers there is an anxiety regarding the risk of alkali-silica reaction in structures. The alkalis and hydroxyl ions from Portland cement paste react with reactive siliceous minerals in aggregates and form an expansive gel that causes cracks. However, no free hydroxyls or alkalis occur in geopolymerization as they are locked in the stable 3-D geopolymer network; in this manner, the siliceous minerals of aggregates are enhancing the chemical bonding of aggregates with the binder, thereby eliminating the risk of ASR. The fear of ASR combined with the lack of in-depth knowledge of geopolymer chemistry in the civil engineering research community delayed the development of geopolymer concrete.

This is why, even though the first applications are dated back to the 70s, organized research on geopolymer concrete started just a few years ago.

To address the concerns of concrete industry about the risk of ASR in geopolymer concrete, Davidovits et al. (1997) performed accelerated expansion tests in saturated NaCl baths. Based on the reported results, geopolymer binders outperformed conventional OPC; even with alkali content as high as 10% the exposed geopolymer materials did not generate dangerous ASR. Additional supporting evidence may be found in the experimental study of (K. L. Li et al., 2005), where a geopolymer mortar and OPC mortar were tested according to ASTM C441, (1997) standard test for ASR. Results showed a maximum change in dimension by -0.02% for the geopolymer mortar while cement mortars exhibited an expansion of almost 0.1% at the same age of 300 days.

- *Corrosion*

Geopolymer concrete is not as highly caustic as AAMs which develop pH values between 13-14. In fact, the range of geopolymer cement pH is between 11.5 to 12.5, which is very close to that of Portland cement's (12-13), and therefore the two materials are usually effective means of reinforcement protection. Several researchers have found that a stable passive protective film is formed around the reinforcement when embedded in a geopolymer matrix with high alkalinity (around pH 12) once dissolved silicates stabilize (Morris & Hodges, 2005).

- *Carbonation*

Carbonation increases the risk of corrosion by lowering the alkalinity of OPC. The reaction of carbonation occurs when certain hydrates (Ca(OH)_2) along with free hydroxyl ions react in presence of CO_2 and form CaCO_3 . Carbonation occurs also in geopolymer concrete but is different from OPC in terms of the formed products. Whereas in the case of OPC the product is calcium carbonate with a pH around 7-8, in case of geopolymer concrete the carbonation products are potassium or sodium carbonate, which are more alkaline than CaCO_3 , as their pH ranges between 10-10.5 (J. Davidovits, 2020).

- *Freeze-Thaw and Wet-Dry Behavior*

Besides the aggressive chemical conditions that highly affect the durability of building materials, structures in cold-climate regions undergo freeze-thaw during the winter periods. This effect has not been examined thoroughly in the literature. However, one study has illustrated that geopolymer concrete with Micro-encapsulated Phase Change Materials (PCMs) show better performance when undergoing freeze-thaw cycles as compared to the counterpart conventional concrete (Pilehvar, 2018). J. Davidovits (2020) proposed the freeze-thaw and wet-dry characterizations as mandatory tests for evaluating the durability of geopolymer concrete. Experiments on MK-750-based geopolymer concrete have shown that after 50 freeze-thaw cycles and 50 wet-dry cycles the strength loss was by 22% and 11%, respectively.

2.1.14. Structural Performance

Determination of structural behaviour of geopolymer concrete is essential for assessing the suitability of the material in structural members. This process will assist the comparison with conventional concrete and the development of codes if it is found that the material responds differently from OPC concrete. Research studies on structural behaviour of geopolymer concrete indicate that the response does not differ a lot from that of conventional concrete with the exception of bond strength which is found in some cases to be higher than in OPC concrete. Reviewing the current literature on structural response of geopolymer concrete is crucial for proving that geopolymer concrete could indeed replace conventional concrete in terms of performance.

- *Bond Strength of Reinforced Geopolymer Concrete*

Bond strength is one of the most crucial mechanisms that govern the structural behaviour of concrete. Through bond development, composite action between concrete and reinforcement is enabled allowing the transfer of forces between the two materials. Studies of bond in conventional concrete are focused on parameters such as the effect of corrosion, effect of compressive strength, bar diameter, cover, volume of steel fibers, embedded length, and splice length. Studies on bond development in reinforced geopolymer concrete include the aforementioned plus some additional parameters such as the effect of soluble silicate concentration, or the type of curing. Table 2.5 summarizes investigations on bond strength of reinforced geopolymer concrete. The most

common method to evaluate bond strength is the standard direct pull-out test and has been adapted by many Testing Standards around the world. In the RILEM RC 6 test, the specimen comprises a cubic block with side equal to 10 times the diameter of the concentrically placed embedded bar (RILEM/CEB/FIB, 1978). The bar is pulled against a fixed supporting plate under the test surface. Despite the tests' simplicity, it is known to produce unconservative estimates of bond strength since the longitudinal compressive stresses exerted by the supporting base on the cover around the bar spuriously enhances the bond strength thereby leading to overestimated bond strength values. Other alternative tests that have been performed for evaluation of bond is the beam-end test (RILEM, 1982) and the splice test (where the longitudinal tension reinforcement of a flexural beam occurs in the central constant moment region under 4-point loading of the beam); both of these tests are considered as more conservative than the standard pullout test since the region around the bar is under tensile longitudinal stresses. Again, there are drawbacks however, either because the response is affected by the vertical reaction over the support where the bar is anchored, or by flexural curvature in the splice test. Figure 2.7 shows the three tests along with their developed stress states. Table 2.5 lists experimental bond stress values indicating the vast range of the results depending on the test conducted.

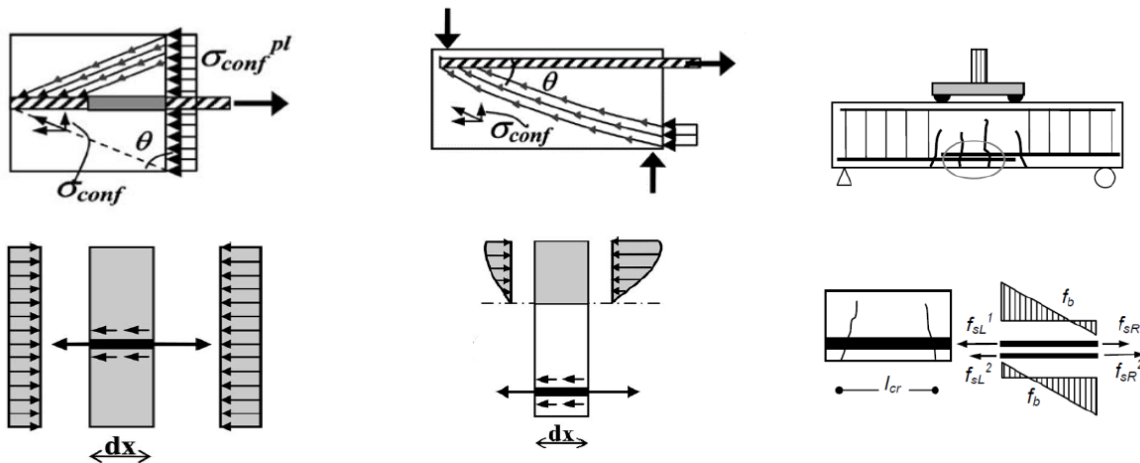


Figure 2.7: From left to right: Standard pull-out test, beam end test and lap splices tests (Tastani & Pantazopoulou, 2006)

Table 2.5: Bond tests studies

Reference	Bond Strength f_b (MPa)	Bond Strength $f_b/f_c^{0.5}$	Details/Parameters	Test Method	Observations/Contributions
(Chang et al., 2009)	3.59-8.77	0.65-1.34	Cover/Bar Diameter	Splice Test	Failure mode and crack patterns similar to conventional RC

			Concrete Strength		Bond strength related to tensile strength
			Splice Length		Current design provisions and analytical models are applicable
(Sarker, 2011)	10.61-19.42	2.10-3.09	Cover/Bar Diameter	Beam-end test	Failure mode and crack patterns similar to conventional RC
			Water Content		Geopolymer concrete yield higher bond strength than conventional concrete for the same parameters
			Embedded Length		Current design provisions and analytical models are applicable
(Ganesan et al., 2015)	12.73-16.57	1.95-2.58	Bar Diameter	Standard Pull-Out Test	Increase of fiber volume increases the bond stress
			Steel Fiber Volume		Bond stress is higher in specimens with larger bars even if the steel fiber percentage was low
			Embedded Length		
(Castel & Foster, 2015)	24.10-31.90	3.53-4.16	Curing Condition	Standard Pull-Out Test	Heat curing is beneficial for bond development
					Chemical adhesion of geopolymer matrix to steel reinforcement is similar to conventional concrete
					10% increase in bond strength of geopolymer concrete compared to conventional
(Al-Azzawi et al., 2018)	7.5-30	1.88-3.93	Alkaline Solution Content	Standard Pull-Out Test	High concentration of SiO ₂ in the alkaline reagent increases bond strength
			Fly Ash Content		Fly Ash reactivity highly affects the bond development
			Fly Ash Type		High Content of Fly Ash increases the bond strength
(Farhan et al., 2018)	16.46-27.32 ² 5.85-16.71 ³	2.57-3.98 0.91-2.43	Corrosion	Standard Pull-Out Test	Increase of fiber volume increases the bond stress
			Steel Fiber Type		Steel fiber reinforced specimens showed better corrosion resistance.
			Steel Fiber Volume		Addition of fibers improved bond strength
					Combination of different fibers yields higher bond strength

The results of the aforementioned investigations show the same trend, namely that development of bond strength in geopolymer concrete is similar to conventional concrete. However, a slightly better performance of geopolymer concrete's bond strength as compared to OPC concrete was reported by Castel and Foster (2015) for the same compressive strength. They also discovered that curing time and curing temperature have a strong impact on bond strength's development.

² Non-corroded specimens

³ Corroded specimens

Except from studies on bond strength of steel reinforced geopolymer concrete, there have been investigations on the bond strength between geopolymer concrete and Glass Fiber Reinforced Polymer (GFRP) bars. More details of these studies are summarized in Table 2.6.

Table 2.6: Bond tests on GFRP reinforced geopolymer concrete

Reference	Bond Strength f_b (MPa)	Bond Strength $f_b/f_c^{0.5}$	Details/Parameters	Test Method	Observations/Contributions
(G. Maranan et al., 2015)	19.39-23.96	3.37-4.17	Bar diameter	Standard Pull-Out Test	Increase in bar diameter reduces average bond strength
			Embedded Length		Shorter embedment length lead to pull-out failure while longer embedded lengths cause concrete splitting
					Similar bond strength to that of steel-reinforced geopolymer
(Tekle et al., 2016)	9.60-19.60	1.51-2.79	Bar Diameter	Standard Pull-Out Test	Increase of embedment length results in decrease of bond strength
			Compressive Strength		Similar bond strength to that of GFRP-reinforced OPC concrete
			Embedded Length		

- *Reinforced Geopolymer Concrete Beams-Behaviour in Flexure & Shear*

Flexural and shear strength are two basic indicators of structural behaviour of concrete and essential for the design of a structural member. Several experimental studies on reinforced geopolymer concrete beams have been conducted investigating flexure and shear through altering parameters such as reinforcement ratio, presence or absence of stirrups, span/depth ratio, use of microfibers and GFRP bars. The majority of observations show high similarity to conventional reinforced concrete flexural and shear response. A series of investigations along with their details and observations are enlisted in Table 2.7.

Table 2.7: Structural behaviour of reinforced geopolymer concrete beams

Reference	Details/Parameters	Observations/Contributions
Flexure		
(D. M. J. Sumajouw et al., 2005)	Longitudinal Tensile Reinforcement Ratio	Increase of longitudinal reinforcement ratio and compressive strength lead to increase of flexural capacity
	Concrete strength	Similar flexural behaviour to OPC concrete beams
		Design provisions of Australian Standard for concrete structures apply
(Ferdous et al., 2015)	GPC filled pultruded composite beam for use as light-duty railway sleeper	Failure is governed by compressive failure of concrete and buckling in the pultruded composites
		Satisfaction of minimum flexural requirements for railway sleeper
		Similar and even better performance than some existing composite and timber sleepers

(G. B. Maranan et al., 2015b)	Bar Diameter	Cracking pattern, resistance curve, bending moment and deflection capacity similar to conventional concrete with GFRP-RGPC showing higher strength than GFRP-RC
	GFRP bars	Flexural performance is independent of the nominal diameter of bottom longitudinal reinforcement
(Junaid et al., 2020)	GFRP bars	Similar behaviour of Fiber reinforced and plain GPC up to peak load with increased post-peak ductility in case of Fiber Reinforced GPC
	Polypropylene macrofibres	CFRP sheets restores the original moment capacity in cases of sufficient development length
	Four-point bending	CFRP strengthening enhanced stiffness and reduced ultimate deflection at CFRP failure
	Half beams undergone further damage and were repaired using CFRP sheets	For flat and U scheme, the failure was CFRP rupture
Shear		
(G. B. Maranan et al., 2015a)	Span/effective depth=1.8	GFRP stirrups improved shear strength by 200%
	Stirrup Spacing	Stirrup spacing influenced initiation of shear cracking with narrow spacing to be the most beneficial
	GFRP stirrups	Excess of web reinforcement did not have an impact on shear strength
	Slender beam (shear span ratio=4.7)	GFRP stirrups could replace steel stirrups Short beams showed higher shear strength compared to the slender beam due to strength enhancing effects of arching action in short beams
(Visintin et al., 2017)	No stirrups	Similar shear-friction properties to conventional concrete
	Span/Depth Ratio	Development of mechanics-based analysis for predicting concrete contribution to shear capacity
	Reinforcement Ratio	

- *Reinforced Geopolymer Concrete Columns and Walls*

Another less commonly investigated member response type in the field of structural behaviour of geopolymer concrete is that of columns. Fewer studies have been conducted as compared to beams and generally the results regarding the failure pattern and response resemble that of conventional concrete. Table 2.8 shows the most representative studies on reinforced geopolymer concrete columns. It is remarkable that again the members reinforced with GFRP showed superior performance as compared to OPC reinforced concrete (Maranan et al., 2015a). This could be explained by high adhesion of GFRP bars to the geopolymeric matrix.

Table 2.8: Structural behaviour of reinforced geopolymer concrete columns

Reference	Details/Parameters	Observations/Contributions
(M. D. J. Sumajouw & Rangan, 2006)	Monotonic Loading with Specified eccentricity	High load capacity results from reduced load eccentricity, high compressive strength, and high longitudinal reinforcement ratio
	Concrete strength	Similar crack patterns and failure modes with OPC reinforced concrete
	Longitudinal Reinforcement Ratio	Design provisions of Australian Standard for concrete structures apply

(D. M. J. Sumajouw et al., 2005)	Axial load and Uniaxial Bending Single Curvature Mode	High load capacity results from reduced load eccentricity, high compressive strength, and high longitudinal reinforcement ratio
	Compressive Strength	Similar crack patterns and failure modes with OPC reinforced concrete
	Longitudinal Reinforcement Ratio	Design provisions of Australian Standard for concrete structures apply
	Load Eccentricity	
(G. B. Maranan et al., 2015a)	Concentric loading	Hoop-confined slender columns failed at 66% of the strength of their counterpart short columns, while spiral-confined failed at 82%
	GFRP bars	GFRP bars contributed 7.6% of the total capacity
	Configuration of transverse reinforcement (no, circular hoop and spiral)	High volumetric ratio yielded better compressive behaviour regardless of the type of transverse reinforcement
	Short and Slender Columns	Spiral type demonstrated higher ductility compared to hoop type
		Superior performance compared to OPC GFRP reinforced columns
(Albitar et al., 2017)	Concentric and Eccentric Loading	Similar behaviour with Reinforced Concrete Columns
	Slenderness effect	Lateral deflection at midheight increases as the eccentricity increases
	Axial Load-Moment Interaction Diagrams	Both slender and short columns when loaded concentrically exhibit a brittle failure, while eccentric loading leads to initiation of flexural cracks and crushing on the compression face

So far, the aforementioned studies on reinforced geopolymer concrete structural members show agreement that structural response of geopolymer concrete is similar to conventional concrete. However, there are some interesting findings regarding the structural behaviour of reinforced geopolymer walls. In fact, experimental studies have shown that even for same load capacity, the lateral deflection of reinforced GPC walls under axial loading is higher than OPC reinforced concrete walls. In addition, more softening behaviour and compliance was observed in the case of GPC walls. According to the authors, the last is probably owing the high concentration of fine particles in the geopolymer matrix (Ganesan et al., 2014).

2.2. Tension Hardening Fiber Reinforced Composites

With the advancements in concrete technology, a class of novel materials with superior properties that has caught the attention of research community and is slowly incorporated in design procedures around the globe is Tension Hardening Fiber Reinforced Composites (THFRC). This section will explore THFRCs characteristics, constituents, and the importance and recommendations of tensile characterization of such materials.

2.2.1. Historical Background

The first attempt towards the realization of concrete with enhanced strength was made in the 20s with through Abrams' law (Abrams, 1918) which states that lower water to cement ratios result in higher strength due to a denser microstructure with lesser amount of air voids. However, the lack of special admixtures and the limited knowledge on particle packing theories in that time added extra challenges, such as poor workability. Almost two decades later, Freyssinet (1936) achieved improved strength by applying pressure to fresh concrete during setting, while in the 60s, heat combined with high pressure was employed as a curing method that enhances concrete strength (Richard & Cheyrezy, 1995). With the introduction of water-reducing admixtures and the commercialization of silica fume, the prospect of High Strength Concrete with low w/c became a reality. The above finding, combined with advancements in particle packing, and the use of hard calcined bauxite aggregates led to the development of DSP technology (Densified Systems containing homogeneously arranged ultra-fine Particles) by Bache in 1981 (Bache, 1981). DSP technology led to the development of concretes attaining strength above 200 MPa.

However, such high strengths did not eliminate the need for mass reinforcement, as the concrete continued to exhibit brittle failure in compression and tension. Romualdi & Mandel (1964) and Krenchel, (1964), led by the necessity to overcome the problem of low tensile strength and poor ductility, introduced discontinuous fibers randomly dispersed in the concrete matrix paving the way for FRCs. Twenty years later, the concept of SIFCON (Slurry Infiltrated Fibered Concrete) was realized by Lankard (1984) who filled formworks with high content (up to 20%) steel fibers, followed by injecting a fluid mortar slurry.

By combining the available knowledge on optimized particle packing, fine reactive powders and aggregates, water reducing admixtures and steel fibers, Richard and Cheyrezy (1995) introduced the concept of Reactive Powder Concrete (RPC) which paved the way for the development of tension hardening high performance fiber reinforced composites. High performance was achieved through dense microstructure, low water to binder ratio, optimized particle packing, and ductility induced by the fibers.

2.2.2. Definition

THFRCs, which also are characterized as high-performance materials, differ from conventional FRCs in terms of their constitutive tensile behaviour as shown in Figure 2.8. Due to their constituents and their synergy (which will be discussed in a future section) after first cracking which signals the end of the elastic portion of the stress-strain curve, THFRCs demonstrate a strain or pseudo-strain hardening behaviour accompanied with multiple cracking till the ultimate tensile strength. Upon exceedance of the latter, localization of a single crack occurs and softening follows. On the contrary, Tension Softening Fiber Reinforced Composites (TSFRC) lack hardening and after the formation of a single crack, localization occurs and softening follows (Figure 2. 8(a)). The existence of hardening and plateau between cracking and ultimate strength in combination with extended softening equips THFRCs with ductility, increased strain capacity and fracture toughness (Figure 2. 8(b)).

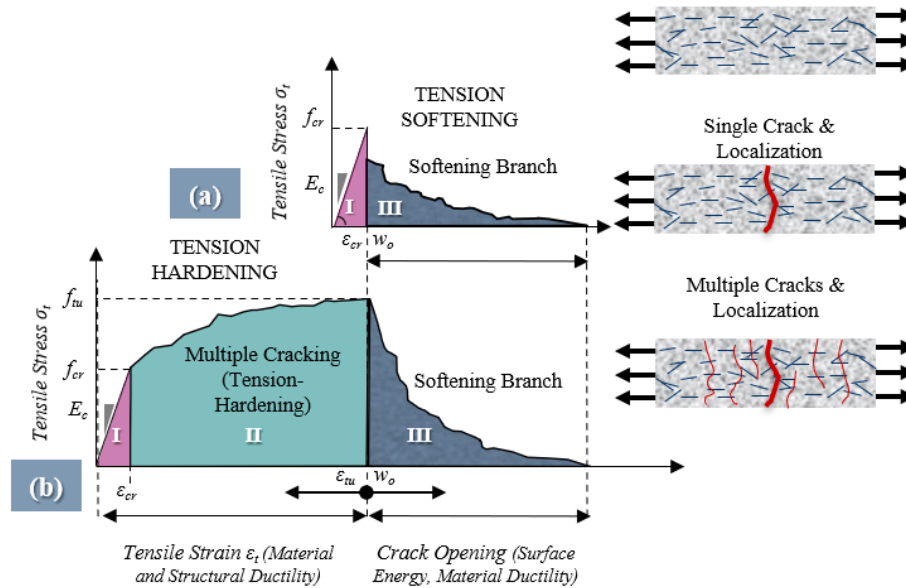


Figure 2.8: Typical constitutive tensile law of FRCs (a) Conventional TSFRCs. (b) THFRCs (modified from(Naaman, 2007))

2.2.3. Constituents & Conceptual Basis

As discussed in the section of historical background of THFRCs, the most important attributes of THFRCs are their constituents which differ from those used in conventional FRCs. To achieve the superior behaviour of such materials, matrix and fiber reinforcement play an important role individually but also synergistically.

- *Fine Reactive Powders*

Besides Ordinary Portland Cement (OPC), common reactive powders used in THFRCs are microsilica, quartz flour/powder and supplementary cementitious materials such as Fly Ash and Slag and Metakaolin. OPC is the main ultra-fine reactive powder usually found in high contents in THFRCs (900 kg/m^3) which has significantly negative impact on the environment. Due to their high fineness, microsilica, quartz powder and metakaolin are highly amorphous and ultra-fine, exhibiting significant pozzolanic activity and also ability to fill smallest voids resulting in dense microstructure. In particular, microsilica enhances the interface between binder, sand, and fibers (Habel et al., 2006). Although Fly Ash is less reactive, because of its morphology described in previous sections (cenospheres) and the low surface adsorption capacity, it enhances the flowability of the mixture, while lowering the demand of extra water that can be created by microsilica (Saikali, 2019). Finally, slag has similar effects in case of conventional cementitious THFRC.

- *Fine Inert Fillers/Aggregates*

One of the main differences from conventional concrete is the exclusive use of fine aggregates with maximum aggregate size in the range of 400-600 microns. Most commercially available and non-proprietary mixes use silica sand as the spherical shape of its particles enhances the fresh-state properties of THFRC mixes. However, there are some other proprietary mixes that contain calcined bauxite or granite aggregates such as Densit that strengthen the binder. Other fine additives commonly used in THFRCs are inert fillers such as limestone that act as partial replacement of the binder densifying the matrix without participating in chemical reactions.

- *Chemical Admixtures*

As discussed previously, the rapid advancement of chemical admixtures over the last decades promoted the development of THFRCs. The most common chemical admixtures used in this class of materials are high range water reducing admixtures (superplasticizers) with polycarboxylate ethers being a new generation of superplasticizers especially designed as the best fit for THFRCs. Another type used is workability-retaining admixtures that allow concrete to maintain its fresh characteristics without adversely affecting the setting time or hardened state properties.

- *Fibers*

In all FRCs, fibers prevent brittle failure, improve ductility and tensile strength while providing toughness by bridging action of tensile cracks and passive confinement in compression. There is a wide range of fiber types used in FRCs, depending on material, shape, size, and aspect ratio. Regardless of the type, the main requirements for fiber selection are (i) compatibility of material properties with the application, (ii) ability for stress transmission through adequate fiber-matrix interaction and (iii) optimum aspect ratio for guaranteed effective post-cracking behaviour (Ranjbar & Zhang, 2020). Along with the aforementioned requirements, volumetric fiber ratio, V_f , is critical in differentiating tension hardening and tension softening FRCs. At low V_f , the collective force carried by fibers in tension is smaller than the capacity of concrete before cracking leading to softening behaviour, while increased fiber content promotes multitracking and thus hardening (Pantazopoulou et al., 2019).

Regarding the material, the most common types of fibers used in THFRCs are steel and synthetic fibers. Their shape and aspect ratio vary, and a summary list of different fibers commonly used in THFRC is given in Figure 2.9.

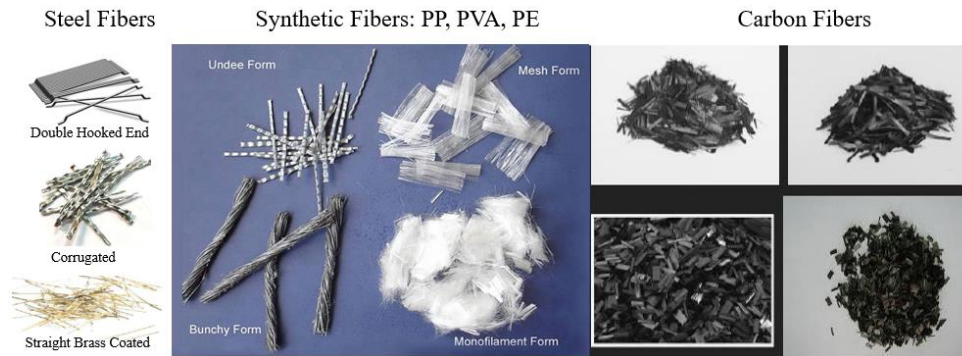


Figure 2.9: Various types of fibers found in THFRCs (Georgiou, 2017)

- *Conceptual Basis*

The combination of ultra-fine dry materials and low water to cement ratio is not enough to achieve a dense microstructure. The latter is achieved through optimization of particle packing density through particle packing theories first developed in the 20th Century. In most particle packing models, the dry material mixtures are modified against predefined continuous grading curves in order to achieve maximum particle packing density. Figure 2.10 shows some of particle packing models available in the literature.

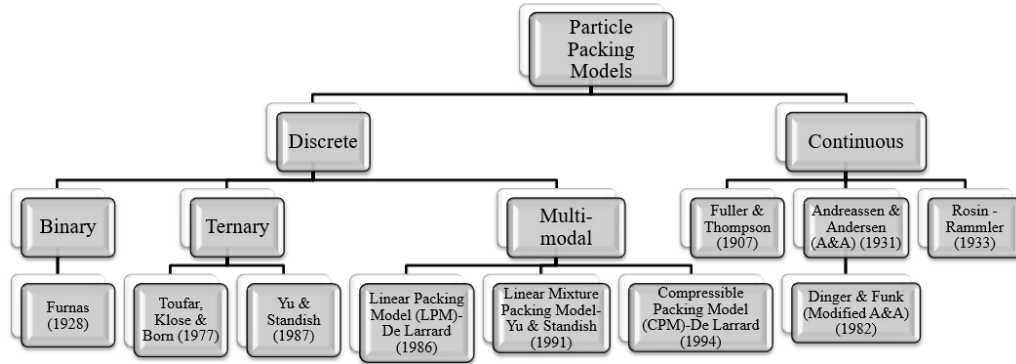


Figure 2.10: Particle Packing Models Published in Scientific Literature (Modified from Sing, 2011)

2.2.4. The Conundrum of Tensile Characterization of THFRCs

Thorough research campaigns are ongoing towards the development of structural design procedures for THFRC, on condition that consensus may be reached in the interim regarding a vetted test procedure to measure experimentally the tensile stress–strain law of the material for characterization of its properties. This is a required ticket for THFRCs to be allowed in engineering practice. It is therefore critical for field use of this novel concrete, for users and producers alike, to have methods of characterization for certification, but also for quality control in the field that are reliable, easy to conduct, and not burdened with uncertainty. After several years of trying to adapt the flexural prism test to the characteristics of THFRC, which led to a great deal of discord and dispute in the academic community, Direct Uniaxial Tension tests were opted for as the main testing option for material qualification (e.g., Graybeal & Baby, 2019), whereas prism flexural testing was kept as an option for quality control in the field. In fact, direct tension tests are considered most conservative. However, a disadvantage of using direct tension tests both for characterization and for quality control is that this test is challenging to conduct by many commercial or university labs. Some test setups require special gripping devices in order to be carried out on specimens of adequate thickness, which is needed in order to achieve an objective fiber distribution and minimal wall effects on fiber orientation. Ideal gripping devices are hydraulic grips; but they are very expensive and unavailable to most labs, whereas fabricated gripping adaptors to the existing equipment introduce a significant degree of uncertainty and randomness. The specimen is usually a prism cast or saw-cut from a bigger structural member and is subjected to uniaxial tension during the test (Figure 2.11(a)). Response is captured through recorded characteristics such as distribution, spacing and width of cracks during the test, needed

for the analysis to extract the stress-strain response. But the presence of unavoidable eccentricities which are intensified after first cracking, the geometrical tolerances, uncertainty in boundary conditions and specimen size, all contribute to hindering the establishment of a single standard test, whereas the direct tension test is not versatile for quality control of field casting. A solution to the limitation of thickness imposed by gripping is the adaptation of dog bone specimens (Georgiou & Pantazopoulou, 2016) undergoing direct tension, by gripping them through bearing action (Figure 2.11(b)). However, the steel frame used in that case is sometimes susceptible to out of plane bending if not fixed properly.

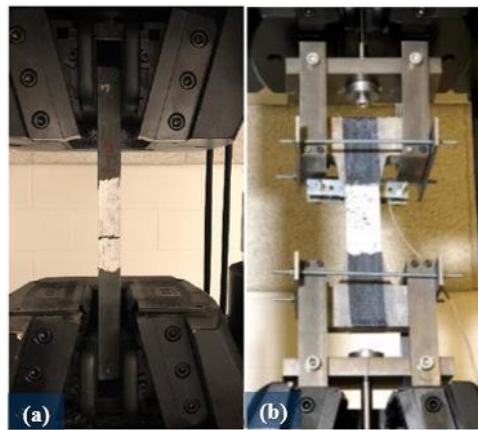


Figure 2.11: Direct tension test setup on (a) modified AASHTO specimen, (b) dog bone specimen (Pantazopoulou et al., 2019)

Flexural tests for experimental characterization of the tensile behavior of THFRC as a concept was seen as an alternative because most Labs can conduct simple prism bending tests. This test draws its origin from the notched beam tests (Figure 2.12 left) that are often used for assessing the tensile strength of conventional concrete (modulus of rupture tests). But because the material is strain-hardening, notches could not be used, as the static system of the three-point bending test in a notched beam does not allow for spread of plasticity. To enable development of multiple cracking, which is the underlying source of the macroscopic strain-hardening characteristic of the tension response of THFRC, four-point bending tests became standard practice (Figure 2.12 right). In the central constant moment region of this specimen form, multiple cracking occurs, leading to significant length change of the specimen. Round robin tests conducted between 3 Canadian Institutions have illustrated that contrary to the initial expectation, the test is much more difficult to interpret: the testing hardware (size and rolling friction of the end support rollers, as well as other practical details during the test) interfere with the experimental values, with a

dispersion as high as 30% been seen in test results from different Universities conducted on otherwise identical specimens (Ralli & Pantazopoulou, 2019; Yang et al., 2021). Uncertainty is compounded due to the recommended analysis procedure used for extraction of the properties of interest from the experimental results; it is noted here that Annex 8.1 of CSA-S6 (2018) and Annex U of CSA A23.1:19/CSA A23.2:19 (2019), do not offer the same methodology for inverse analysis, and there is no evidence that the two alternatives converge to a unique set of values.

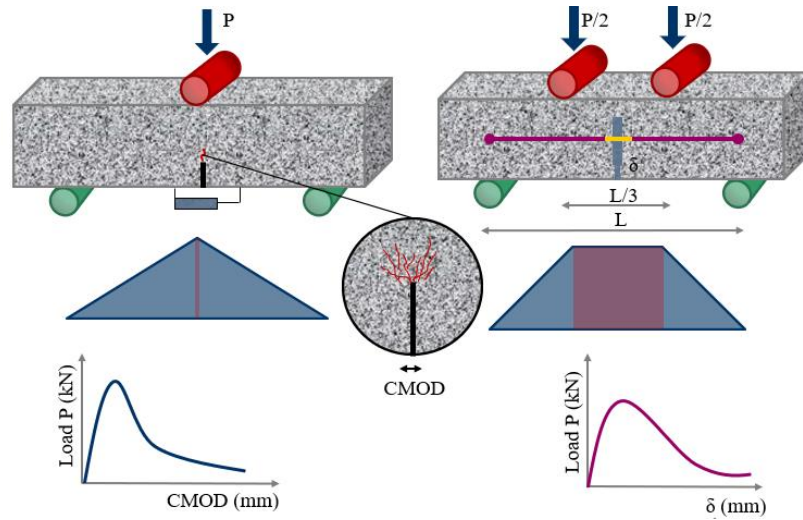


Figure 2.12: Difference between notched three point bending and unnotched four-point bending tests

Other alternatives for indirect evaluation of the tensile characteristics are possible, provided the response is dominated by clearly identifiable, pure tensile failure. In fact, there is a need for a practical alternative that most commercial labs would be able to conduct, as the mere number of tests required for quality control on samples obtained during each field casting escalates to a volume of tests that cannot be handled by the few academic labs that currently conduct most of the characterization tests. As in the case of flexural tests, the motivation here is to come up with a test setup that uses the available equipment and hardware that most labs have access to, without requiring special gripping devices as is the case with the direct tension test. In fact, all labs are equipped with compression testing machines, and it is therefore desirable that the standardized test could be conducted easily using this rudimentary equipment. Variations of such alternatives are the well-known Brazilian splitting test whereby a cylindrical specimen is subjected to compressive load, along two diametrically opposed generating lines of the cylinder. Uniform transverse tensile stress state is generated almost lengthwise and height wise in the diametric plane that is parallel to the applied loads. However, response becomes complicated after diametric

cracking of the THFRC cylinder, because of the ductility in the splitting plane; thus, significant load bearing may occur where complex stress states may develop without being possible to identify and separate the clear contribution of post-cracking pure tensile resistance of the material in the obtained response.

2.3. Self-Sensing Concrete

With self-sensing concrete emerging as the latest advancement in the field of SHM, research gravitates more and more towards the development of self-monitoring cementitious composites by exploiting nanomaterials. In fact, the global consumption for nanomaterials is expected to reach \$100 billion by 2025 where 7% of them are intended to be used in infrastructure (Nguyen-Tri & Nguyen, 2020). At this point, it should be highlighted that in order to be practical, self-sensing concrete should not have any embedded or attached sensors. Instead, the addition of nanomaterial admixtures is intended to modify the concrete so as to act as sensor. The development of such smart materials is critical for special infrastructure projects such as bridges, nuclear plants or structures that are not easily accessible and cannot be assessed by a site visit. In this chapter, two basic self - sensing technologies are being discussed along with nanomaterials used, factors affecting self-sensing properties, their underlying physics, and analytical studies.

2.3.1. Self-Sensing Systems Based on Electromechanical Properties of Carbon Nanomaterials

The most common self-sensing system studied by research community exploits the unique electromechanical properties of Carbon Nanomaterials. The idea begun with studying carbon materials at the micro and macro level, which are known to be semiconductors. Early studies were performed in the 90's and were focused on the electromechanical effects of cementitious materials reinforced with carbon microfibers. Banthia et al. (1992) measured the electrical resistance of the samples through high frequency alternating current and proved that the addition of microfibers in the amount of 5% by weight enhanced the conductivity of the composites. The electrical resistivity, also denoted as specific electrical resistance of the cement composite was reduced from 66117 $\Omega\cdot\text{cm}$ to 78 $\Omega\cdot\text{cm}$. Meanwhile, Fu & Chung, (1996) studied the electromechanical behaviour of cementitious materials containing carbon fibers (CFs) at a lower percentage of 0.24% by volume. The piezoresistive response of the composite was determined through direct

current measurements in dog-bone shaped specimens subjected to tension or compression. The results demonstrated the piezoresistivity effect, because adequate correlation between the induced strain and the change in electrical resistance was observed. Specifically, the resistance was reduced during loading where the cracks closed and increased during unloading where the cracks opened.

- *Underlying Physics*

For better understanding, the definitions of electrical resistivity, electric conductivity and semiconductor are provided. The electrical resistivity or specific resistance (ρ ($\Omega\cdot\text{m}$)) expresses the ability of a material to oppose the flow of electric current, while the electric conductivity, also known as specific conductance (κ (S/m)) is a reciprocal of the electrical resistivity and measures the material's ability to conduct electric current. Semiconductors are materials with electrical conductivity value between that of a conductor (i.e.: copper) and an insulator (i.e.: glass).

The basic concept of self-sensing systems based on electromechanical properties is the piezoresistive effect, which is the change in relative dimensions and resistance of semiconductors or metal due to applied mechanical stress/strain, as illustrated in Figure 2.13.

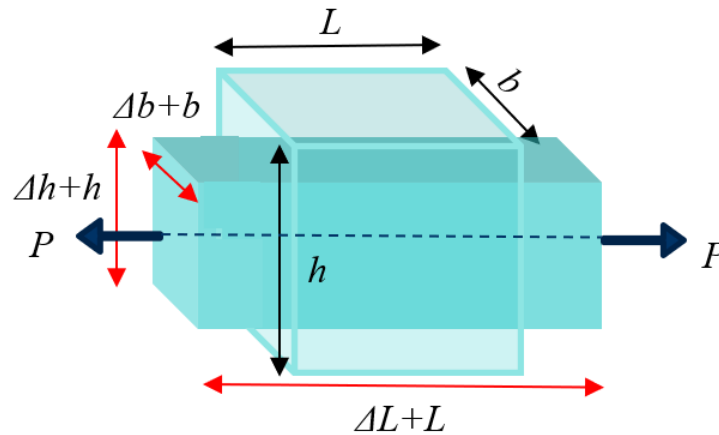


Figure 2.13: Changes in dimension of an element subjected to tensile stress.

Piezoresistivity and stress are related through the piezoresistance coefficients (π coefficients) and are defined by:

$$\pi = \frac{\Delta R/R_0}{P} \quad (2.1)$$

Where R_o is the material's resistance related to the dimensions of the sample, $R_o = \rho \frac{1}{bh}$ (w is the width and h is the height), ΔR is the change of resistance, and P is the applied mechanical load. The relative change in resistance compared to the original ($\Delta R/R_o$) also known as Fractional Change of Resistance (FCR) is the results of the relative change in dimensions of height, width, length (h, b, L) and in resistivity (ρ) and is expressed by the equation:

$$\frac{\Delta R}{R_o} = \frac{\Delta L}{L} - \frac{\Delta b}{b} - \frac{\Delta h}{h} + \frac{\Delta \rho}{\rho} \quad (2.2)$$

The change in geometry due to applied stress is expressed through the first three terms of Equation (2.2), while the last term shows the electromechanical relationship of mechanical stress and electrical resistivity. However, in most semiconductors the change in dimensions is negligible compared to the change in resistivity (Sun et al., 2010).

The same mechanism applies to carbon nanocomposites. The electrical resistance changes when the carbon nanofiller is deformed under loading. This change again is due to geometry alteration but mainly due to change in contact resistance. Contact resistance⁴ depends on the diameter of carbon nanomaterial, tunneling gap⁵ at junctions and matrix filling⁶ at the gap between the fillers (Han et al., 2011). As the composite undergoes loading, the thickness of the matrix is deformed closing the gap between adjacent carbon nanofillers with ultimate result the reduction of contact resistance.

Since the geometrical changes do not have the same impact as resistivity in altering material's resistance, a deeper understanding on the relation between resistivity and mechanical stress is needed. Most of the semiconductors possess a crystal lattice deformation, which deforms under application of stress. As the lattice deforms, the energy band curves undergo a change of shape resulting in change of the effective mass which is related to conductivity. The alteration of energy band curves under different stress states is depicted in Figure 2.14.

⁴ Contact resistance: the electrical resistance resulting from the interface between a CNT and a metal contact when a CNT is connected to a pair of electrodes. Also, the resistance of a CNT-CNT intersection when an overlapped joint occurs between two CNTs

⁵ Tunneling gap: the gap between hopping electrons that are transferring between adjacent carbon nanofillers

⁶ Matrix filling: the insulating matrix of the composite, in case of self-sensing concrete, the concrete matrix.

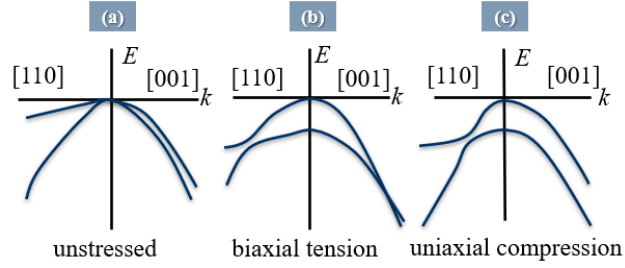


Figure 2.14: Band curves for Si along the [110] and [001] lattice indices (modified from Sun et al., 2010)

- *Carbon Nanomaterials*

Carbon Nanotubes (CNTs)

Carbon Nanotubes (CNTs) were first studied and described in 1952 (Radushkevich & Lukyanovich, 1952). Later, Oberlin et al., (1976) studied a type of CNTs, known as Single-Walled CNTs (SWCNTs). Iijima, (1991) discovered the most commonly used type of CNTs in self-sensing applications, called Multi-walled CNTs (MWCNTs). They are graphite layers rolled up into an extremely thin tube structure (Iijima & Ichihashi, 1993). Their length to diameter ratio could reach $1 \cdot 10^7:1$ (Wang et al., 2009). Their diameter could be less than a nanometer and as large as 50 nm (Gullapalli & Wong, 2011). The nature of atomic bonds in a CNT is described by quantum chemistry. More specifically, the chemical bonds of nanotubes are sp^2 bonds similar to those of graphite. This bond type is stronger than sp^3 bonds found in alkanes and provides unique strength. Based on their geometry and structure there are two types of CNTs which were mentioned earlier: (i) Single-Walled Carbon Nanotubes (SWCNTs) and (ii) Multi-Walled Carbon Nanotubes (MWCNTs) (Gullapalli & Wong, 2011). The structure of the former could be explained as a result of folding a single graphene sheet as Figure 2.15 shows.

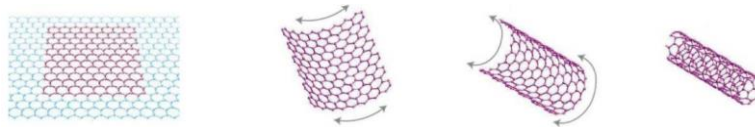


Figure 2.15: Carbon Nanotube formation after folding a graphene sheet (Ibrahim, 2013)

MWCNTs consist of multiple layers of graphite rolled up into a multi-walled tube structure. The construction of such CNTs could be described based on two models. The first model is known as Russian Doll, where graphite layers are organized in homocentric cylinders. The second model is called Parchment, and in this case a single graphite layer is rolled in around itself like parchment (Ibrahim, 2013). The MWCNTs resulting from these models are illustrated in Figure 2.16.

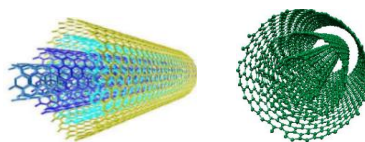


Figure 2.16: Structure of MWCNTs according to Russian doll model (left) and Parchment model (right)

Carbon Nanofibers (CNFs)

Carbon Nanofibers (CNFs) also known as Graphene Nanofibers (GNFs) similarly to CNTs form a hollow cylindrical nanostructure with diameter ranging from 50nm to 200nm and a length of 50 to 100 μ m, consisting of consecutive graphite layers that demonstrate a hybrid structure. The graphite layers are stacked up in conical and tubular elements. These arrangement styles result in well-organized structures of conical shape or parallel layers. The understanding of the morphology of such nanomaterial is challenging due to the complexity of the nanostructure.

CNFs demonstrate excellent mechanical properties, high electrical and thermal conductivity, i.e., properties that are exploited in a wide range of applications as elastomers, ceramics, and metals (Burton *et al.*). A unique feature of CNFs is their surface. The edges of the graphitic layers are formed throughout the surface of the nanofiber and could be used to increase their adhesion in the matrix and avoid interfacial slip. As a result, the interfacial transitional zone in the matrix is enhanced promoting better transfer of forces and electricity.

The most common type of CNF has a conical shape. Initially it was considered that this type of structure, also known as stacked up structure, consists of cones in nano-level stacked up in a sequence, forming in that way the fiber (Figure 2.17) (Endo *et al.*, 2002, 2003). It is highly possible for the conical nanofibers to form a cone-helix structure (Ekşioğlu & Nadarajah, 2006). There is a strong resemblance in their structure that hinders their being distinguished from each other even with the use of Transmission Electron Microscope (TEM). However, the presence of cones with wide range of angles is visible due to the helix morphology.

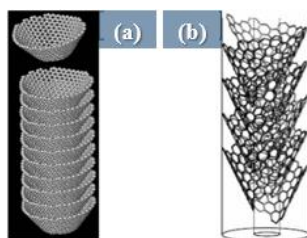


Figure 2.17: (a) Cone shaped CNF and (b) cone-helix CNF (Monthioux *et al.*, 2007)

Two other usual forms of nanofibers are platelet CNFs, and ribbon/tubular CNFs. In the first case, the graphene layers are placed perpendicular to the axis of the fiber, while in the ribbon structure the layers are parallel to the axis as illustrated in Figure 2.18.

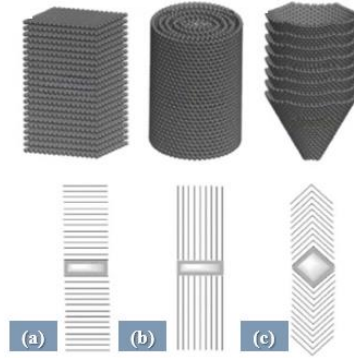


Figure 2.18: (a) platelet CNF, (b) ribbon/tubular CNF and (c) cone shaped CNF (Pérez-Mayoral et al., 2016)

Although this section focuses only on CNTs and CNFs a brief description is given on other carbon nanofillers. Other types of carbon nanomaterials available for the development of self-sensing concrete are carbon black (CB) and graphene nanoplatelets (GNPs). CB is typically composed of pure elemental carbon in the form of colloidal spherical particles with average particle size of 30 nm, while GNPs are chemically exfoliated from natural graphite. Their structure resembles the structure of CNF platelets as they consist of graphene layers stacked together yielding a structure with thickness of 3-10nm (Pisello et al., 2017). Figure 2.19 shows the structure of CB and GNPs. It should be noted here that regarding cost, GNPs are the most expensive with MWCNTs, CNFs and CB following (Ubertini & D'Alessandro, 2018).

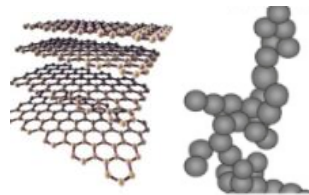


Figure 2.19: GNP structure (left), Carbon Black structure (right) (Pisello et al., 2017)

2.3.2. Factors Affecting Self Sensing Properties

Over the last decades the research community, being impressed by the availability of the novel Carbon composites and their potential applications, conducted several experimental studies focusing on the fabrication and investigating its candidacy as a self-sensing tool in concrete. Most of the experimental investigations involved wired probes embedded in the specimens in order to

monitor changes in resistivity with stress; parametric studies were conducted exploring how different factors such as the type of carbon nanomaterial, the dispersion method, or the concentration in the cementitious matrix affect the resistivity response of conventional pastes and concrete.

- *Type of Carbon Nanomaterial*

Establishing the type of the Carbon Nanomaterial that performs better as a filler in terms of electrical properties has always attracted a lot of attention. So far, various materials in the carbon/graphene family have been tested. Research has shown that CNTs have the highest sensitivity, with graphene oxide and Graphene Nanofibers following, while graphene showed the lowest values (Yoo et al., 2019)/ Similar study was conducted by Pisello et al. (2017) investigating the performance of four different types of carbon nanofillers, namely MWCNTs, CB, CNFs, and GNPs. More specifically, GNPs proved to possess the highest electrical conductivity and capacitance with MWCNTs showing the best strain-sensing capabilities, despite their lower contribution to electrical conductivity. However, other researchers claim that CNFs show better piezoresistive strain sensitivity due to their length (Sasmal et al., 2017). Researchers also investigated the piezoresistivity of cementitious materials reinforced with MWCNTs and carbon black (CB) at percentages of 0.1% and 0.5% by weight, respectively. The results indicate that NCB enhance the sensitivity but lacks the high repeatability that the MWCNT samples demonstrated (Luo et al., 2010).

Besides testing each material alone as a filler, there have been studies investigating hybrid nanocomposites containing CNTs and CNFs or SWCNTs and MWCNTs (Azhari & Banthia, 2012; Han et al., 2012). It has been found that the CNT/CNF hybrid yields low resistivity but the SWCNTs/MWCNTs system shows even better resistive sensitivity.

- *Dispersion Method*

The development of advanced self-sensing nanocomposites with enhanced electromechanical properties is based on exploiting the unique properties of carbon nanomaterials. However, such an exploitation is based on a homogeneous dispersion of the nanomaterials in the matrix, which has proven to be challenging. The challenge lies on the formation of agglomerates and bundles at the nanoscale due to Van der Waals attractions between carbon molecules (Grobert, 2007).

Dispersion of carbon nanomaterials has attracted research interest and numerous investigations have been conducted establishing mechanical and chemical methods or a combination of both. Figure shows the dispersion mechanisms available in the literature.

So far, the most successful mechanical method uses ultra-sonication energy and requires low viscosity fluids such as water, acetone, and ethanol. The released energy creates waves that break the microbubbles and the formed bundles (Cheaburu-Yilmaz et al., 2019). Other mechanical but less common methods are shear mixing, extrusion, calendaring process, and ball milling. The last is used in conjunction with chemicals and reduces the length of the nanomaterials by chopping them.

Chemical dispersion methods are based on the utilization of solvents or surfactants in order to achieve functionalization of the nanomaterial's surface. In this way, the tendency for agglomeration is weakened and thus the dispersion and the bond with the matrix as well are enhanced. Two basic chemical methods are, the covalent and the non-covalent treatment. In covalent treatment, the functionalization is performed either on the walls or on possible defects such as open ends or holes. The first type targets the alteration of sp^2 bonds to sp^3 in order to increase the bond with the matrix and hinder the formation of bundles while the second eliminates the defects. The chemicals used in this method are usually highly reactive acids such as carboxylic acid, nitride acid, sulfuric acid, or a combination of those. Regarding non-covalent methods, they are not preferred as they tend to destroy the nanomaterial's structure. In this method surfactants and polymers are mainly used. Finally, a less common method is the endohedral functionalization, where the internal hollow structure of SWCNTs is used as a storage space of fullerenes⁷ (Hirsch, 2002).

Functionalization of carbon nanomaterials usually alters their structure and thus their properties. C. Li et al. (2007) conducted experiments on cementitious materials reinforced with 0.5% by weight acid treated CNTs (SPCNTs) and untreated CNTs (PCNTs). The dispersion of the CNTs was performed via ultrasonication. Both types of CNTs significantly reduced the resistivity of the

⁷ Fullerene: allotrope of carbon whose molecule comprise carbon atoms connected by single and double bonds in order to form a closed or partially closed mesh

nanocomposite cement-based materials. A remarkable observation was that the treated CNTs had a significant effect on the enhancement of piezoresistivity, while the untreated had a stronger impact on the reduction of the electrical resistivity. This phenomenon was explained through SEM, where a formed three-dimensional meshwork was observed on the untreated CNTs.

Yu & Kwon, (2009) conducted an experimental parametric investigation studying the effect of different amounts of CNTs and different dispersions methods on the piezoresistivity. According to their findings, the use of superplasticizers in the dispersion could hinder the contact between CNTs altering the piezoresistive response, while higher concentration of nanomaterials could enhance the strain sensitivity. It was also observed that the electrical resistance of the non-polarized surface modified CNTs changes as the compressive stress changes and the rate of change depends on the loading rate.

However, the dispersion methods such as sonication and ultrasound are impractical for the case of large-scale production and more efficient and easy applicable methods should be found. D'Alessandro et al., (2016) proposed and tested a scalable procedure of nanocomposites based on water suspensions containing different types of dispersants that yield electromechanical properties as good as sonicated composites. The dispersants were mainly superplasticizers such as polycarboxylate ether, sulfonates and lignosulfonic acid sodium salt with the latter proving to be the most efficient. The quality of the dispersant was evaluated via measurements of MWCNTs settling time and SEM inspections

- *Concentration of Filler*

Generally, the unique electromechanical properties of carbon nanofillers are activated when their properly dispersed concentration reaches a critical value known as percolation threshold. It represents the point at which the matrix composite transforms from insulating to conductive. The change of piezoresistive sensitivity when the concentration alters from 0.05%wt. to 0.1%wt. is drastic. At 0.05% wt. of the binder, the large thickness of the insulating matrix hinders the connection of conducted tunneling junctions and thus the conductive path is not formed, resulting in low sensitivity (Figure 2.20). However, when the concentration of the filler increases to double percentage, the thickness of the matrix is reduced and the transition of electrons via the tunneling effect is easier. In that way the material becomes conductive. However, as the concentration of

filler increases to more than 0.1%wt the tunneling gap shortens and the CNT network becomes stiffer and more difficult to deform under loading, thus the piezoresistive sensitivity is lost (Han et al., 2011). This is why the concentration of the carbon nanofiller should be close to the percolation threshold. The most common concentration in the literature that yields the best performance is around 0.1%wt for MWCNTs (Sasmal et al., 2017).

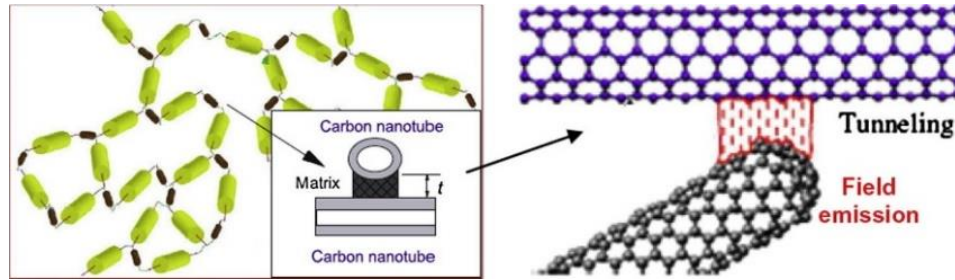


Figure 2.20: Schematic diagram of basic conductive element in conductive network of CNTs cementitious composites (Li and Chu, 2008)

- *Water to Cement Ratio*

Water to cement ratio is also a parameter that affects the electromechanical properties. It is found that higher w/c ratio (i.e., 0.6) yields more sensitive nanocomposites compared to lower ratios (i.e., 0.45). This could be attributed to better dispersion in the matrix in presence of extra water (Han et al., 2011).

- *Curing Conditions and Hydration Age*

It has been shown that curing type and duration highly affects the electromechanical properties of carbon nanocomposites. In fact, CNT-modified materials show higher sensitivity when subjected to water curing than to air curing (Jia, 2009). Generally, long periods of curing increased the resistivity (Sasmal et al., 2017). This peculiar phenomenon could be explained through investigating the hydration process. Between 14-28 days, several changes in the microstructure occur such as, the capillary pores lose their continuity and the concentration of pores' fluid changes (Fu and Chung, 1996).

2.3.3. Sensing Mechanism

Self-sensing concrete comprises an insulating matrix (concrete) and a semi-conductive filler (carbon nanomaterials). This combination results in conductive but also in dielectric properties. The latter originate from double layer phenomena around electrodes and nanoparticles and cause

high complexity in the understanding and assessing the electromechanical response of the composite. In other words, the material is characterized by its resistive and capacitive nature at the same time. It is believed by the research community that this situation leads to polarization. This refers to charge accumulation under applied constant field, which subsequently may lead to a time-based drift of electrical resistance, perplexing more the strain sensing (Ubertini & D' Alessandro, 2018). A typical time drift due to polarization is shown in Figure 2.21. However, some researchers state that the material's resistance is more influenced by the application of mechanical deformation compared to the capacitive constants (Han et al., 2012).

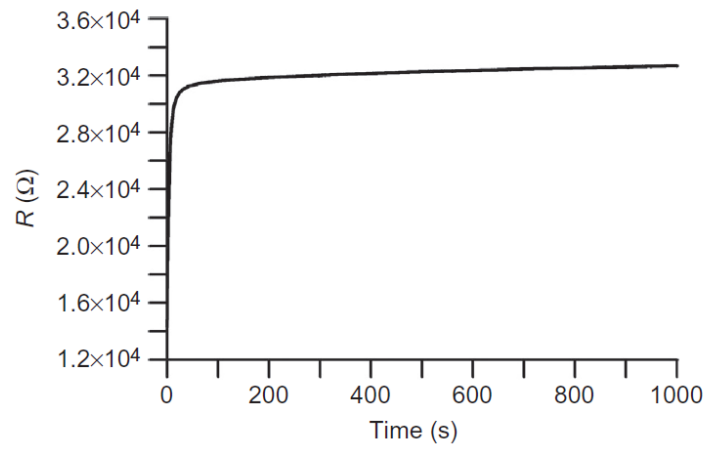


Figure 2.21: Polarization curve in a self-sensing concrete (Ubertini & D' Alessandro, 2018)

The underlying physics explained earlier are introduced again here for defining another significant property of self-sensing concrete, namely, the gauge factor GF . Assuming small deformations, Equation (2.3) could be expressed as:

$$\frac{\Delta R}{R_o} = - GF \cdot \varepsilon \quad (2.3)$$

In Equation (2.3), R_o is the material's resistance related to the dimensions of the sample, ΔR is the change of resistance, GF is the gauge factor, and ε is the axial strain due to the application of external load. Smart concretes possess a gauge factor around 200-400 which is at least 2 orders of magnitude greater than conventional strain gauges (Azhari & Banthia, 2012). The strain sensitivity S that was mentioned earlier is an index of self-sensing performance and could be expressed as:

$$S = \frac{\Delta R}{\varepsilon} = -GF \cdot R_o \quad (2.4)$$

2.3.4. Measurement and Assessment of Sensing Properties

The measurement of electrical properties is made using either AC or DC current. DC current has prevailed as it is easy and inexpensive to work with, despite the more profound time drift. Possible causes and solutions for accounting time drift are shown on Table 2.9. On the other hand, AC, despite the accuracy it enables, is not so commonly used owing to the complex LCR meters and the high cost of the equipment. Table 2.10 shows a comparison between performance under the two currents. In both cases (DC or AC) two methods have been used, i.e., the two-probe method and the four-probe method. The first method comprises an easy configuration of two probes, where each probe is responsible for either sourcing the current and measuring the voltage or sinking the current and providing a voltage reference. In the more accurate case of 4 probes this is done by 2 pairs of electrodes.

Table 2.9: Time Drift: reasons and solutions

Possible Causes of Time Drift	Proposed Solutions
Polarization effect (Chung, 2002; Turner et al., 1994)	Control sample for comparison (Azhari & Banthia, 2017)
Change in material's dielectric constant (Wen & Chung, 2002)	Delayed measurement till drift fades out (Materazzi et al., 2013)
Direct piezoelectric effect (Sun et al., 2010b)	High pass filter in dynamic strain measurement (D'Alessandro et al., 2016)

Table 2.10: Comparison between AC and DC current for measuring self-sensing properties

Characteristics	AC	DC
Measured Electrical Properties	Reactance	Voltage
	Impedance	Resistance
	Phase Angle	Conductivity
Advantages	Time drift rarely occurs	Easier configuration
	Accuracy	Inexpensive
Disadvantages	Complex LCR meters	Less accurate
	High cost	Intense time drift
	Not applicable in large scale	

Studies so far have shown that self-sensing concrete's resistivity measurement can efficiently describe the damage evolution (Han et al., 2015; Liu et al., 2019). It is found that the change in resistivity-strain curve is more in agreement with theoretical material behaviour of concrete

compared to stress-strain curves and ultrasonic velocity curves. In fact, the resistivity – strain curve could be described firstly by a slight decrease in the elastic region followed by a slow increase right before the strength is reached. Then the increase is faster till the load drops to 50% of the peak load. The final stage, after reaching 50% of the post peak strength exhibits a gradual increase (Liu et al., 2019). This is very similar to the pattern of volumetric strain – vs. axial strain concept. Figure 2.22 illustrates the typical sensing behaviour of self-sensing concrete under different types of loading, where the horizontal axis is either load, stress, or strain. It is interesting that macrocracks lose their ability to transfer force, but not the ability to transfer electricity. This could imply the existence of descending branch is the case of uniaxial tension although it is not macroscopically visible and thus is ignored.

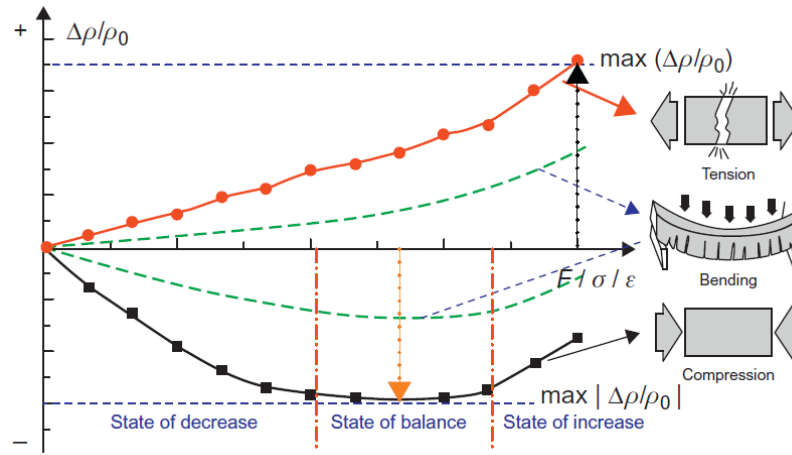


Figure 2.22: Typical sensing behaviour of Intrinsic Self-Sensing Concrete under loading (Han et al., 2015)

2.4. Conclusions

There is an abundance of aluminosilicate source materials that could be used as binders in geopolymer concrete. Most of them are industrial by-products but also natural rock minerals or products such as calcined clays. However, the suitability of the material should be tested before introducing them in a geopolymer mix, by examining reactivity, mineralogy, pH value, gradation, fineness, and glassy content.

- The only hardener that yields geopolymer products is soluble alkali silicate and not hydroxides alone. Molar Ratio is the most critical parameter for soluble silicates as it determines a variety of properties and thus the applications of the silicate. A MR around 2 is reactive and stable enough for geopolymer concrete of high strength.

- Geopolymer concrete could be used in applications where early strength is needed, because almost after 24 hr from casting, it yields half of the strength observed at 28 days. However, the fast setting of the material could be a concern and further research is needed in the field of additives and admixtures because those that are already available are not very effective in eliminating flash setting, since they were designed for conventional concrete and target the OPC hydration mechanism.
- Geopolymers and AAMs are two totally different technologies and should not be treated as same. The two technologies differ in terms of chemistry, materials, mixing method and final solid products.
- Geopolymer concrete outperforms conventional concrete and alkali activated concrete in terms of sustainability, cost, and performance. Conventional concrete demands great amounts of energy, natural resources, and expensive additives to exhibit good performance. AAMs may exhibit higher mechanical strength but the free alkalinity present in the system could cause material stability and durability issues.

As discussed in a previous section of this chapter, tension hardening behaviour is an important differentiating attribute for FRC materials. To achieve a sustainable alternative of this class of materials, the characteristic features along with constituents and conceptual basis of OPC THFRCs were reviewed. More importantly, since tensile strength and strain capacity are the vehicle to design with THFRC, the available tension characterization techniques in international codes and standards presented in this chapter are related to various complications and disadvantages such as overestimation of tensile strength, or requirement of special gripping setup that is not available to most testing facilities. It is therefore desirable to develop an alternative setup that avoid all the issues discussed in this section, being easy to carry out and leading to unequivocal results that may be repeatable in a round robin exercise.

Regarding self-sensing ability of nanoengineered concrete, the chapter explored the underlying physics, reviewed the carbon nanomaterials commonly used for this purpose and the factors that affect piezoresistivity. It is evident that MWCNTs and CNFs are the most suitable candidates for self-sensing due to their high sensitivity. Also, after exploring the current measurement and assessment of self-sensing properties it is clearly indicated that the gap in the literature regarding measuring resistivity relies on future development of wireless measurements that will eventually

promote the development of wireless self-sensing concrete structures. Therefore, a research priority lies in the development of pertinent techniques that remove the barrier of wiring and could be practically used in large scale applications.

2.5. References

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Chapter 3: Synthesis of a Novel, Cementless Geopolymer Concrete Matrix¹

As already mentioned, geopolymer concrete differs from conventional OPC concrete and alkali activated materials. The development of strength and stability is not based on a hydration mechanism, but rather on a rigid three-dimensional network formation (geopolymerization) and is rapid compared to conventional concrete. So far, scientific literature proposes techniques for geopolymer concrete that are used in the OPC concrete technology and are not suitable for optimized geopolymer composites. Thus, the underlying chemistry and reaction kinetics that govern the geopolymer formation need to be fully comprehended and then used to develop stable, high performance and repeatable geopolymer formulations. The first step to develop such a formulation for the needs of the present thesis, is a detailed characterization of raw materials in terms of particle size distribution, mineralogy, chemical composition, and reactivity index. In this way, the suitability of the precursors is determined, and their properties can be exploited to yield the optimized results. In this chapter, the development of a new geopolymer concrete material is pursued. The material is intended to contain no cement, to comprise primarily of waste powders, and to be eventually adapted to tension hardening response (THFRG) through the addition of fibers. To achieve this objective, besides the characterization of raw materials and the interpretation of their effect on properties in fresh and hardened state, the concept of optimum packing density is explored, along with the effect of various chemical admixtures on the synthesis of the geopolymer concrete. Finally, based on the above, an optimized mix design is proposed along with a sustainability analysis of the proposed cementless concrete mix.

3.1. Characterization of Raw Materials

The right selection of the alumino-silicate source (precursors) is a vital part of a stable geopolymer concrete formulation. For optimized results, a precursor material mainly should be highly amorphous and rich in alumina and silica. Several studies have been conducted on different types

¹ Parts of this chapter have been used in the Author's publications with her supervisor, titled: "Microstructural Characterization of High-Performance Steel Fiber Reinforced Geopolymer Concrete" *Acta Polytechnica CTU Proceedings* pp.480-488 and "Effect of Aggregate Skeleton and Admixtures on Fresh and Hardened Properties of High Strength Geopolymer Mortars" *Proceedings of SynerCrete23': The International RILEM Conference on Synergising expertise towards sustainability and robustness of cement-based materials and concrete structures*.

of binders and how these affect the properties of the geopolymer concrete. In literature, a variety of aluminosilicate raw or processed natural materials, such as metakaolin or industrial by-products such as fly ash, as well as granulated blast furnace slag, have been used alone or in combinations as binders for geopolymer concrete and their mechanical and physical properties along with their microstructure have been studied. Despite the growing interest of the research community on the characterization of geopolymer concrete, there is a limited number of investigations that explore the effect of precursor's properties on the properties of the geopolymer concrete. In this section, fly ash type F (FA-F), blast furnace slag (GGBFS), two different types of silica fume (undensified/densified) (USF and DSF), metakaolin-750 (MK-750), sieved trap rock dust (TRD), Feldspar Powder (FP), Pumice (PM) and Granite Sand (GS) have been considered. The raw materials are illustrated in Figure 3.1.



Figure 3.1: Dry raw materials used

3.1.1. Particle Size Analysis

The particle size analysis was performed on a Laser Diffraction Particle size analyser (Beckman Coulter LS 13 320). For very fine particulate materials, surfactants were used for deagglomeration. Figure 3.2 shows the particle size distribution of the analysed materials. At this point, it should be noted that the high coarseness of GS and TRD as received from the supplier, imposed limitations for their use in the development of geopolymer self-consolidating matrix for a THFRG application and for this reason they were manually sieved to fit the particle range of the other raw materials. The fineness of precursor materials is an indicator of their reactivity. The finest materials are USF, MK-750 and GGBFS with D_{90} to be 0.55, 5 and 27 μm , respectively. Generally, precursors with grain size less than 50 μm are highly amorphous and thus more reactive and favourable for geopolymerization. FA-F and DSF seem to be the coarsest precursors

and thus less reactive. This could be attributed to high amount of residual unburnt coal that in DSF is evident from the deep dark grey colour (Figure 3.1) while coal grains in FA-F with size greater than 200 μm can be seen in Figure 3.3. The unburnt coal residue is not favourable as it inhibits the geopolymerization.

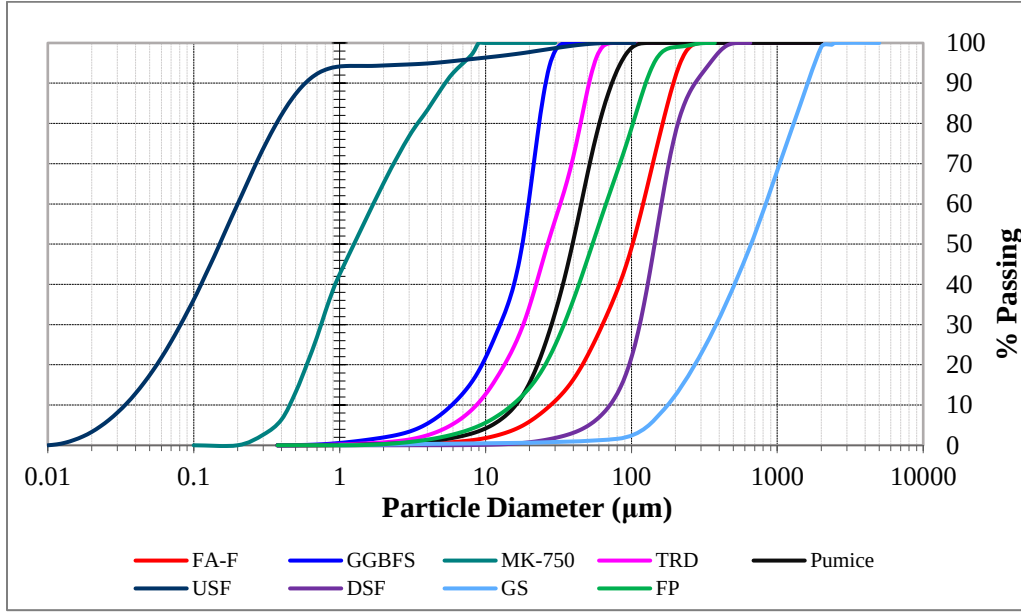


Figure 3.2: Particle Size Analysis of raw materials



Figure 3.3: Unburnt coal grain residues in FA-F

3.1.2. Chemical Analysis

X-ray Fluorescence (XRF) was performed to get the chemical composition of the precursors. The data given in Table 3.1 confirm that the aluminosilicate source materials could be potential candidates as geopolymer binders as they contain elevated amounts of alumina and silica, with MK-750 to possess the ideal analogy of those oxides. Although USF and DSF are rich in silica and not in alumina, they can work either as precursors that provide the silicon in the binder or as partially reactive fillers that densify the microstructure.

The high amount of iron oxide in TRD indicates that the dust could be an ideal candidate for ferro-sialate (-Fe-O-Si-O-Al-O) geopolymers. More specifically, a rock dust with 5-40% by weight Fe_2O_3 and 15-60% by weight Al_2O_3 could be used for the production of rock-based geopolymer concrete, while raw materials with Fe_3O_4 , $\text{FeO}(\text{OH})$ or Fe_2O_3 higher than 40% could be used as inert fillers (Davidovits, 2020). As already mentioned in previous chapters, Fe^{3+} can partially replace Al^{3+} in the geopolymer microstructure.

The relatively high amount of MgO in GGBFS poses concerns regarding setting time, as the majority of Mg precipitates in form of $\text{Mg}(\text{OH})_2$ and carbonation products with CO_2 rather than participating in geopolymerization. The same risk comes also from the CaO content in precursors. Although it does not necessarily mean that it exists in free lime form, rather than being part of other minerals (such as melilite in case of GGBFS), the high content of this oxide could provide early strengths, but also precipitate and speed up the setting time. GGBFS is an aluminosilicate glass mainly with covalent bonds of Si-O, Al-O and Ca-O. The bond energy is respectively 444, 221-280 and 134 kJ/mol. During the first step of alkalination, the bond with the lowest energy, namely Ca-O undergoes cleavage releasing Ca^{2+} , followed by gradual deconstruction of Al-O and then Si-O bonds (N. Li et al., 2018). On the other hand, the negative charge of every Al atom must be balanced with a corresponding positive charge from one K^+ or Na^+ - silicate or half Ca^{2+} from Ca-rich precursors. The other half Ca^{2+} precipitates or reacts with atmospheric CO_2 forming CaCO_3 .

The last important information that the chemical analysis could provide is the Loss on Ignition (LOI), which represents volatile materials lost that comprise of hydrates, hydroxyl compounds and carbon dioxide from carbonates. LOI could be used as a quality test and for by-products such as those examined here it usually consists of contaminant unburnt fuel or carbon. It is a commonly used method to assess the quantity of the organic and carbonate content (Dean, 1974). For FA-F, LOI usually represents residual unburnt carbon which may have detrimental effects on the properties of the produced concrete. The organic matter due to its high specific surface area, absorbs water and chemical admixtures such as water reducing admixtures or air-entraining agents, hindering their efficiency (Chen et al., 2021). In the case of GGBFS, due to the high CaO content LOI can be associated with calcium forms of volatile components such as CaCO_3 rather than residual organic carbon. For Microsilica or Silica Fume LOI represents mainly the unburnt

carbon and that is evident also in the difference of the colour between USF which has a significantly lower LOI than DSF which is darker as depicted in Figure 3.3. As in the case of FA-F, the residual carbon due to its high specific surface area absorbs water while it might also hinder the reaction of silica with the free lime in the system. The latter reaction forms a secondary C-S-H phase along with geopolymerization that enhances the strength of the produced geopolymer while it delays setting as silica consumes the free lime reducing precipitation. Regarding MK, LOI value could be attributed to the presence of water released during dehydroxylation of residual kaolinite. For natural weathered rocks (TRD, FP and GS) and volcanic ashes (PM) the LOI represents hydrated compounds rather than unburnt carbon and should not raise concerns.

Table 3.1: Chemical composition of precursors in wt. %

Chemical Composition (%)	FA-F	GGBFS	USF	DSF	MK-750	PM	TRD	FP	GS
SiO₂	56.5	35.2	98	94.5	52.1	76.2	49	72.1	62.4
Al₂O₃	23.5	8.37	0.3	0.41	43.3	13.5	13.88	15.1	17.2
Fe₂O₃	3.55	0.68	0.1	0.16	0.47	1.1	14.9	0.47	3.49
FeO	-	-	-	-	-	0.1	-	-	-
MgO	1.08	10.7	0.1	0.3	0.05	0.05	4.73	0.1	1
CaO	9.1	38.9	0.3	0.49	0.08	0.8	7.61	0.36	2.51
Na₂O	2.13	0.31	0.1	0.15	0.35	1.6	3.94	3.86	4.57
K₂O	0.84	0.59	0.2	0.59	0.19	1.8	0.6	6.35	5.66
TiO₂	0.63	0.34	-	<0.01	1.42	0.2	2.41	0.01	0.77
P₂O₅	0.09	0.02	0.03	0.08	0.06	-	0.34	0.24	0.18
MnO	0.06	0.27	-	0.04	<0.01	-	0.18	0.03	0.07
Cr₂O₃	0.02	0.02	-	0.02	0.03	-	0.02	0.02	<0.01
V₂O₅	0.01	<0.01	-	<0.01	0.03	-	0.06	0.01	<0.01
SO₃	-	2.93	0.1	-	-	-	-	-	-
LOI	1.89	1.32	0.6	3.5	2.01	5	3.33	0.97	1.23

3.1.3. Mineralogical Analysis

The mineralogy of the aluminosilicate precursors plays a vital role in geopolymerization. For this reason, measurements were taken to determine through X-Ray Diffraction (XRD) the mineralogy and degree of crystallinity of the raw materials. Table 3.2 illustrates the results of the analysis. The presence of mullite and quartz in FA-F in combination with the relatively lower amorphous content (compared to the rest) suggests that the combustion temperature of FA-F was higher than 850 °C but lower than 1500 °C. GGBFS was found highly amorphous with the presence of gehlenite which is favourable in terms of geopolymerization. However, previous studies have shown that it cannot be used alone as a geopolymer precursor. During alkalination, gehlenite

depolymerizes into hydrates and precipitates alkalis. The resulting product is not a geopolymer, but an alkali activated slag, which despite its high strength, presents problems of stability and durability in case the material comes in contact with water. This is because geopolymerization is incomplete and the alkali cation from the hardener is not chemically bonded to the structure. MK-750 and PM are highly amorphous and thus highly reactive and could be used as precursors. Meanwhile for TRD using the XRD and XRF analysis combined it can be concluded that the precursor is Meta-Gabbro (MG), a type of intrusive igneous rocks, product of low-grade metamorphism of gabbroic rocks. Due to relatively low amorphous content FP and GS could be used as partially reactive aggregates which will create a better bond with the geopolymer paste, as their surface is amorphous while their core remains crystalline.

Table 3.2: Mineralogical composition of the studied precursors in wt. %

Mineral	Mineral Formula	FA-F	GG BFS	USF	DSF	MK-750	PM	TRD	FP	GS
Quartz	SiO ₂	11.1	-	-	-	1.4	-	7.5	72.1	13.4
Mullite	2Al ₂ O ₃ · SiO ₂	19.4	-	-	-	-	-	-	15.1	-
Calcite	CaCO ₃	0.5	-	-	-	-	-	-	0.47	-
Silicon Carbide	SiC	-	-	1	2.8	-	-	-	-	-
Gehlenite	Ca ₂ Al(AlSi)O ₇	-	3.2	-	-	-	-	-	0.1	-
Alite	Ca ₃ SiO ₅	-	1.2	-	-	-	-	-	0.36	-
Larnite	Ca ₂ SiO ₄	-	0.2	-	-	-	-	-	3.86	-
Albite	NaAlSi ₃ O ₈	-	-	-	-	-	-	31.9	6.35	37.3
Muscovite	KAl ₂ (AlSi ₃ O ₁₀)(OH) ₂	-	-	-	-	-	-	-	0.01	2.8
Magnetite	Fe ₃ O ₄	-	-	-	-	-	-	5.6	0.24	0.18
Chlorite	(Fe,(Mg,Mn) ₅ ,Al)(Si ₃ Al)O ₁₀ (OH) ₈	-	-	-	-	-	-	11.0	0.03	0.9
Diopside	CaMgSi ₂ O ₆	-	-	-	-	-	-	1.5	0.02	3.4
Pyrite	FeS ₂	-	-	-	-	-	-	0.3	-	0.3
Phlogopite	KMg ₃ (AlSi ₃ O ₁₀)(OH) ₂	-	-	-	-	-	-	-	0.1	16.8
Microcline	KAlSi ₃ O ₈	-	-	-	-	-	-	5	0.97	23.3
Hematite	Fe ₂ O ₃	-	-	-	-	-	-	0.8	-	-
Actinolite	Ca ₂ (Mg,Fe) ₅ Si ₈ O ₂₂ (OH) ₂	-	-	-	-	-	-	9.6	-	-
Epidote	Ca ₂ (Al,Fe) ₅ Si ₈ O ₂₂ (OH) ₂	-	-	-	-	-	-	10	-	-
Augite	(Ca,Na)(Mg,Fe,Al)(Si,Al) ₂ O ₆	-	-	-	-	-	-	2.1	-	-
Titanite	CaTi(SiO ₄)O	-	-	-	-	-	-	5.2	-	-
Plagioclase	(Na,Ca)(Si,Al) ₄ O ₈	-	-	-	-	-	2	-	-	-
Amorphous		69	95.4	99	97.2	98.6	98	10.5	-	*

* The weight percent quantities indicated have been normalized to a sum of 100%. The quantity of amorphous material has not been determined

3.1.4. Reactivity Index

Another reactivity and suitability index was determined through pH measurements. This measurement determines whether there is a risk of flash setting. The pH of aqueous solutions consisting of 5 g of raw material in 50 mL of distilled water was measured after 5 minutes of preparation. Generally, values lower than 8 do not indicate danger for instant hardening, whereas fast hardening occurs for values between 8 and 11 and flash-set is highly possible for values above 11. The recorded values of pH of the materials are listed in Table 3.3.

Table 3.3: Reactivity Index of precursors

pH value after 5min	FA-F (aq.)	GGBFS (aq.)	USF (aq.)	DSF (aq.)	MK-750 (aq.)	PM (aq.)	TRD (aq.)	FP (aq.)	GS (aq.)
	11.5	11.8	6.5	8.1	6.3	7.2	6.1	7	8.5

3.1.5. Alkaline Reagents/Soluble Silicates

One of the most crucial ingredients in geopolymer chemistry is soluble alkali silicates, as they serve as the trigger of the geopolymerization mechanism. The composition, important parameters and the role of alkaline reagents are explained in detail in Chapter 2. A significant parameter in choosing the alkaline soluble silicate is the molar ratio MR which refers to the moles of SiO₂ to moles of Na₂O or K₂O contained in the solution. However, the industry of silicates uses the weight ratio WR to label their products, which is not the same with MR. To convert the WR to MR the following equation is used for sodium (SS) and potassium silicate (PS), respectively:

$$\begin{aligned}
 MR_{SS} &= \frac{M_{Na_2O}}{M_{SiO_2}} \cdot WR = \frac{61.98 \text{ (g/mol)}}{60.08 \text{ (g/mol)}} \cdot WR = 1.032 \cdot WR \\
 MR_{PS} &= \frac{M_{K_2O}}{M_{SiO_2}} \cdot WR = \frac{94.2 \text{ (g/mol)}}{60.08 \text{ (g/mol)}} \cdot WR = 1.568 \cdot WR
 \end{aligned}
 \tag{3.1}$$

In Equation (3.1) M_{SiO_2} , M_{Na_2O} and M_{K_2O} are the molar masses of SiO₂, Na₂O and K₂O, respectively. Commercially available silicates have a MR ranging from 0.4 to 4.0 with silicates that have values under 1.45 being very corrosive and unstable due to the high causticity and the lack of three-dimensional structure. Meanwhile high MR values such as 3 and 4, which are most commonly found are less reactive and could be lowered by dissolving NaOH or KOH pellets into the silicates.

In this project, the following alkaline reagents have been tested: (i) a solution of soluble Sodium Silicate of MR=3.32 and NaOH flakes to produce a Sodium Silicate solution of MR=1.5 (SS-SH-1.5), (ii) a solution of anhydrous Potassium Silicate of MR=3.92, distilled water and KOH flakes to produce a Potassium Silicate of MR=2 (PS-PH-2.0), (iii) a solution of soluble Potassium Silicate of MR=3.29 and KOH flakes to produce a Potassium Silicate of MR=1.5 (PS-PH-1.5), (iv) a solution of soluble Potassium Silicate of MR=3.29 and KOH flakes to produce a Potassium Silicate of MR=1.5 (PS-PH-1.7) and (v) a commercially available Soluble Potassium Silicate with MR=1.7 (PS-1.7). The name convention for the alkaline reagents used in this section is given in Figure 3.4.

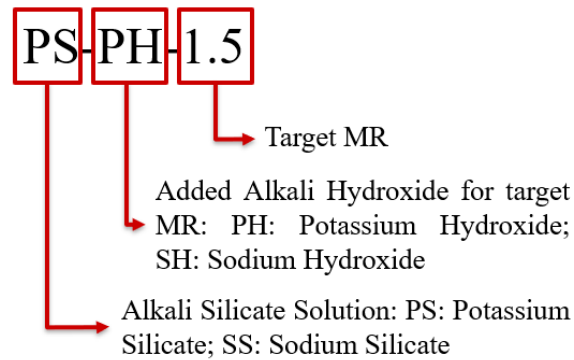


Figure 3.4: Overview of Alkaline Reagents Naming Convention

Solutions SS-SH-1.8 and PS-PH-1.5 and PS-PH-1.7 were fabricated by dissolving NaOH or KOH pellets in distilled water, and then after 24hr the NaOH or KOH solutions were mixed with Sodium and Potassium Silicate, respectively. The resulting mixtures were left to cool for another 24h prior to mixing. For solution PS-PH-2.0, the anhydrous Potassium Silicate was mixed with distilled water to obtain a soluble potassium silicate solution with 60% wt. water and when the solution stabilized, a KOH solution fabricated as prescribed above was used to lower the MR. Finally, for PS-1.7 24hr prior to casting, tap water was added in the solution to increase fluidity. It is important to add water in the alkaline reagents in advance, so that it is chemically bound to alkali silicate, otherwise it will not be incorporated in the system resulting in evaporation, macro-porosity, and cracks.

The suitability of each alkaline reagent was tested through trial-and-error while related literature verified the process. (A total of 9 mixes were tried before arriving at a final formula). More details about the trial mixes can be found in Appendix A. Table 3.4 summarizes the properties of each

tested reagent. In all cases of lab-made silicates (SS-SH-1.5, PS-PH-1.5 and PS-PH-1.7), the results in mechanical performance were lacking repeatability, while erratic behaviour was also observed in terms of workability and setting time. More specifically mixes where SS-SH-1.7 was used, required a high amount of extra water in their system for workability while during curing they developed efflorescence. The findings are in accordance with the literature, as Sodium Silicates are generally more viscous than Potassium Silicates, thus require more water which, in combination with Na ions being lighter than K ions, increase the risk of leaching and thus efflorescence. The issues of repeatability could be attributed to the fact that sodium silicates performance is generally temperature dependent as described in Chapter 2. In case of PS-PH-2.0 the produced solution is not stable as a glassy precipitate was observed after mixing the anhydrous silicate with distilled water, so it was not used in any mix at all.

According to the literature, there are different technologies used to produce soluble silicates in the industry and along with the different types of raw materials, these methods affect the reactivity and the type of molecular structure (Davidovits, 2020). Besides the instability of the reagent, it is unsure if any geopolymerization occurred in the mixes, as low strengths and high dispersion was observed in the results. Although PS-PH-1.5 was the most reactive in terms of MR, optimum strength was not achieved, as the high causticity hindered the workability and thus extra water was required, while flash setting occurred in several mixes.

To tackle the above challenges, a commercially available Potassium Silicate with MR=1.7 was used in the remainder of the study. PS-1.7 as an industrially produced silicate was stable and did not cause any workability or setting issues, while high strengths were achieved with repeatability. Finally, a lab-fabricated potassium silicate with MR=1.7 was tested, too in order to compare with the industrial silicate. The latter was found to be the best option of the lab-fabricated reagents. However, issues with workability, repeatability and higher water demand, thus higher porosity were inevitable. For the above reasons, PS1.7 was selected as the most suitable hardener for the experimental program.

Table 3.4: Alkaline Reagents

Reagents	SS-SH-1.5	PS-PH-1.5	PS-PH-2.0	PS-PH-1.7	PS-1.7
Sodium Silicate (aq.)	✓	-	-	-	-
Sodium Hydroxide pellets	✓	-	-	-	-
Potassium Silicate (aq.)	-	✓	-	✓	✓

Potassium Hydroxide pellets	-	✓	✓	✓	-
Anhydrous Potassium Silicate	-	-	✓	-	-
Distilled water	-	-	✓	-	-
Initial MR	3.32	3.32	3.92	3.32	1.7
New MR	1.5	1.5	2.0	1.7	1.7
New SiO ₂ (%wt.)	29	19.5	22.43	23.52	23.52
New Na ₂ O (%wt.)	19.95	-	-	-	-
New K ₂ O (%wt.)	-	20.5	17.57	21.48	21.48
New H ₂ O (%wt.)	51.05	60	60	55	55
High Water Demand	✓	✓		✓	-
Stability Issues	✓	✓	✓	✓	-
Poor workability	✓	✓		-	-
High Risk of Flash Setting	✓	✓	-	-	-
High Risk of Efflorescence	✓	-	-	-	-
High Strength	✓	✓	-	-	✓
Repeatability in Results	-	-	-	-	✓

3.2. Geopolymer Binder Formulations

Geopolymerization is a mechanism of advanced alumino-silicate inorganic polymer chemistry that in order to produce stable and high-performance concrete with repeatability, chemical ratios and equilibrium must be attended to in the microstructure (Davidovits, 2020). Such material should have an atomic ratio (Na,K)/Al=1 and Si/Al=3 in the geopolymer binder, excluding the fillers and aggregates. The first ratio is to prevent free alkalinity, and efflorescence while the second is for the formation of a rigid 3-D macromolecular structure. When (Na,K)/Al is less than 1, the alumino-silicate oxides are in excess and might not polycondense; as a result, they remain as white powder inside the hardened geopolymer paste. It should be noted here that the above ratio applies for precursors rich mainly in silica and alumina. In case of presence of high amounts of other ions that participate in geopolymerization (i.e., Ca, Fe) the ratio is adjusted. Regarding the second rule, a low Si/Al of 1, 2,3,4 initiates a rigid 3D-Network, while higher order (greater than 15) confers a linear polymeric character. In this section, different formulas are given for different aluminosilicate precursors. In this section, several geopolymer formulas on different precursors will be discussed and since the alkaline reagent selected in this project is PS1.7 the atomic ratio K/Al=1 will be used. To construct the formulas, the chemical composition of the precursors and soluble potassium silicate PS1.7 will be used from Table 3.1 and 3.4, respectively.

3.2.1. Formula for Pure Aluminosilicate Precursors (MK-750): K/Al=1

Among the commonly used raw materials for geopolymer binders, MK-750 is the most suitable main precursor, as it is rich in alumina and silica, highly amorphous and since it is an industrial

product, it is pure with consistent properties. According to Davidovits (2020), Equation (3.2) can be used to determine the amount of MK-750 (powder) that will completely react with 100gr of alkaline reagent (liquid silicate) to form a geopolymer binder with K/Al=1:

$$Q_{\text{powder}} = \frac{100 \text{ gr liquid silicate} \times (\% \text{wt. } K_2O \text{ in liquid}) \times 101.96 (\text{gr/mol } Al_2O_3)}{94.19 (\text{gr/mol } K_2O) \times (\% \text{ wt. } Al_2O_3 \text{ in powder})} \quad (3.2)$$

Numerals in the equation, i.e., 101.96 gr/mol and 94.19gr/mol, are the molar masses of Al_2O_3 and K_2O , respectively. This formula can be also used for highly amorphous aluminosilicate materials such as PM.

3.2.2. Formula for Ca-rich Aluminosilicate Precursors (GGBFS): (K+Ca)/Al=1

Ca-rich aluminosilicate raw materials such as GGBFS induce hardening at room temperature while enhancing the early strengths. However, as discussed in Chapter 2, due to its mineralogy GGBFS cannot be used as the main precursor. It should be noted here that half of the Al_2O_3 participates in geopolymerization while the other half precipitates in the form of $Al(OH)_3$ (Davidovits, 2020). For the amount involved in geopolymerization, the negative charge of every Al^- atom must be balanced with a corresponding positive charge of one K^+ or half of Ca^{2+} . In that sense the formula for Ca-based geopolymers is further simplified to K/Al=0.67 and Ca/Al=0.33. Moreover, it has been observed in the literature that only two thirds of Ca from GGBFS participates in geopolymerization, while the rest precipitates in the form of $Ca(OH)_2$ or reacts with CO_2 to produce carbonation products. Taking into consideration the above, Equation (3.3) which is a modification of Equation (3.2), determines the amount of GGBFS needed to react with 100gr of soluble silicate and create a balanced geopolymer binder.

$$Q_{\text{powder}} = \frac{100 \text{ gr liquid silicate} \times (\% \text{wt. } K_2O \text{ in liquid}) \times 101.96 (\text{gr/mol } Al_2O_3)}{94.19 (\text{gr/mol } K_2O) \times (\% \text{ wt. } Al_2O_3 \text{ in powder}) \cdot \frac{2}{3}} \quad (3.3)$$

3.2.3. Formula for FA-F: K/Al=0.2

As explained in Chapter 2, the difference between a FA-F geopolymer binder and alkali activated FA-F is that in case of the later, total dissolution of fly ash is the goal to produce an alkali activated binder. This is achieved with highly caustic alkaline reagents that will attack the FA cenospheres.

However, in geopolymer chemistry, the amount of FA-F is calculated based on depolymerization of the cenospheres' surface, to achieve high strength (Davidovits, 2020). It is assumed that only one fifth of the available Al_2O_3 in FA-F reacts superficially with the soluble silicate, thus instead of $K/Al=1$, $K/Al=0.2$ is used, and Eq. (3.2) transforms to Equation (3.4):

$$Q_{powder} = \frac{100 \text{ gr liquid silicate} \times (\%wt. K_2O \text{ in liquid}) \times 101.96(\text{gr/mol } Al_2O_3)}{94.19(\text{gr/mol } K_2O) \times 0.2(\%wt. Al_2O_3 \text{ in powder})} \quad (3.4)$$

3.2.4. Formula for Fe-rich Aluminosilicate Precursors (TRD): $K/Al=1$ and $K/Fe=0.2$

One of the latest advancements in geopolymer technology is the rock-based or ferro-sialate [Fe-O-Si-O-Al-O]-based geopolymer systems. In such geopolymers trivalent Fe^{3+} from geological elements rich in iron oxides substitutes up to 25% for the Al. Previously, TRD using the information of XRD and XRF analysis was identified as meta-gabbro (MG), a mafic intrusive igneous rock that is rich in iron minerals, and thus qualifies for precursor of ferro-sialate-based geopolymer binders. According to Davidovits (2020), Fe substitutes part of Al the formulas for such geopolymers are transformed to $K/Al=1$ and $K/Fe=0.2$. More specifically:

$$Q_{powder} = \frac{100 \text{ gr liquid silicate} \times (\% \text{ wt. } K_2O \text{ in liquid}) \times 101.96(\text{gr/mol } Al_2O_3)}{94.19(\text{gr/mol } K_2O) \times \left\{ \left[\frac{101.96(\text{gr/mol } Al_2O_3)}{159.69(\text{gr/mol } Fe_2O_3)} \times \frac{(\%wt. Fe_2O_3 \text{ in powder})}{5} \right] + (\%wt. Al_2O_3 \text{ in powder}) \right\}} \quad (3.5)$$

Where c is the percentage by weight of Fe_2O_3 in the iron-rich powder, which here is MG, whereas 159.69 gr/mol is the molar mass of Fe_2O_3 .

3.3. Effect of Most Commonly Used Source Materials on Mechanical Properties & Microstructure

A preliminary FA-F-based mix design based on the above geopolymer formulations was used to validate the produced geopolymer in terms of mineralogy and assess the effect of the most commonly used source materials in the microstructure. Subsequently, the geopolymer was cured at an ambient temperature and was then characterized in terms of fresh, hardened, physical, microstructural, and mineralogical properties. A relation between the latter and the raw materials used was also identified.

3.3.1. Materials and Methods – Preliminary THFRG Mix Design

The materials used in this preliminary study are FA-F, DSF, GGBFS, MK-750, PS-1.7 used as alkaline reagent, a mix of coarse grained TRD ($D_{max}=600\text{ }\mu\text{m}$) and GS as aggregates, short straight steel fibers with $l_f=12\text{ mm}$ and $d_f=0.2\text{ mm}$ while a polycarboxylate HRWRA was used to increase workability. In this preliminary study three cases are examined varying the amount of GGBFS and MK-750, while keeping FA-F amount constant. In all three cases 60% of the alkali silicate is assigned to FA-F while for mix (i) 35% is assigned to MK-750 and the rest 5% to GGBFS (Mix ID is GPC1-S5), for mix (ii) 37.5% to MK-750 and 2.5% to GGBFS (Mix ID is GPC1-S2.5) and for mix (iii) 40% MK-750 (Mix ID is GPC1-S0). DSF, FP, coarse TRD and GS amounts were kept constant, with a sand to wet binder ratio of 0.47. An overview of mix naming convention is given in Figure 3.5. In all mixes Equations 3.2- 3.4 were modified based on the corresponding silicate amount that is assumed to react with the aforementioned precursors. The available soluble silicate amount is 355 gr per Liter of fresh geopolymer concrete mix. Based on the above information, the calculated mix proportions are shown in Table 3.5.

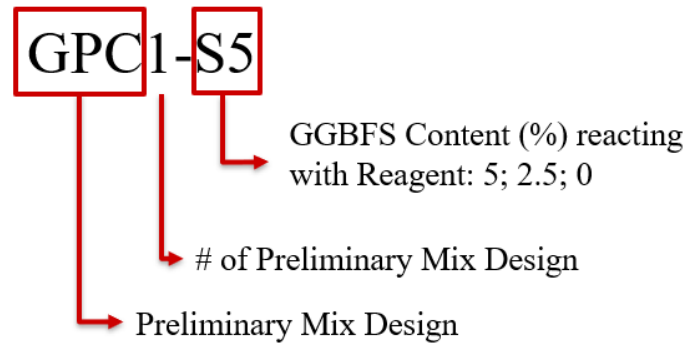


Figure 3.5: Overview of Preliminary Mix Designs with varying GGBFS content

Table 3.5: Mixture Design GPC1

Materials	Proportions (kg/m ³)		
	GPC1-S5	GPC1-S2.5	GPC1-S0
FFA	907	907	907
DSF	145	145	145
MK-750	75	80	92
GGBFS	94	47	-
PS-1.7	355	355	355
Water	71	71	71
Coarse TRD	574	574	574
GS	203	203	203
HRWRA	18	18	18
Steel Fibers	160	160	160

For the mixing of geopolymer concrete, a 5 L Hobart planetary mixer was used. The mixing process comprises of dry mixing of FFA with DSF for 3 minutes, followed by the addition of the alkaline reagent which was mixed with water and high-speed mixing (200 rpm) for 10 minutes. Next, MK-750 is added and mixed for 5 minutes. The last binder material added was GGBFS and was mixed for another 3 minutes. Subsequently the sand mixture was added to the system and mixing continued for 2 min. At this point HRWRA was introduced to lower the viscosity and prepare the mix for fiber impregnation. When the mix gained the desired fluidity (using the testing procedure described in Section 3.3.2), fibers were added, and mixing continued for 2 more minutes. The concrete was then cast in cubic moulds of 50 mm size (minimum dimension had to be about 4-5 times the fibre length). Mild vibration was then performed through the use of tamping rods. Finally, the specimens were sealed with a plastic sheet to prevent water evaporation. The specimens were demoulded after 24 h and cured in water at ambient temperature until the day of testing.

3.3.2. Fresh Properties

Flowability tests were conducted to evaluate the fresh properties of the developed concrete. The test was performed in accordance with ASTM C1856 (2017). The flowability of each mixture was measured immediately after mixing and right before casting. During this measurement, the conical mould of the flow table was filled with a single layer of fresh geopolymer concrete. Then, the mould was lifted and after one minute, the reported flow value was calculated as the average of maximum and minimum diameter of the spread. The flow values were 157, 172 and 181 mm for the mixtures GPC1-S5, GPC1-S2.5, and GPC1-S0, respectively. The increase in flowability with the decrease of GGBFS even in such small proportions was evident and linked to the fineness and high CaO content of GGBFS.

The final setting time was also measured empirically as the time from the start of casting to the time the geopolymer completely hardens. The final setting times were 15, 22 and 30 min for the mixtures GPC1-S5, GPC1-S2.5, and GPC1-S0, respectively. The short setting time was attributed to the high pH of the precursors and particularly the high content of CaO in both GGBFS and FFA.

3.3.3. Compressive Strength

A force-controlled compression-testing machine was used to test the 50 mm cubes under compression at a loading rate of 0.259 MPa/s in accordance with ASTM C39, (2021). To study the age effect on compressive results, the specimens were tested at the age of 1, 7 and 28 days. The compressive strength results of each mixture at different age is presented in Figure 3.6. Note that the reduction in the GGBFS did not affect the strength at early ages owing to the substitution by MK-750. However, the absence of GGBFS had a more prominent effect on the final strength of GPC1-S0.

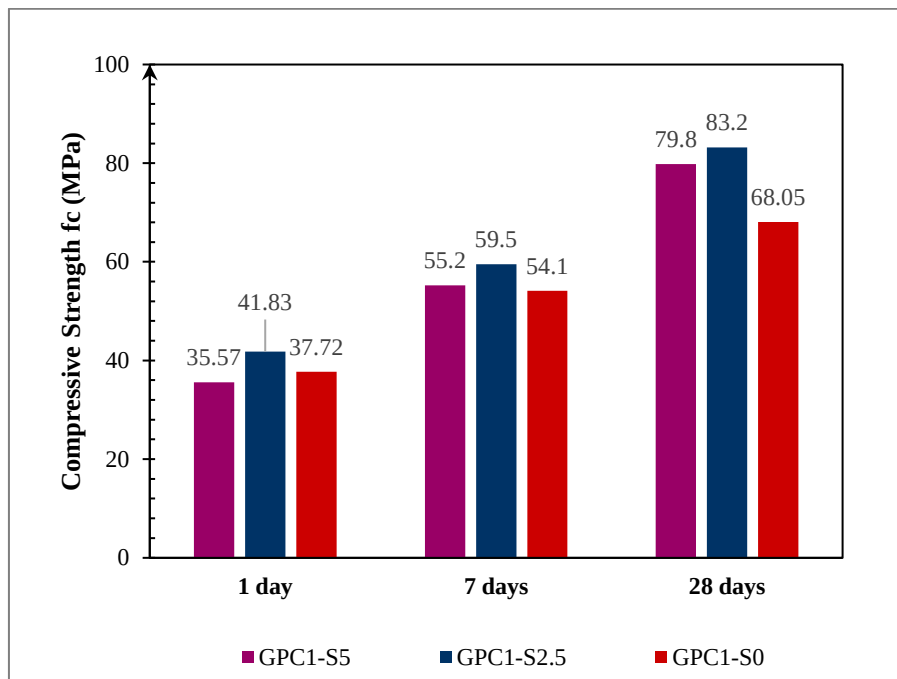


Figure 3.6: Compressive strength development of GPC1

3.3.4. Mineral Analysis

In order to study the microstructure and mineralogy of the produced geopolymer concrete a QEMSCAN (Quantitative Evaluation of Materials by Scanning Electron Microscopy) analysis was conducted. The system is widely used in the minerals industry as it provides phase and mineral identification at micrometre-scale. Polished thin sections were taken from each cube sample. The QEMSCAN results are illustrated in Figure 3.7(a). The resolution of the produced images is 15 μm . Figure 3.7(b) also shows the quantitative composition of the geopolymer mixtures. The mineral maps of all mixtures contain the same major mineral, K-feldspar. The

second prevailing mineral is albite. The results agree with what is expected from a geopolymerization process as the K-feldspar and albite are frameworks with 3-dimensional networks. More specifically the produced concrete is a K, Na-Poly(sialate-disiloxo) with atomic ratio Si/Al=3 whereas its geological analogue is sanidine and albite (Davidovits, 2020). The white parts of the images represent the porosity. The blue parts correspond to fibres with their brass coating intact while the black represent the steel fibres that have lost their coating during hardening.

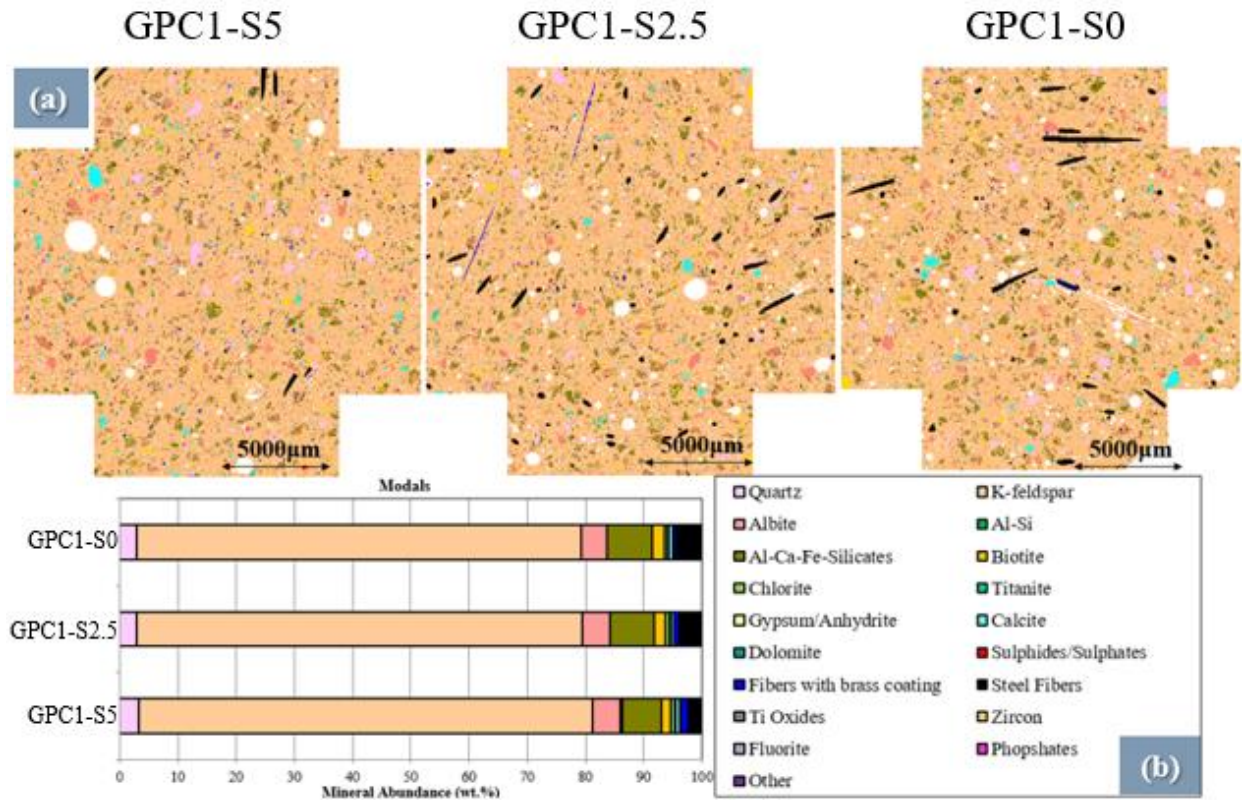


Figure 3.7: QEMSCAN images of GPC1 thin sections. (a) Phase assemblage maps (b) total mineral contents of each sample by mass

3.3.5. Fiber Distribution

QEMSCAN images are used as a tool to interpret the distribution of fibres in the thin sections. A schematic (Figure 3.8) is used to assess the number of fibres crossing an arbitrary plane, a parameter that is critical for assessing their contribution to the mechanical properties of the solid material. The particles with black and blue colour having a dimension in their short axis at least equal to the fibre diameter $d_f = 0.2$ mm were identified as fibres (the typical fiber is a cylinder

with a diameter of 0.2 mm oriented randomly with respect to the plane of the cross section, therefore in general the intersection has an elliptical shape).

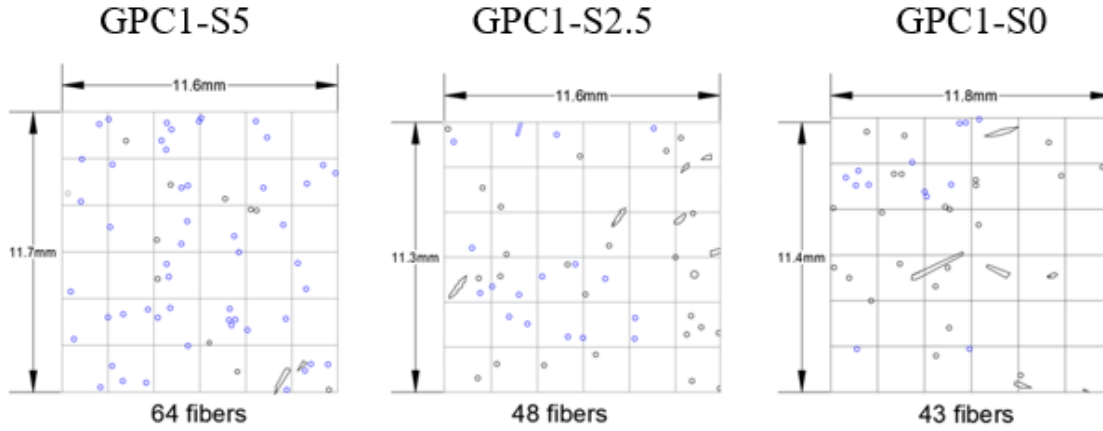


Figure 3.8: Schematic of thin section QEMSCAN images and the number of fibres per cross section of GPC1 specimens.

In a random arrangement of fibres, the number of fibres, n_f , crossing a unit area is estimated from the cross-sectional area of a single fibre having a diameter (d_f) and the volumetric ratio of the fibres, V_f (Archontas & Pantazopoulou, 2015).

$$n_f = 0.64 \frac{V_f}{d_f^2} \quad (3.6)$$

Equation 3.6 has been derived assuming only 50% of the fibers to be effective on account of random spatial orientation; for preferential arrangement of the fibers effected by the casting procedure this factor may be higher.

For $V_f = 2.5\%$ and $d_f = 0.2$ mm, the n_f should be 0.4 fibers per mm^2 . Based on the aforementioned data, ideally the number of fibers at the given randomly cut 11×11 mm cross-sections should have been around 54; on average the three samples shown in Figure 3.8 agree quite well with this estimation.

3.3.6. Porosity

QEMSCAN images were further used to provide a rough estimation of porosity and its distribution at the analysed thin sections. Figure 3.9 shows the porosity as a function of size and surface area. The porosity can be attributed to three different mechanisms: (a) the production of

gas, (b) the fast increase of viscosity with time during casting, and (c) the compaction of the material resulting from simple casting rather than extrusion (Prud'homme et al., 2010).

The first mechanism is a result of incorporation of SF in the mix design. It is worth noting that studies have shown that even small content (<1%) of elemental silicon in SF could generate gas approximately 1 to 11 mL/gr of SF (Zhang et al., 2000). More specifically, this elemental silicon is oxidized in an alkaline environment (Equation (3.7)). H₂ is generated as a result of water reduction and acts as a foaming agent (Equation (3.8)). The overall reaction forms Si(OH)₄ as shown in Equation (3.9). The chemically induced porosity seems to yield micropores in the range of 0-50 µm. This is clearly shown in Figure 3.7 where the porosity distribution is depicted demonstrating that all mixtures have similar percentages of 0-50 µm micropores.



Another reason for gas production could be attributed also to the high elemental carbon content in FA-F. This is further explained in a following parametric study later on in this chapter.

The increased viscosity due to the addition of very fine and high-density particles such as SF, MK-750 and GGBFS and the incorporation of the fibres also contributes to the pore formation as the diffusion of the generated gas is enhanced (Prud'homme et al., 2010).

Finally, compaction also affects the porosity, as it depends on the viscosity of the mixture. In case of S5, the mixture with the highest amount of GGBFS was difficult to compact as much as the other two. This is evident from the high content of pores with maximum size greater than 500 µm in S5, as shown in Figure 3.9.

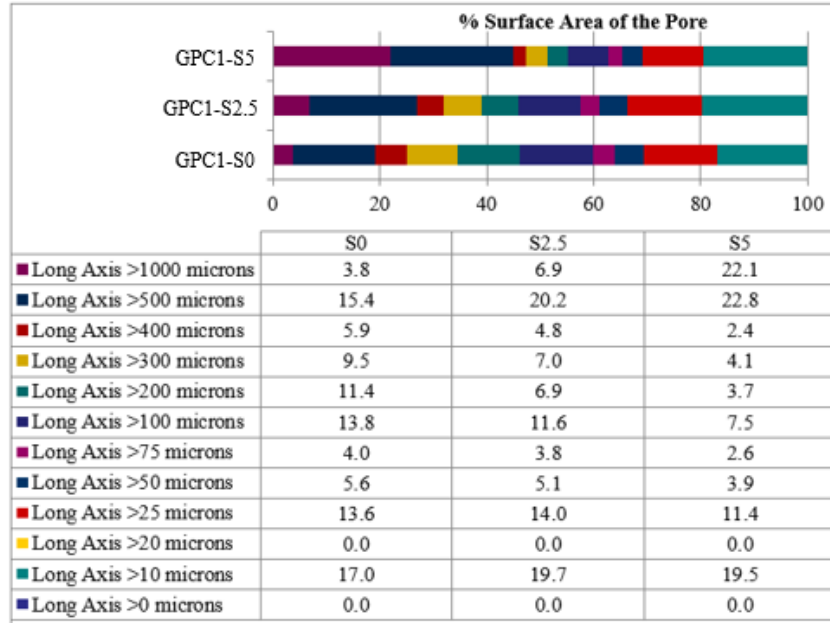


Figure 3.9: Porosity distribution measured through QEMSCAN analysis on GPC1 specimens

3.4. Behaviour of Geopolymer Mortars Based on Different Geopolymer Formulations

All of the above formulas were used alone to further understand the role of every precursor tried in the preliminary study individually, and in order to assess their potential for large scale applications. The formulations were implemented in a geopolymer mortar with coarse TRD ($D_{\max}=600 \mu\text{m}$), and GS used in lieu of sand. The mix proportions of the geopolymer matrices are shown in Table 3.6.

Table 3.6: Geopolymer Mortars based on formulations

Materials	Proportions (gr)		
	MK-750	GGBFS	FA-F
FA-F	-	-	495
MK-750	53.7	-	-
GGBFS	-	833	-
PS-1.7	100	100	100
Water	50	50	50
coarse TRD	162	162	162
GS	57	57	57

3.4.1. MK-750-based Geopolymer Matrix

The mixing procedure prescribed in the previous section was followed in preparing a geopolymer matrix with MK-750. In this case the mix was very viscous. Due to the high fineness of MK-750,

it is common to add extra amount of soluble silicate till sufficient workability is achieved (Borges *et al.*, 2014). In this case, another 50 gr of PS-1.7 were added, and mixing continued till the desired workability was achieved. Then, coarse TRD and GS were added and the geopolymer concrete was mixed further, for another 3 minutes. When mixing was complete, the material was cast in 50 mm cubes. Although the mix demonstrated eventually increased flowability, the specimens could not harden in room temperature even after 48 hr, while segregation was evident as shown in Figure 3.10. The above issues are posing limitations for MK-750 based geopolymer concrete in large applications.



Figure 3.10: MK-750 based geopolymer matrix specimen demonstrating setting and segregation issues

3.4.2. GGBFS-based Geopolymer Matrix

Although it is already known that using GGBFS as the only precursor leads to alkali activated materials which have completely different mechanism of strength development, a GGBFS-based mortar matrix was fabricated. The mix was highly viscous and extra amount of silicate (100 gr) was needed to improve the properties. The produced mortar showed flash setting, minutes after it was poured into the moulds. As illustrated in Figure 3.11, the hardened specimens showed cracking patterns that could be attributed to the flash setting. The characteristic blue/ green colour is assumed to be linked with the presence of sulphur in GGBFS. More specifically, the literature assumes this coloration to be the result of the formation of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ and/or $\text{FeSO}_4 \cdot 5\text{H}_2\text{O}$ compounds (Chaouche *et al.*, 2017).



Figure 3.11: Drying Shrinkage cracking patterns on a discolored GGBFS-based mortar

3.4.3. FA-F-based Geopolymer Matrix

Finally, a trial geopolymer binder with only FA-F used as precursor was developed. During the resting period between mixing and casting, bubbles were formed with a distinct black outline that could be linked to the residual unburnt coal. This phenomenon indicates the production of a gas. Those bubbles are only present in mixes with FA-F content and can also be distinguished during assessment of fresh properties, such as flowability measurement as illustrated in Figure 3.12(a) and (b). Figure 3.12(c) shows the large, rounded pores of the fractured surface of FA-F based geopolymer mortar. Further research is needed to validate this theory.

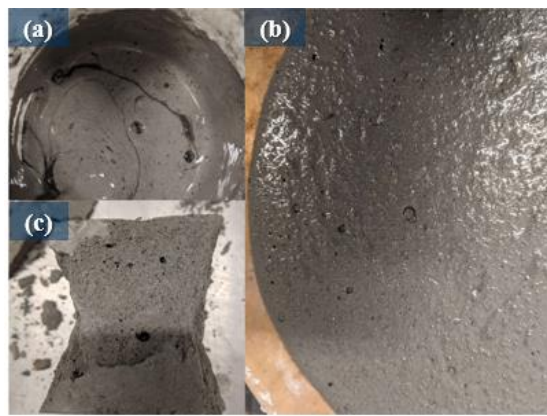


Figure 3.12: (a) Unburnt coal “bleeding” and bubble formation, (b) bubbles with black outline in a flowability measurement, (c) fractured FA-F-based geopolymer mortar specimen with large, rounded pores

All of the above precursors, when used alone in geopolymer systems do not yield the best performance in terms of workability, setting time or physical and mechanical properties and thus an optimized mix design with combination of the available precursors is recommended to achieve best results.

3.5. Aggregate Skeleton

3.5.1. Particle Packing Density

Porosity and level of capillary voids interconnectivity are two major factors that significantly affect the performance of cementitious materials. The development of a concrete of high performance is a challenge for it is a material with highly nonhomogeneous nature that comprises of a wide range of different particles in shape and size. Instead of following the tedious trial and error method, particle packing density theory that is based on physical mechanisms, could be used to optimize a concrete mix design. Despite being proposed in the 1900s, recent advancements and

emerging trends in concrete such as ultra-high-performance mixes brought the theory back to the spotlight of concrete research interest. The optimization of particle packing density is efficiently used to design advanced sustainable concrete mixtures with improved rheological properties and workability characteristics. Packing density is defined as the ratio of solid volume of particles over the bulk volume occupied by these particles. In the 1960's, Powers (1969) investigated the effects of packing density of aggregate on the fresh properties of concrete. Earlier, Kennedy (1940) proposed the excess paste theory. According to that theory, concrete shall be envisioned as a mix of cement paste and aggregates, where the former has twofold purpose; the dispersion and lubrication of concrete mix and filling up the voids between aggregates. In essence, higher packing density of aggregates results in reduction of paste demand, while part of the excessive paste is now available to improve the rheological properties of the concrete mix. What is more, higher dimensional stability, less porosity and thus higher strength can be achieved with high packing density. This is evident in Figure 3.13, where different packing density scenarios are illustrated. Generally, the schematic figures of this section represent coarse aggregates as blue particles, fine aggregates as dark gray, cementitious particles as light gray and ultra-fine fillers as black. Factors affecting the packing density are particle gradation, way of placement and casting, method and degree of compaction, shape, and size of particles (Table 3.7).

Table 3.7: Parameters influencing the packing density (Gray, 1968)

Particle	Boundary	Placement / Casting	After placement
Shape	Shape	Intensity	Vibratory compaction
Texture	Size	Velocity of placement	Pressure compaction
Size Distribution	Elasticity	Method	
Density	Surface properties		
Elasticity			
Resilience			
Surface properties			

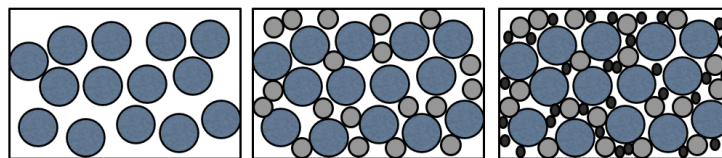


Figure 3.13: Typical representation of particle packing definition, from left to right: low, medium, and high packing density

Concrete is a suspension of aggregate in paste, where the size of the granular particles defines the magnitude of inter-particle forces and hence the predisposition of particles to agglomerate. The solid particles in cementitious suspensions are classified as (i) non-colloidal particles such as

aggregates, where only mechanical interactions occur (Figure 3.14(a)), (ii) dispersed colloidal particles where repulsive forces keep them separated (Figure 3.14(b)) and (iii) colloidal particles such as cementitious materials where particle attractive forces prevail over gravitational shear forces (Genovese, 2012) (Figure 3.14(c)). The optimization of particle packing density also depends on a series of structural effects and parameters. Particle interactions result also in (i) loosening effects, (ii) wall effects and (iii) wedging effects. The loosening effect which occurs in mixes with dominant in amount coarse particles, is the disturbance in packing density due to the presence of larger sized small particle which cannot fit in the void spaces of coarse aggregate (Figure 3.15(a)) while the wall effect is the disturbance in packing density due to the presence of isolated coarse particle in the network of fine aggregates (Alexander & Mindess, 2005; de Larrard, 1999) as idealized in Figure 3.15(b). Finally, the wedging effect, which is more common for angular particles, takes place when fine particles are slightly not enough to occupy the voids between coarse particles as illustrated in Figure 3.15(c) (Kwan et al., 2015).

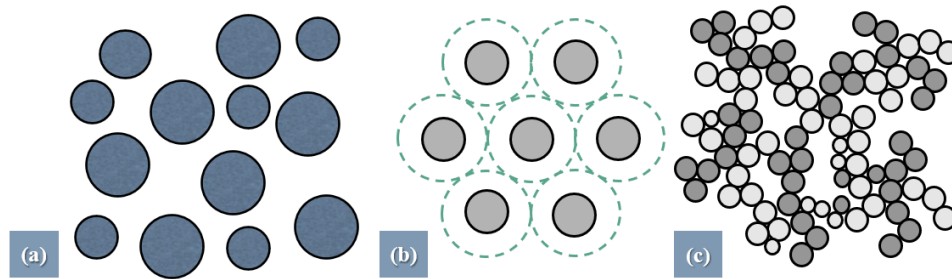


Figure 3.14: (a) Non-colloidal particles, (b) dispersed colloidal particles (repulsive potential forces are represented by dashed lines) and (c) agglomerated colloidal particles (modified from (Genovese, 2012))

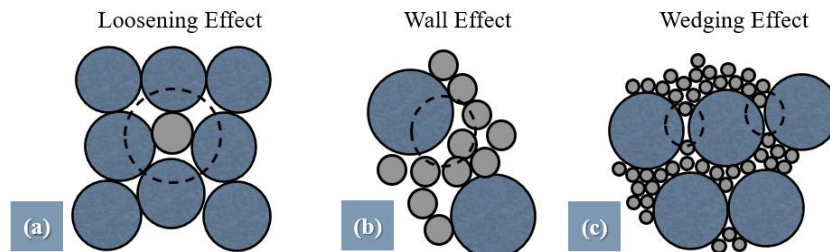


Figure 3.15: Schematic representation of structural effects due to particle interactions: (a) loosening effect, (b) wall effect, and (c) wedging effect (modified from (Kwan et al., 2015))

3.5.2. Theoretical Particle Packing Models

The optimization of aggregate gradation as a means to enhance concrete properties was first proposed by (Féret, 1897) who believed that maximum strength could be achieved when minimum amount and size of voids exist in the matrix. Since then, several models have been

proposed and used to develop the concept of particle packing. Advances in the theory and its application can be seen through the consideration of the structural effects discussed above. For example, the early model of Powers (1968) considers only the loosening effect of the fine particles, while Toufar et al., (1977) considered only the wall effect of the coarse particles. Later on, more advanced models considered both effects (de Larrard, 1999; Dewar, 1999; A. B. Yu et al., 1996), whereas the wedging effect was introduced later by Kwan et al., (2015). The development is also evident through the transition from continuous models to discrete, for more accurate particle packing simulations. Continuous models, as the name suggests, employ a continuous input, such as particle size distribution curves and attempt to model the maximum achievable packing density of the particles. On the other hand, discrete models are developed by discretization of particles into several groups based on their size and models them using binary, ternary, or multi-modal models. In this chapter, focus is placed primarily on continuous models.

As mentioned above, in optimized aggregate distributions, the aggregate mixtures are modified against predefined grading curves. The first of such grading curves was presented by Fuller & Thompson, (1907) and is also known as the maximum density theory. The Fuller & Thompson distribution is described by Equation (3.10):

$$P(D_i) = \left(\frac{D_i}{D_{\max}} \right)^q \quad \forall D_i \in [0, D_{\max}] \quad (3.10)$$

where P is the fraction that passes the sieve with opening D_i , so, it represents the total volume in the aggregate mix with maximum grain size D_i ; and $D_{\max}$ is the maximum particle size of the mixture. The distribution modulus q has a value between 0 and 1, although Fuller & Thompson (1907) used a value of q equal to 0.5. Later, Powers (1968) used the same expression (Equation (3-5)) but with a distribution modulus other than 0.5, thus other parabolic particle size distributions were possible. Andreasen & Andersen (1930) (A&A) further explored the Fuller & Thompson model and introduced a semiempirical packing model of continuous particle size distributions (PSDs), which is also described by Equation (310). In order to achieve higher packing density Andreasen & Andersen (1930) proposed a value of q in the range of 0.33 to 0.5 and observed that optimum packing is achieved when q is 0.37. This PSD curve, with a q value of 0.37, is known as the A&A curve. Distribution modulus is determined experimentally and

depends on particle characteristics. However, in case of self-compacting concretes and generally concretes and mortars with a high amount of fines (<250 μm), the model fails to achieve a high packing density (Brouwers & Radix, 2005). In case of angular coarse particles, the ideal curve would be best described with a lower q , meaning that more fine particles are present in the mix to fill the voids between larger particles (Kumar & Santhanam, 2003). However, both theories assume particles of infinite fineness, meaning that the minimum size of aggregate is zero which is practically not true.

On a way to improve the Fuller and A&A curve, Funk & Dinger (1993) suggested that any real size distribution of particles must have a finite lower size limit and modified A&A curve. This modified version of the model incorporated the minimum particle size in the mixture as:

$$P(D_i) = \frac{D_i^q - D_{\min}^q}{D_{\max}^q - D_{\min}^q} \quad \forall D_i \in [D_{\min}, D_{\max}] \quad (3.11)$$

where $D_{\min}$ is the minimum particle size in the mix whereas all the other parameters are defined previously.

In order to obtain the optimum particle packing density, researchers have followed the modified A&A curve for particle size and distribution (Brouwers & Radix, 2005; Hunger M., 2010; R. Yu et al., 2014, 2015). Figure 3.16 shows the modified A&A, A&A and Fuller curves for PSD with q values of 0.25, 0.37 and 0.5, respectively. The modified A&A curve was used to obtain dense packing in the case of a mixture with a high proportion of fine particles.

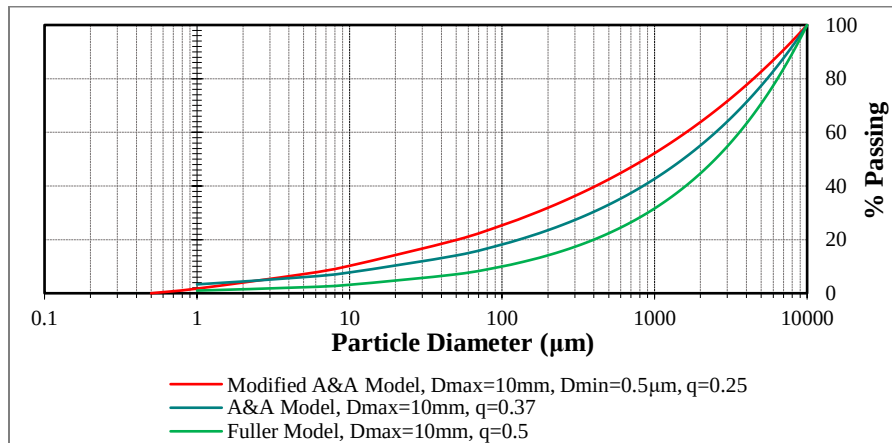


Figure 3.16: Particle size distribution for modified A&A from Funk and Dinger (1993), A&A from Andreasen and Andersen (1930), and Fuller from Fuller and Thompson (1907) models.

Distribution modulus, q , in the above theories could be used as an indicator of fresh properties such as workability. As shown in Table 3.8, higher values of q indicate lower workability as coarse aggregates occupy higher volume in the mix. On the other hand, lower q values indicate higher amounts of fine particles that control the water demand and the ability to hold water. Thus, the selection of q value is critical for obtaining the desired properties of the mix, as higher values of q could lead to segregation while lower values may result in high cohesion. Generally, values between 0.21 and 0.25 are adopted for Self-Compacting Concrete and, especially a value of 0.22 has been used for UHPC mixtures (P. P. Li et al., 2018)

Table 3.8: Relative values of compaction factor and distribution modulus

Degree of workability	Distribution Modulus q	Compaction Factor
Very Low	0.37	0.8
Low	0.32	0.85
Medium	0.27	0.9
High	0.22	0.95

Brouwers & Radix (2005) and Hüsken & Brouwers, (2008) employed the modified A&A to adjust the entire PSD of all solid materials in order to achieve the ideal (target) grading. More specifically, the Residual Sum of Squares (RSS) method was used to achieve the target grading curve and is given by Equation 3.12.

$$RSS = \sum_{i=1}^n e_i^2 = \sum_{i=1}^n [P_{actual}(D_i) - P_{target}(D_i)]^2 \rightarrow \min \quad (3.12)$$

Where P_{actual} is the actual PSD curve and P_{target} the target particle size distribution curve (A&A model curve). The gradation with higher particle packing density and optimum mix ingredients is represented by the “ideal” curve. But it has been reported that gradation of aggregates alone will not yield an optimized mix with greater density; rather, the fine particles like cement and other mineral admixtures mixed with water will also play a role in particle packing by accommodating the finer pores.

3.5.3. Application and Implications of Modified Andreasen & Andersen Packing Model in High Performance Geopolymer Concrete Design

As mentioned above, particle-size optimization is gaining more and more ground with the introduction of novel and advanced concretes such as SCC and UHPC. The high strength and

durability of such concretes is achievable by increased packing leading to less porosity and thus enhanced performance. In recent years, several studies have employed particle packing models, especially the modified A&A model for the optimization of different concrete mixture proportioning. A summary of the studies conducted to optimize the particle packing density using the modified A&A model is presented in Table 3.9.

Table 3.9: Summary of investigations on particle packing optimization using modified A&A (Extended from Mehdipour, 2017)

Reference	Concrete Type	Cementitious Materials (kg/m ³)	w/b ratio	Optimized Granular Material	Distribution Modulus q
(Brouwers & Radix, 2005)	SCC	315	0.55	Aggregate & Binder	0.25
(Wang et al., 2014)		380-450	0.4	Aggregate & Binder	0.23-0.29
(Khayat & Mehdipour, 2014)		315	0.45	Aggregate	0.29
(Mueller et al., 2014)		317	0.6	Aggregate & Binder	0.27
(R. Yu et al., 2014)	UHPC	650	0.33	Aggregate & Binder	0.23
(R. Yu et al., 2015)		840	0.2	Aggregate & Binder	0.23
(Meng et al., 2016)		712	0.18-0.23	Aggregate & Binder	0.23
(P. P. Li et al., 2018)		487.5-675	0.27-0.3	Aggregate & Binder	0.19-0.2
(Khayat & Libre, 2014)	RCC	300	0.39	Aggregate	0.35
(Q. L. Yu et al., 2013)	LAC	423	0.54	Aggregate & Binder	0.25
(Borges et al., 2014)	GPC		1.3-1.5 ⁸	Aggregate	0.21-0.26

As it can be seen, modified A&A has been extensively used for UHPC mixes, while limited research has been performed on geopolymer concrete mix design using the aforementioned model. This can be attributed to the increased complexity of geopolymer systems where the high viscosity of alkaline solutions and the high fineness of precursors interfere with geopolymerization and the fresh properties of the mix leading to stiff mixes with higher porosity and permeability due to poor compaction.

Borges et al., (2014) employed the modified A&A particle packing model to optimize MK based geopolymer concrete formulations. In this study the parameters and their effect on particle packing that were examined were distribution modulus, alkaline solution to dry binder ratio (AS/DB) and aggregate type. More specifically, three different distribution moduli, i.e., 0.21, 0.235 and 0.26 with AS/DB of 1.3, 1.4 and 1.5 were used. Regarding the aggregate type, glass and quartz aggregates have also been considered. It is worth noting here that the differences

⁸ In case of geopolymer concrete this value corresponds to the amount of alkaline solution over the dry binder and not to the conventional w/b ratio

between the two aggregate types are specific surface area and particle morphology, with glass aggregates to be more elongated with lower surface area. As in the case of conventional OPC concrete, lower q values demonstrated enhanced workability while a combination of lower AS/DB and high q (above 0.3) demonstrated higher viscosity and compressive strength with lower workability. Other properties that were investigated were flexural strength, apparent porosity, and apparent density. In the case of quartz aggregates, the observations in flexural strength and apparent density agree with those regarding compressive strength. However, the lowest apparent porosity in case of quartz aggregates was observed for $q=0.235$ and AS/DB=1.4. In case of glass aggregates, the combination of $q=0.26$ and AS/DB=1.3 yields the highest compressive strength and apparent density, while the highest flexural strength is achieved in the case of $q=0.235$ and AS/DB=1.4. Finally, the apparent porosity is less for $q=0.6$ and AS/DB=1.4. The above observations indicate that there are more implications of the modified A&A model in case of geopolymer concrete formulations as beside particle morphology and surface area, the underlying chemical reactions, and the combination of highly viscous alkaline solutions with extremely fine precursors add extra complexity in the particle packing optimization and the yielding properties.

Based on the aforementioned literature, one of the main objectives of this chapter aims is to optimize the granular skeleton and characteristics of high performance geopolymer concrete based on the particle packing density approach and to tackle the challenges that arise from the complexity of the geopolymer systems.

As already explained in detail in previous chapters, geopolymer formulations are not primarily dependent on physical concepts such as particle packing, as strength development and durability are linked to the degree of geopolymerization that is controlled mainly by mineralogy and chemistry. For this reason, the geopolymer paste, namely the precursors along with the alkaline reagents were kept constant in order to yield always the same geopolymer three-dimensional network with Si/Al=3 and K/Al=1. The proportions for the binder are listed in Table 3.10, while the aggregate content varies from 0 to 0.7. Due to lower free silicon and unburnt content USF was utilized instead of DSF. In all cases, the aggregate content consists of 75% GS, 22% coarse TRD and 3% FP. Using the particle size analysis of the precursors and the modified A&A model with $q=0.25$, the target and the actual curve of geopolymer paste are plotted in Figure 3.17. The minimum particle size is $D_{min}=0.01 \mu\text{m}$ from micro silica, while the maximum particle size of the

paste is $D_{max}=256\text{ }\mu\text{m}$ from fly ash. The deviation of the actual paste from the target indicates that the actual mix needs more precursor material, especially in the range of 1 to 100 μm . In the same Figure, mixes with different Sand to Binder (S/B) ratio are plotted along with the target modified A&A curve of the concrete mixture. The increase of S/B ratio highly affects the upper range of particle size distribution (greater than 100 μm) with higher S/B ratios to match the upper part curve better. The reverse phenomenon is slightly evident in the fine particle range (1-100 μm).

Table 3.10: Binder/Geopolymer Paste Mix Design GPC2

Materials	Proportions (kg/m ³)
FA-F	880
USF	146
MK-750	92
GGBFS	90
PS1.7	378
Water	72
Extra Water	5

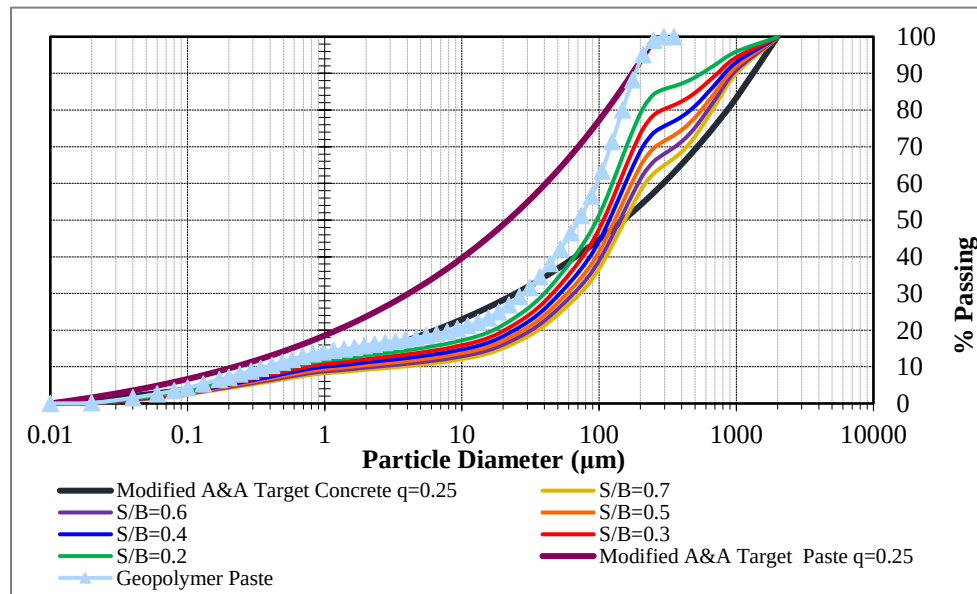


Figure 3.17: Particle size distribution for the geopolymer paste and concrete with different aggregate content related to their ideal curves for GPC2 mix

The mixing procedure is the same as described in section 3.3.1. Geopolymer Concrete was cast in 50 mm cubic molds and covered with a lid or a plastic sheet for 24 hr to prevent moisture loss. Upon demolding, the specimens were cured in water at ambient temperature until the age of testing. Figure 3.18 shows the corresponding compressive strength (per ASTM C39, 2021) and apparent density. As expected, the density is proportional to the aggregate content, while higher strength being obtained in cases of higher aggregate content. The enhanced strength should also

be attributed to the particle morphology of the GS which demonstrates higher degree of interlocking due to angularity and rough surface. However, it is also observed that in case of 0.4 and 0.5 the strength slightly decreases compared to lower ratios. This paradox could be attributed to the double effect of fine aggregates. More specifically, for 0.4 and 0.5 ratios, the high SSA could reduce the paste thickness, while larger amount of Interfacial Transition Zone occurs due to the increased aggregate content leading to a slight reduction of the strength.

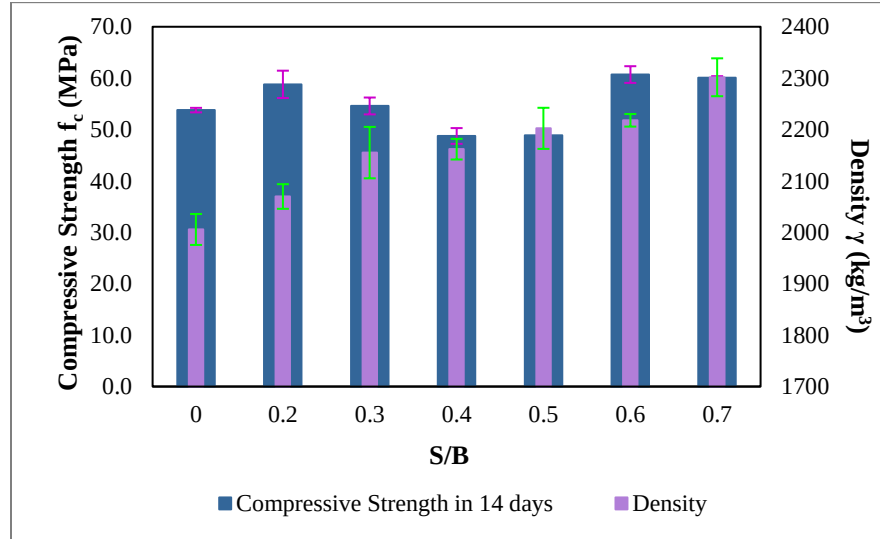


Figure 3.18: Compressive Strength and Density for different aggregate content in GPC2 mixes (average of three specimens)

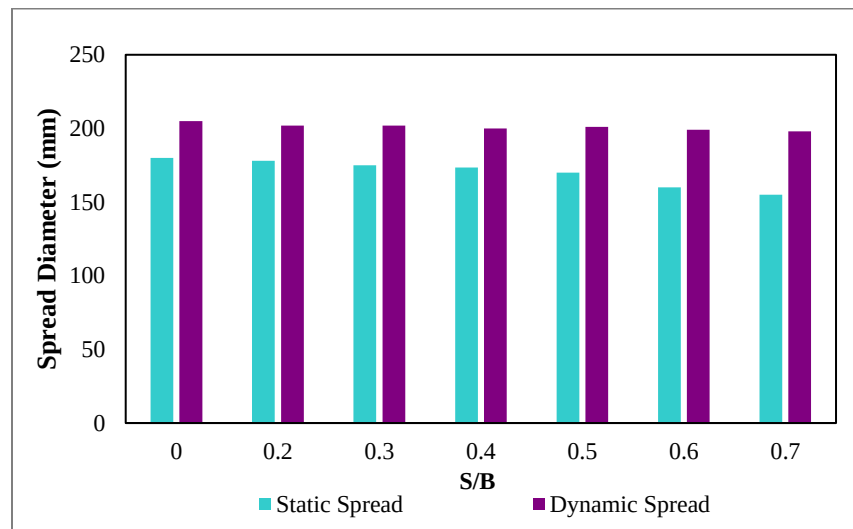


Figure 3.19: Flowability measurements for different aggregate content in GPC2 mixes

The effect of aggregate content was also studied for fresh properties such as workability. Figure 3.19 shows flowability measurements (per ASTM C1856, 2017) of the different geopolymer

concrete specimens. The flowability of each mixture was measured immediately after mixing and right before casting. During this measurement, the conical mold of the flow table was filled with a single layer of fresh geopolymer concrete. Then, the mold was lifted and after two minutes, the reported flow value was measured as the average of maximum and minimum diameter of the spread. This measurement corresponds to the static spread (Figure 3.20(b)). Spread was also measured as the average of maximum and minimum diameter after lifting the table for 20 times in 15 seconds according to the standard (Figure 3.20(c)). The latter corresponds to the dynamic spread. The results demonstrate that with increase of the aggregate content, the static flow decreases. This is because with higher aggregate contents, no extra water is added, while the angularity of the GS particles hinders the workability. On the other hand, the dynamic flow is independent. This indicates that the latter is dominated by the kinetics of the chemical reactions in the geopolymer system, as in all cases the binder, in which the aforementioned reactions occur is the same for all mixes. Finally, although the distribution modulus (q) was selected for high-flowability, the hypothesis about viscous chemicals interacting with extremely fine particles complicates the model is true.

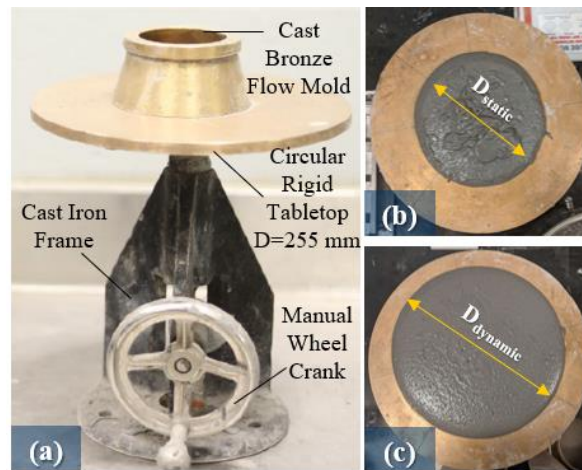


Figure 3.20: (a) Flow Test Apparatus (b) Static Spread (c) Dynamic Spread after tapping

3.6. Effect of Different Admixtures

During the last 50 years, chemical admixtures have been used to manipulate concrete properties in both the fresh and hardened states. More specifically, admixtures are added in concrete to enhance workability or rheology, reduce water demand, and thus improve strength development, accelerate or delay setting time, increase or decrease air-content and enhance durability against

aggressive chemical conditions (sulphate attack, corrosion etc.) Based on their applications, their chemical composition varies from that of surfactants and soluble salts to polymers and insoluble minerals (Mehta & Monteiro, 2014). However, currently available admixtures have been developed to control the aforementioned properties of OPC concretes, as their mechanisms target to alter the kinetic/chemical reactions related to hydration of OPC. As already mentioned in Chapter 2, most of them are organic polymers that not only might be less efficient but could hinder geopolymerization and thus strength development. For this reason, further research is needed to select the suitable admixture based on composition, target property and applications of the developed geopolymer concrete.

3.6.1. Materials & Methods

So far, in the literature but also in this project, certain issues have arisen with regards to fresh state of geopolymer concretes. The high viscosity and density of the soluble silicates with combination with the high alkalinity of some raw materials (fly ash, slag) resulted in poor workability and accelerated setting time. Poor workability can also be attributed to the high SSA of metakaolin and silica fume in combination with the angularity of the crushed granite sand. Meanwhile, other raw materials that contain free silicon (silica fume) or unburnt carbon content (fly ash) were found to generate hydrogen gas resulting in porosity and thus lower strength. To tackle those challenges a variety of chemical admixtures were used, and their efficiency was assessed in both the fresh and hardened state. The admixtures used along with their properties and main chemical substance are listed in Table 3.11. The admixtures were studied individually, and the dosage for all of them was 0.5% wt. with respect to the binder, with the exception of HRWRA-P4, RCA and RA-TA. For HRWRA-P4 and RCA the added amount was 0.25% wt. as recommended by the manufacturer, while 0.2% wt was selected for RA-TA. Besides intervention in the chemical reaction of the system through use of chemical admixtures, three other methods were used: (i) addition of 25% extra water (Mix ID 1.25W), (ii) thermal pre-treatment of all ingredients at 2° C 24 hr before mixing (F), and (iii) substitution of all sand with GS (G). The mix design is the case of S/B=0.6 from the section 3.5.5. Table 3.12 shows the proportions of the control mix design. The mixing process is described in Section 3.3.1. After testing the fresh properties, the concrete was cast in 50 mm cubic molds and covered for 24 hr to prevent moisture loss. Upon demolding, the specimens were cured in water at ambient temperature until the age of testing.

Table 3.11: Chemical Admixtures used in GPC3 mixes

Admixture ID	Category	Chemical Base	Physical appearance	pH (20° C)	Density (g/cm ³)
ADA	Air-Detraining	N/A	Brown Liquid	4.8	0.997
HRWRA-P1	High-Range Water Reducing	Polycarboxylate Ether	Brown Liquid	7.8	1.1
HRWRA-P2	High-Range Water Reducing	Polycarboxylate Ether	Brown-Gray Liquid	4.6	1.05
HRWRA-P3	High-Range Water Reducing	Modified Polycarboxylate Ether	Yellow Liquid	5	1.06
HRWRA-P4	High-Range Water Reducing	Polycarboxylate Ether	Brown Liquid	5.8	1.05
HRWRA-N	High-Range Water Reducing	Naphthalene sulphonates	Brown Liquid	6.5	1
WRA	Workability Retaining	Acrylate Polymer/Vinyl Ether	Brown Liquid	6	1.04
RCA	Rheology Controlling	N/A	Brown Liquid	7.2	1.01
RA	Hydration Controlling /Retarder	N/A	Red Liquid	3.5	1.075
RA-TA	Retarding	Tartaric Acid	White Powder	3.2	1.79

Table 3.12: Control mix design GPC3

Materials	Proportions (kg/m ³)
	Control (C)
FA-F	880
USF	146
MK-750	92
GGBFS	90
PS1.7	378
Water	72
coarse TRD	170
GS	570
FS	10
Extra Water	5

3.6.2. Fresh State Properties

- *Workability*

The first property used to assess the efficiency of the studied chemical admixtures was the flow spread as an index for workability. As described in Section 3.5.5, the workability was measured through the flow test and the spread was recorded both statically and dynamically. Figures 3.21 and 3.22 show the static and dynamic spread results for all 14 mixtures described above, respectively. All admixtures and interventions have a more prominent effect on static flow, indicating that dynamic flow is dominantly influenced by the geopolymerization. The viscosity modifying agent had an adverse effect on both flowability measurements (-10%) as expected. In fact, the mix was so thixotropic that it could be extruded and stay in the given shape as shown in

Figure 3.23. The findings indicate that RCA could be used for the development of 3D printing concrete. Generally, the efficiency of the chemical admixtures is more evident in case of static flow where an increase of 11%-13% was observed for superplasticizers, workability, and air detrainers admixtures, except HRWRA-P3 and HRWRA-N with 19% and 22% increase, respectively. The retarders seem to be more effective in static flow, indicating that their mechanism has also some plasticizing effect. As expected, the addition of 25% more water (1.25W) is the most effective with 30% and 13% increase in static and dynamic spread, respectively. This implies that the mechanisms of the above admixtures do not apply on geopolymeric systems, as most of them are organic based. Cooling all mixing constituents at a temperature of 2 °C (mix F) was also relatively effective in terms of achieving flowability, although no extra liquid was added to the system. The lowered temperature of the system seems to have an impact on the kinetics of the reaction leading to prolonged workability retention. Finally, the substitution of all sand with GS (mix G) had the least impact in the case of static flow (+5%), while in the case of dynamic flow, a slight increase was also observed (+3%). Both increases can be attributed to the change of the grading curve of the system as GS is less fine than coarse TRD.

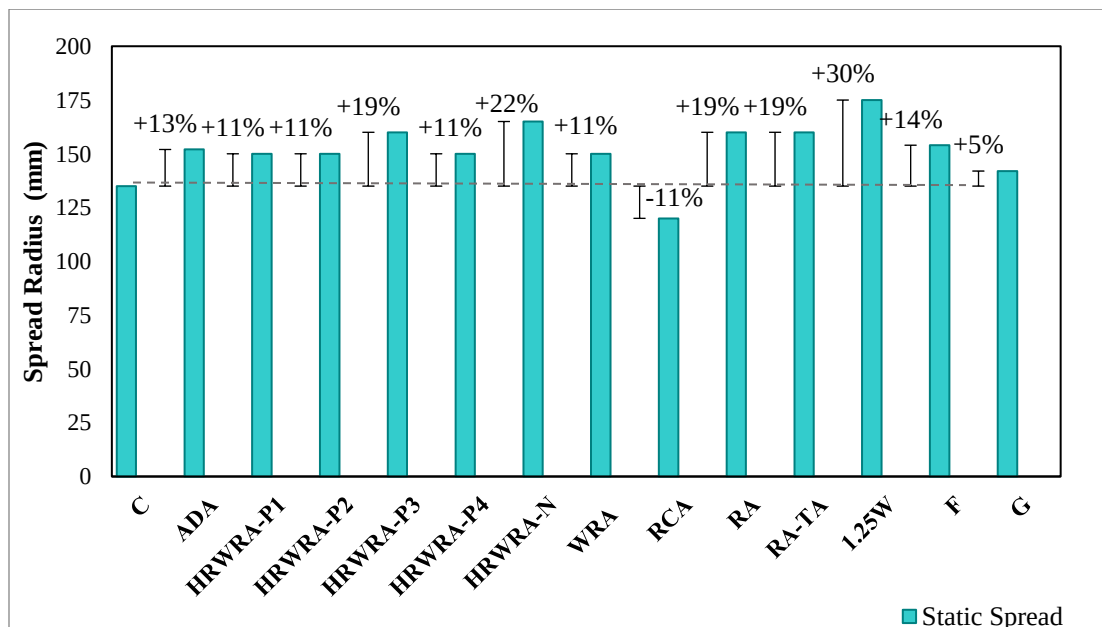


Figure 3.21: Static Flow measurements of geopolymer concrete with different admixtures for GPC3 mix series

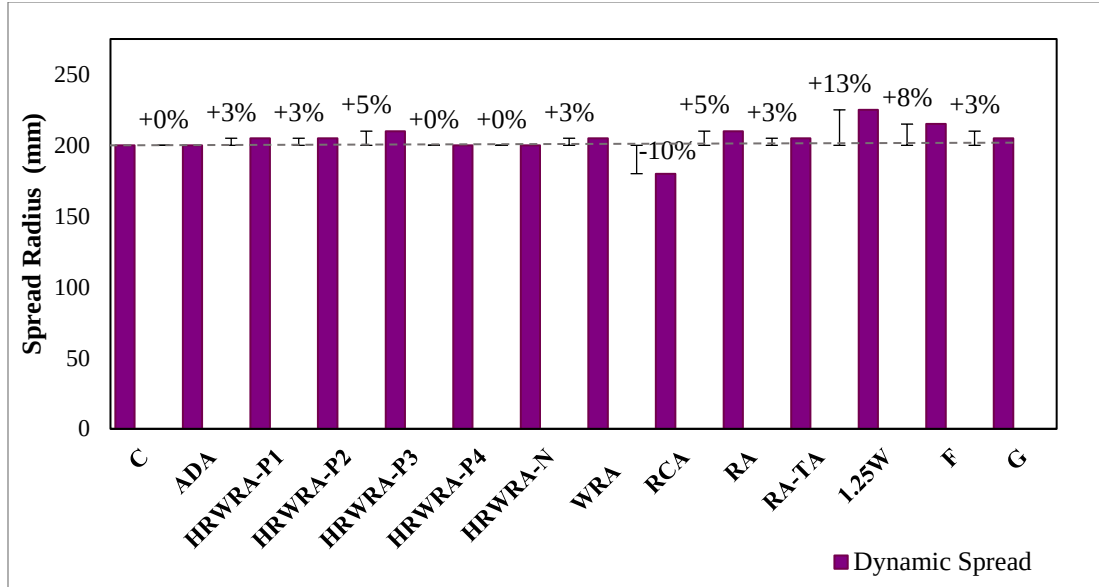


Figure 3.22: Dynamic Flow measurements of geopolymer concrete with different admixtures for GPC3 mixes



Figure 3.23: Highly thixotropic concrete through use of RCA in GPC3 mixes

- *Setting Time*

The setting time of the geopolymer concrete mixes was measured according to ASTM C191, (2021) using Vicat needle measurements; a method that is commonly used for determination of setting time in hydraulic cements. The test is based on the resistance to penetration of a needle with 1 mm diameter and 50 mm length into a fresh paste under a mass of 300 ± 0.5 g at ambient temperature (Figure 3.24). The determination of initial and final setting time is available through this procedure. According to ASTM C191, the initial setting time is defined as the time elapsed between the start of hydration (water is added to cement; in case of geopolymers, the addition of the alkali silicate to the precursors) and the time of first penetration being less than 25 mm (Equation (3.13)). E is the time (minutes) of last penetration greater than 25 mm, H is the time (minutes) of first penetration less than 25 mm, while C and D the corresponding penetration readings at E and H , respectively.

$$\left[\left(\frac{H-E}{C-D} \right) \times (C-25) \right] + E \quad (3.13)$$

Final setting time is defined as the time elapsed between the start of hydration and the first time the needle has 0 mm penetration. To obtain the setting time, measurements should be taken every 15 minutes, if possible. For fast setting concretes, the interval is reduced to 5 min. Inability to measure the hardening rate of the material before initial setting time and the discontinuous nature of the measurements are some of the implications of the test (Ranjbar et al., 2020). Despite these limitations, the Vicat needle is easy to operate and sufficiently reproducible for characterization of geopolymer concrete's setting time.

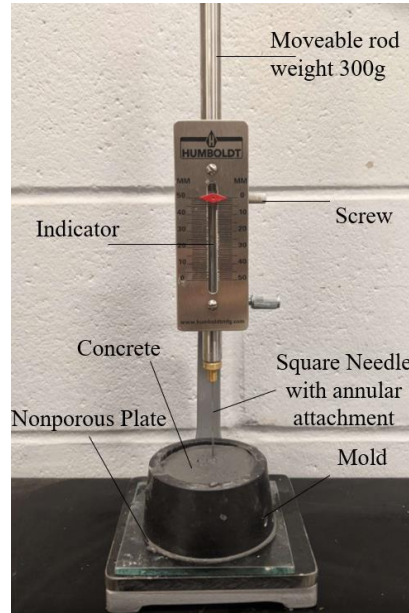


Figure 3.24: Vicat needle apparatus

For all 14 mixtures both initial and final setting time was measured according to ASTM C191, and the results are shown in Figures 3.25 and 3.26, respectively. It is observed that the admixtures had little to no impact in delaying the setting time, while adverse effects were observed especially in the case of RCA (shorter setting time by almost 40%). The commercial retarder RA achieved a 9% increase of the initial setting time but accelerated the final setting time by 3%. Tartaric acid (RA-TA) accelerated setting initial and final time by 22% and 20%, respectively. As was expected, the increase of water content by 25% (1.25W) and the cooling of raw constituents at 2 °C (F) effectively delayed the setting time. Finally, substituting all aggregates with GS had a small retarding effect (4-6%). This could be attributed to the reduced fineness and thus SSA of the

system. As already mentioned above, GS is partially reactive and a smaller SSA could lower the dissolution rate and thus the hardening evolution.

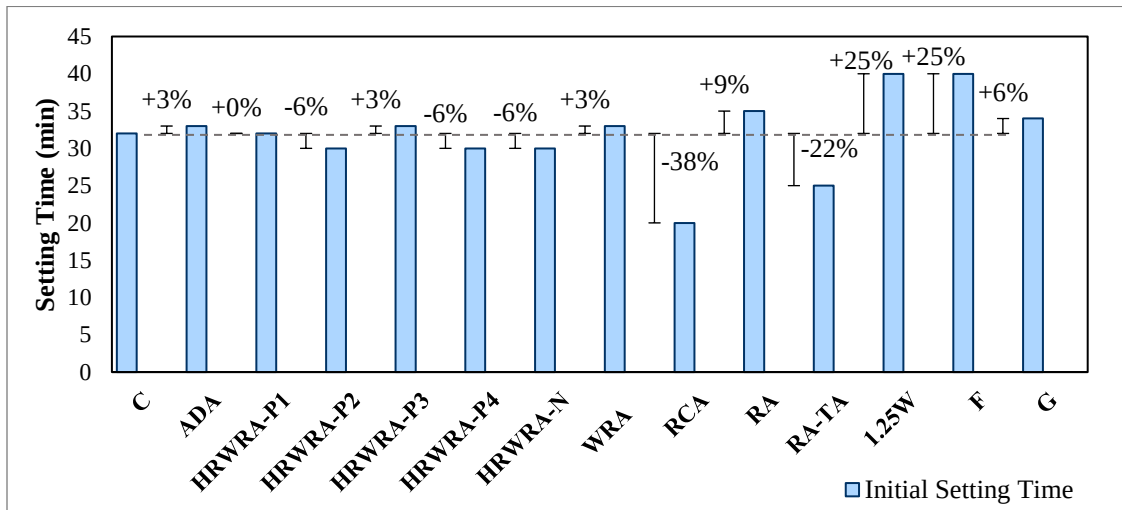


Figure 3.25: Initial Setting Time for geopolymer concretes with different admixtures for GPC3 mixes

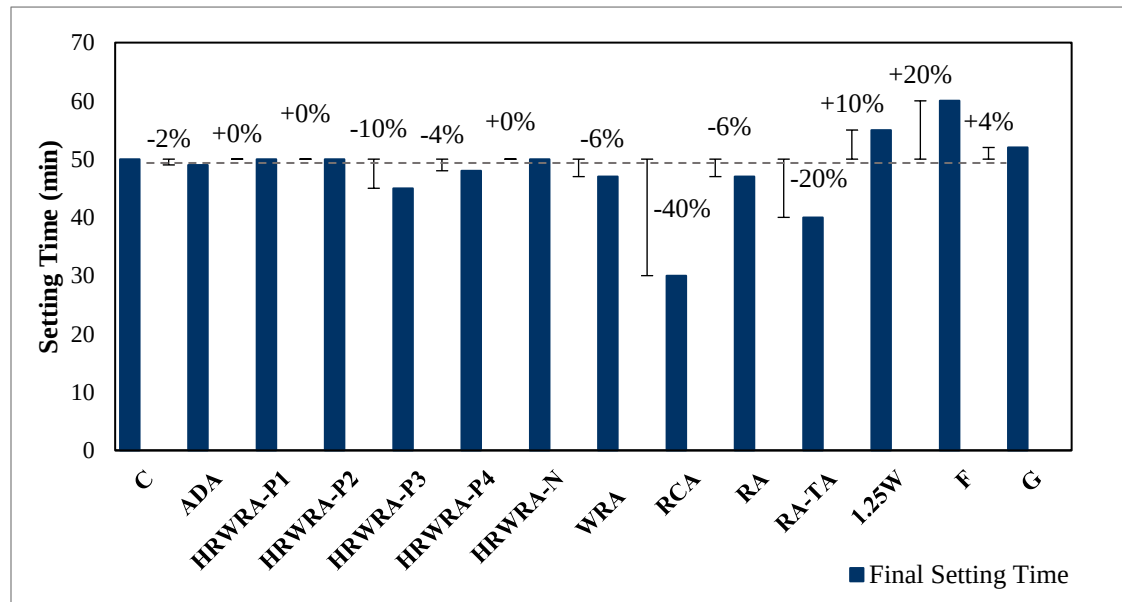


Figure 3.26: Final Setting Time for geopolymer concretes with different admixtures for GPC3 mixes

3.6.3. Compressive Strength

Finally, the effect of the chemical admixtures on compressive strength was also studied. A force-controlled compression-testing machine was used to test the 50 mm cubes under compression at a loading rate of 0.259 MPa/s in accordance with ASTM C39 (2021) at 14 and 28 days. For each testing age, three specimens were tested and their average compressive strength with the

corresponding standard deviation were obtained and presented in Figure 3.27. It is observed that compared to the reference mix (C) for all cases of admixtures the compressive strength at 14 days is decreased, with higher reduction in cases of HRWRA-N, RA-TA, and RA (23, 18 and 15%, respectively). For 28 days, the lower strength was observed in case of HRWRA-N (16% reduction) while a small increase of strength was noted for RA-TA. Generally, with all admixtures high standard deviations are observed, raising concerns about the reproducibility of mixes containing currently available chemical admixtures. On the other hand, lowest deviations in cases of 1.25W, F and G indicate that these interventions are more preferable as they don't interfere significantly with reaction kinetics of the system.

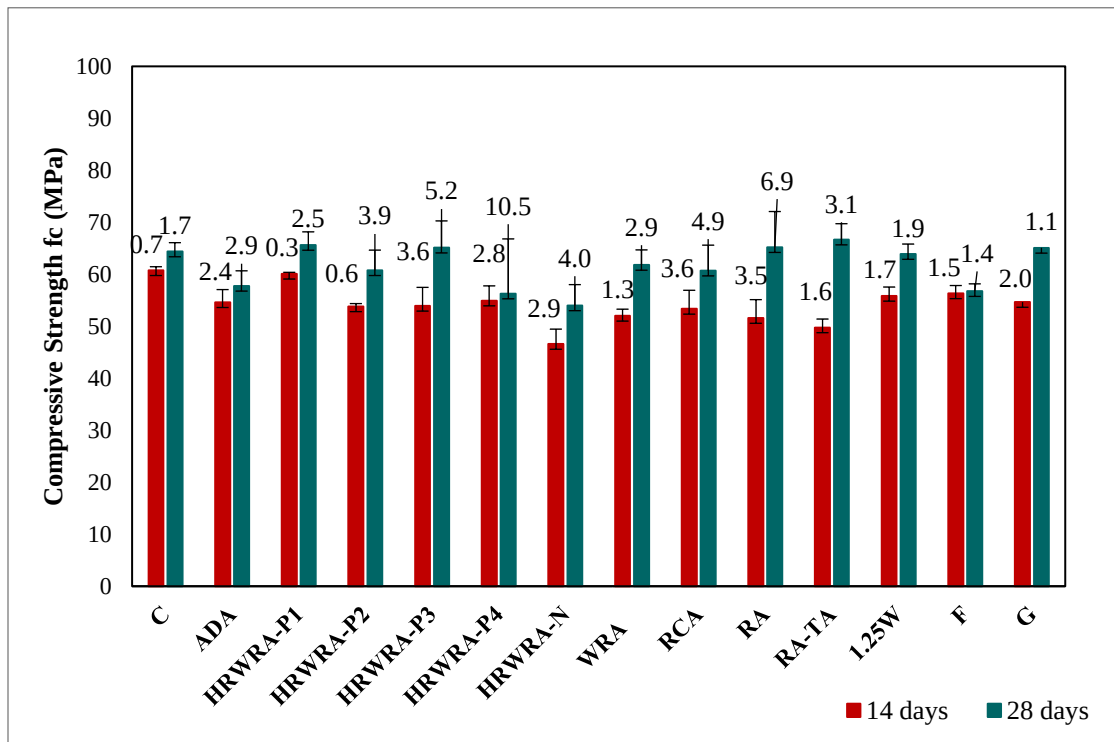


Figure 3.27: Compressive Strength for geopolymer concretes from GPC3 mixes with different admixtures at 14 and 28 days (average of three specimens)

3.7. Optimized Mix Design

After running an exhaustive series of trial mixes with various raw materials targeted for high strength, high packing density, workability, stability, extended setting time and repeatability, an optimized mix was arrived at, and is presented in this section. The optimization was achieved through synergy of the geopolymer formulas for obtaining a rigid 3-D network in the microstructure, combined with the particle packing density model and accounting for the lessons

learned from previous sections regarding the actual behaviour of each constituent. For this reason, large part of FFA_F was substituted with the iron-rich quarry waste powder MG and a rock-based geopolymer concrete was designed.

3.7.1. Determination of Geopolymer Paste Formula

The first step to develop the optimized mix design is the determination of geopolymer binder formula, taking into consideration not only the fundamentals of geopolymer chemistry (Si/Al=3 and K,Ca/Al=1) but also the implications of the available raw materials in fresh and hardened state. Besides the reactivity, it is also important, the main precursor to be the most viable and sustainable option. Main precursor is not necessarily the precursor with the highest amount in the binder, rather than the raw material that is assumed to react with most of the available alkali silicate. Based on these criteria, the Equations in Section 3.2. were implemented to determine the mix proportions of the binder. The amount of potassium silicate was set to 400 kg/m³, and it was assumed that around 44 % by weight of the liquid will react with MG, 37 % with MK-750, 6.4 % with P, 5.4% with GGBFS and 2% with FA-F. The rest 5% was left to react with the weathered surface of partially reactive GS. USF was not assumed to react with the system because of its lack of reactive alumina.

Using Equation (3.5) and chemical composition of PS1.7 and MG, the amount of MG (gr/L) was determined:

$$MG = \frac{44\% \times 400 \text{ gr liquid silicate} \times 21.48 (\% K_2O \text{ in liquid}) \times 101.96 (\text{gr/mol } Al_2O_3)}{94.19 (\text{gr/mol } K_2O) \times \left\{ \left[\frac{101.96 (\text{gr/mol } Al_2O_3)}{159.69 (\text{gr/mol } Fe_2O_3)} \times \frac{14.9 (\% Fe_2O_3 \text{ in powder})}{5} \right] + 13.8 (\% Al_2O_3 \text{ in powder}) \right\}} \quad (3.14)$$

Using Equation (3.2) and the chemical composition of PS1.7, MK-750 and P, the amount of MK-750 and P (gr/L) was obtained:

$$MK-750 = \frac{37\% \times 400 \text{ gr liquid silicate} \times 21.48 (\% K_2O \text{ in liquid}) \times 101.96 (\text{gr/mol } Al_2O_3)}{94.19 (\text{gr/mol } K_2O) \times 43.3 (\% Al_2O_3 \text{ in powder})} \quad (3.15)$$

$$P = \frac{6.4\% \times 400 \text{ gr liquid silicate} \times 21.48 (\% K_2O \text{ in liquid}) \times 101.96 (\text{gr/mol } Al_2O_3)}{94.19 (\text{gr/mol } K_2O) \times 13.5 (\% Al_2O_3 \text{ in powder})} \quad (3.16)$$

Using Equation (3.3) and the chemical composition of PS1.7 and GGBFS, the amount of GGBFS (gr/L) could be found:

$$\text{GGBFS} = \frac{5.4\% \times 400 \text{ gr liquid silicate } 21.48 (\% \text{ K}_2\text{O in liquid}) \times 101.96 (\text{gr/mol Al}_2\text{O}_3)}{94.19 (\text{gr/mol K}_2\text{O}) \times 0.5 \cdot 8.37 (\% \text{ Al}_2\text{O}_3 \text{ in powder}) \cdot \frac{2}{3}} \quad (3.17)$$

Next, using Equation (3.4) and the chemical composition of PS1.7 and FA-F, the amount of FA-F (gr/L) was determined:

$$\text{FA-F} = \frac{2\% \times 400 \text{ gr liquid silicate } \times 21.48 (\% \text{ K}_2\text{O in liquid}) \times 101.96 (\text{gr/mol Al}_2\text{O}_3)}{94.19 (\text{gr/mol K}_2\text{O}) \times 0.2 \cdot 23.5 (\% \text{ Al}_2\text{O}_3 \text{ in powder})} \quad (3.18)$$

A detailed comparison of the stoichiometry of the proposed high performance geopolymer concrete with strain-hardening geopolymer concretes available in the literature is given in Appendix A.

3.7.2. Determination of Optimum Gradation Based on Particle Packing Density

After determining the optimized geopolymer paste in terms of geopolymer chemistry, to produce a high performance geopolymer concrete, the modified A & A model, described in section 3.5.2 and applied in 3.5.5 was used. To increase the flowability of the mix, the distribution modulus was set to $q=0.22$. Figure 3.28 illustrates the modified A&A Target curve for $q=0.22$ and the Mix Curve for both Chemically Optimized Paste (COP) and Optimized Concrete (OC). As it is demonstrated, the COP does not yield the optimum particle packing and thus manipulation of the mix is needed through addition of GS and USF. The former balances the curve in the coarse range while the latter covers the range of very fine particles. For the Modified A&A Target COP curve, $D_{\max}=250 \mu\text{m}$ from FA-F, while $D_{\min}=0.2 \mu\text{m}$ from MK-750. The modified A&A Target OC curve is determined by taking into consideration the size of the coarsest grain (GS, $D_{\max}=1200 \mu\text{m}$) and the size of the finest grain (USF, $D_{\min}=0.01 \mu\text{m}$). For the optimized concrete mix, USF is employed at 23% by weight of the binder, to densify the system and delay the setting time. The latter is achieved through the constant supply of silicate in the binder as discussed in Chapter 2. GS was chosen for sand while the Aggregate to Wet Binder (dry binder and silicate) ratio was set to 0.79. The proposed optimized mix design with and without steel fibers is given in Table 3.13.

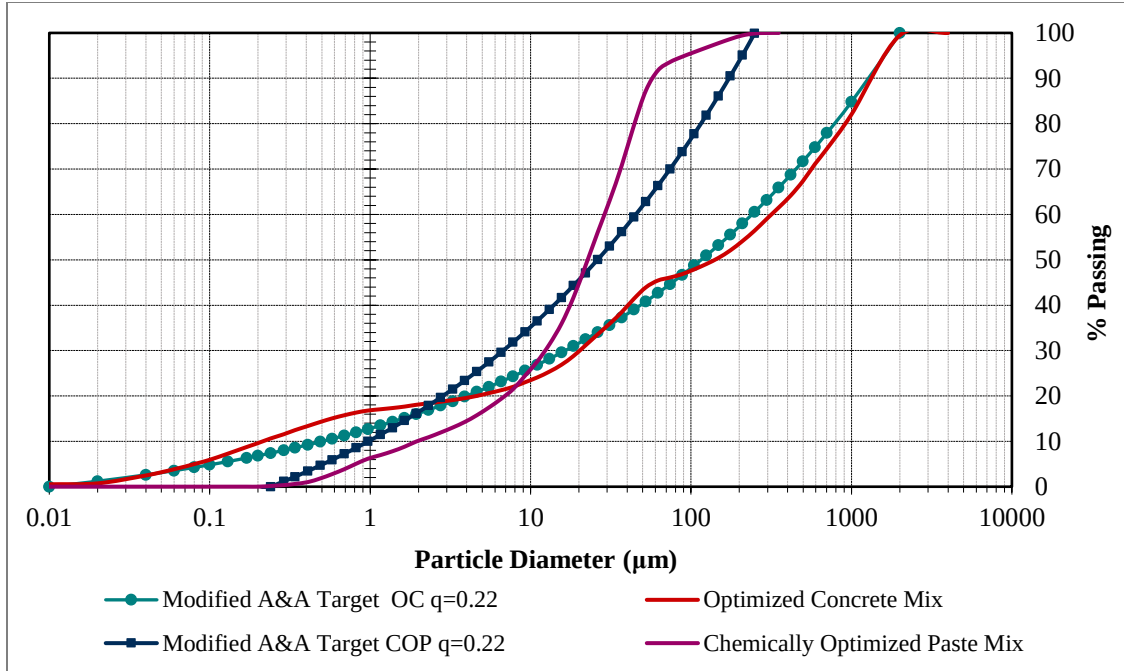


Figure 3.28: Particle size distribution for optimized geopolymer paste and concrete with related to their ideal curves

Table 3.13: Optimized Geopolymer Concrete Mix Design for with (GPC4-F) and without fibers (GPC4-NF)

Materials	Proportions (kg/m ³)	
	GPC4-NF	GPC4-F
MG	260	260
USF	230	230
GGBFS	180	180
MK-750	80	80
P	45	45
FA-F	40	40
PS-1.7	400	400
W	80	80
GS	980	980
Extra Water	22	22
Steel Fibers	-	156
Fresh Density	2317	2463

3.8. Sustainability Analysis of the Proposed Geopolymer Matrix

Finally, a sustainability analysis of the proposed fiberless geopolymer concrete is conducted in order to assess the CO₂ mitigation. The sustainability indices used are the energy intensity and the CO₂ emissions generated by the production of the materials used in the mix. Table 3.14 shows the energy intensity and CO₂ emissions for different components used in the proposed concrete but also usually found in construction materials. It is worth noting here than MG is a local quarry

by-product, not commercialized as Fly Ash and Slag, and thus its energy intensity and CO₂ emissions for its production are assumed to be zero.

Table 3.14: Energy intensity, and CO₂ emissions for materials used in the proposed geopolymer concrete and generally in construction

Material	Energy Intensity (MJ/kg)	CO ₂ emissions (kg/kg)	Reference
Ordinary Portland Cement	4.7	1.02	(Davidovits, 2020)
Fly Ash	0.1	0.009	(Hammond & Jones, 2008)
Silica Fume	0.1	0.008	(Hammond & Jones, 2008)
River Sand	0.081	0.0048	(Hammond & Jones, 2008)
Granite Sand	0.81	0.0459	(Bascetin et al., 2017)
Slag	1.33	0.07	(Hammond & Jones, 2008)
Metakaolin-750	2.5	0.37	(Hammond & Jones, 2008)
Potassium Silicate Solution	2.1	0.28	Estimated (Davidovits, 2020)
MG waste powder	0	0	Estimated
Pumice	1.681	0.0022	(Lava Mining & Quarrying SA, 2021)
HRWRA	11.47	0.6	(Zheng, 2009)
Water	0.01	0.001	(Hammond & Jones, 2008)

The above sustainability indicators were used to calculate the total energy intensity and CO₂ emissions of the geopolymer matrix (GPC4-NF as proposed in Table 3.13). The results are compared with 4 different concrete mixtures available in the literature with same strength of 70 MPa (Table 3.15). In addition to that an equivalent mixture (EQ-OPC) with substituting the total amount of MK-750, MG, P and PS-1.7 with the equivalent in OPC is also examined. The comparative results are illustrated in Figure 3.29. As it can be seen (Figure 3.29(a)), the proposed geopolymer matrix leads to a CO₂ emission reduction in the range of 56-87% compared to high performance and engineering cementitious composites. The highest reduction is compared to a HPC made of 100% OPC while the lowest reduction (56%) corresponds to an ECC where 70% of the OPC is substituted by FA. The energy intensity reduction is also significant with the highest value to be 70%.

Table 3.15: Different concrete mixes used for sustainability analysis

Reference	Mix Design Label
Present Study Substitution with OPC	EQ-PC
(Kobayashi et al., 2010)	HPFRC-PC
(Yao et al., 2012)	ECC1-0.5PC
(Eshghi, 2018)	ECC2-0.4PC
(Yao et al., 2012)	ECC1-0.3PC
Present Study	GPC4-NF

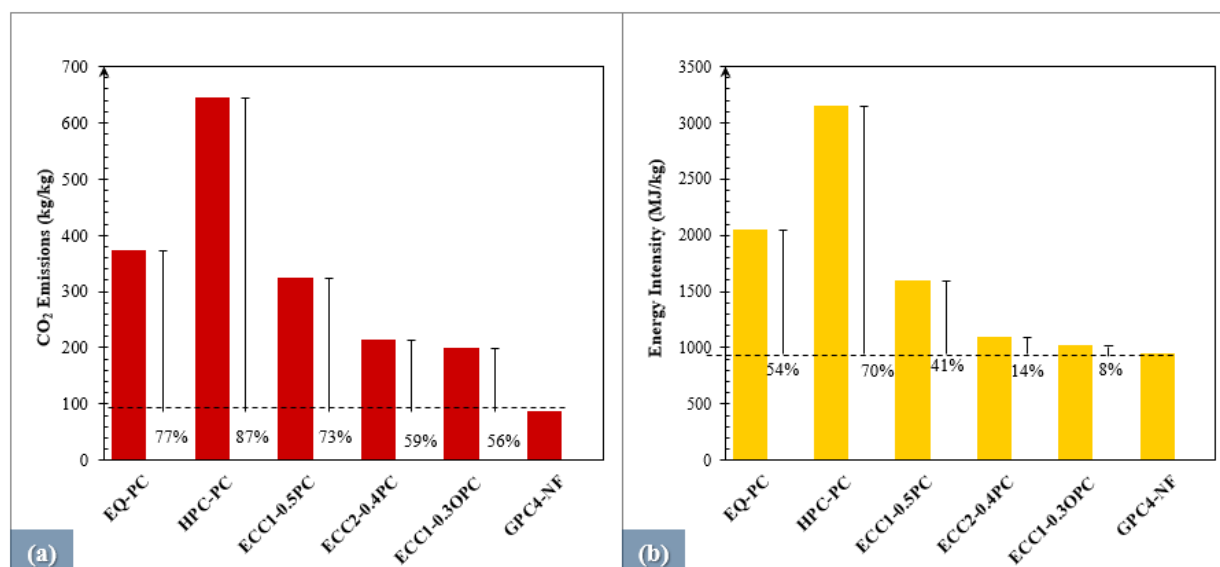


Figure 3.29: Material sustainability indicators for 1 tonne of different concrete mixtures (a) CO₂ emissions, and (b) Energy Intensity

3.9. Conclusions

This chapter explored the synthesis of geopolymer concrete matrix utilizing a variety of industrial by-products, low temperature calcined clays and quarry wastes. From the above experimental investigation, the following conclusions are drawn:

- A full characterization in terms of physical, chemical, and mineral properties is needed to evaluate the suitability of locally available aluminosilicate source materials as geopolymer binders. The determination of the main precursor should be made, taking into consideration the above characterization tests.
- Besides the main oxidal composition, the LOI content is significant, as it is related to hydrate compounds or unburnt carbon content in the wastes. The latter which can be found in higher amounts in Fly Ash and Densified Silica fume has an adverse effect in fresh properties but also in durability.
- Regarding setting time, precursors should have neutral to slightly basic pH. The high content of CaO and MgO that attribute to high pH could provide early strengths, but also precipitate and progressively decrease the setting time.
- Careful formulation that combines different precursors should be used to produce a stable mix suitable for the application designed, as each precursor has its own advantages and disadvantages.

- Besides chemical optimization regarding the Si/Al and Al/K ratio, particle packing models should be employed for physical optimization in terms of density. Although the available particle packing models that were originally designed for OPC concretes are applicable in case of geopolymer concrete, they should be carefully implemented for optimal packing density rather than designing for fresh properties, such as design for flowability. The implications are related to the thixotropic nature of the alkaline reagents, the particle morphology and specific surface area of dry materials that are not taken into consideration in most of the available particle packing models.
- The chemical admixtures developed for OPC concretes are not ideal for geopolymer concretes, as they are specifically designed to target the hydration mechanism which is different from geopolymerization. Use of such admixtures might demonstrate a degree of efficiency in some cases, whereas in others erratic behaviour is observed in fresh state while lack of repeatability in the mechanical properties and loss of strength are observed.
- The phase assemblage of the developed geopolymer matrix agrees with the stoichiometric design of the precursors' proportions with respect to the alkaline reagent. This indicates successful geopolymerization, as the matrix composition is similar to rocks such as K-feldspar and albite.
- The porosity of the studied geopolymer matrices is induced by chemical reactions, viscosity, and consolidation. For the chemically induced porosity, pretreatment of aluminosilicate materials to eliminate the unburnt carbon and free silicon is needed.
- The proposed geopolymer matrix leads to 56-87% carbon footprint reduction compared to HPCs and ECCs of same strength available in the literature

3.10. References

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Chapter 4: Fiber Distribution and Orientation in THFRC

Fiber reinforced concrete is an evolution of reinforced concrete, where conventional reinforcement bars are substituted by steel fibers. The addition of the latter enhances the tensile strength and ductility, while the residual strength is governed by the presence of fibers in the cracked surface. Fiber distribution is a crucial parameter affecting fresh and hardened-state properties. For example, in fresh state, the fiber count which is related to the distribution has impact on the self-consolidating ability of the concrete. Conversely, in the hardened state, fiber distribution not only influences the mechanical strength but also the post-cracking behaviour. The efficacy of the fibers reduces from 100% when aligned along the direction of tensile stress to as low as 30% when randomly distributed (Martinie & Roussel, 2011). For this reason, it is important not only to determine the factors influencing fiber positioning but also review and apply the currently available techniques used to estimate or predict the fiber distribution and orientation. Apart from that, the current chapter investigates experimentally the impact of rheology on the distribution and orientation of the fibers through the method of cuts and manual counting and translucent fluid model concrete, while it explores their effect on the mechanical properties of the hardened THFRC.

4.1. Factors Affecting Fiber Distribution and Orientation

Fiber positioning in the hardened state is the final outcome of a series of phases that THFRC passes through from mixing till hardening inside the formwork. Those phases combined, influence fiber orientation and distribution and usually are examined independently in the literature. In this section factors causing the change of fiber position along with their synergistic effect are studied.

4.1.1. Fiber Type

At the early stages, in mix design the fiber type has a strong influence on fiber distribution. Different types of fibers are based on material (i.e., steel, or synthetic), diameter or length. Synthetic fibers tend to form clusters whereas some fibers that are also hydrophilic absorb part of the mix water hindering the workability. Same workability issues can be observed with long fibers that also agglomerate resulting in a less homogeneous material and thus anisotropic properties. Another parameter dictated by the material of the fibers and is important for their

spatial distribution is density. Depending on their density, fibers can create some extra anisotropy (Stähli et al., 2008).

4.1.2. Fresh State Properties/Flowability

After mixing, in the fresh state, high flowability, a characteristic feature of SCC is a beneficial factor for fiber alignment and thus enhanced mechanical properties. More specifically, with the assumption that concrete flows in layers, the fibers are forced to align with those layers (Laranjeira et al., 2012). This is because in cases of flow where shear stresses are prevailing, fibers have the tendency to place themselves in the minimum energy position which coincides with the boundary of the layer. Nevertheless, this positioning is not the final one attained, as there is high instability due to continuous movement and rotation of the fibers due to the flow till alignment (Martinie & Roussel, 2011). This phenomenon is known as flux effect and is illustrated in Figure 4.1, which also shows the layering assumption in SCC along with the wall effect imposed by the boundaries of the formwork.

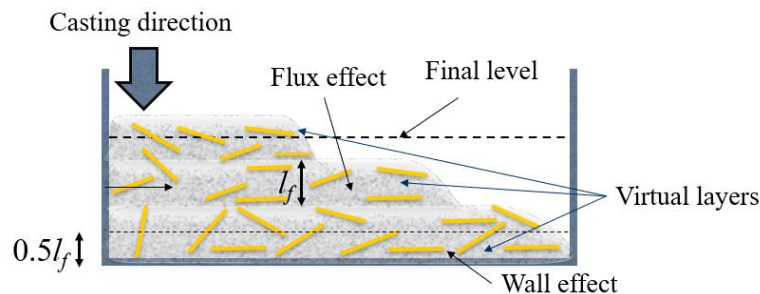


Figure 4.1: Layering theory due to the high flowability of SCC (modified from Alberti et al., 2018; after Laranjeira et al. 2012)

4.1.3. Casting Method

Casting method is also a crucial parameter affecting the fiber positioning and should be considered during structural design. There are numerous studies available in the literature investigating the effect of casting on mechanical properties of UHPFRC and FRCs in general. Figure 4.2 illustrates the most common casting method found in the literature. In one particular study, Torrijos et al., (2010) examined different placing methods of FRC containing steel and polymeric fibers. Three different casting methods were investigated: (i) vertical flow, in the center of the beam, and flow from one end through a 5 m tube with a diameter of 150 mm and inclined at 20 °. After conducting fracture tests in notched specimens of dimensions 600 mm x 150 mm x

150 mm, the authors also performed cuts in the three main perpendicular planes and counted the number of fibers. All beams were tested horizontally, and it was observed that the least efficiency in fiber orientation was the case of the vertical placement while the tube which was used to simulate a pumping pipeline showed optimum results for long steel fibers. This can be attributed to the fact that the point of drop which is at higher risk of local turbulence during pouring was not in the area of interest in the flexure tests. The improvement of fiber orientation and thus mechanical properties with the use of an inclined chute was also reported by (Meng & Khayat, 2017).

Shao (2016) investigated the effect of casting conditions on flexural strength of UHPFRC. Namely the casting methods considered were layered, flow from the center and flow from one end. The beams were tested in flexure via third point loading. It was found that the layered method led to significantly enhanced flexural strength due to favourable fiber alignment, while for the flow methods, large portion of fibers found to be aligned diagonally or parallel to the transverse plane. Finally, between the two flow methods, flow from the end produced slightly better results as the turbulence in flow at the point of pouring was not in the area of interest.

Although layer by layer method is proven to yield the most favourable results in terms of fiber alignment, it is difficult to be achieved as there are many factors that have significant impact to the final distribution and depend on the person that performs the casting (i.e., speed or thickness of the layer). To tackle the issue of non-uniform layering, Huang et al. (2018) proposed a flow controlling method using an L-shape device to pour flowable THFRC with enhanced fiber orientation. This was achieved due to the height of the horizontal channel being 10 mm which is less than the length of typical short fibers (13 mm) forcing the latter to rotate to a horizontal state in the flux. Indeed, the new method yielded enhanced flexural behaviour compared to direct casting. Generally, many codes and standards recommend the flow from one end as a method with improved fiber alignment and less uncertainty. The latest casting method that showed promising results and can be an indicator of fiber distribution in additive manufacturing is the extrusion via which enhanced fiber alignment was achieved (Ge et al., 2021).

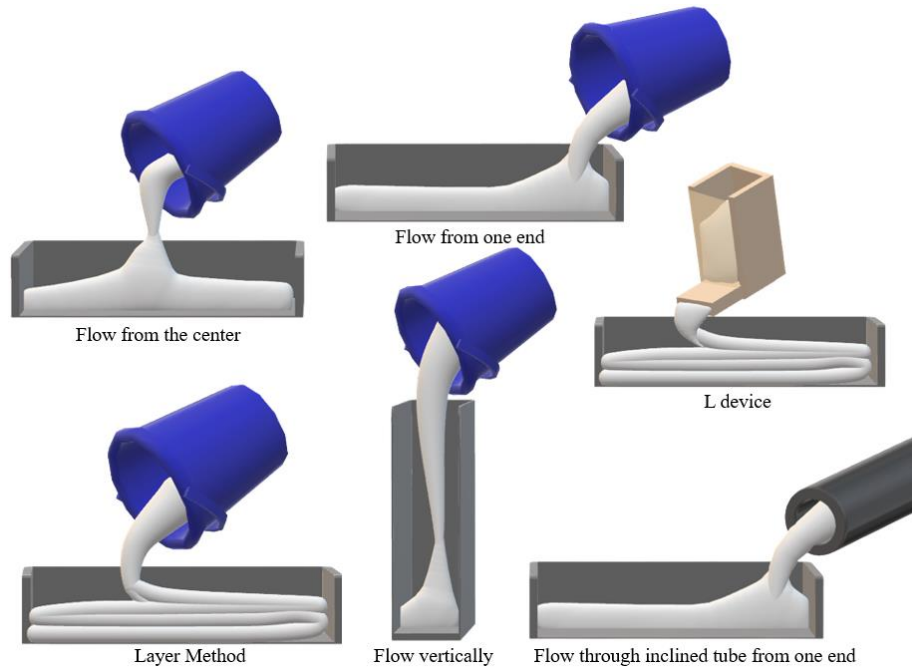


Figure 4.2: Casting methods found in the literature

4.1.4. Rheology

Despite being one of the major aspects dominating fiber positioning, only recently characterization of rheology through standardized equipment has gained interest in the research community. Before this, the correlation of rheology with fiber orientation was studied by simply distinguishing the behaviour of conventional and self-consolidating FRC in fresh state. Through the use of a rheometer at standard shear rate in combination with fluid models, yield stress, plastic viscosity and even thixotropy of THFRC can be determined and correlated to fiber positioning obtained by techniques that will be discussed in forthcoming sections. Although there is limited information on the rheological behaviour of UHPFRC mixes, as it is expected the inclusion of fibers increase the yield stress and plastic viscosity. Indeed, there is a critical concentration of fibers beyond which, the rheological properties of THFRC increases several orders of magnitude over that of the mortar or paste mixtures (Martinie & Roussel, 2011). Moderate rheological properties are important for achieving a uniform fiber distribution. On the one hand, considerably low yield stress and plastic viscosity drive dynamic fiber segregation which results to intense variation in steel fiber concentration and mix inhomogeneity (Jasiūnienė et al., 2018; R. Wang et al., 2017). Dynamic segregation mainly is associated with gravitational forces and shear-induced particle migration. In contrast, significantly high yield stress and plastic viscosity hinder the fiber

dispersion during mixing, having an adverse effect on workability and finally on the mechanical properties. This is because higher air content is entrapped, reducing the packing density whereas fiber alignment is not optimal. However, it is observed that the high cohesion created stabilizes the fibers. Generally, adequate rheological properties are required to prevent fiber segregation and guarantee optimized fiber positioning.

4.1.5. Vibration

UHPFRC mixes are usually self-consolidating eliminating the need of internal vibration that is used in the conventional concrete. However, there are some UHPFRC mixes with higher viscosity that do not qualify as SCC and some form of compaction is needed. In this case, internal vibration is not recommended as the areas where the vibrator is placed is found to be lacking fibers when such methods are used (Døssland, 2008). Generally external vibration is preferred, with some caution as it could have an impact on the orientation along the gravity axis (Laranjeira et al., 2012). The latter phenomenon depends also on the density of the fibers, for levitation of synthetic fibers in ECC mixes has been reported in the literature (Pantazopoulou et al., 2019).

4.1.6. Formwork & Reinforcing Bars

Formwork geometry causes changes in fibre positioning. Previous research has been focused on geometry of the formwork and wall effects. The geometrical restraints cause the fiber distribution in the vicinity of the formwork boundary to become nonhomogeneous, resulting in significant variation in the flexural performance with the distance from the formwork boundary. The effect is more pronounced in thin elements where the depth of the element is comparable to the influenced region (Song et al., 2018). Note that in the field, the wall effects diminish due to the considerably larger size of the formwork and flux effect dominates during casting. Besides the geometry of the molds, an interesting finding is related to their surface. As illustrated in Figure 4.3, a rough surface creates a higher shear zone and a smaller constant speed zone which changes the fiber orientation to vary in the range of depth. In other words, in the constant speed zone the fibers are flow-orientated while in the shear zone they have a more isotropic orientation (Žirgulis et al., 2016). This phenomenon is especially critical for thin structural elements.

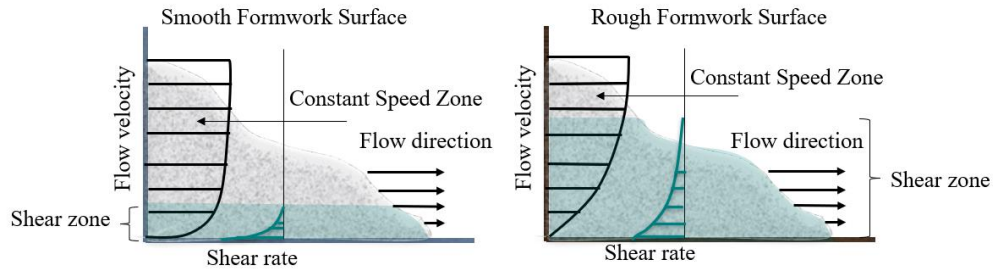


Figure 4.3: Shear rate and flow velocity profile in the vertical plane over the depth of the formwork for a smooth and rough surface (modified from Žirgulis et al., 2016)

Another factor that is capable of causing uneven fiber distribution is the presence of reinforcing bars. Research on how the latter may affect fiber distribution is limited. Žirgulis et al., (2016) investigated the effect of reinforcing bars in fiber positioning and mechanical performance of slabs. Bars caused a fibre blockage, while forcing the fibers to rotate around them as shown in Figure 4.4. In the meantime, shear in the vertical plane was observed similarly to the case of rough framework surfaces explained above. To eliminate the fiber clustering around reinforcement, a bar spacing of at least three times the fiber length has been recommended (Groth, 2000).

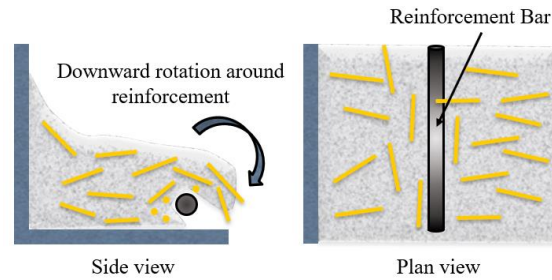


Figure 4.4: Fiber orientation and obstruction in the vicinity of reinforcing bar: side view (left), plan view (right) (modified from Žirgulis et al., 2016)

4.2. Techniques for Estimation and Prediction of Fiber Distribution & Orientation

Fiber positioning is essential for the mechanical response of THFRC, especially in tension. As already discussed above, along the manufacture process of the material there can be a chain of effects that impact the final distribution and orientation of the fibers in the cementitious matrix. Knowing these factors and how they influence the mechanical properties by affecting the positioning of fibers is one step towards better comprehension of, and design with THFRC. Structural modelling with THFRC can only be realized through validation of the above using

techniques to estimate and predict the fiber positioning in the material. During the last two decades, research interest gravitates towards advanced methodologies that enable the determination of fiber orientation and distribution in both fresh and hardened state. The spectrum of methods varies from simple and cost-effective to time-consuming and expensive, while they can be destructive or non-destructive and direct or indirect, as illustrated in Figure 4.5 (Alberti et al., 2018).

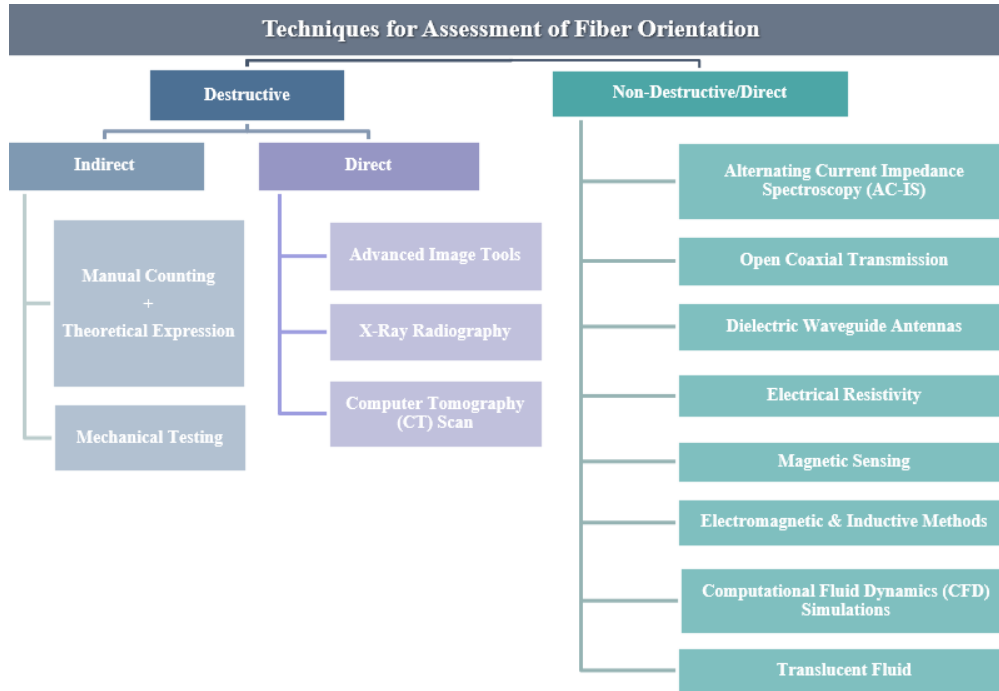


Figure 4.5: Techniques for estimation and prediction of fiber positioning (extended from Alberti et al., 2018).

4.2.1. Destructive Methods for Fiber Counting

The most common and intuitive destructive technique is manual counting of fibers on sawn surfaces with a drawn grid (Saikali, 2019). The actual number of fibers is then correlated against the ideal number through theoretical expressions or a pertinent orientation factor. Although analysis in the three principal directions is possible, the technique is spatially limited. The method of counting can be also performed through advanced digital image tools that are equipped with the capability to analyze the orientation of the fibers. It is also possible to combine the results from different cuts, while spatial limitations are still an issue.

X-ray images of cores or thinly sliced sections have also been used extensively and proved to be effective in visualization of fiber orientation. However, the procedure is cumbersome, especially

in case of high fiber content of short fibers. An interesting technique is X-Ray radiography, where real time flow monitoring is possible during casting. Despite the fiber clustering, the X-Ray techniques are proven to be accurate (Marks et al., 2021). In the hardened state, high accuracy can be obtained with Computed Tomography (CT) scanning. However, the clustering of fibers may hinder the analysis of the 3-D images. Note that because of the long scanning times, small-scale specimens cored from different locations of the structure are often used.

4.2.2. Non-Destructive Methods for Fiber Counting

These techniques are mainly based on the exploitation of the fiber properties, electromagnetic mostly. Alternate Current Impedance Spectroscopy (AC-IS) has been used non-destructively to that end. The fundamentals of the procedure rely on the dual arc behaviour with the inclusion of conductive fibers. It is mostly used to assess the fiber dispersion as besides the orientation it is capable of detecting clumping and segregation (Ferrara et al., 2011). Another practice is the Open Coaxial Transmission Line, where an electromagnetic wave propagates through a cable. In case of abrupt end of the transmission line in open circuit, the wave is reflected. The analysis of the reflected wave provides information about the material in contact while correlation of the sensor with the fiber content is possible. However, the sensitivity of the technique is related to the depth as the sensing of electromagnetic waves deteriorates with the penetration depth and fibers near the surface have higher response (Torrents et al., 2009). This practically means that the method has limited application to thin concrete slices only and cannot be used in large scale practices.

Dielectric waveguide antennas have shown promise for field applications. In this technique, fibers are treated as electrical dipoles and through the Maxwell-Garnett mixing rule for dielectric mixtures, the estimation of fiber density is possible (Roqueta et al., 2011). Finally on the methods that are exploiting strictly the electrical properties of the fibers, measurement of electrical resistivity has been used extensively in literature as an indicator of content and distribution of conductive fibers. It has been shown to accurately produce maps of the fiber orientation. Nevertheless, limitations include specimens with small thicknesses, and as is the case of all methods based on the electrical properties, there is sensitivity to aging, moisture content and presence of electrolytes in the pores (Ferrara et al., 2011).

Magnetic monitoring is an alternative option, because the presence and relative position of fibers modify the magnetic field lines resulting in variation in measured inductance (Künzel *et al.*, 2021).

Numerical models have been also developed to predict the fiber distribution and orientation, especially in the fresh state (Kang & Kim, 2012). Early numerical frameworks are based on Smoothed Particle Hydrodynamics (SPH) (Huang *et al.*, 2020) or on the Lattice Boltzmann Method (LBM) (Švec *et al.*, 2014). Modern simulation techniques may use 3-D computational Finite Volume Method (FVM) models to simulate the behaviour of FRC in fresh state. Computational Fluid Dynamic (CFD) modeling provides insight into the fiber positioning and has been extensively used by researchers towards comprehension of fiber distribution in FRC. In CFD, usually fresh concrete is simulated as a homogeneous non-Newtonian fluid and more specifically can be described by the Bingham model which is explained in detail in future sections. Because of high flowability, thixotropic effects are usually overlooked. The fibers are simulated as particles and regarded as rigid bodies that undergo only translation and rotation (Bi *et al.*, 2017). Generally, CFD modeling is proved to be an accurate tool for prediction of fiber distribution as usually the simulations agree with experimental results (Zhao *et al.*, 2021).

Recently, a translucent fluid model of concrete with consistent rheological properties have been used to predict fiber positioning. For example, polyacrylic Acid (also known as Carbomer or Carbopol) has been used as model concrete for determination of SCC behaviour in the fresh state (Neophytou *et al.*, 2010). The suitability of the material to model concrete in the fresh state is based on the transparency of the fluid but most importantly on the nature of the suspension which is characterized as non-Newtonian fluid described by the Bingham model. An extra advantage is the lack of thixotropy and the recyclability of the material.

Translucent fluid concrete models can be used for the prediction of fiber distribution in FRCs. The suspension is designed and produced with yield stress and viscosity values in the range of FRC matrices and the behaviour in fresh state is simulated. It is generally concluded that fiber positioning is substantially sensitive to yield stress, viscosity but also framework geometry (Boulekbache *et al.*, 2010; Zhou & Uchida, 2017). The high absorption polymer and fiber suspension in it can provide insight about the effects of casting method. In fact, in case of one end

flow the fibers were found to be oriented diagonally upward due to the flow disturbance near the casting point instead of horizontal alignment with the longitudinal direction (Zhou et al., 2013). However, all the available using translucent fluid in the literature studies investigate the effect of considerably low fiber content than the usual contents used in FRC (i.e:1-2.5% steel fibers by volume), while the interference of reinforcing bars has neither being explored.

4.3. *Fiber Distribution Assessment Through Cut Sections, Theoretical Expressions and RVE Computational Modeling*

To better comprehend the capabilities of FRC and develop design guidelines, the effect of fiber distribution and orientation on the mechanical properties of FRCs need be understood. Particularly important to that end is the number of fibers crossing a plane. Theoretical models that are based on modern stereometry or statistics have been developed to determine the so-called orientation factor α . This number represents the ratio between the actual number of fibers crossing a plane, n_{rf} and the theoretical number $n_{th,f}$ (Equation 4.1). The latter is related to the volume fraction (V_f) and the cross-sectional area of the fibers (A_f) per unit area. Assuming the fibers are rigid cylinders with diameter d_f , n_{rf} is defined by Equation (4.1), or Equation (4.2) after explicit substitution of the fiber area. In the ideal situation where the fibers are aligned, uniformly spaced, perfectly positioned, and cross perpendicularly a fracture surface, the coefficient $\alpha = 1$.

$$n_{r,f} = \alpha \cdot n_{th,f} = \alpha \cdot \frac{V_f}{A_f} \quad (4.1)$$

$$n_{r,f} = \alpha \cdot \frac{4V_f}{\pi d_f^2} \quad (4.2)$$

The aforementioned fiber orientation factor also expresses the average ratio, for all possible fiber orientations, of the projected fiber length in the tensile stress direction to the embedded fiber length (Figure 4.6). However, there is no consensus in the research community about the orientation factor, α . In this section, various theoretical models found in literature have been used in combination with manual counting and image analysis of sawn UHPFRC specimen surfaces tested at York. Specimens from several commercial mixes obtained with different casting method, fiber type and fiber percentage have been collected and analyzed to determine which theoretical models are more accurate and under which circumstances. With the aim of addressing this matter,

the most appropriate orientation factor will be used later for the definition of the material constitutive response in tension.

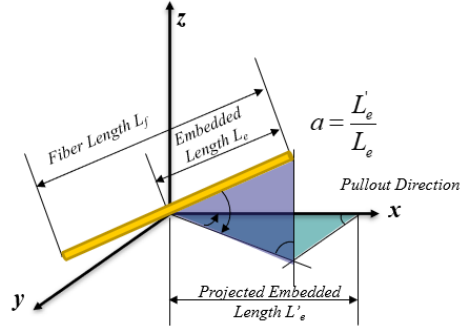


Figure 4.6: Schematic illustration for determining the fiber orientation α (modified from(Holschemacher et al., 2006))

4.3.1. A Review of Theoretical Approaches to Fiber Counting

The geometrical probability problem and the random orientation of short linear elements seems to be a longstanding problem, possibly first established by Buffon, (1777), known as Buffon's needle problem. It is proven to be directly linked with modern stereometry and therefore applicable to concrete technology (Alberti et al., 2018). The problem entails the determination of the probability that a needle randomly thrown on a horizontal plane (a wooden floor with parallel equidistant strips) would come to rest by intersecting one of the lines (Figure 4.7). The length of the needle l is assumed to be equal to the distance between the strips d . The two variables of the problem are the distance of the centroid of the needle from the nearest line and the acute angle θ , that is formed between the former and the parallel lines. The marginal and joined Probability Density Functions (PDF) of the above random variables are given in Equations 4.3-4.5. Buffon's needle problem is used to describe a two-dimensional situation for a fiber randomly distributed in a cell of dimension d equal to the fiber length l and thus resulting in a 2-D probability of $2/\pi$.

$$x : P(x) = \frac{2}{d}, \quad 0 \leq x \leq \frac{d}{2} \quad (4.3)$$

$$\theta : P(\theta) = \frac{2}{\pi}, \quad 0 \leq \theta \leq \frac{\pi}{2} \quad (4.4)$$

$$P\left(x \leq \frac{1}{2} \sin \theta\right) = \int_0^{\frac{\pi}{2}} \int_0^{\frac{l}{2} \sin \theta} \frac{4\pi}{d} dx d\theta = \frac{2l}{\pi d} = \frac{2}{\pi} \quad (4.5)$$

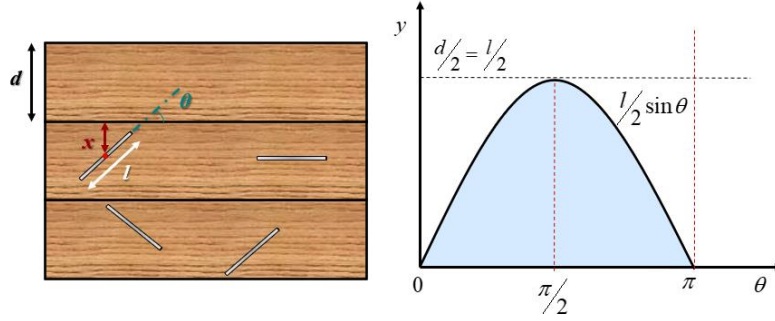


Figure 4.7: Buffon's needle problem

The majority of the derived orientation factors are based on the concept that the three-dimensional condition could be defined by determining two angles which describe the orientation of the fiber. These angles are the rotation angles inside the planes that contain the fibers in its longitudinal axis. Namely, assuming fibers are rigid cylinders, their orientation is defined with a vector $\mathbf{f}$ which is a function of angles φ and θ , as illustrated in Figure 4.8 (Alberti et al., 2018). After determination of fiber positioning, some researchers have used probabilistic models to derive the orientation factors. Table 4.1 provides a range of orientation factor models found in the literature along with their references. Most of them describe 1, 2 and 3-D orientation situations, while recent models take into consideration the anisotropy of casting and formwork or even the aspect ratio of the fibers. It should be noted that 3-D distribution as random orientation with a factor of 0.5 is usually considered for the development of tensile behaviour models of FRCs in the literature such as the Variable Engagement Model (VEM) (Voo & Foster, 2003).

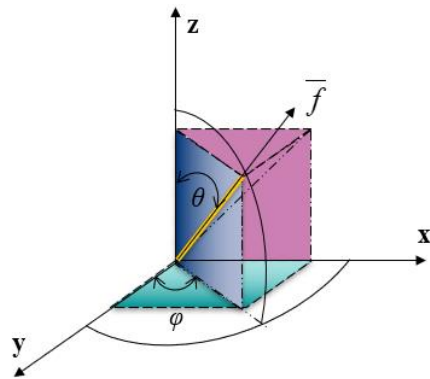


Figure 4.8: Potential rotation of an isolated fiber (modified from Alberti et al., 2018)

Table 4.1: Orientation Factors found in the literature

References	Orientation Factor α	Remarks	Label
Buffon's needle problem (Buffon, 1777)	$2/\pi = 0.6367$	2-D	A2D
(Romualdi & Mandel, 1964)	$(2/\pi)^2 = 0.405$	Random	A3D

(Aveston & Kelly, 1973)	$2/\pi = 0.6367$	2-D	A2D
	0.5	3-D	B3D
(Antoine Naaman et al., 1974)	0.5	3-D	B3D
(Kameswara Rao, 1979)	$(2/\pi)^2 = 0.405$	Vertical	A3D
	$2/\pi = 0.6367$	Horizontal	A2D
	0.5	3-D	B3D
(Nammur & Naaman, 1986)	1	1-D	A1D
	$2/\pi = 0.6367$	2-D	A2D
	0.5	3-D	B3D
(Soroushian & Bayasi, 1987)	$(2/\pi)^2 = 0.405$	3-D	A3D
(Li et al., 1990)	0.5	3-D	B3D
(Soroushian & Lee, 1990)	$2/\pi = 0.6367$	2-D	A2D
	$\frac{l_f}{h} \arctan\left(\frac{h}{l_f \sqrt{6}}\right) \cdot \left(1.56 + 0.766 \cdot \frac{l_f}{b}\right)$ $h \leq l_f$	3-D & Boundary Effect	D3D
	$0.098 \cdot \frac{l_f^2}{b \cdot h} + 0.2l_f \cdot \frac{(b+h)}{b \cdot h} + 0.405$ $h > l_f$		
(Lin & Li, 1997)	$0.382e^{-0.0033h} + 0.37$	Boundary Effect	BH1
(Barr et al., 2003)	0.6	2-D	B2D
(Dupont & Vandewalle, 2005)	0.5	3-D	B3D
(Martinie & Roussel, 2011)	$0.5 + (l_f/20h) \quad h < b$	Slabs	BE1
	$0.5 + (l_f/10b)$	Any other Specimens	
(Michels et al., 2012)	$0.699e^{-0.0024h}$	Boundary Effect	BH2
(Laranjeira et al., 2012)	$0.465 + 0.27 \cdot 0.5$	Layer Effect	BL1
(Alberti et al., 2015)	0.667	2-D	C2D
	0.444	3-D	C3D
(Georgiou & Pantazopoulou, 2016)	0.5	3-D	B3D
(Alberti et al., 2018)	$2/\pi = 0.6367$	2-D	A2D
	0.5	3-D	B3D

4.3.2. Materials and Methods

To assess the accuracy of the theoretical models for the number of fibers crossing a plane, a total of 64 sawn UHPFRC specimens after testing have been analyzed from different mixes, cross-sections, casting methods, and fiber types (Figure 4.9). Three main placement methods were studied: (i) flow from one end (OW), (ii) random placement (R), and (iii) layered (L). More information about the mechanical properties and the proprietary mixes of the tested specimens can be found in Pantazopoulou et al. (2019) and Husain (2021). The commercial mix designs from which the specimens were cast are listed in Table 4.2 while details about the fibers can be found in Table 4.3. Note that for all the theoretical models, the actual dimensions of the fibers were used as they differentiate significantly from the nominal values. The average measurements

of 33 fibers which corresponds to 1% of the maximum theoretical number found in the largest cross-section (50 mm x 150 mm) were used to obtain average fiber length and diameter. The measurements were performed using a digital caliper (Figure 4.10).

Table 4.2: Mix Design of proprietary UHPFRC mixes cast and tested in house

Mix Details & Proportions (kg/m ³)	FD	DE-1	DE-2	LD-1	KI-2	FD-1
Portland Cement	928	1956.2	1956.2	712	1087	928
Silica Fume	223			231	163	223
Ground Quartz	-	-	-	211	-	
Sand	928	443.75	443.75	1020	652	928
D _{max} (µm)	600	2000	2000	600	500	400
Accelerator	-	-	-	30	-	-
Superplasticizer	39	*	*	30	46	39
Water	193	313	313	109	200	193
Fiber Type	S1	S2A	S2A	S2B	S2B	S1+S3
Fiber Content (% vol)	2	1	2	2	2	1+1
Flow Spread D (mm)	210	183	233	228	213	197



Figure 4.9: Types of steel fibers used

Table 4.3: Fiber's geometry and properties

Fiber Type	S1	S2A	S2B	S3
Shape	Straight	Straight	Straight	Double Hooked End
Average Actual Diameter d_f (mm)	0.204	0.21	0.21	0.31
Nominal Diameter d_{f0} (mm)	0.2	0.2	0.2	0.3
Average Actual Length l_f (mm)	20.7	12.9	13.2	25.6
Nominal Length l_{f0} (mm)	20	13	13	25
Actual Aspect Ratio	98	61.4	62.9	82.6
Nominal Aspect ratio	100	65	65	83.3
Material	HCS	LCS	HCS	HCS
Density (gr/cm ³)	7.85	7.87	7.85	7.85
Tensile Strength	≥2500	1900	2750	≥2500
Coating	Copper	-	-	Copper

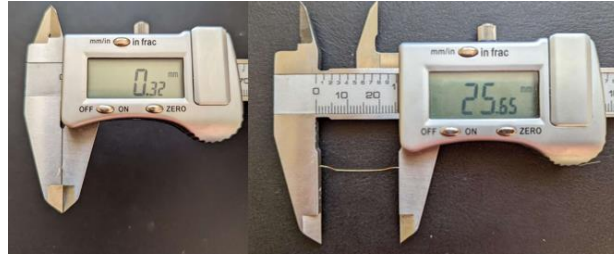


Figure 4.10: Measurement of diameter (left) and length of a S3 fiber (right) using digital caliper.

The UHPFRC specimens were cut after testing at the section near their developed failure crack via a concrete saw. However, when the blade of the concrete saw cuts the specimen, it bends the fibers crossing the section, resulting in a deformation of their cross section, where the diameter of the cut fibers seems to be even 3-4 times higher than the actual (Figure 4.11). The presence of bright fine aggregates such as calcined bauxite in the case of DE mixes, hinders even more the identification of the fibers (Figure 4.12). These two phenomena prevented the use of Advanced Image Analysis as they interfere with the actual number of fibers crossing the surface resulting skewed results (Figure 4.18). For this reason, manual counting was chosen via a drawn grid on the cut surface (Figure 4.19).



Figure 4.11: Deformation of fiber trace due to saw blade

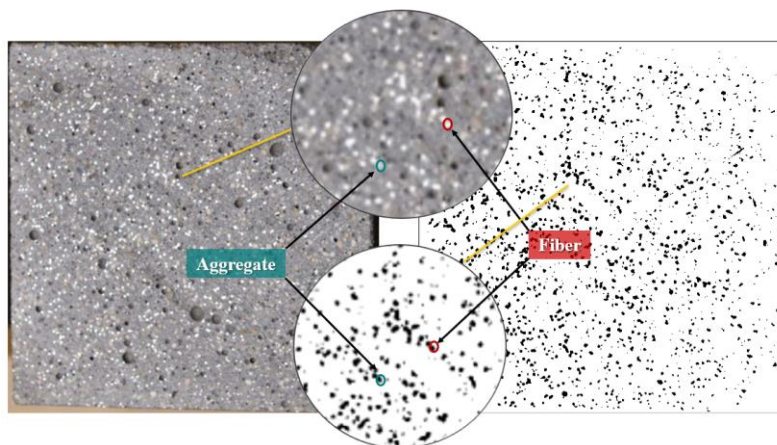


Figure 4.12: The challenge in distinguishing fibers from aggregates via Advanced Image Analysis: Sawn UHPFRC cross section (left) and image analysis (right)

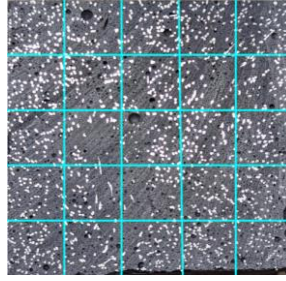


Figure 4.13: Cross section of a sawn UHPFRC specimen with drawn grid

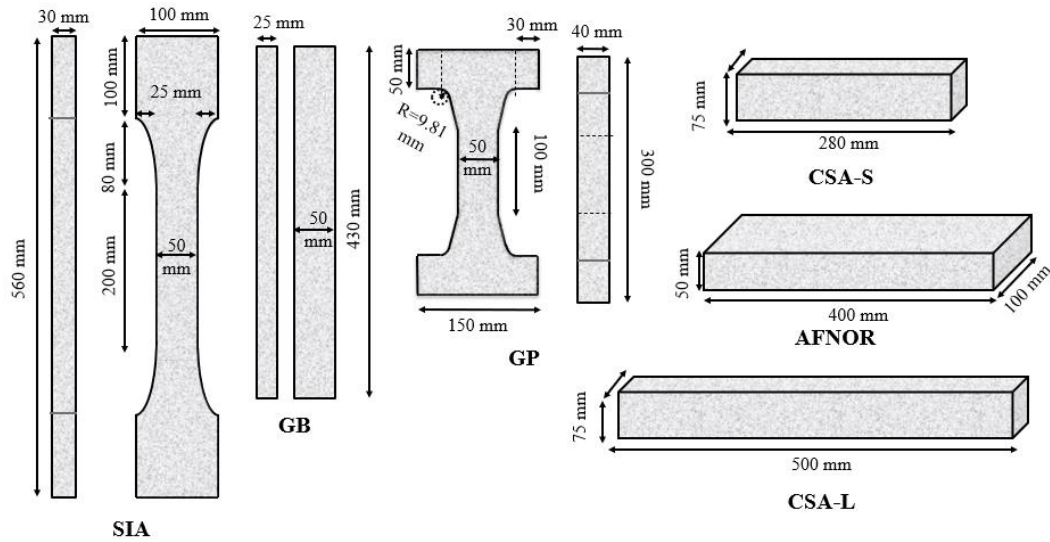


Figure 4.14: Geometry of different specimen types used for manual fiber counting on cut sections

Since different specimen geometries (Figure 4.14) with different fiber types and casting methods were examined, Figure 4.15 illustrates an overview of the naming convention of each examined cut.

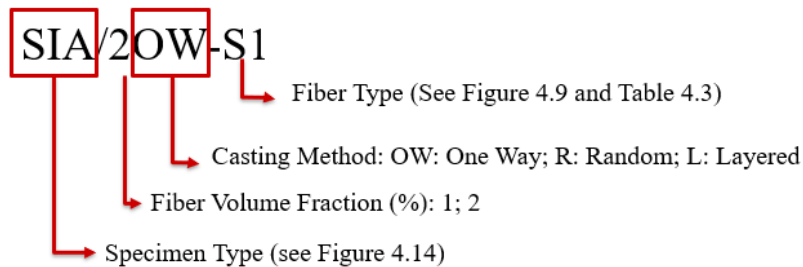


Figure 4.15: Overview of cut naming convention for fiber counting

4.3.3. The Effect of Formwork Geometry

Formwork geometry has a strong influence on fiber positioning. For this reason, the numbers of fibers from FD specimens with different geometry cast with flow from one end were compared

4.3.4. The Effect of Casting Method in Low Fiber Contents

In low fiber contents (1%), fiber clustering and fiber alignment are less expected. Cuts from DE1 flexural prisms with dimensions 75 mm x 75 mm x 280 mm cast with flow either from one end or randomly, were analyzed and their results are shown in Figure 4.17. For OW specimens, the actual number of fibers is found to be around 0.16 per mm² and can be determined more accurately if 2-D orientation and wall effects are assumed. In case of random casting where generally lower number of fibers is expected (0.104 per mm², namely 35% less), it is clear that 3-D orientation prevails with the most conservative orientation factor $\alpha=0.5$. This proves the lack of fiber alignment in lower fiber contents. It is noted that for low fiber content, the theoretical estimates tend to systematically overestimate the fiber numbers crossing an arbitrary plane.

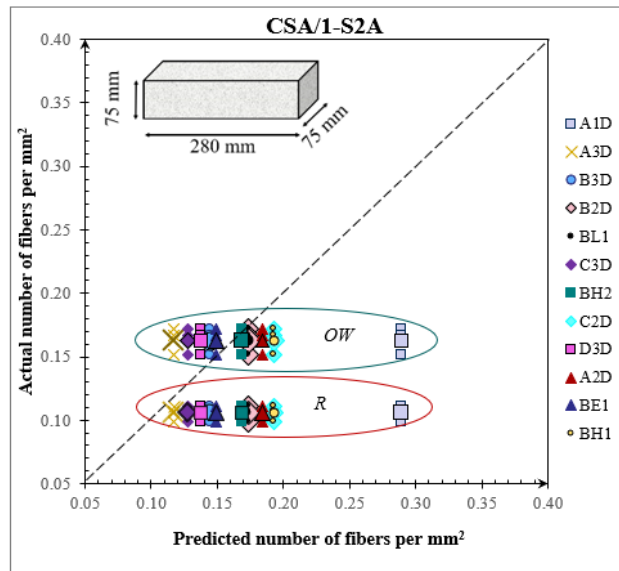


Figure 4.17: Comparison between the actual number of fibers crossing a plane per unit area with predictive models for 1% vol fibers for random and one-way casting

4.3.5. The Effect of Specimen's Length for Different Casting Methods

Increasing the formwork length is an effective method to optimize fiber alignment when flow from one end is utilized (Zhao *et al.*, 2021). To investigate further this effect, cuts from prisms with same cross section (75 mm x 75 mm) but with different length (280 mm and 500 mm) were analyzed. The specimens are selected from castings the DE2, LD1, KI2 and FD1. All castings have 2% vol. ratio of fiber while OW and R casting methods are also investigated. Indeed, as illustrated in Figure 4.16, in both casting methods the long prisms showed an increase by 10% of fibers crossing the section as compared to short ones, while for both prism types, flow from one

end results in 11-12% more fibers compared to random casting. The higher dispersion in data for Figure 4.18(a) can be attributed to the turbulence created from the one end flow which is more prominent in short prisms. In all cases a 2-D orientation dominates. More specifically the fiber number in OW short beams is more accurately described by A2D model, while for the same casting and long beams C2D seems more appropriate. This difference could be attributed to the probability density distribution variation from a nearly uniform distribution to an obviously left-skewed distribution decreasing the range of possible fiber revolutions (Zhao *et al.*, 2021). In random casting, the number of fibers in short prisms is best predicted using models that take into consideration wall effects (BH2) while for long prisms B2D fits better.

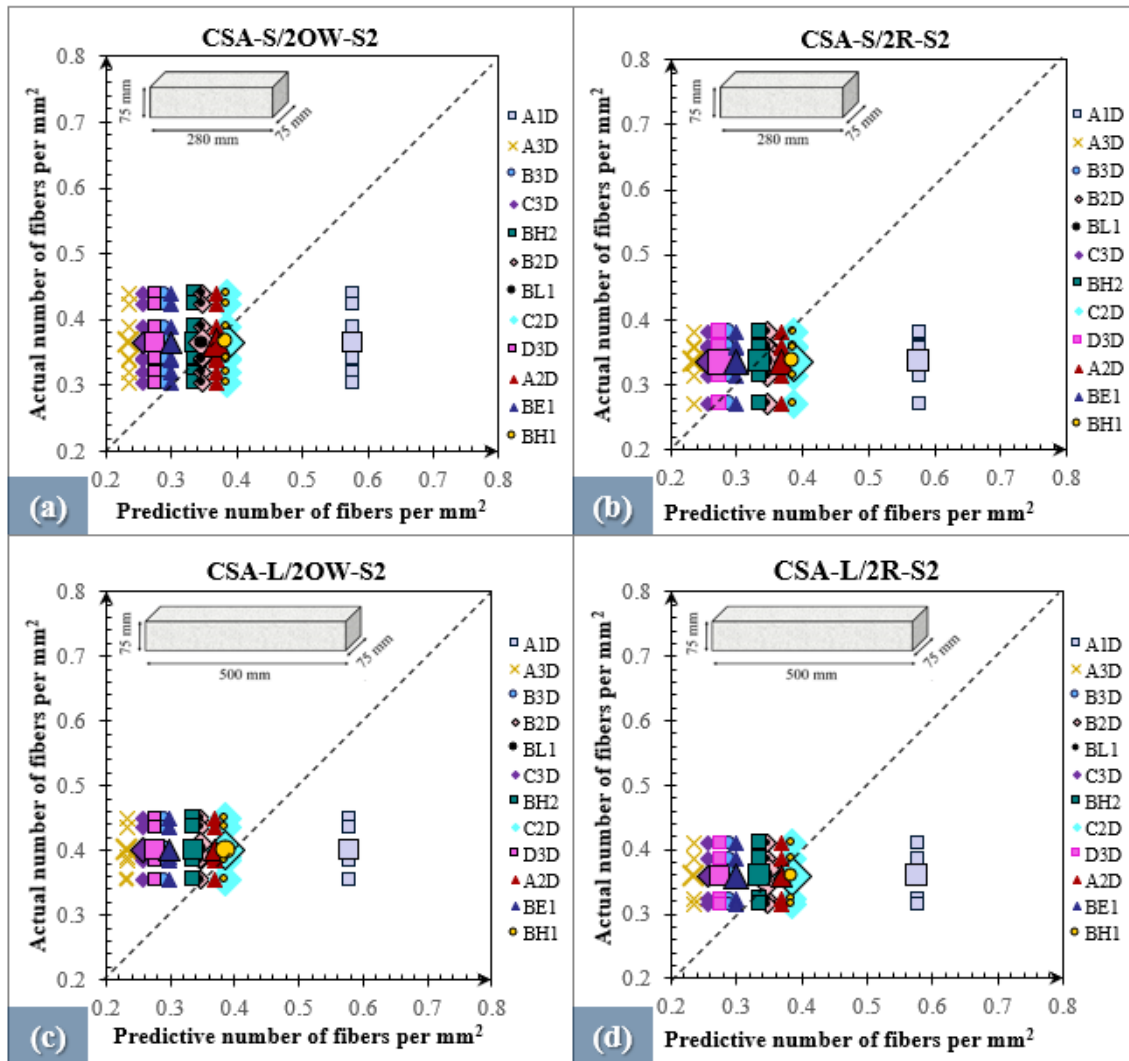


Figure 4.18: Comparison between the actual number of fibers crossing a plane per unit area with predictive models for short and long prisms and random and one way castings from DE2, LD1 and KI2 (Enlarged data points represent the average)

FD1 is examined individually because of the combination of different types of fibers incorporated in the mix. Due to the increased diameter of S3, the actual number of fibers for OW casting is lower compared to the corresponding prisms with short fibers by 20 and 27% for long and short prisms, respectively. The higher reduction in case of short prisms is attributed to the increased length of S1 and S3 fibers (20 and 25 mm, respectively). 2-D orientation is observed again with C2D model to predict with higher accuracy the fiber count. For the random, C2D seems appropriate model. As it can be easily observed from Figure 4.19, there is no significant difference caused by the length of the formwork, but it is notable that short beams cast randomly demonstrated increased fiber counts compared to one way casting, while the contrary is observed in case of long beams. This could be explained as an error in casting, where in case of short beams cast randomly a layered pattern was followed leading to enhanced fiber alignment.

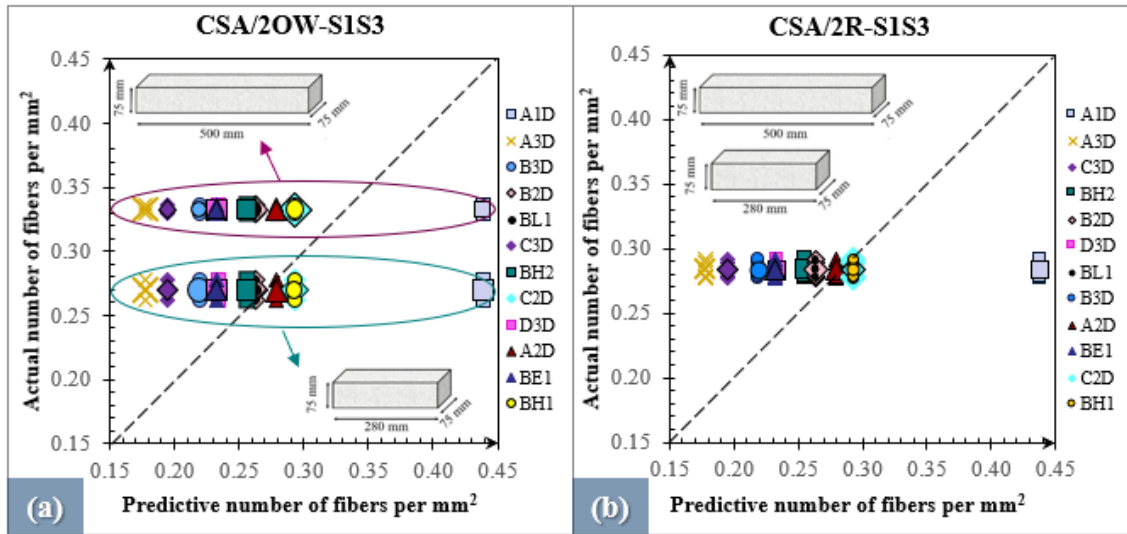


Figure 4.19: Comparison between the actual number of fibers crossing a plane per unit area with predictive models for short and long prisms and random and one way castings from FD1 (Enlarged data points represent the average)

For better insight regarding the fiber distribution in the cross-section, the drawn grids with the manual counting were further analyzed to produce contour plots showing the intensity of fiber count in the cross section and the resulting wall effect. KI2 and LD1 were selected due to their slightly higher difference in fiber count between them, despite the use of the same fiber type. More specifically one way prisms from DE2 have 0.35 fibers per mm^2 , while LD1 and KI2 have 0.37 and 0.44, respectively. As illustrated in Figure 4.20, higher fiber count is generally observed at the bottom of the cross section with exception of the short random specimen KI2R-S (Figure

4.20(a)). This phenomenon may be attributed to potential fiber segregation and wall effects. In both OW specimens an area with less fiber count is observed in the middle, which is linked to the turbulence in flow close to the casting point. The difference in the intensity of the turbulence might be due to different position of cutting planes. In case of LD mix (Figure 4.21), similar phenomena are observed with higher dispersion in case of flow from one end, which can possibly explain the lower number of fibers compared to KI2. Another observation for LD is the reduced number of fibers near the casting side. The trend could be explained by the fact that the casting side was scraped after casting to produce a smoother finishing (Figure 4.22). These contour plots further prove the intense anisotropy and uncertainty prevailing in FRC mixes and validate the high dispersion in results from flexural tests found in literature (Pantazopoulou et al., 2019; Hussain, 2021).

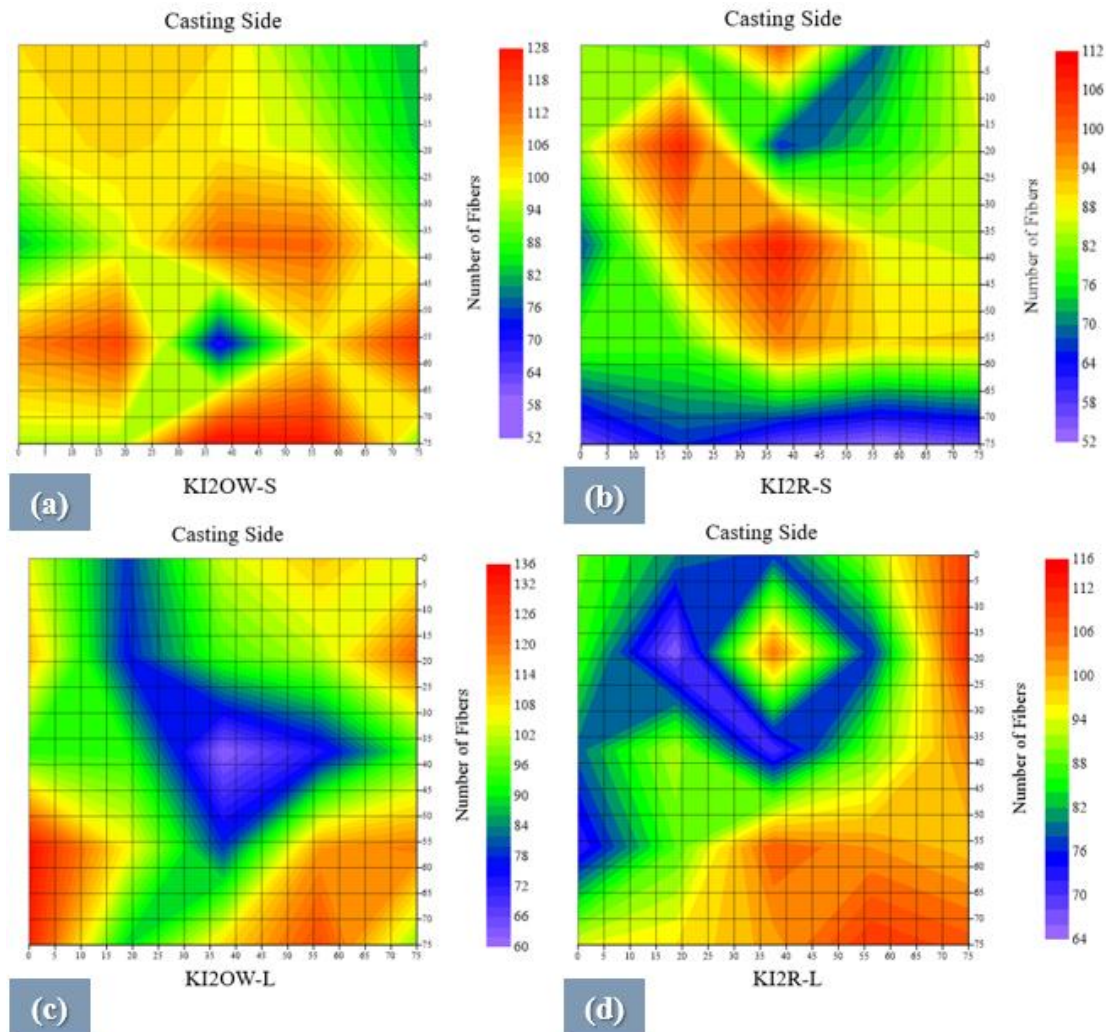


Figure 4.20: Fiber distribution for short and long beams from random and one way casting of KI2

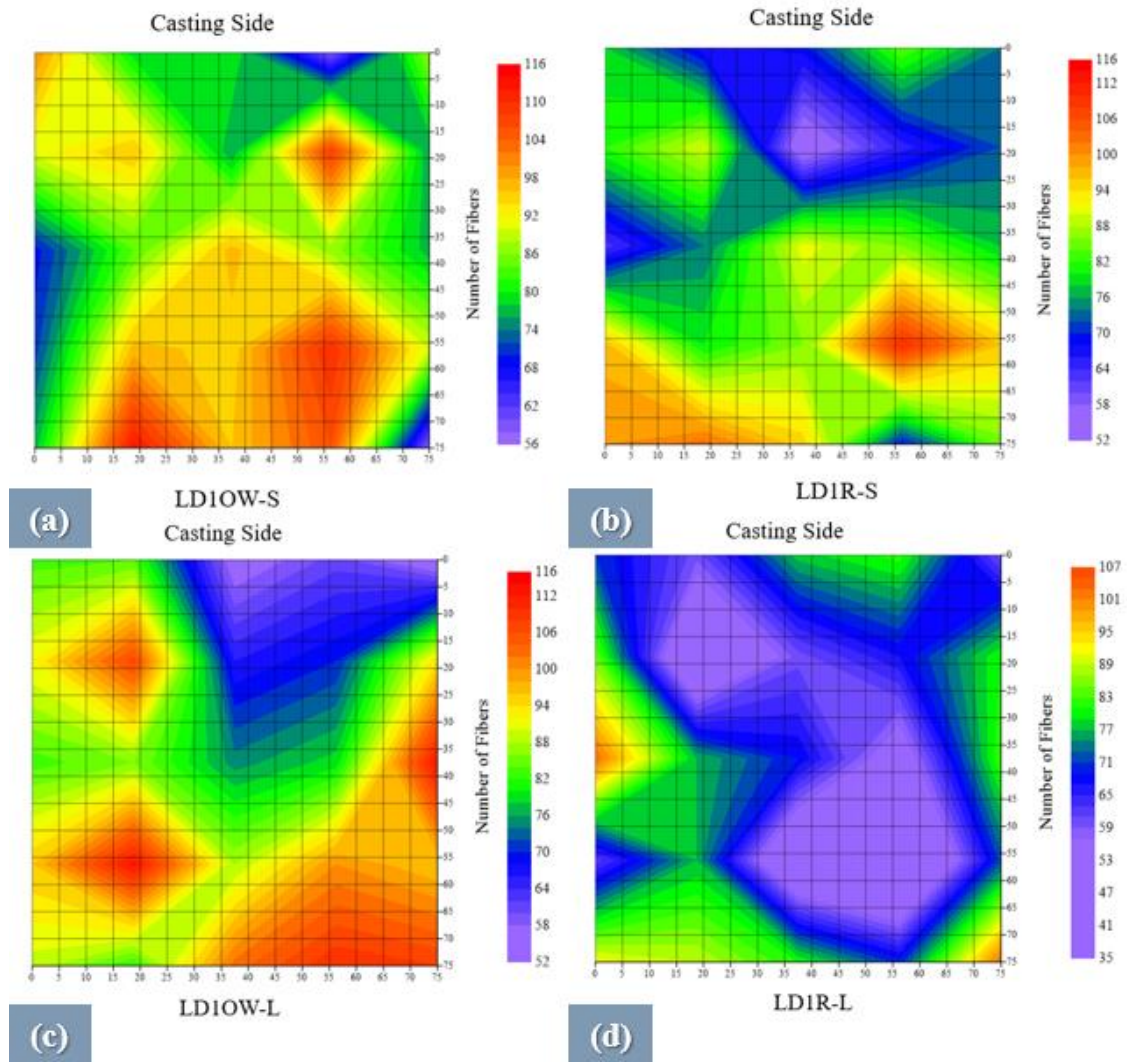


Figure 4.21: Fiber distribution for short and long beams from random and one way casting of LD1



Figure 4.22: Surface finishing by scrapping excess UHPFRC for LD1 mix

4.3.6. The Effect of Flowability

The effect of flowability on fiber distribution is also investigated. For all mixes, flowability was recorded in accordance with ASTM C1856. Figure 4.23 shows the effect of flowability on fiber distribution for each mix as the maximum difference of fiber count between specimens of the same cross section and from the same mix. It is observed that highly flowable mixes (such as DE2) demonstrate higher dispersion in fiber counts. The latter underscores the increased risk of segregation that accompanies high flowability. The opposite trend is observed for more stiff mixes (FD1 and DE1).

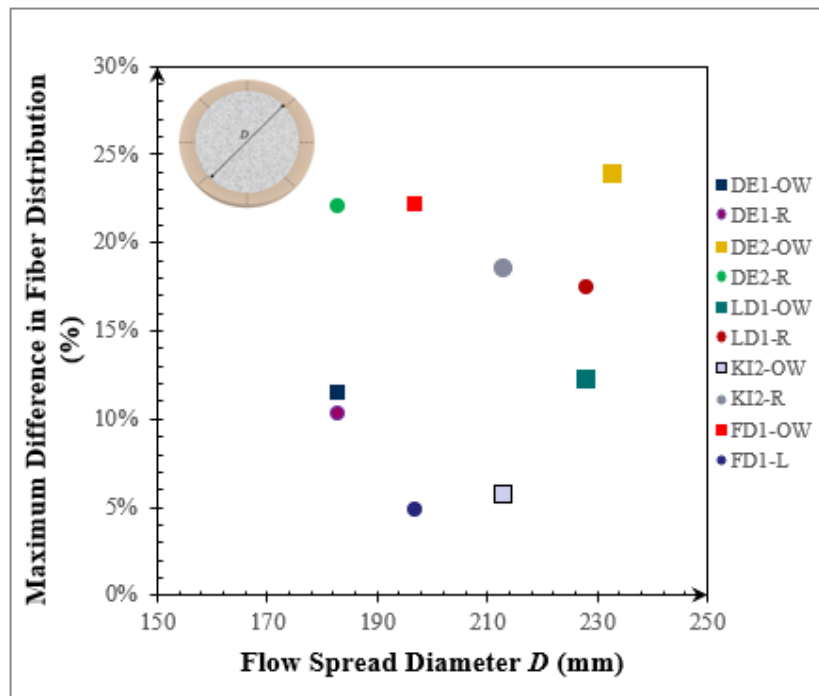


Figure 4.23: Effect of flowability in fiber distribution

4.3.7. Fiber Counting Using RVE Computational Modeling

For a better insight on the correlation of number of fibers crossing a plane with their orientation, a Finite Element based Representative Volume Element (RVE) model was used. The analysis was performed in Material Designer, a relatively new feature of ANSYS software using RVE for short fiber composites. A total of 10 different orientation cases were examined with range from 3-D to 1-D for 2 different seed numbers. These cases were based on varying the diagonal components of the orientation tensor, which has a trace of 1. As Material Designer tries to create an RVE where the principal axes of the orientation tensor match the global coordinate system,

the diagonal components represent these axes. An example of orientation tensor for a 1-D distribution in X direction is given in Equation (4.6). Table 4.4 and 4.5 list the options used for generating the RVE and the different orientation cases, respectively. The extrapolated RVEs can be further analyzed to yield a homogenized material with different material properties according to the fiber orientation. This analysis is out of the scope of this chapter, and thus not presented.

$$A = \begin{pmatrix} 1 & 0 & 0 \\ 0 & 0 & 0 \\ 0 & 0 & 0 \end{pmatrix} \quad (4.6)$$

Table 4.4: RVE geometrical properties

RVE Geometry	
Volume Fraction (%)	2
RVE Size (mm)	20
Fiber Length (mm)	13
Fiber diameter (mm)	0.2

Table 4.5: Different orientation cases

Label	α_{11}	α_{22}	α_{33}
3D-333	1/3	1/3	1/3
3D-442	0.4	0.4	0.2
3D-622	0.6	0.2	0.2
3D-631	0.6	0.3	0.1
2D-550	0.5	0.5	0.0
2D-640	0.6	0.4	0.0
2D-730	0.7	0.3	0.0
2D-820	0.8	0.2	0.0
2D-910	0.9	0.1	0.0
1D-100	1.0	0.0	0.0

Figure 4.24 illustrates the number of fibers measured using RVE versus the theoretical number obtained from Equation (4.1) in different planes and orientations. It is observed that the orientation factor, which is the ratio of the real fiber number to the theoretical ranges from 0.5 to 1, 0 to 0.7 and 0 to 0.5 for the X, Y and Z directions, respectively. In the isotropic case of 3D-333, equal number of fibers are observed in all directions as expected and the orientation factor is around 0.5. For cases with 2-D dimension the orientation factor in the principal directions (here X and Y) is between 0.6-0.7, while values above 0.7 indicate higher alignment in one direction and are closer to 1-D distribution (i.e., 2D-730, 2D-820, 2D-910). It is worth noting here that in 2D orientations, there were fibers in the XY plane. However, they were not considered as they were parallel to the plane (Figure 4.24(c)), resulting in a 0-orientation factor in that plane. Finally Figure 4.25 shows the RVEs of selected orientation cases.

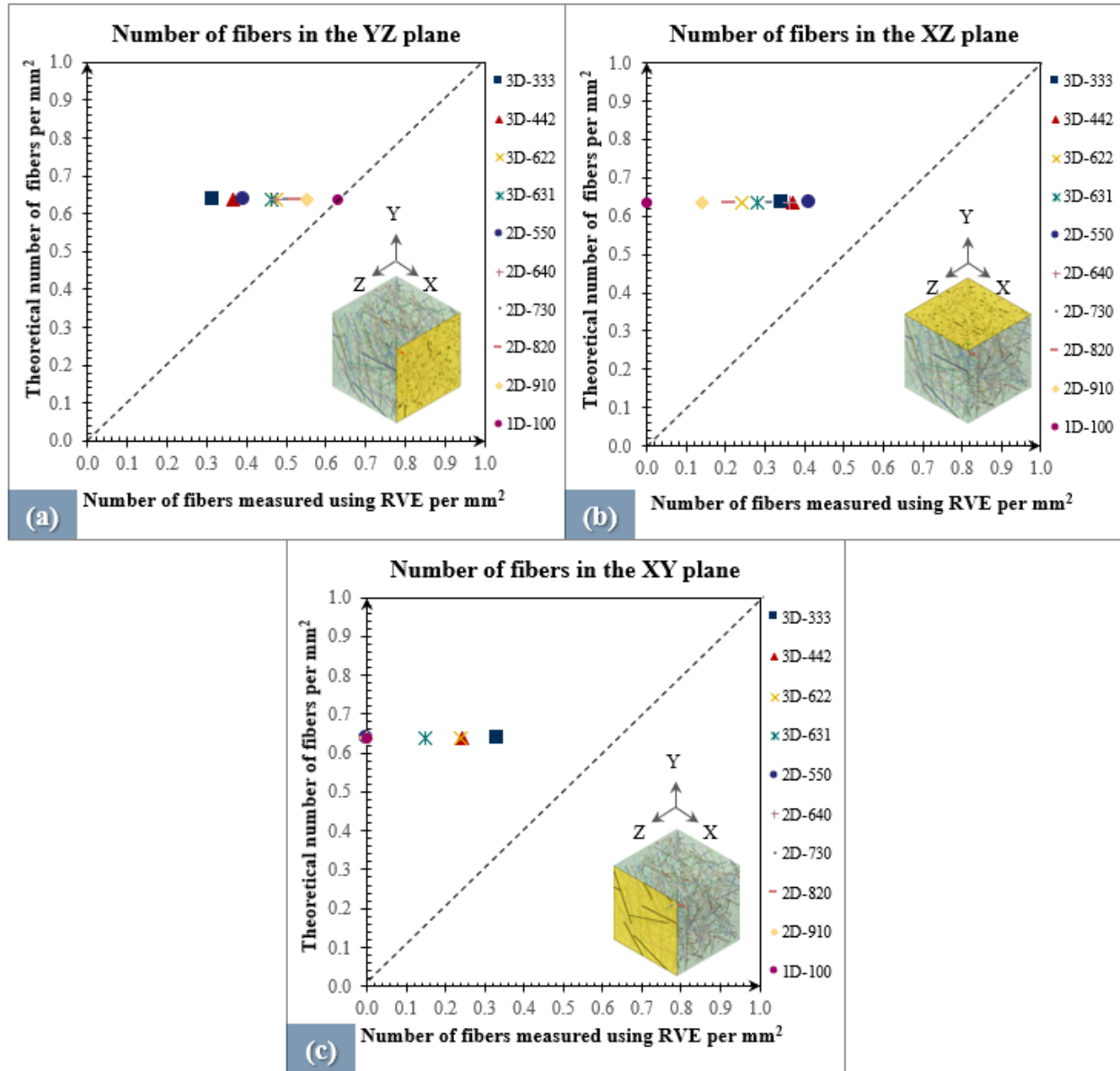


Figure 4.24: Comparison between the theoretical number of fibers crossing a plane per unit area with the number of fibers measured using RVE for (a) YZ, (b) XZ and (c) XY planes.

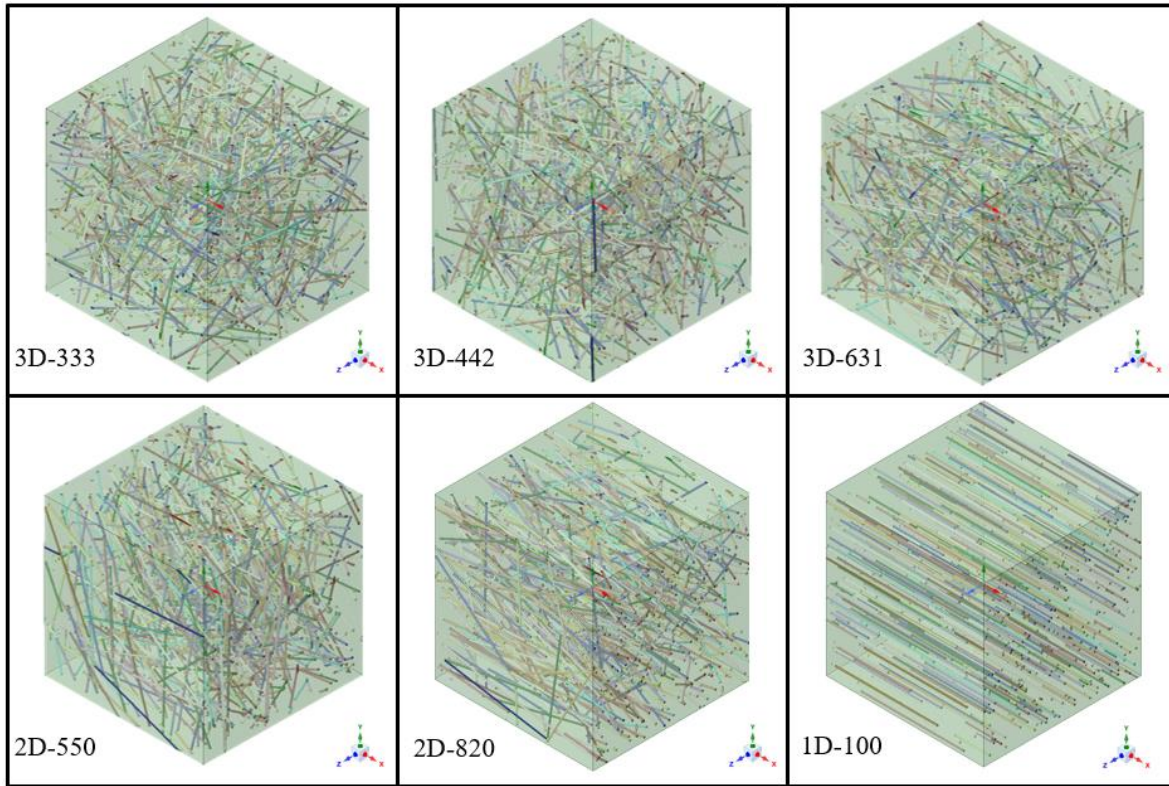


Figure 4.25: RVEs of different orientation cases

4.3.8. Proposed Theoretical Expression for Prediction of Number of Fibers Crossing a Plane

It is evident from the above investigation, that there is no single orientation factor available in the literature that can be used with confidence in predicting the number of fibers crossing a plane. It is also obvious that since fiber distribution is highly influenced by boundary effects, the ratio of fiber length to the dimensions of the specimen should be taken into consideration for the orientation factor. As illustrated in Figure 4.26, in case of very thin prisms where long fibers were used with a length almost the same as the height of the specimen, the fiber distribution was 1-D with the actual number of fibers being very close to the theoretical with orientation factor of 0.9. However, in an opposite scenario, namely a specimen with height magnitudes higher than the fiber length, the distribution of fibers regardless the casting method should be 3-D and an orientation factor of 0.4 as prescribed by CSA Annex S6 should be used.

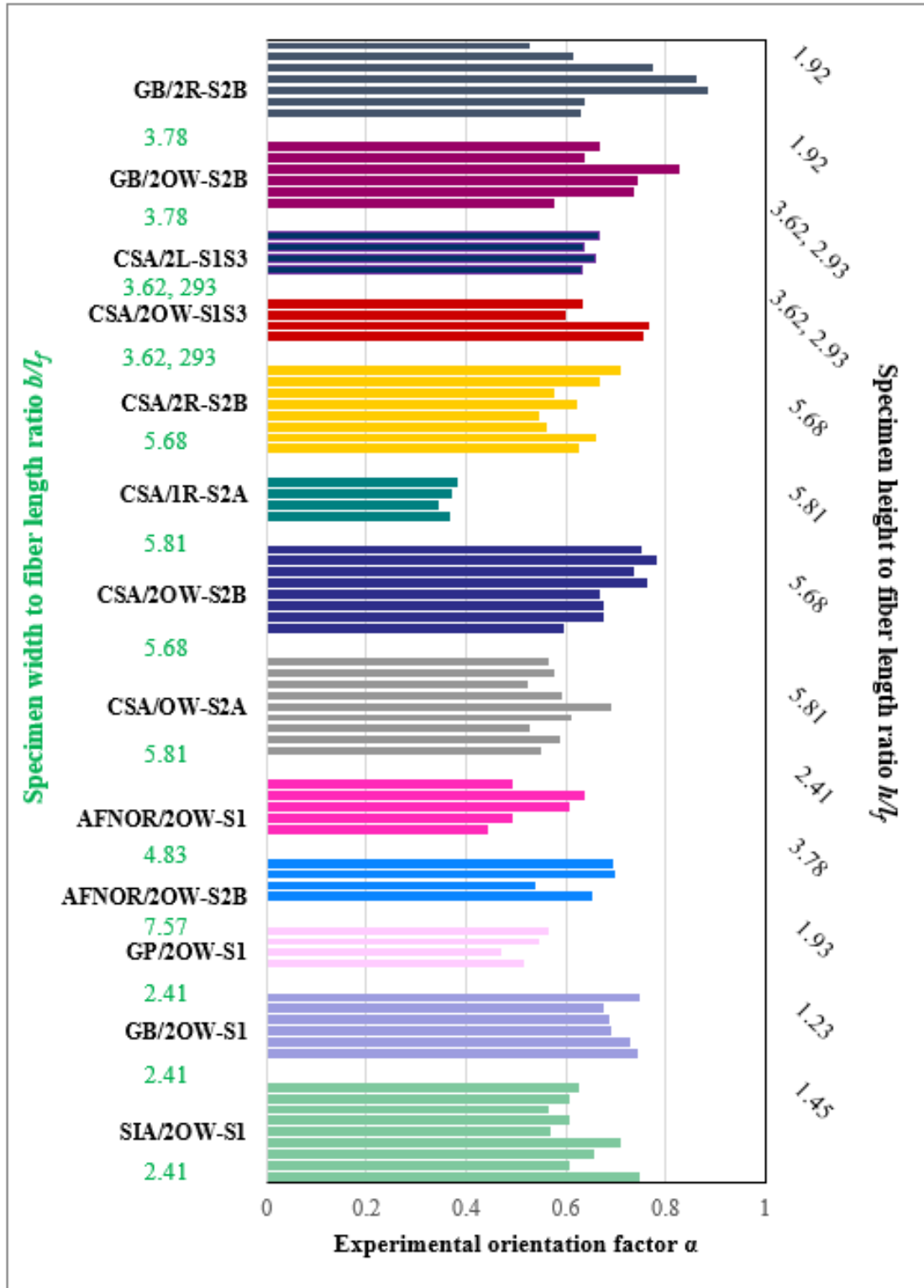


Figure 4.26: Experimental orientation factor for different cross-sections, fiber lengths and placement methods

Taking into consideration the above observations and assumptions a mathematical expression for the orientation factor is proposed. The latter is determined through curve fitting of the

experimental data plotted in Figure 4.27. The theoretical expression of the orientation factor proposed is given in Equation (4.7). The function agrees well with the average of the experimental data with exception to the case of low fiber content and random casting where the dispersion is significant, and the predicted orientation factor should be further multiplied with 0.5.

$$\alpha = 0.4 + \frac{l_f}{h} + \frac{l_f^2}{bh} \text{ for } l_f/h < 0.4$$

$$\alpha = 0.4 + 0.25 \left(\frac{l_f}{h} + \frac{l_f^2}{bh} \right) \text{ for } l_f/h \geq 0.4 \quad (4.7)$$

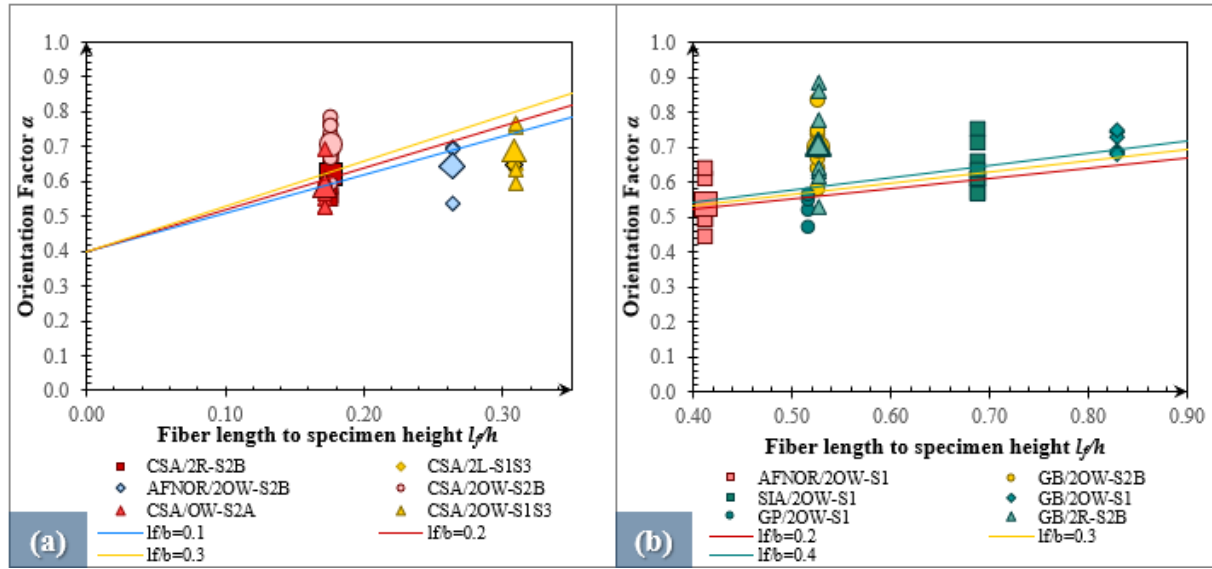


Figure 4.27: Correlation of experimental orientation factor with the fiber length and specimen's cross section and curve fitting of proposed theoretical expression for orientation factor for (a) $l_f/h < 0.4$ and (b) $l_f/h \geq 0.4$

Finally, the proposed theoretical expression is correlated with the fiber efficiency due to size effect. Table 4.6 lists the different specimen types from different proprietary UHPFRC mixes along with their prediction factor. Table 4.7 summarizes the fiber efficiency on linearized tensile properties obtained through inverse analysis per CSA S6 Annex 8 and the corresponding ratio of orientation factors. The linearized tensile properties of FD1, FD and FD2 can be found in Pantazopoulou et al., (2019) and Hussain, (2021). Results for DE3 and KI2 are taken from other projects performed by the author. Fiber efficiency is directly linked to size effect in the same manner that orientation factor is correlated to the geometry of the specimen. Indeed, the ratio of orientation factors between specimens with different scale agrees with the ratio of cracking and post cracking tensile strength for the corresponding specimens.

Table 4.6: Predicted orientation factor and details of flexural prisms with varying size

Specimen Type	Fiber Type	Mix	h (mm)	b (mm)	A (mm ²)	l_f/h	l_f/b	Orientation Factor a
AFNOR-50	S2B	DE3	50	100	5000	0.26	0.13	0.70
AFNOR-100	S2B	DE3	100	150	15000	0.13	0.09	0.54
CSA-75	S1S3	FD1	75	75	5625	0.31	0.31	0.80
CSA-75	S2B	KI2	75	75	5625	0.17	0.17	0.60
CSA-100	S2B	FD2	100	100	10000	0.13	0.13	0.55
CSA-100	S1S3	FD1	100	100	10000	0.23	0.23	0.69
CSA-100	S1	FD	100	100	10000	0.21	0.21	0.65
CSA-150	S1	FD	150	150	22500	0.14	0.14	0.56
CSA-150	S2B	KI2	150	150	22500	0.09	0.09	0.50
CSA-150	S2B	FD2	150	150	22500	0.09	0.09	0.49

Table 4.7: Fiber efficiency due to size effect and proposed orientation factor

Size Ratio (A_B/A_S)	$f_{cr,m,B}/f_{cr,m,S}$	$f_{Fu,B}/f_{Fu,S}$	α_B/α_S
1.78	0.77	0.65	0.85
2.25	0.79	0.91	0.90
2.25	0.82	0.67	0.86
3.00	0.73	0.73	0.78
4.00	0.97	0.77	0.82

4.4. Visualization of Fiber Distribution via Translucent Fluid Model Concrete Based on Yield Stress

As already discussed in previous sections, the placement method along with wall effects generated from formwork geometry and the fresh material's rheology, especially yield stress, are considered to be crucial factors impacting fiber distribution and orientation in FRC. So far, by means of manual counting and theoretical models wall effects and placement method were studied, but with 2-D spatial limitations imposed by the method. Better insight on rheology and the aforementioned factors influencing fiber distribution is possible through a translucent fluid model concrete based on yield stress. The method has been used in the literature before either, by simulating the yield stress (Boulekbache *et al.*, 2011) or the flow spread (Zhou & Uchida, 2013). In this section a translucent fluid with yield stress and flow spread similar to UHPFRC is employed to visualize the effect of yield stress, fiber content, casting method, formwork geometry and presence of reinforcing bars on fiber distribution of UHPFRC.

4.4.1. Conceptual Basis

Fresh concrete is a concentrated suspension of solid particles (aggregates) in a viscous medium (cement paste). Similarly, cement paste is a concentrated suspension of cement grains in water. In fresh state, cementitious materials behave as a viscous non-Newtonian fluid with yield stress and plastic viscosity. Differences in the material constituents of fiberless matrix of UHPC (compared to CC) results in the different rheological properties for UHPC as indicated in Table 4.8. There is a considerable number of different models used to describe the rheological behaviour of non-Newtonian liquids such as cement-based materials. Some of them are found in Figure 4.28.

Table 4.8: Typical rheological properties range for different concrete mixtures (Khayat et al., 2019)

Rheological Property	CC	SCC	HPC	UHPC
Yield stress (Pa)	500-2000	5-50	50-2000	10-100
Plastic viscosity (Pa.s)	50-100	100-400	50-550	20-200

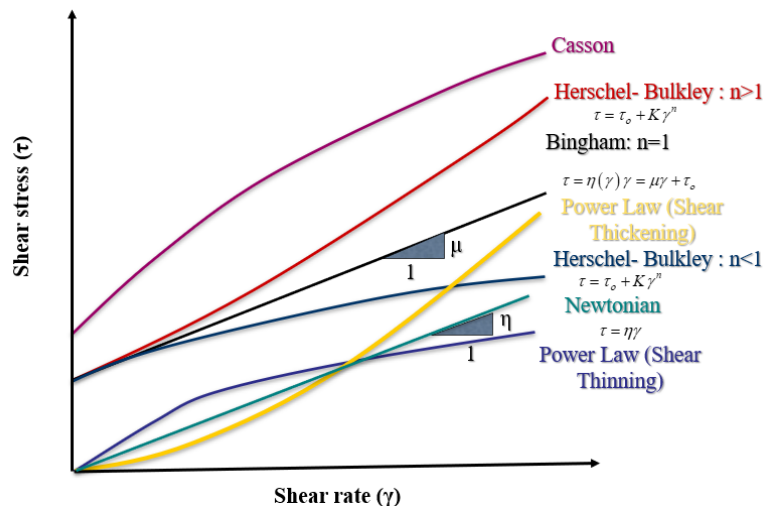


Figure 4.28: Models used for characterization of the rheological behaviour of cementitious materials in the fresh state (modified from W. Wang et al., 2021)

The most common is the Bingham model which is linear and described by Equation (4.8):

$$\tau = \tau_o + \mu_o \cdot \dot{\gamma} \quad (4.8)$$

In Equation (4.8), variable τ is the shear stress, τ_o is the yield stress, μ_o is the plastic viscosity and $\dot{\gamma}$ is the shear strain rate. Yield stress is defined as the critical shear stress value required to initiate deformation, where in this case this represents the flow and is a result of interactions between particles. Fresh concrete behaves as an elastic solid when the shear stress is less than the yield, and when it becomes equal or surpasses it, concrete starts to flow. Plastic viscosity describes the resistance of the material to flow. Yield stress and plastic viscosity are time-dependent properties

which are also affected by thixotropic effects due to hydration (Neophytou et al., 2010). For zero yield stress, the Bingham model becomes Newtonian where the viscosity is constant. Although the Newtonian model represents basic fluid flow, it fails to describe the rheological behaviour of cementitious materials in the fresh state, as most fluids behave non-linearly. For this reason, non-linear models are frequently used; for example, the Herschel-Bulkley model (Equation (4.9)), is the generalization of Bingham model and is more suitable to represent the rheology of fresh concrete.

$$\tau = \tau_o + K \cdot \dot{\gamma}^n \quad (4.9)$$

In Herschel-Bulkley model, a consistency (K) and a flow index (n) are introduced. For Bingham model $n=1$. Flow index values greater than one represent shear thickening, while values less than one, shear thinning. While UHPC paste mixtures often show a shear-thickening behaviour, the addition of the fibers, reverses into shear thinning. Meng & Khayat (2018) evaluated the rheological flow behaviour of fiberless UHPC mixtures and observed a linear variation in the relation between the shear stress and strain rate. Arora et al., (2018) and W. Wang et al. (2021) also reported the linear flow behaviour for UHPC paste and mortar mixtures, respectively. The difference in the flow behaviour (linear/nonlinear) can be attributed to the difference in shear rate for UHPC mixtures indicating that it is important to know the shear rate ranges that are expected during a particular material batch to accurately estimate rheological properties and to predict flow behaviour.

Practically, yield stress might be the most critical rheological property, regarding fiber orientation, according to shear tests found in literature (Sepehr et al., 2004) whereas plastic viscosity is associated to the velocity of the flow but in high values it might hinder a uniform fiber distribution (Boulekbache et al., 2011). For this reason, a translucent fluid model concrete based on yield stress is chosen to investigate visually the correlation between rheology and fiber distribution. The studied parameters include fiber content, yield stress, placement method and height, formwork geometry and presence of reinforcement.

4.4.2. Materials and Methods

The translucent fluid used in this study is a suspension of an acrylic polymer frequently used in pharmaceutical and cosmetic industry, also known as Carbomer. Carbomer 940 dispersion is an

excellent candidate for the model concrete as it can be described by yield-pseudoplastic rheological models like the Herschel-Bulkley model. The yield stress develops due to the formation of a continuous particles network which interact with each other forming a continuously interacting hydrogel (Kelessidis & Hatzistamou, 2011). The preparation of the solution involves two stages: (i) dispersion of Carbomer powder in distilled water and (ii) neutralization of the obtained solution. Figure 4.29 shows in steps the preparation process of the model concrete. The polymer was first weighed and then added in small quantities in distilled water while the solution was continuously mixed with a magnetic stirrer at slow speed to achieve uniform distribution of Carbomer particles and in order to eliminate the formation of bubbles. In this study the neutralizer that is used to raise the pH to be around 7 is triethanolamine (TEA). At this point the thickening increased progressively and the liquid is transformed to a gel. The addition of TEA was performed gradually as trials showed that adding the neutralizer in one dosage increases abruptly the rheological properties. After neutralization, a few drops of coloring were added to color-code the different model concretes. Red was used for the highly flowable mix (0.2CM) and blue for the less flowable (0.3CM). Then the mixtures were left to settle for 48 hr.

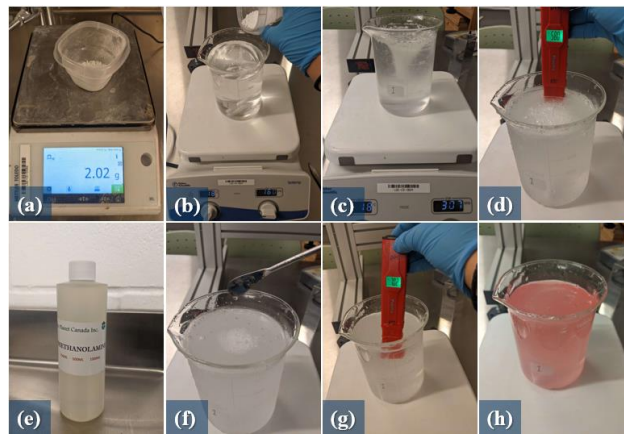


Figure 4.29: Preparation of Carbomer Suspensions

In this study two different Carbomer concentrations were used to simulate a highly flowable and a viscous mix. Namely, the two ratios of water over polymer powder are 0.2% and 0.3% (grams of dry powder dispersed in 100 ml distilled water) simulating the flowable mix, respectively. As stated above Carbomer solutions if prepared properly are very stable and no change in rheological properties is expected with time. For this reason, right after their production and several days after the resting period their yield stress was recorded via software running rheology tests in the ICAR

rheometer (Figure 4.30 (a)). As expected, yield stress is proportional to the concentration of the polymer, with 0.2% exhibiting a yield stress of 35 Pa, while in case of 0.3%, yield stress was 58 Pa (Table 4.9).

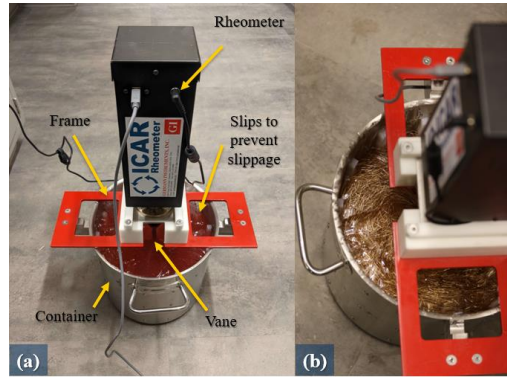


Figure 4.30: Rheometer used to measure yield stress of model concrete (a) without fibers and (b) with fibers.

The fiber type used in this investigation is S2A as described in detail above. For 0.2CM suspension two fiber contents by volume were investigated, namely 1% (0.2CM1) and 2% (0.2CM2); similarly, for the case of 0.3CM suspensions of 1% (0.3CM1) and 2% (0.3CM2) were studied. To verify the mass of fibers added to the suspension, fibers were added slowly in a volumetric tube with 500 mL of water till the volume changes to 510 mL. It is noteworthy that the change in the volume of the water in the cylinder after the addition of 2% fibers is imperceptible when considering the number of fibers added as illustrated in Figure 4.31.



Figure 4.31: Change in volume with addition of 2% by vol. fibers

Table 4.9: Rheological and flowability properties of the studied suspensions

Rheological Property	0.2CM	0.2CM1	0.2CM2	0.3CM	0.3CM1	0.3CM2
Yield Stress (Pa)	35	43	-	58	-	-
Static Flow Spread (mm)	178	180	190	163	145	138
Dynamic Flow Spread (mm)	-	-	-		185	170

The fibers were added slowly to the suspension while mixing to achieve a uniform distribution in the model concrete. The mixing stopped when homogeneity and elimination of entrapped air were attained. After the mixing, a flow table was used and the flowability of the suspensions with the fibers along with the time needed for flow were measured according to ASTM C1856 (Table 4.7). For the 0.3CM1 and 0.3CM2 suspensions, dynamic flow spread was also recorded (Figure 4.32). Then the rheometer was also used to measure the yield stress of the suspension 0.3CA-1.

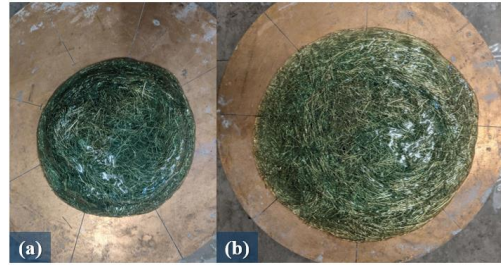


Figure 4.32: Flow spread for 0.3CM1: (a) static, (b) dynamic

Finally, the model concrete was poured into transparent plexiglass formworks of various geometries, some mimicking the characterization tests conducted for THFRC materials (Figure 4.33) using different placement methods.

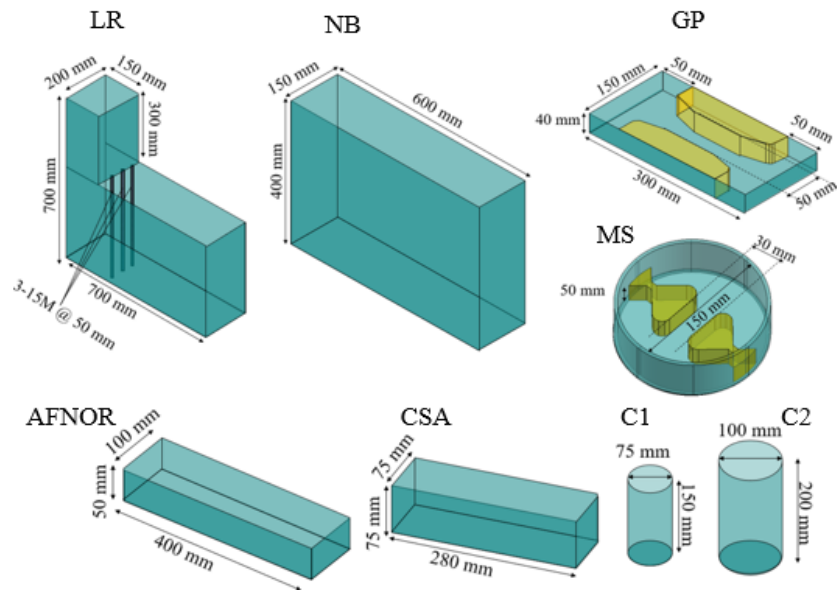


Figure 4.33: Plexiglass formworks used

4.4.3. Effect of formwork geometry and presence of reinforcement

The objective of the tests was to see how the fiber alignment is affected by the formwork geometry and presence of reinforcement bars. For this reason, formworks LR and NB were investigated.

Mold type LR is slightly wider and longer than type NB. Figure 4.34(a)-(d) illustrates the time history of one-way casting performed in the NB formwork. At the start of the casting the flow is turbulent, transitioning to laminar as the model concrete approaches the end of the mold. During casting, eddies, which are swirls observed in turbulent flow are formed around the casting point. The eddies have high kinetic energy drifting the fibers and leaving at the end of the flow the stagnant zone containing fewer fibers and thus creating a weak point with lower homogeneity. The swirls at the end of the flow create a 45-degree plane that would coincide with a potential shear crack in flexural prisms. This is consistent with UHPFRC shallow prisms cast with flow from one end and tested in flexure. Meanwhile at the opposite side of the formwork, the fibers tend to change their orientation due to wall effect. More specifically, the fibres re-orient themselves at 45-degree angle relative to the longitudinal axis.

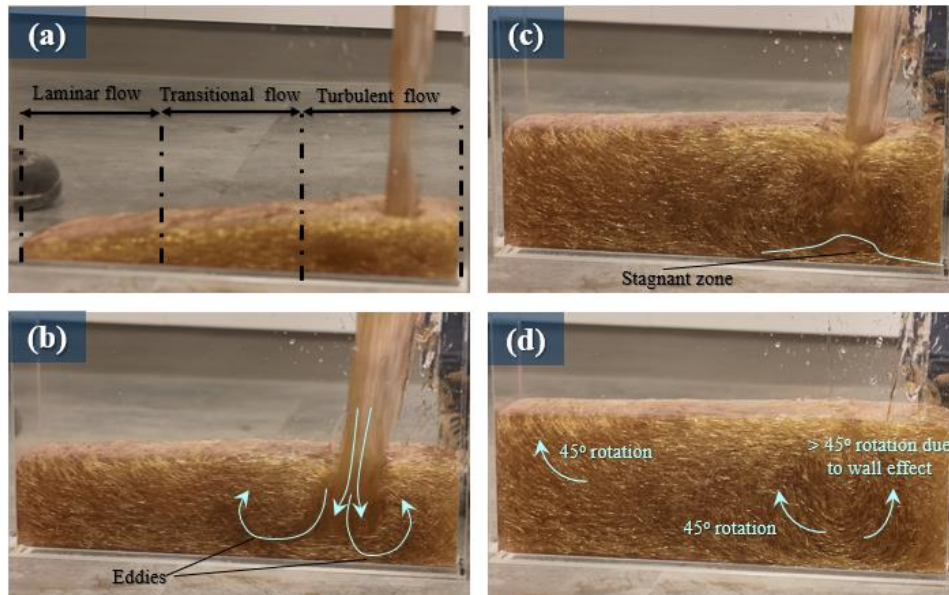


Figure 4.34: Flow timeline of one-way casting 0.2CM1 in NB formwork and fluid dynamics effects on fiber distribution and orientation

Similar behaviour with 1% fiber content is observed for 2% (Figure 4.35). However, because of the larger number of fibers, the effects are detected in a clearer way. The increase in the fiber content suggests that eddies under the casting point engage more fibers than before, and the turbulence does not have such a detrimental effect on the distribution affecting the homogeneity of the area. Nevertheless, still the swirl drifts the fibers in a circular orientation, diminishing the longitudinal distribution, and thus resulting in a region of weakness.

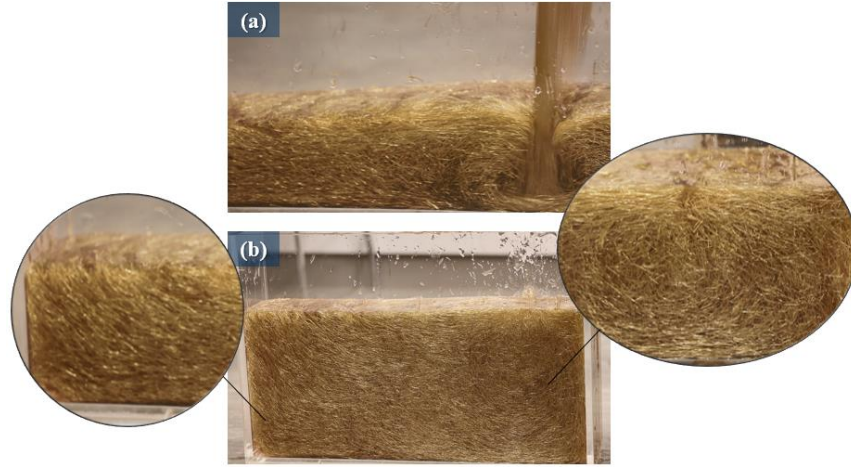


Figure 4.35: Fiber distribution of 0.2CM2 one way casting in NB formwork

Fiber distribution and orientation in formwork type LB with flow from end was also examined. Figure 4.36(a)-(c) shows the flow timeline. The starting of the casting is similar to type LB where turbulent flow at the casting point transitions to laminar towards the end of the mold. The eddies and disturbance around the casting point is not as prominent as in the case of LB and this could be correlated to the slightly increased dimensions of the formwork. Meanwhile at the opposite side of the formwork, similar to LB, the fibers tend to change orientation due to wall effect. It is also observed again that the increase of fiber content does not significantly change the effects that take place in the concretes with lesser fiber volume fraction.

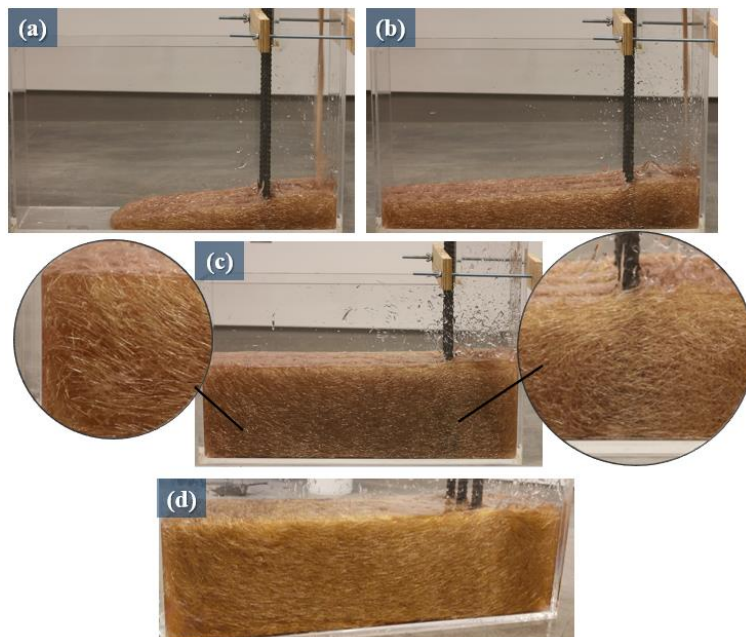


Figure 4.36:(a-c) Flow timeline of one-way casting 0.2CM1 in LR mold and fiber re-orientation, (d) fiber distribution of 0.2CM2

Another parameter that was studied with formwork LR is the presence of reinforcement bars. As illustrated in Figure 4.37, the flow creates separation planes which could potentially cause longitudinal splitting (Figure 4.37(a) and (b)). However, with higher fiber content, the separation planes are less distinct resulting to better homogeneity (Figure 4.37(c) and (d)). Although the bar spacing was chosen to be 3.8 times the length of the fiber, some clustering is detected with higher fiber content.

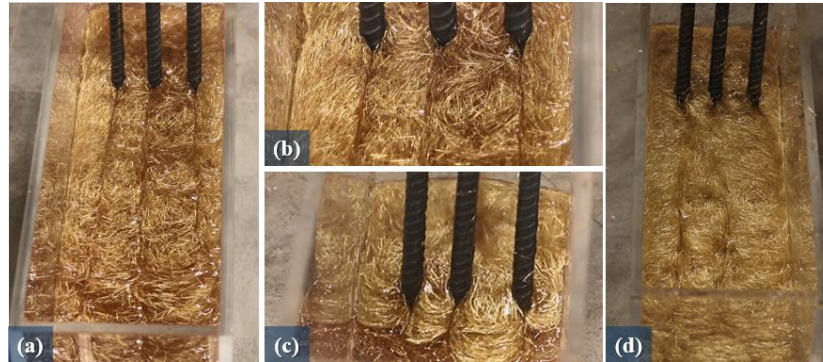


Figure 4.37: Separation planes around reinforcement for flow from one end for (a) 0.2CM1 and (b) 0.2CM2

Model concrete was also poured in one layer (Figure 4.36) in cylindrical molds of two different sizes (C1 and C2) to assess the distribution in cylindrical specimens used to test the compressive strength. Random orientation is predominant in the case of the bigger cylinder (Figure 4.38(b)). However, in the case of the smaller specimen, because of the wall effect, fiber align in the circumference (Figure 4.38(c)). The difference in the fiber distribution and orientation between the two sizes indicated that strength difference and deformation capacity would be expected when testing large vs small specimens in compression (Note here that Annex U requires compression testing to be conducted on 75 mm diameter specimens; clearly the results would be expected to be overly favorable in these experiments compared to what might actually occur in the field if the fiber orientation is different).

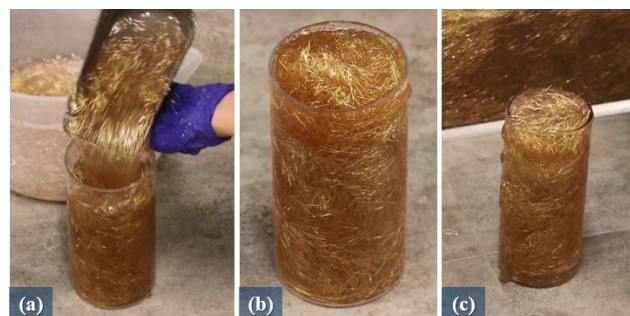


Figure 4.38: (a) One layer casting of cylinder, (b) fiber distribution in C2 and (c) fiber distribution in C1

4.4.4. Effect of Casting Method

To study the effect of placement method in shallow formworks, type AFNOR mold was used, and three different methods were investigated, namely flow from one end, flow from the center and random. In case of flow from one end, it is clearly demonstrated that eddies are generated due to turbulent flow close to the casting point but also spreading towards the midspan (Figure 4.39(a)). The re-orientation of fibers at a 45 degree angle in the vertical plane is clear again around the casting point. However, in the horizontal plane at the end of the mold they seem to turn around to 90 degrees as compared to the longitudinal axis. Another observation is that in case of highly flowable mixes, segregation is evident, as a significant number of fibers is concentrated in the plug flow on top of the stagnant zone compared to the end of the formwork. Another sign of segregation is the higher amount of fibers concentrated in the bottom layer than in the top. For flow from the center, eddies are evident around the casting point. The fibers separate from the center outwards creating a wake region underneath. However, casting from the center created a more symmetrical distribution of fibers near the ends of the mold compared to one way placement (Figure 4.39(b)). Finally for random casting, there are some fibers that are aligned with the longitudinal axis and no significant eddies are observed (Figure 4.39(c)). Nevertheless a 2-D orientation prevails due to the randomness of casting. Usually in random placement, 3-D orientation is dominant, but in case of shallow specimens, due to wall effects the orientation is more two-dimensional.

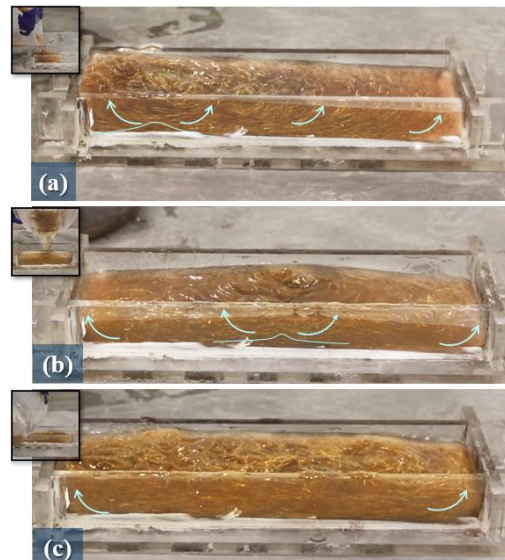


Figure 4.39: Different Casting Methods for FR specimens: (a) flow from one end, (b) flow from center, and (c) random flow

The effect of casting method is also explored in the CS prismatic molds which have higher aspect ratio of cross-sectional dimensions ($h/b=1$) compared to AFNOR formwork ($b/h=0.5$), but also reduced length. As illustrated in Figure 4.40(a), the eddies are still present for flow from one end. The boundary effect close to the casting point is slightly less obvious. This could be explained due to the increased height to width ratio (1 instead of 0.5). Flow from the center (Figure 4.40(b)) and random flow (Figure 4.40(c)) seem to be similar to the corresponding flows of AFNOR formworks. However, in all cases the reduced length hinders the longitudinal fiber alignment for all cases, resulting to a more 3-D orientation compared to AFNOR specimens. This observation is consistent with computational simulations from the literature but also fiber counting that was performed in short and long prisms in a previous section of this chapter.

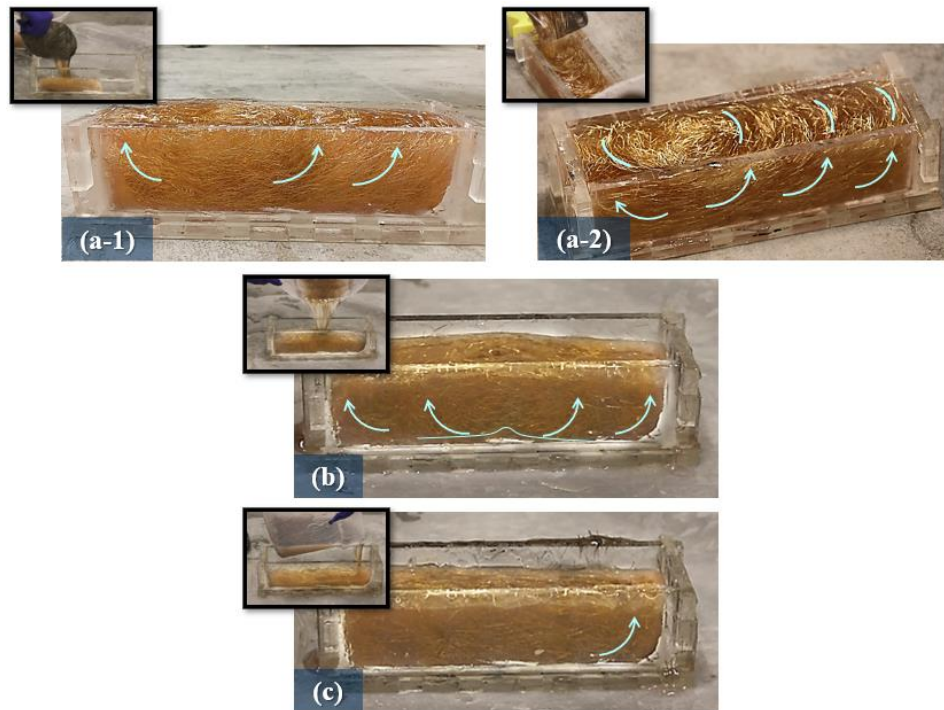


Figure 4.40: Different Casting Methods for CS specimens: (a) flow from one end (1: side view, 2: perspective view), (b) flow from center, and (c) random flow

Besides flexural prismatic molds, the influence of flow was also studied for tension specimens (Type GP). The flow from one end concentrates more fibers close to the point of casting and fewer towards the other end. This type of flow results in the formation of a weakness plane on the thin neck near the opposite end of the specimen (Figure 4.41(a)). This is consistent with experimental evidence that shows the tension crack in case of one-way castings to occur closer to the neck opposite to the point of casting. Moreover, fiber alignment is observed in the area of

interest in tension but closer to the casting point. On the contrary, in case of random casting, the distribution is more homogeneous, but fibers in the constant cross section are less aligned (Figure 4.41(b)).

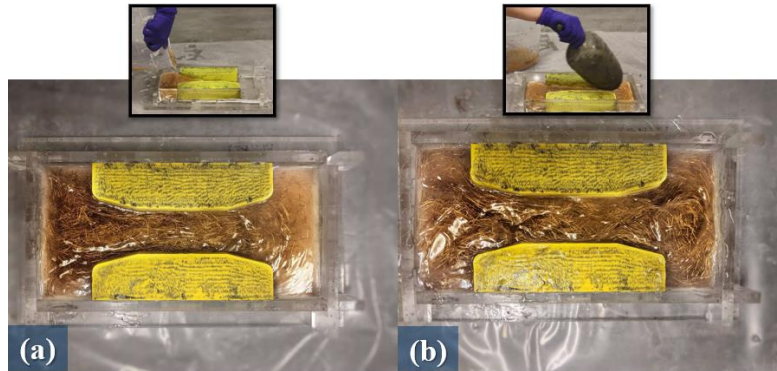


Figure 4.41: Different Casting Methods for GP specimens: (a) flow from one end (b) random flow

Finally, for MS specimen, only flow from one end was explored. As shown in Figure 4.42, a formation of a weakness plane with fewer fibers and not aligned is observed on the diametrically opposed end. This coincides with the location of crack in tested UHPFRC specimens of that formwork geometry. Besides the weak plane, there is a discontinuity of fiber flow close to the curved parts due to abrupt change of geometry.



Figure 4.42: Formation of weak plane in MS specimens cast with flow from one end

4.4.5. Effect of Rheology

Regarding the effect of rheology, higher concentration suspension was used to simulate less flowable mixes (0.3CM). As observed above, the high concentration of Carbomer leads to higher yield stresses and reduced spread flows (Table 4.7). In case of one way placement, because there is no flowability, the suspension cannot spread easily from the casting point towards the end of the formwork. For this reason, external vibration by lifting the mold from the ground and tapping it on the sides was used to help the spread. As it is apparent from Figure 4.43 which depicts the timeline of flow from one end for 0.3M, the reduced flowability does not yield eddies around the

casting point, but also no fiber alignment is evident before the external vibration. The yielded orientation is mostly a combination of 2-D and 3-D.

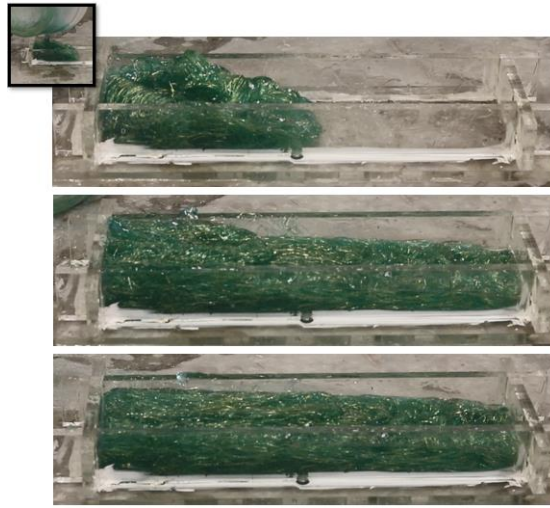


Figure 4.43: Flow timeline of flow from one end for 0.3CM1 in FR formwork

For the case of one-way casting, increasing the fiber content from 1 to 2%, the mix becomes stiffer and the same effects as in the case of lower volume fraction are observed (Figure 4.44). Due to poor workability of the suspension, the casting surface has to be scraped resulting in a different orientation on the top layer.



Figure 4.44: Flow timeline of flow from one end for 0.3CM2 in FR formwork

Random casting for 0.3CM1 is also explored in FR specimens. The casting procedure is rather challenging, and it results in an orientation that could be better characterized as 3-D with little fiber alignment due to wall effects (Figure 4.45).

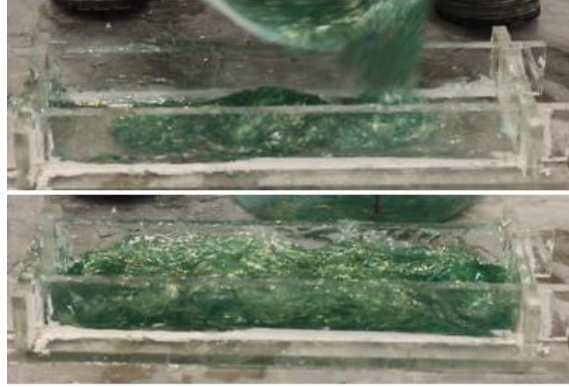


Figure 4.45: Flow timeline of random casting for 0.3CM1 in FR formwork

Finally, the 0.3CM2 suspension was also poured in CS molds one end casting in the C1 molds. Figure 4.46(a)-(c) shows the timeline of casting CS from one end. No disturbance around the casting point is observed as the mix is rather viscous and not flowable, while scraping was used at the end to achieve a smoother finishing (Figure 4.46(c)). In the case of the cylindrical mold, the vertical orientation seen in the more flowable mixes owing to the prevalence of the gravitational forces seems to have diminished here, whereas more fibers are aligned in the circumference (Figure 4.46(d)) – thus a more ductile behavior would be expected in the more viscous THFRC materials.



Figure 4.46: One way casting of 0.3CM2 (a-c) Flow timeline in CS formwork, (d) fiber distribution in C1

4.5. Conclusions

This chapter explored the fiber distribution and orientation in THFRC, along with influential factors and techniques for estimation and assessment. From these methods destructive/indirect

and non-destructive/direct approaches were utilized to explore experimentally the effects of a variety of factors impacting fiber positioning in UHPFRC.

The chosen destructive method is manual fiber counting on sawn UHPFRC surfaces combined with theoretical expressions regarding the number of fibers crossing a plane. The actual number of fibers on a series of cuts performed on flexural and tension specimens from the literature is compared with theoretical numbers from predictive mathematical models found in literature. Since there is no available model that predicts accurately the number of fiber crossing a plane, an orientation factor as function of the fiber length and specimen's height (i.e., the height of the cast member) is proposed for one way and random casting, through curve fitting of the manual counting on a variety of cross-sections.

Besides manual counting, transparent fluid model concrete was used as a nondestructive method to visualize the effects that various phenomena related to casting in the fresh state and have on the fiber distribution. The FRC in fresh state was described by the Bingham model which is the conceptual base for Carbopol suspensions. Through this model concrete, it is shown that:

- Highly flowable mixes in case of one-end casting create areas of turbulence near the casting point, while the wall effect is prominent especially in small sizes of formwork. Other than the point of turbulence, the fibers show better alignment in the specimen, except towards the ends where a re-orientation at 45 angle is formed due to wall effects.
- Flow from the center is also linked with the creation of a plane of weakness due to local disturbance around the casting point, but because the point is in the center, a more symmetrical distribution throughout the length of the specimen is observed; note that a wake region with thinner fiber concentration forms under the casting point; this, in a flexurally tested prism specimen would be the tension zone at midspan and would lead to significantly lower strength estimates.
- For random casting, despite the more homogenous distribution as there is not a single casting point which would suffer from turbulence, less fiber alignment is observed.
- Splitting planes can be created when one way casting is used in formwork that contains longitudinal reinforcement.
- Regarding casting of cylinders in one layer, fibers in small cylinders tend to align in the circumference while in case of larger cylinders, they align themselves parallel to the axis

of the cylinder due to gravitational forces. This difference might explain the higher compressive strength found in smaller cylinders.

- The fiber distribution in less flowable/thixotropic mixes is independent of the placement method. In this case, a more isotropic/3-D orientation is observed while fiber alignment can be achieved to a certain degree through external vibration.

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Chapter 5: Material Characterization of the Developed THFRGC

5.1. Introduction

In order to promote the use of geopolymer FRCs in low carbon structural applications, it is necessary to fully characterize the material in terms of mechanical, physical and durability properties. This chapter presents the results of an extensive material characterization campaign of the developed Tension Hardening Fiber Reinforced Geopolymer Concrete (THFRGC) following North American Standards (ASTM and CSA) for conventional Concrete. The mechanical behaviour was characterized in terms of Compressive, Tensile, and Flexural resistance, modulus of Elasticity and Poisson's ratio, while physical properties characterization tests include Density, Absorption, Drying Shrinkage, Abrasion, and the Coefficient of Thermal Expansion. Regarding durability performance, Chloride Ion Penetration resistance, Sulphate and Salt-Scaling resistances were determined.

5.2. Specimen Preparation-Mixing and Casting Procedure

For the material characterization of the developed optimized THFRGC three batches were prepared. Table 5.1 shows the details of the material characterization specimens. For Coefficient of Thermal Expansion and Salt Scaling tests, two additional 75 mm x 150 mm cylinders with embedded probe thermometers were also cast. As Figure 5.1 depicts, wood and plexiglass molds were prepared for direct tension and flexural specimens, while standard plastic molds were prepared for cylinder specimens for Batch 1. The modified splitting specimens comprise of 150 mm x 100 mm cylinder formwork with glued 3-D printed inserts. As discussed in Chapter 9 in further detail, the pertinently placed inclusions isolate a central chord which undergoes direct tension when the cylinder is subjected to splitting. Prior to casting, sunflower oil was used as a releasing agent to all specimens unless otherwise specified by a particular standard (i.e., CSA A23.2-11C). Due to the strong adhesion of geopolymer concrete with wood and metal surfaces observed in previous castings, the wooden formworks for direct tension are covered with duct tape and then sunflower oil is applied on top in order to facilitate the demolding.

Table 5.1: Material Characterization of geopolymer THFRC

Property	Test Method	# of Specimens	Fibers	Type of Specimen	Batch No.
Compressive Strength	(CSA-A23.2-9C, 2014)	6	Yes	75 mm x 150 mm cylinder	1
		3	No		3
Static Modulus of Elasticity & Poisson's Ratio	(ASTM C469, 2022; ASTM C1856, 2017)	6 [×]	Yes		1
Uniaxial Tensile Strength	(AASHTO T 397, 2022) [§]	3	Yes	25 mm x 50 mm x 430 mm prism	1
Splitting Tensile Strength	(ASTM C496, 2017)	3	Yes	100 mm x 100 mm cylinder	1
Indirect Tensile Strength (Modified Splitting)	-	3	Yes	150 mm x 100 mm modified cylinder	1
Flexural Strength	(ASTM C1609, 2019)	3	Yes	75 mm x 75 mm x 280 mm prism	1
Absorption	(CSA-A23.2-11C, 2014)	3	Yes	75 mm x 150 mm cylinder	2
Shrinkage	(CSA A23.2-21C, 2014)	4	Yes	25 mm x 25 mm x 285 mm prism	2
Coefficient of Thermal Expansion	-	3	Yes	75 mm x 150 mm cylinder	2
Abrasion	(ASTM C944, 2019)	2	Yes	100 mm x 100 mm cylinder	2
Sulphate Resistance	(ASTM C1012, 2018)	4	No	25 mm x 25 mm x 285 mm prism	3
Chloride Ion Penetration	(CSA-A23.2-23C, 2014)	2	No	100 mm x 200 mm cylinder	3
Freeze Thaw and Deicing Salt-scaling	(CSA A23.2-22C, 2014)	1	Yes	240mm x 200 mm x 75 mm slab	2



Figure 5.1: Formwork used for mechanical testing

[×] Measured Concurrent with compressive strength

[§] Modified

Prior to batching, the alkaline reagent PS-1.7 mixed with half of the water was stored in the fridge at a temperature of 2 °C for at least one day. The batching process comprised of dry mixing of the powdered precursors for 3-4 minutes, followed by the addition of the cold solution of alkaline reagent with water and further mixing at high speed for 7 minutes. Subsequently the sand was added to the system and the mixing continued for 3 minutes. After that the remaining amount of water was added to increase workability and further mixed for 2 minutes. When the mix gained the desired fluidity, fibers were added, and the mixing continued for 2 extra minutes. The mix design is the GPC4-F and GPC4-NF from Table 3.13 of Chapter 3 and is also presented in Table 5.2. The steel fibers used are S2B discussed in Table 4.3 of Chapter 4.

Table 5.2: Mix Design for Batches 1-3 (Table 3.13)

Materials	Proportions (kg/m³)	
	Batch 1, 2	Batch 3
MG	260	260
USF	230	230
GGBFS	180	180
MK-750	80	80
P	45	45
FA-F	40	40
PS-1.7	400	400
W	80	80
GS	980	980
Extra Water	22	22
Steel Fibers S2B	-	156
Fresh Density	2317	2463

For Batch 1 which is used for mechanical testing, the prisms for flexure, direct tension and modified splitting are filled with flow from one end as depicted in Figure 5.2, in order to achieve preferential fiber alignment which has proven to be significant for the mechanical properties. The excess material was used to fill 50 mm cube specimens which were tested in compression at different ages to evaluate the development of compressive strength due to maturity. After casting external vibration was applied by slightly hitting the sides with rubber mallets. Then the specimens were covered with plexiglass sheets and placed in big plastic bags to prevent evaporation. After 24 hr the specimens were demolded and cured till the age of testing according to the specifications of the prescribed standards for OPC concrete. For Batch 1 which was to be tested for mechanical properties moist curing was done, with the specimens wrapped in wet burlap sheets, placed in curing tanks which were sealed with plastic sheets to prevent water evaporation. This type of curing was selected to avoid the risk of potential drying shrinkage cracks.

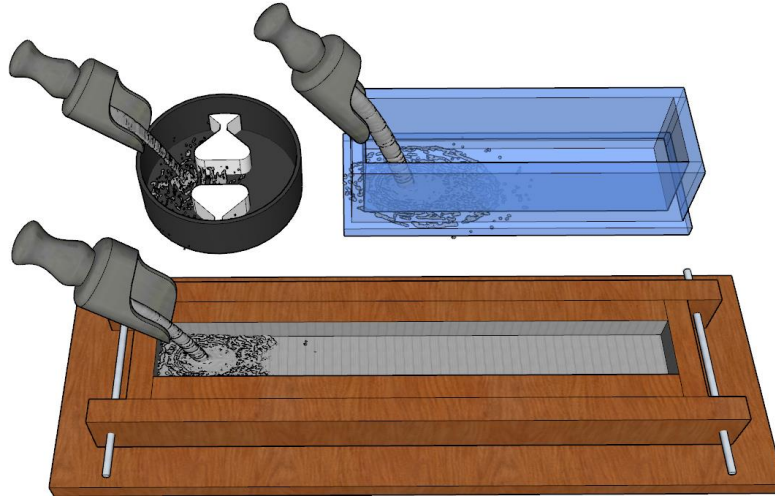


Figure 5.2: Schematic representation of flow from one end placement method

5.3. Fresh Properties

Right after mixing and before casting, the temperature of the mix was recorded using a probe thermometer and flowability test per ASTM C1856 (2017) was conducted to evaluate the fresh properties of the material. The conical mold of the flow table was filled with a single layer of fresh geopolymer concrete (Figure 5.3(a)). Subsequently, the mold was lifted and after 2 minutes, the static flow spread was measured as the diameter of the spread (average of two orthogonal diameters, Figure 5.3(b)). In addition, the dynamic spread was measured, and it corresponded to the diameter of the spread after lifting and dropping the table 20 times in 15 minutes (Figure 5.3(c)).



Figure 5.3: (a) Filling the mold with one layer of fresh geopolymer, (b) static spread after 2 minutes, and (c) dynamic spread after tapping

Besides temperature and flowability, the setting time of each batch was measured according to ASTM C191 (ASTM C191) with the Vicat needle apparatus. The method was described in detail

in Chapter 4 since a design parameter for the geopolymer matrix. The measurements of fresh properties and temperature for each batch are listed in Table 5.3.

Table 5.3: Summary of fresh properties of Batches 1-3

Properties	Batch 1	Batch 2	Batch 3
Temperature (°C)	21.8	22.1	20.5
Static Spread (mm)	134	140	126
Dynamic Spread (mm)	190	200	184
Initial Setting Time (min)	91	94	100
Final Setting Time (min)	>120	>120	>120

5.4. Mechanical Properties

The tests of mechanical properties were conducted at the age of 63 days. For the compressive response, 6 cylinders were tested at 196 days, namely unreinforced cylinders C1, C2, C3 and fiber reinforced cylinders F1, F4 and F5 as it is mentioned in the literature that the structure is more stable after 90 days for most geopolymers (Ranjbar & Zhang, 2020).

5.4.1. Compressive Response, Static Modulus of Elasticity and Poisson's Ratio

Nine cylinders with a size of 75 mm x 150 mm were cast for determination of the constitutive law in compression; six of these were fiber reinforced while the remaining three were used only for determination of compressive strength of the unreinforced matrix. All cylinders were cast in a one way pouring using a large scoop. Although the material is self-compacting, external consolidation was used by tapping the circumference with a tamping rod. Prior to testing, the cylinders were ground using a concrete grinder until the top and bottom surfaces were flat and smooth. Moreover, four different measurements of length and diameter were recorded to determine the average dimensions for each specimen.

- *Plain Geopolymer Concrete*

The unreinforced specimens were tested according to CSA-A23.2-9C (2014) under force control with a loading rate of 0.25 MPa/s. The compression test setup is presented in Figure 5.4 and conducted on a Controls Pilot testing machine. On account of the nature of loading type, post-peak response could not be recorded, as the failure was brittle. For this reason, the compressive strength of the geopolymer matrix was only recorded and the results are given in Table 5.4. Specimen C2 resulted in significantly higher strength than C1 and C3 and for this reason it is

considered an outlier. For this reason, the compressive strength of the unreinforced geopolymer concrete is taken as the average value of the lower strengths of C1 and C3, equal to 46 MPa.

Table 5.4: Compressive strength results for plain geopolymer concrete

Cylinder	1	2	3
Average Height h (mm)	150	142.8	150
Average Diameter D (mm)	76.3	76.4	76.6
Peak Load (kN)	216.3	295.7	205.7
Compressive Strength (MPa)	47.3	64.6	44.6
Average Compressive Strength (MPa)	52		
Standard Deviation (SD)	10.85		



Figure 5.4: Controls Compression Machine used for uniaxial compression of unreinforced geopolymer concrete cylinders and cubes

As illustrated in Figure 5.5, the cylinders collapse into fragments beyond attainment of the peak load. C1 and C2 specimens developed a Type 3 (CSA A23.2-9C, 2014) fracture where columnar vertical cracking goes through both ends and no well-formed cones are visible. These types of failure indicate normal or reduced friction between the loading plates and the specimens. Specimen C3 exhibited a Type 2 (CSA A23.2-9C, 2014) failure where only one end has a well-defined cone and there are vertical cracks funning through caps. Finally compared to previously developed mixes, the texture of the fractured core is more cohesive, less porosity is observed, and the white dust disappeared indicating that no reactive material was left unreacted.



Figure 5.5: Fracture pattern of failed unreinforced cylindrical specimens

- *Fiber Reinforced Geopolymer Concrete*

To obtain the response into the post-peak range, uniaxial compression tests were performed under displacement control using a closed-loop, servo hydraulic controlled testing machine at a loading rate as per CSA-A23.2-9C (2014). The axial deformations and strains were measured employing virtual extensometers over a gauge length of 100 mm through Digital Image Correlation (DIC) Analysis (Figure 5.6a). For some specimens, circumferential elongation was also measured utilizing a circumferential extensometer (chain), attached at the midheight. Finally, four strain gauges were mounted on diametrically opposed sides (a pair of a parallel and transverse to the loading direction on each side) for the calculation of Poisson's ratio as shown in Figure 5.6(b). The combination of the virtual extensometers, strain gauges and chain are important for the determination of the constitutive law in compression. While strain gauges are more sensitive than the chain and virtual extensometers and can detect accurately the strains at low loads, they lose their ability to measure strain after cracking.

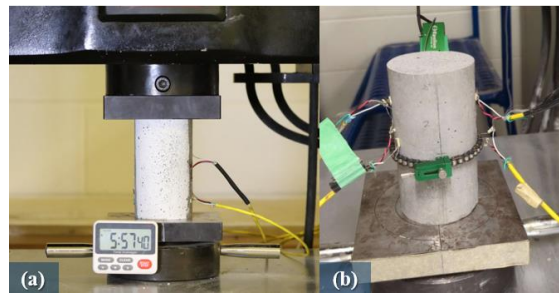


Figure 5.6: (a) painted face of cylinder for DIC analysis (b) hardware components: strain gauges and circumferential extensometer

Five of the specimens were subjected to monotonic uniaxial compression while one was subjected to cyclic compression. Figure 5.7 shows the monotonic stress-axial/lateral strain response for the tested cylinders, while Table 5.5 listed the characteristic values of the constitutive law. The

continuous curves correspond to cylinders tested at the age of 63 days, while the dashed curves correspond to cylinders tested at the age of 196 days. As mentioned before, a cyclic monotonic uniaxial compression test was performed on specimen F6 and is illustrated in Figure 5.8. The compressive stress of the geopolymer THFRG reached an average ultimate stress value of 47.7 MPa and 51.9 MPa for the age of 63 and 196 days, respectively. The axial deformation that corresponds to the compressive strength ϵ_{co} , is observed to be in all cases higher than 0.003, while the axial deformation corresponding to $0.5f_c$ post-peak is in the range of 0.016-0.034. On account of the lateral strains, large values are attained with a less steep descending branch, owing to the passive confinement provided by the fibers. The response of the material which is also marked by a prolonged descending branch for the axial deformation combined with the high deformations post-peak indicate the highly ductile nature of the developed geopolymer material.

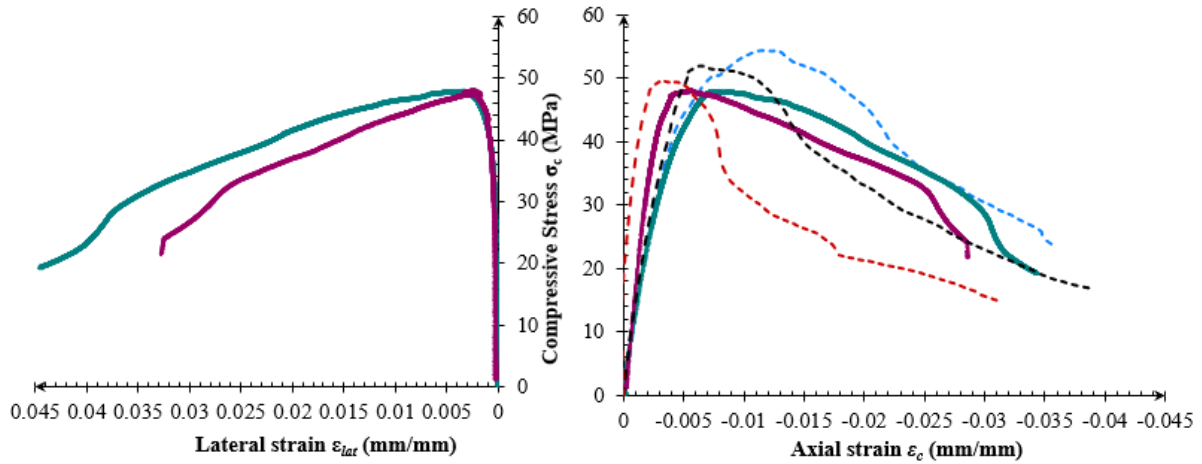


Figure 5.7: Stress-axial/lateral strain of THFRGC under monotonic uniaxial compression (repetition of two and three specimens; continuous lines: specimens of 63 days of age; dashed lines: specimens of 196 days of age)

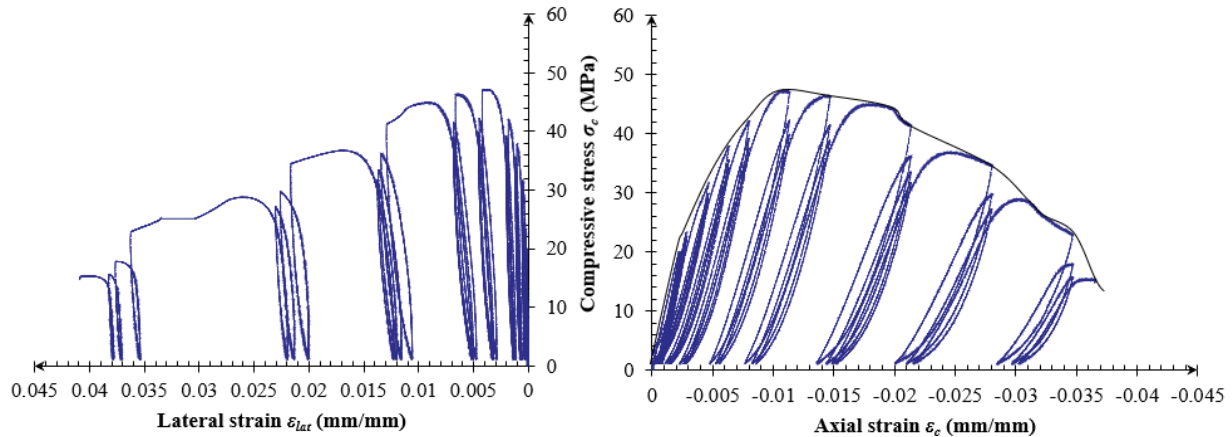


Figure 5.8: Stress-axial/lateral strain of THFRGC (#6 specimen) under cyclic uniaxial compression

Table 5.5: Material properties and characteristic points of stress-strain curves of THFRGC under uniaxial compression

Specimen	ν	E_c (MPa)	f_c (MPa)	$\epsilon_{c,o}$ (mm/mm)	$\epsilon_{c,50}$ (50% f_c) (mm/mm)
1	0.16	16589	51.9	0.00631	0.02673
2	0.17	16370	47.9	0.00732	0.0310
3	0.16	16693	48.0	0.00584	0.02864
4	0.16	16325	54.3	0.01234	0.03382
5	0.17	32698	49.5	0.00335	0.01671
6	0.16	15384	47.1	0.01092	0.03463
Average	0.16	19010	49.8	0.00768	0.02856
SD	0.00)	6721.8	2.79	0.00335	0.00654

Table 5.5 also presents the static modulus of Elasticity and Poisson's ratio obtained according to ASTM C469 (2022). Although the standard prescribes that the modulus of Elasticity should be measured at 40% of the maximum load, it was observed that the THFRGC lost its linearity earlier than 40% and thus 30% was used. Compared to an OPC fiber reinforced concrete of the same strength, the investigated material demonstrates lower modulus of Elasticity and Poisson's ratio. This is attributed to the different molecular structure of the material and the possibly shorter range of Van-der-Waals attractions in the molecular structure. The average elastic modulus is 16.27 GPa and the average Poisson's ratio is 0.163.

The fracture pattern of THFRGC cylinders under uniaxial compression differentiates the material from the plain geopolymers concrete. As illustrated in Figure 5.9, the cylinders hold their integrity beyond the peak load even after undergoing such high deformations. The cracks are parallel to the loading direction indicating the bridging effect of the fibers crossing the cracks. The fracture pattern resembles a failure Type 3.



Figure 5.9: Fracture mode of cylindrical geopolymers THFRG specimens

- *Effect of Maturity*

Besides the cylinders, 50 mm cubes were also tested at different ages in order to determine the cylinder to cube compressive strength ratio. The cylinders were tested in a Controls Compression Machine under force control with a rate of 0.259 MPa/s. Prior to testing, their dimensions and

mass were recorded to determine their density. The results of 24 (3 cubes at each age) cubes tested in compression along with their densities are presented in detail in the Appendix A while Figure 5.10 shows the development of cubic compressive strength and density due to maturity. Indeed, after 28 days the strength stabilizes, while after 60 days it seems that the material has reached a plateau. At this time the density has dropped by 4% while at 90 days it drops further by 5.3% and then at 200 days, the difference is less than 2% from the last measurement. The drop in the density is due to the water evaporation of the specimen. This observation is in accordance with the literature for geopolymyer concrete.

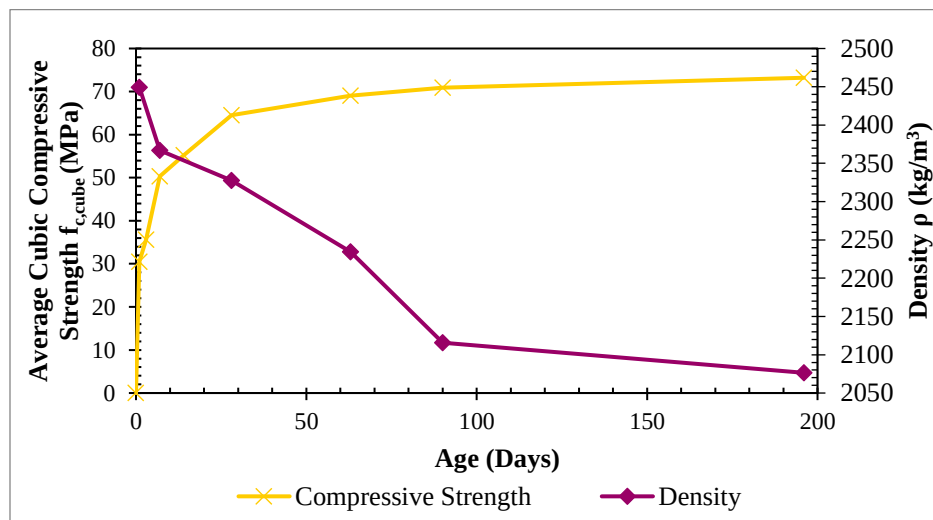


Figure 5.10: Compressive Strength and density development of THFRGC cubes over time(average of three specimens)

Furthermore, when comparing the results in the average cubic compressive strength to the cylindrical compressive strength at the corresponding age, a strong triaxial state is suggested for the cubic tests. More specifically, the cubic compressive strength to cylindrical compressive strength at 63 and 196 days of age is 1.41 and 1.45, respectively.



Figure 5.11: Fractured THFRGC cube after uniaxial compression test

- *Effect of curing method*

The effect of curing method on compressive strength was investigated by comparing three groups of specimens: 3 cubes with size 50 mm were submerged in water, 3 cubes were stored in plastic bags to prevent any evaporation and 3 cubes were covered with a wet burlap and a plastic sheet until their age of testing which was 40 days. The results are shown in Figure 5.12. The average compressive strength at 40 days for water curing is 64.3 MPa, for air curing 76 MPa and for moist curing it is 72 MPa. A 15% difference is observed between water curing and air curing. The lowest strength of water curing can be attributed to potential leaching.

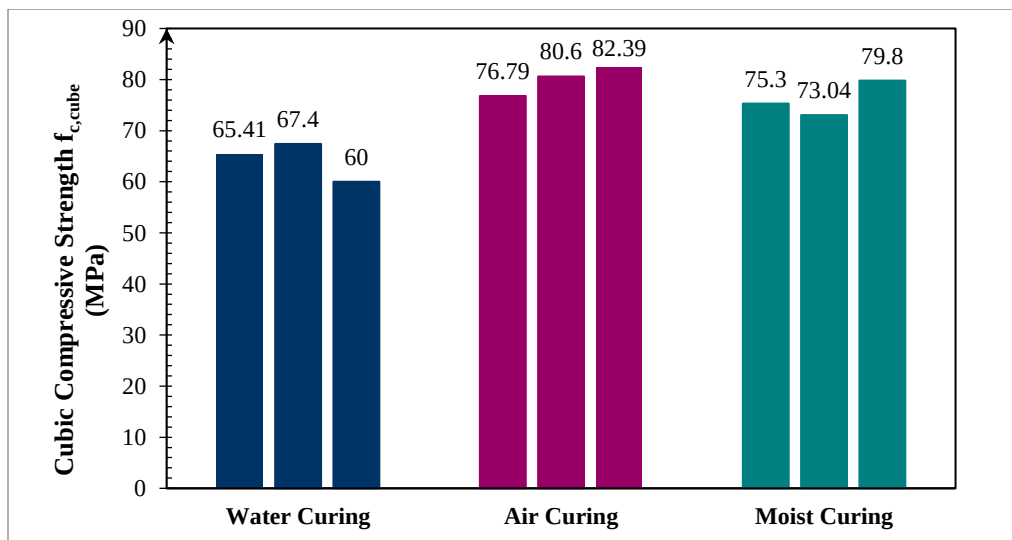


Figure 5.12: Effect of curing on the compressive strength at the age of 40 days (repetition of three specimens)

5.4.2. Flexural Response

The prisms for determination of flexural performance were tested at the age of 63 days. A stepwise loading rate in light of ASTM C1609 was adopted in conducting the flexural tests. More specifically there are two stages: a loading rate of 0.15 mm/min till the specimen reaches 70% post peak load and then 0.2 mm/min for the rest of the experiment until the post-peak residual strength has dropped to 20% of the peak load. Figure 5.13 illustrates the schematic of the experimental set-up. The distance between the loading rollers and the shear span is equal to the height of the specimen height, namely 75 mm. The loading rollers are free to rotate around their axis and have a diameter of 63.5 mm. This specific type of four-point bending is known as third point bending test. The supports which are placed in transverse C channels are rollers with a diameter of 30 mm and are free to roll along the span of the beam and rotate around their axis. A

rectangular frame consisting of aluminum pieces surrounding the prism is clamped at midheight directly over the supports. The horizontal beams of the frame are pinned at one end and have a roller connection on the other. On both of the horizontal parts of the instrumentation jig, at midspan and midheight, Linear Variable Differential Transformers (LVDTs) are mounted to measure the true deflection of the prism, which is the mid-height distance between the elastica from the chord defined between the support points. In this way, effects of seating and twisting of the specimens on the supports are minimized. The LVDTs are bearing on metal angles mounted on the midspan of the specimen on both sides with hot glue. The frames on which the horizontal beams of the jig are connected are clamped over the supports through threaded rods in order to prevent instability of the jig during the experiment.

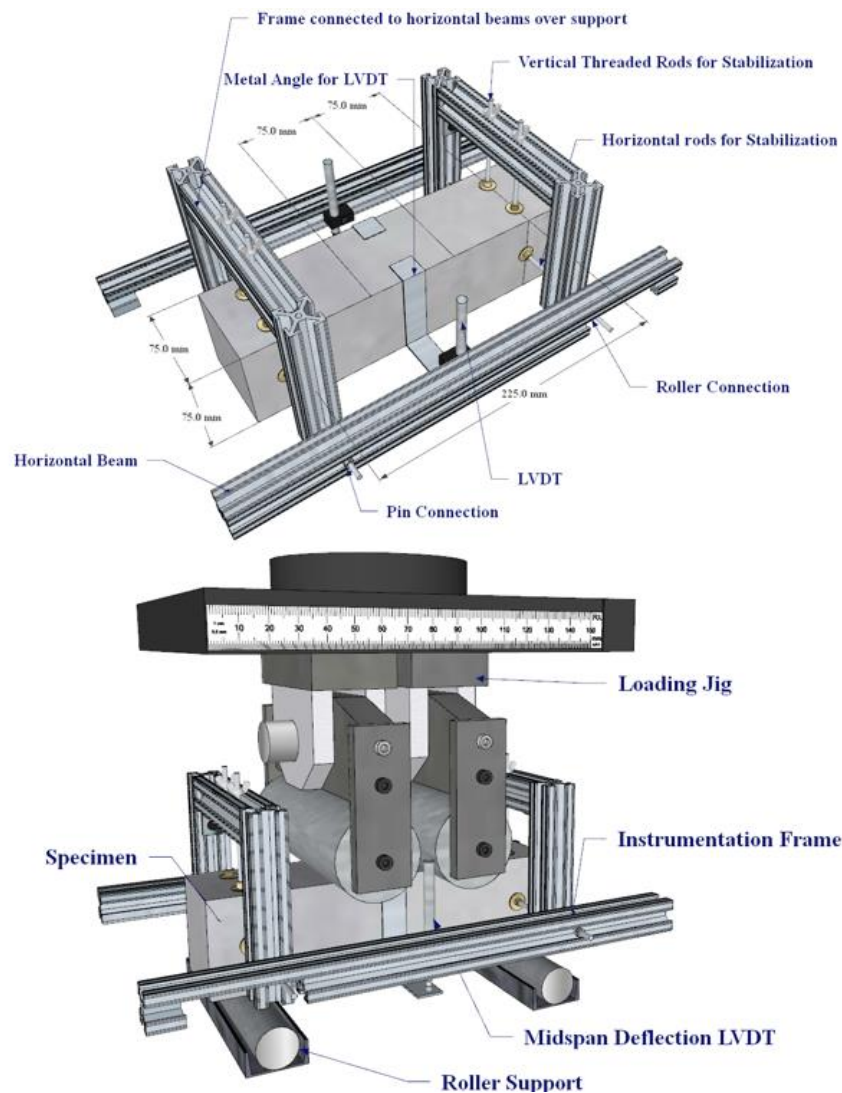


Figure 5.13: Schematic representation of the instrumentation jig and experimental set-up

Figure 5.14 illustrates the resistance curves of the tested specimens. The average maximum load is 20.7 kN which corresponds to a maximum bending stress of 10.6 MPa. The maximum bending stress σ_f is calculated using Equation (5.1) where P_{max} is the ultimate load, L is the span, b the width and h the height of the specimen.

$$\sigma_f = \frac{P_{max} L}{bh^2} \quad (5.1)$$

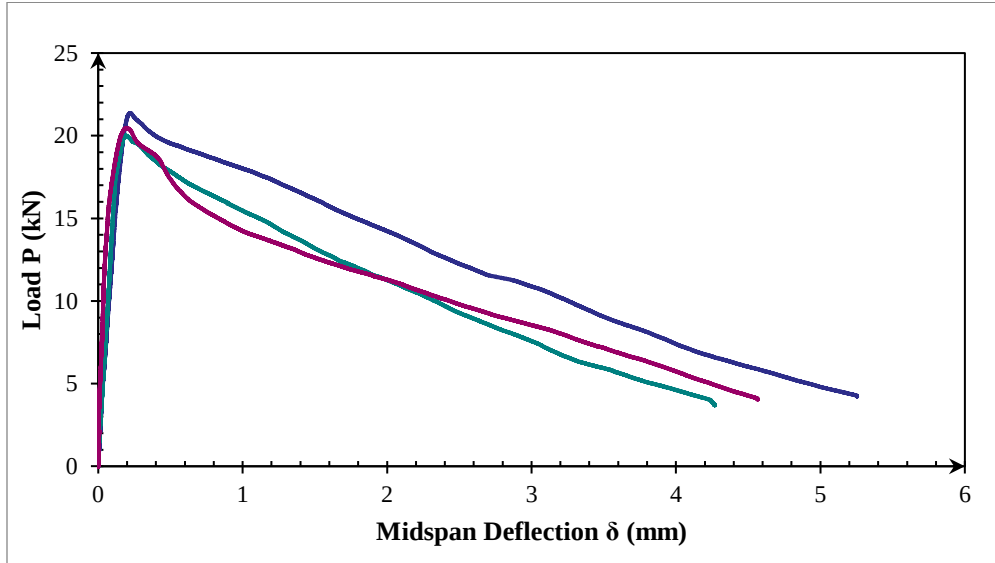


Figure 5.14: Resistance Curves of tested geopolymer THFRGC flexural prisms (repetition of three specimens)

The Modulus of Rupture f_r is marked by the loss of linearity in the ascending branch and can be related to the first cracking strength. It is observed to be 80% of the peak stress proving that the material is strain hardening. Table 5.6 summarizes the flexural performance properties of the geopolymer THFRGC. Besides the maximum bending stress and the modulus of rupture, the drifts at various loading stages are also measured to quantify the hardening behaviour of the material. Note that the drift β is defined as the ratio of the measured midspan deflection to the distance between the midspan and the support. As illustrated in Figure 5.15, this distance corresponds to 1.5α , where α is the shear span ratio. The average drift at 50% post-peak is 2.36% while at 20% residual strength it is 4.24%. This is an indicator of high ductility. Finally, the flexural properties according to ASTM C1609 are also presented in Table 5.7. First peak (cracking) strength is considered to be the modulus of rupture.

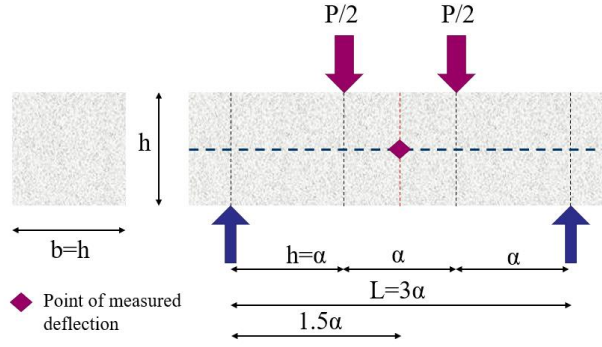


Figure 5.15: Third point loading illustration

Table 5.6: Summary of Experimental Flexural Performance Properties of THFRGC

Specimen	b (mm)	h (mm)	L (mm)	P_{cr} (kN)	P_{max} (kN)	$\sigma_{f,max}$ (MPa)	f_r (MPa)	$\beta_{.50}$ (50% $\sigma_{f,max}$) (%)	$\beta_{.20}$ (20% $\sigma_{f,max}$) (%)
1	75.7	77	231	17.5	20.01	10.47	9.16	1.97	3.68
2	77.8	76.9	231	16.75	21.37	10.73	8.41	3.04	5.03
3	76.4	75.8	228	15.83	20.48	10.64	8.23	2.06	4.00
Average						10.61	8.60	2.36	4.24
SD						0.13	0.50	0.59	0.70

Table 5.7: Flexural Properties of THFRGC according to ASTM C1609

Sample	1	2	3	Average	(SD)
First Peak Load P_1 (kN)	17.5	17	15.83	16.69	0.84
Peak Load P_p (kN)	20.01	21.37	20.48	20.62	0.69
First-peak deflection δ_1 (mm)	0.11	0.09	0.07	0.09	0.02
Peak Load deflection δ_p (mm)	0.19	0.17	0.19	0.18	0.01
Residual Load at $L/600$ P_{600}^D (kN)	18.53	19.98	18.93	19.15	0.75
Residual Load at $L/150$ P_{150}^D (kN)	13.05	16.47	12.58	14.03	2.12
First Peak Strength f_1 (MPa)	9.16	8.41	8.23	8.60	0.50
Peak Strength f_p (MPa)	10.47	10.73	10.64	10.61	0.13
Residual Strength at $L/600$ f_{600}^D (MPa)	9.70	10.03	9.84	9.86	0.17
Residual Strength at $L/150$ f_{150}^D (MPa)	6.83	8.27	6.54	7.21	0.93
Toughness T_{150}^D (J)	24.77	28.77	24.41	25.98	2.42
Equivalent Flexural Strength Ratio $R_{D,150}^T$ (%)	9.4	11.2	10.1	10.2	0.009

To obtain a better insight regarding the response of the material, namely if the formed cracks are purely flexural, or include shear and torsion contributions, the failure mode of the specimens was also studied, by examining the location and the path of the crack. In all cases the crack was vertical and propagated perpendicular in the region of the constant moment area or under one of the loading rollers as illustrated in Figure 5.16(a). For specimens #1 and #3 the flexural crack initiated at the cross section that was under one loading roller. This unsymmetric crack pattern is attributed to unsymmetric fiber distribution in case of flow from one end due to the disturbance in flow around the casting point as was demonstrated in Chapter 4. In case of OW3 some

microcracks were formed close to the main crack and inside the constant moment region (Figure 5.16(b)), while the average crack width at a residual load of 20% of the peak, was 6.5 mm which is half the fiber length (Figure 5.16(c)).



Figure 5.16: (a) Fractured specimens (b) multi-cracking in the constant moment region for specimen #3 and (c) measured crack width

5.4.3. Tensile Response

As there is no universally established test for tensile response of THFRGC in the available standards, for the determination of tensile strength and strain capacity different testing procedures were followed. These include direct tension tests on prisms, indirect splitting test on cylinders and a modified splitting test which was developed and proposed as an alternative in the course of the present work; design of this testing alternative and verification thereof are presented in Chapter 9.

- *Direct Tension Test (DTT)*

Three prisms with dimensions 25.4 mm x 50 mm x 430 mm were tested at a mature age (63 days) under direct tension by means of a servo-controlled, closed loop Universal Testing Machine (UTM) at a loading rate of 0.15 mm/min (Husain, 2021; Yang, 2019). As illustrated in Figure 5.17, in order to prevent premature failure near the gripped surfaces and promote the formation of the critical cracking plane within the gauge length, Carbon Fiber Reinforced Polymer (CFRP) sheets (SikaWrap Hex-230c supplied by Sika-Canada) were mounted on the gripping surfaces through Sikadur-330 epoxy laminating resin which is a thixotropic two-component epoxy resin. The carbon fiber properties are listed in Table 5.8. Due to the reduced thickness of the specimen,

hardware instrumentation was challenging to be installed and for this reason Virtual Extensometers (VE) from DIC analysis were employed.

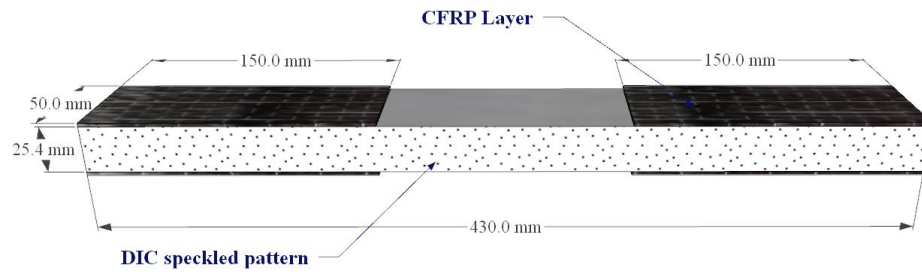


Figure 5.17: Nominal dimensions of DT specimens and specimen preparation prior to testing

Table 5.8: SikaWrap Hex-230c material properties

Carbon Fiber Properties	
Tensile Strength (GPa)	5.45
Tensile Young's Modulus (GPa)	230
Elongation (%)	1.5
Density (gr/cm³)	1.8
Area weight (gr/m²)	228
Nominal fiber thickness (mm)	0.13

Despite that AASHTO T39-22 prescribes the specimens to have a cross-section of 50 mm x 50 mm, the height of the specimen is reduced to 25.4 mm in order to be able for the specimen to fit in the available UTM. This is because the maximum allowable spacing between the hydraulic grips is 30 mm and a few millimeters clearance is required to facilitate the external CFRP jacketing of the surfaces that are gripped. Note that according to the standard (AASHTO T 397, 2022) instead of CFRP layers, aluminum transfer plates should be glued to the parallel faces at each end of the specimen. In this context, the CFRP layers at the ends have the same function as the aluminum plates.

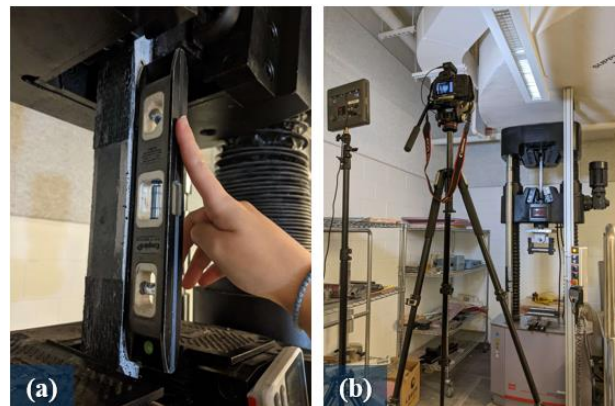


Figure 5.18: (a) Check for vertical alignment of the specimen, (b) experimental setup.

Before the specimens were placed in UTM, measurements of height and width were taken within the gauge length using a digital caliper. To avoid eccentricities and out of plane bending, a magnetic level was used along the axis of the specimen to ensure symmetry and perfect alignment with the grips (Figure 5.18(a)). The experimental setup (Figure 5.18(b)) also includes a standing light, a clock and a camera facing the speckled surface of the specimen and taking photos every 10 sec to capture the deformation of the specimen over time. In DIC analysis the average of three VE with gauge length of 100 mm was used to establish the axial deformation and thus the axial strain in the region of interest. The positioning of the VE adopted in DIC analysis is shown in Figure 5.19.

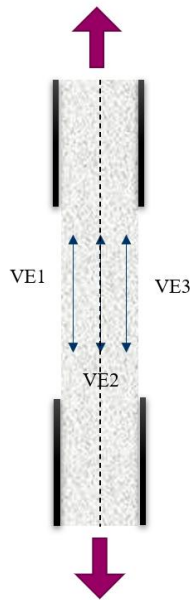


Figure 5.19: Virtual Extensometers from DIC.

Figure 5.20 shows the tensile stress σ_t versus the corresponding axial strain of the tested specimens. A zoom in at the ascending branch of the response is shown to highlight the tension hardening behaviour. All specimens lost their linearity before reaching the ultimate load, indicating first apparent cracking at an average strain of 0.00093 which was also observed in the DIC analysis. Table 5.9 summarizes the tensile properties of the geopolymer THFRGC as derived from the DTT. The average cracking strength is 6.79 MPa while the ultimate tensile strength is 7.7 MPa. This means that the hardening ratio is 1.13 which along with a strain of 0.002 corresponding to ultimate strength satisfy the requirements for classification as THFRGCs imposed by CSA Annex S6.

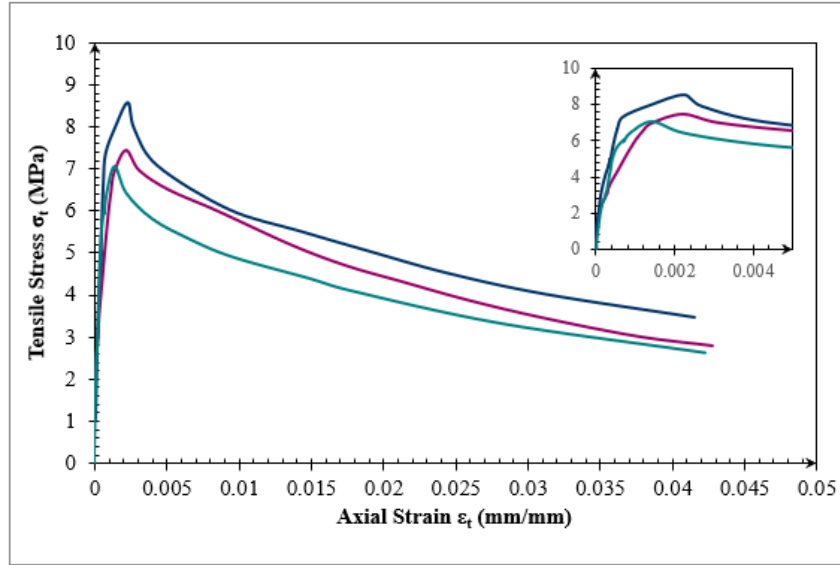


Figure 5.20: Tensile stress-axial strain of THFRGC under direct tension (repetition of three specimens)

Table 5.9: Tensile properties of geopolymer THFRGC

Specimen	f_{cr} (MPa)	f_{tu} (MPa)	ϵ_{cr} (mm/mm)	ϵ_{tu} (mm/mm)	E_t (GPa)
1	6.02	7.07	0.0007	0.0015	21.74
2	7.42	8.62	0.0007	0.0023	25.55
3	6.44	7.45	0.0014	0.0022	15.80
Average	6.62	7.7	0.00093	0.002	22.74
(SD)	0.7	0.8	0.0004	0.000436	4.91

Regarding the failure mode, all three specimens developed failure planes inside the gauge length, while upon localization of the crack and the non-symmetry created, some slight bending is also observed. The latter is inevitable as stress concentrations take place with the initiation of the crack from one side. The failure planes of the tested direct tension specimens are illustrated in Figure 5.21.



Figure 5.21: Failure planes of THFRGC direct-tension specimens

To extract the axial deformation of the specimens, DIC Analysis was performed. For every specimen 10% of the total pictures taken were used to minimize the computational time. The pictures selected corresponded to specific points of the response curve in order to capture the complete behaviour (Figure 5.22). Out of those photos, 6 are used to show the surface principal strains. P4 is the ultimate load corresponding to the ultimate tensile strength. P3 is the load corresponding to the first cracking tensile strength. P2 and P5 correspond to 0.8 and 0.7 of P4, respectively, while P1 and P6 represent 0.4 of the maximum load. P1-P3 are in the ascending branch of the resistance curve, while P5 and P6 are post-peak. Figure 5.23 illustrates the maximum principal strain at different stress states for each specimen.

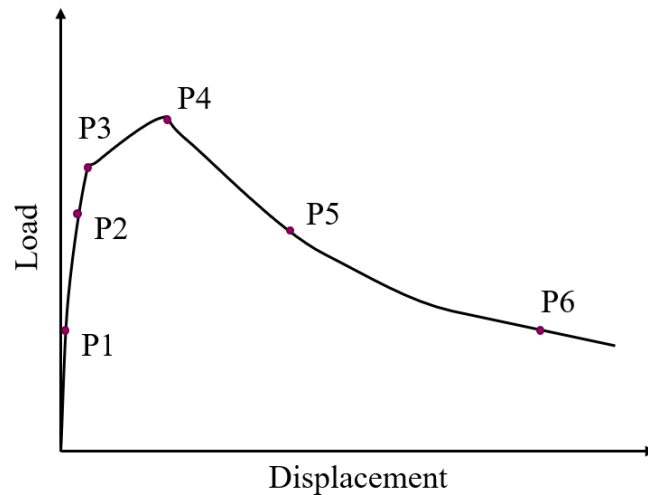


Figure 5.22: Selection of characteristic points of direct tension resistance curve for DIC strains

At early stages in the elastic region (P1 and P2) there is no clear stress concentration as the specimen is still settling in the setup – this is true for specimens G1 and G2 whereas for G3 there is concentration of stresses around the location of the crack that will form later. At P3 for all specimens the crack initiation is obvious as a distinct region with orange-red colour while the rest of the specimen had significantly lower strains as indicated by the green and blue colour. The local strains at that stage and regions are in the range of 0.002-0.005 indicating cracking. For P4 which corresponds to the maximum load and thus ultimate strength, localization of cracking is manifested by the contrast of small regions with significantly high strains with the rest of the specimen unloading. The multiple regions with red colour isolated from the rest specimen indicate multitasking. However, as it seen in P5 and P6 one of these cracks prevails as the weakest region and keeps spreading (localization of damage), while the rest of cracks are slowly closing.

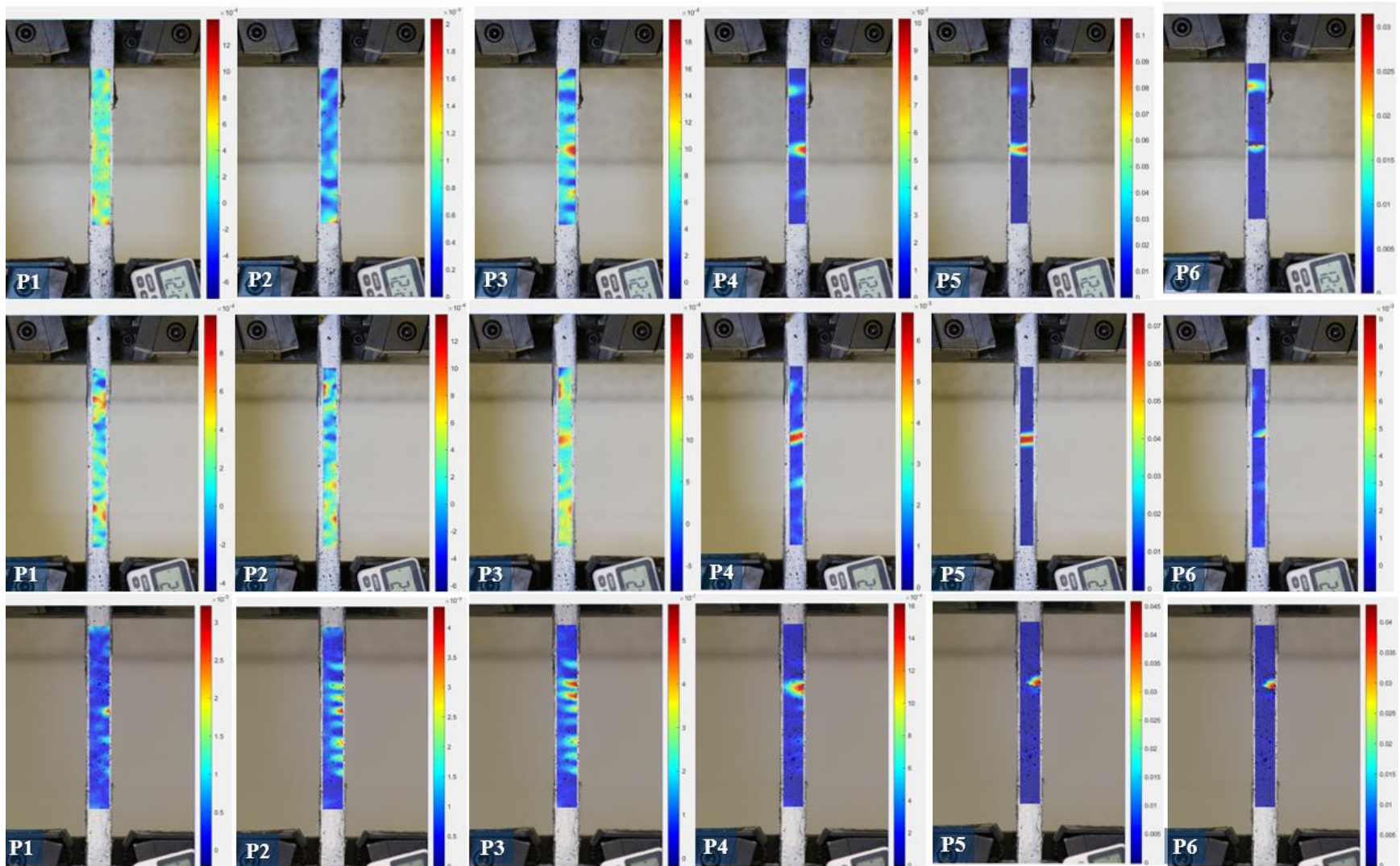


Figure 5.23: Maximum Principal strains for characteristics points, Specimen 1 (top), Specimen 2 (middle) and Specimen 3 (bottom) specimens

- *Splitting Test (ST)*

Since direct tension tests are generally difficult to conduct and most concrete testing labs do not have the suitable equipment and experimental setups to perform such tests, splitting test has been adopted as an alternative for testing FRCs in tension. In this test, a cylindrical specimen is subjected to compressive load, along two diametrically opposed generating lines of the cylinder. Uniform transverse tensile stress state is generated almost lengthwise the diameter plane that is parallel to the applied loads. Failure occurs once the tensile strength of the material is reached. Despite the simplicity of the experimental setup and procedure, uncertainty arises regarding the actual stress distribution particularly after cracking in tension hardening materials. because the mode of failure changes to compression in a direction parallel to vertical crack, leading to a compressive response rather than tensile with an unreasonably high strength increase post-cracking. Due to the above reasons, this test has been abandoned for strain hardening materials (Husain, 2021). However, for sake of completeness and to further prove the above observations, 3 cylinders with dimensions 100 mm x 100 mm were subjected to splitting. According to ASTM C496, the specimen should be placed in an instrumentation jig (Controls Group, Fig. 5.24(a)) that transfers the compression load along the diametrically opposed line of the cylinder. To record the horizontal displacement induced by the splitting, an LVDT is placed transversely to the application of the load on the centre of the back face of the cylinder from one side as illustrated in Figure 5.24(b). Besides recording of the lateral expansion of the specimen, DIC Analysis was also implemented on the front face of the cylinders. However, due to the excessive deformation and cracking the results could not be used quantitatively. To ensure alignment a magnetic level was placed on the top of the specimen (Figure 5.24(c)). The loading rate was selected to be 0.00254 mm/s.

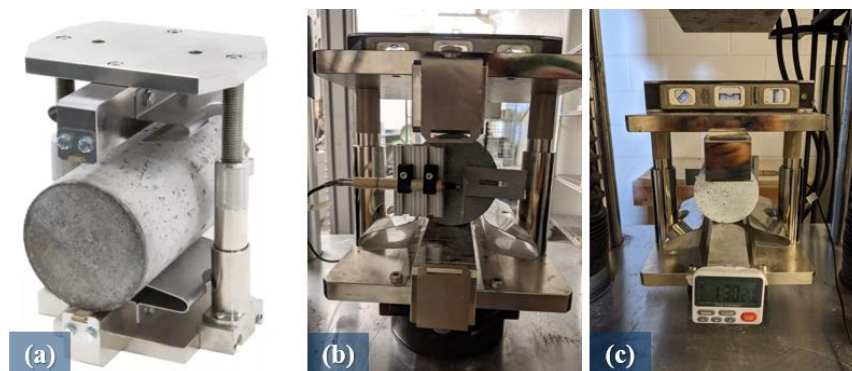


Figure 5.24: (a) *Splitting Tensile Test Device (Controls Group)* (b) *instrumentation* (c) *leveling of test device*

The cracking splitting stress, σ_{sp} , is calculated by means of the Load P , the diameter of the cylinder D and its length L , using Equation (5.2):

$$\sigma_{sp} = \frac{2P}{\pi DL} \quad (5.2)$$

Figure 5.25 illustrates the splitting load versus the lateral deformation recorded by the LVDT during the splitting test of specimens S1, S2 and S3 while Table 5.9 summarize the splitting strengths of the specimens at cracking and ultimate. The dispersion in the values of the load is partially owing to the different lengths of the tested specimens as $L_1=97.51$ mm, $L_2=94.75$ mm and $L_3= 73.25$ mm. However, the main reason for the dispersion is the unknown fiber distribution. More specifically as seen in Chapter 5, in cylinders of 100 mm diameter, often gravity effects are more prominent leading to a fiber positioning parallel to the length of the cylinder. This effect leaves the splitting plane without active fibers leading to lower ultimate strength. Meanwhile the loss of linearity in the graph corresponds to cracking which is strongly dependent on the matrix and is consistent in three specimens. The dispersion of ultimate splitting is evident from Table 5.10 which also lists the number of fibers crossing transversely the plane and splitting planes in fractured specimens in Figure 5.26. Finally, the latter illustrates the excessive deformation sustained in the specimens by fibers due to the formation of vertical compressive struts and the crushing of the diametrically opposed loading lines.

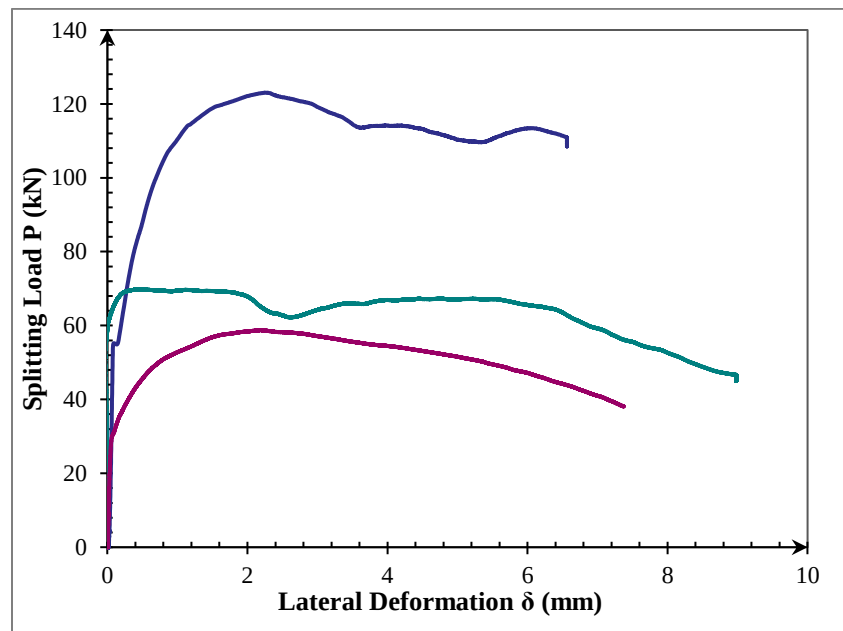


Figure 5.25: Splitting load versus lateral deformation measured by LVDT for splitting THFRGC specimens (repetition of three specimens)

Table 5.10: Spitting Tensile Results

Specimen	D (mm)	L (mm)	$P_{sp,cr}$ (kN)	$\sigma_{sp,cr}$ (MPa)	P_{max} (kN)	$\sigma_{sp,max}$ (MPa)	No. Fibers
1	101.1	97.51	55	3.6	124	8.0	39
2	100.4	94.75	31	2.1	70	4.7	19
3	99.85	73.25	27.5	2.4	58.7	5.1	23

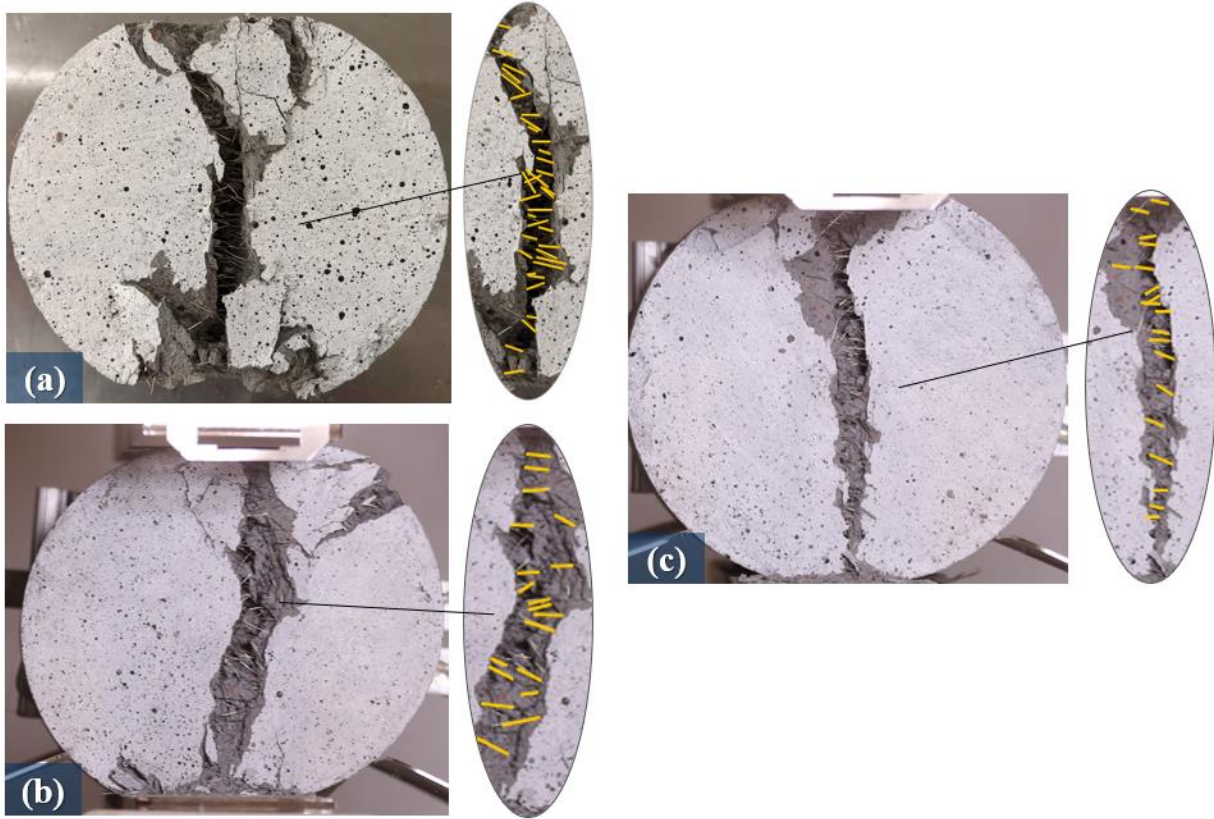


Figure 5.26: Failed splitting specimens and manual counting of fibers crossing transversely the splitting plane

- *Modified Splitting Test (MST)*

To tackle the challenges and limitations of the above tensile testing procedures, the Modified Splitting Test (MST) which was developed, designed, and implemented in this thesis is also conducted for comparison with the aforementioned tensile tests. The test comprises of a 50 mm thick cylindrical specimen with recesses placed in such positions so as to isolate a central chord that under splitting will undergo direct tension. The splitting load is transferred to the specimen through rollers which are placed on the top and bottom inclusions as illustrated in Figure 5.27. Due to the 45° angle that is created in the strut and tie, the tensile force F exerted from the central chord is equal to the applied load P . For further details on the design, formwork production and investigation of the test are discussed in Chapter 9 which is entirely dedicated to MST.

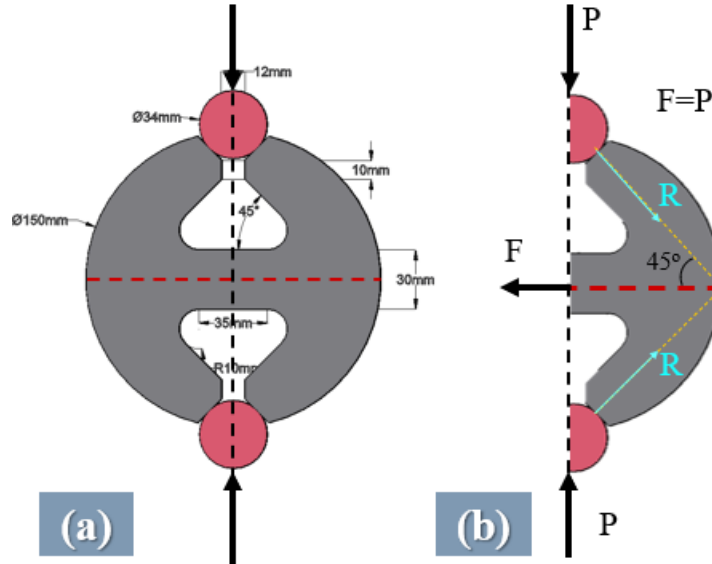


Figure 5.27: Mechanism of the proposed MST

To explore the proposed test, three specimens were tested. Prior to testing, the front face of the specimen was painted white with a black speckle pattern for DIC Analysis, while on the back face a transverse LVDT was attached bearing on a metal angle. Figure 5.28 shows a schematic representation of the set-up.

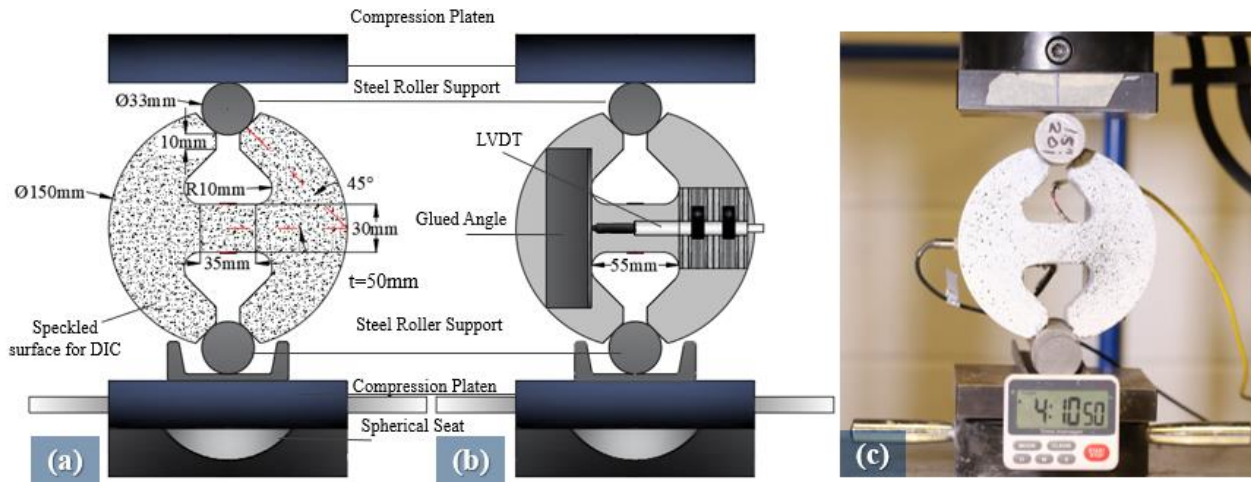


Figure 5.28: Schematic representation of MST setup (a) front, (b) back and (c) experimental setup

The response curves of M1 and M2 are shown in Figure 5.29 (M3 not shown because it experienced premature failure). The summary of the results is presented in Table 5.11. Note that the cracking strength is close to the cracking strength obtained from DTT, indicating the accuracy of the test; the response curve is closer to that obtained from the DTT and avoids the spuriousness of the ultra-ductile post-cracking behavior of the ST. However, the ultimate strength from this test

is higher than the corresponding ultimate from DTT. This is attributed to the development of friction as the roller moves – further details are provided in Chapter 9.

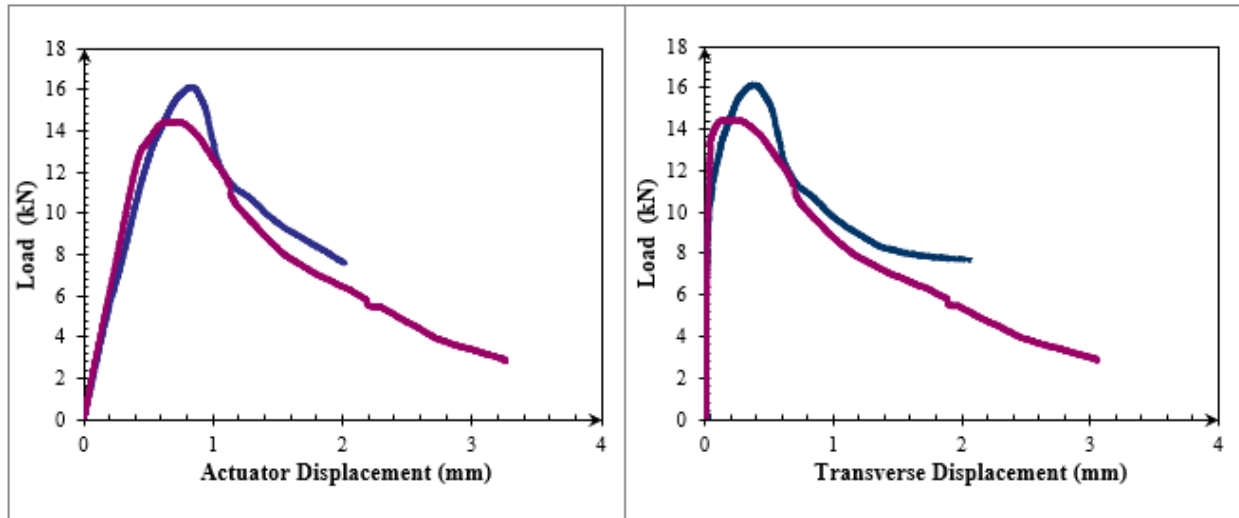


Figure 5.29: Response curves of MST specimens (repetition of two specimens)

Table 5.11: Summary of modified splitting test results

Specimen	b (mm)	t (mm)	P_{cr} (kN)	f_{cr} (MPa)	P_{max} (kN)	f_u (MPa)
1	30	51	10	6.53	16.14	10.54
2	30	49	10.05	6.83	14.43	9.81

Finally, as illustrated in Figure 5.30, the failure plane is within the central chord as designed but closer to the edges. This was anticipated already from Chapter 4 where a disruption of fiber arrangement was observed when casting from one end.

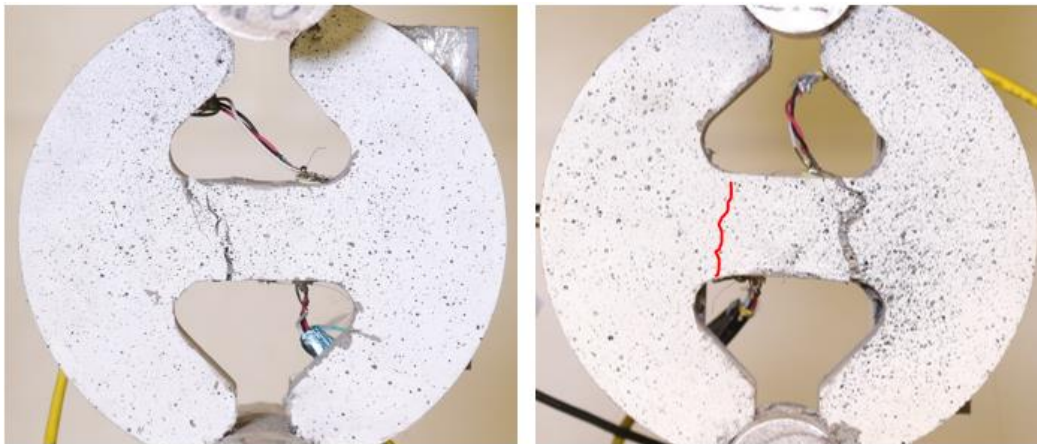


Figure 5.30: Failure pattern on MST specimens

5.5. *Physical Properties*

The experimental investigation of physical properties of the developed THFRGC considered length, mass, and density changes due to exposure to natural phenomena. These include absorption, air dry density, shrinkage, coefficient of thermal expansion and resistance to mechanical abrasion.

5.5.1. Water Content, Density, Absorption and Void Content

Three cylindrical specimens with dimensions 75 mm x 150 mm were tested according to CSA-A 23.2-11C (2014) for determination of water content, density, absorption, and void content. As discussed above the cylinder molds were filled with a single layer of fresh THFRGC and were free of oiling agent as per ASTM C1856 (2017). The mass of the specimens was measured under different conditions which are explained below. For quick reference, the results of designated mass properties are summarized in Table 5.12.

- *Mass after demolding*

One day after casting, the cylinders were demolded using air pressure and were immediately weighed using a digital scale with an accuracy of 0.01 g. The corresponding mass is designated as *I*. Subsequently, the specimens were kept in a fridge with no power which functioned as an insulated chamber. As per the standard the specimens should be cured in an environment with Relative Humidity (RH) $50 \pm 5 \%$ and $23 \pm 2^\circ\text{C}$ room temperature. For this reason, in this insulated chamber, a digital thermometer and an automatic humidifier were placed in order to record the temperature and regulate the humidity, respectively.

- *Mass after controlled air-curing*

After every week of curing the cylinders, they were weighted and then returned back to the insulating chamber till the difference in mass between two consecutive measurements was less than 0.5%. This difference was used as an indicator of mass equilibrium. Mass equilibrium was observed at 35 days and the mass measured at that age is designated as *G*.

- *Mass after oven drying*

Next, the cylinders were put in an oven at 110°C for three days for any physically bounded water left in their microstructure to evaporate and then returned to the chamber for 24 hr in order to cool

down to room temperature. The next day, before and after the new oven dry cycle, the masses of the specimens were recorded, and since their difference was less than 0.5 %, the latest recorded values were designated as mass *A*.

- *Mass after immersion in water*

Following, in order to determine the absorption of the developed material, the cylinders were submerged in a water tank in ambient laboratory temperature for 24 hr., then wiped to remove any water on the surface and weighted. Then, the specimens were returned to the water tank for another cycle and measured again at the end of each cycle till less than 0.5 % difference is observed between two consecutive measurements. The last mass measurement of the wet cycle corresponded to mass *B*.

- *Mass after boiling and surface drying*

For this step, the cylinders were positioned in a container filled with tap water and the latter was placed in an oven at 110 °C for 5 hr. Right after, the specimens were returned in the insulating chamber to cool down at room temperature. The procedure described above corresponds to one cycle of boiling. Between cycles, the mass of the cylinders was measured till two consecutive measurements had less than 0.5 % difference, and that value was designated as mass *C*.

- *Mass after suspension in water*

Finally, the specimens were suspended in water by a wire, while the mass of the cylinders in water was measured with a hanging scale. This measured mass corresponded to mass *D*.

Table 5.12: Designated measured masses of absorption specimens

Specimen	Mass <i>I</i> (g)	Mass <i>G</i> (g)	Mass <i>A</i> (g)	Mass <i>B</i> (g)	Mass <i>C</i> (g)	Mass <i>D</i> (g)
1	1598.38	1466.78	1420.12	1547.38	1553.82	802.8
2	1643.18	1508.44	1460.27	1589.69	1595.24	806.7
3	1615.14	1485.43	1433.70	1558.75	1566.12	802.0

Using the measured masses from Table 5.12, the water content, density absorption and void content can be obtained (Table 5.13). The average water content is 12.6%, absorption after immersion is 8.9%, absorption after immersion and boiling is 9.3%, air-dry density is 1940 kg/ m³, oven dry density is 1870 kg/ m³, density after immersion and boiling is 2050 kg/ m³, and void content is 17.41%. These properties seem to be consistent with the literature, as geopolymers are

known for their hydrophilic character and microporous structure which results in a lower density compared to OPC concrete (Davidovits, 2020).

Table 5.13: Results of water content, density, absorption, and void content for THFRGC.

Property	1	2	3	Average	SD
Water Content = (I-A)/A (%)	12.55	12.53	12.66	12.6	0.07
Absorption after immersion = (B-A)/A (%)	8.96	8.86	8.72	8.9	0.12
Absorption after immersion and Boiling = (C-A)/A (%)	9.41	9.24	9.23	9.30	0.10
Air-dry density = G/(C-D) (kg/m³)	1953.1	1913.05	1944.03	1940	2
Oven-dry density = A/(C-D) (kg/m³)	1890.97	1851.96	1876.33	1870	19
Density after immersion = B/(C-D) (kg/m³)	2060.43	2016.09	2039.98	2040	22
Density after immersion and boiling = C/(C-D) (kg/m³)	2068.97	2023.08	2049.6	2050	23
Void Content = (C-A)/(C-D) (%)	17.80	17.11	17.33	17.4	0.35

5.5.2. Shrinkage

FRC materials are usually mortars and due to the absence of coarse aggregates demonstrate higher shrinkage than conventional concretes. There are three types of shrinkage: chemical, autogenous, and drying. FRC materials often experience the last two. Autogenous shrinkage occurs due to the reduction of volume as the hydration of the cementitious materials evolves, while drying is caused by the loss of water from the material (FHWA, 2013). For determination of linear shrinkage, four prisms with dimensions of 25 mm x 25 mm x 285 mm were cast and tested according to CSA A23.2-21C. Linear shrinkage is related to the length change under controlled conditions of temperature and humidity. The formworks in which the specimens were cast were first treated with a release agent and then the stainless-steel gauge studs were installed. After casting and finishing, the specimens were covered with plexiglass sheets to prevent drying. After 24 hr, the specimens were demolded, then submerged in saturated lime water and left to cure for six days at room temperature of 23 ± 2 °C. On the seventh day, the first measurement was taken using the digital length comparator illustrated in Figure 5.31. First a steel bar was placed for reference and the digital transformer was set to zero. Then the specimen was placed in the setup ensuring the gauge studs on both ends fit properly. The specimen was then spined and the value at the end of the spinning is recorded. This was repeated two more times for extra accuracy. The above measurement is designated as base length measurement and corresponds to 0 days of drying age. Subsequently the prisms were stored in the insulating chamber. Humidity was controlled at 50%, while the door of the chamber was propped enough to allow for air circulation. At the age of 7, 28

and 62 days the specimens' length along with the reference bar were recorded following the procedure described for the measurement of the base length.

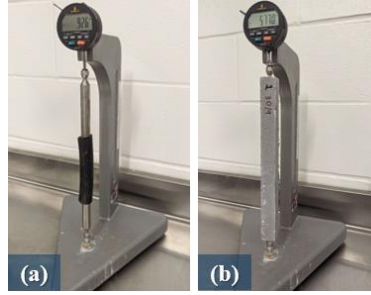


Figure 5.31: Length Comparator device: (a) measurement of reference bar's length, (b) measurement of specimen length

The length change L (%) was calculated according to Equation (5.3), where L_{comp} is the comparator reading of the specimen at the age of interest, L_{base} the base length measurement and GL is the gauge length which is 285 mm.

$$L = \frac{L_{comp} - L_{base}}{GL} \times 100 \quad (5.3)$$

In Figure 5.32 the change in length over time for the four specimens and their average is illustrated. At 28 days of drying age the average shrinkage is 0.22% while at 217 days is 0.253 %. Shrinkage is significantly higher in geopolymer mortars on account of the high capillary pressure generated between wet and dry zones of the micropore network resulting in internal cracking initiation (Ranjbar & Zhang, 2020). This high shrinkage is evident from surface microcracks as illustrated in Figure 5.33.

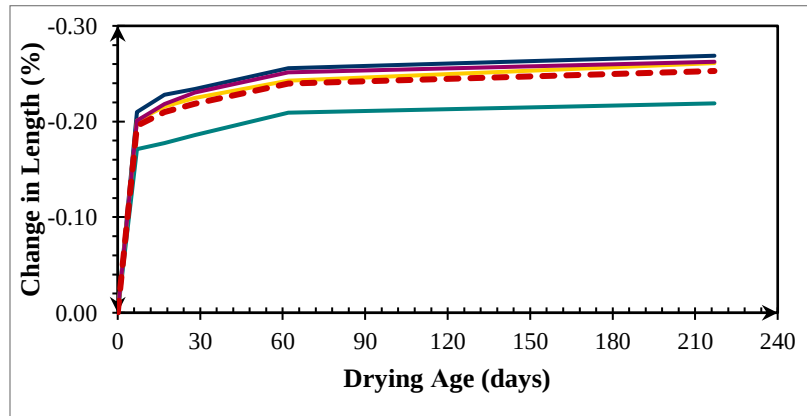


Figure 5.32: Change in length development over time due to shrinkage. The dashed line represents the average (repetition of four specimen)



Figure 5.33: Shrinkage microcracks

5.5.3. Coefficient of Thermal Expansion

Coefficient of thermal expansion (CTE) measures the length change of the material in response to changes in temperature. This parameter is very important for the performance of concrete pavements and bridge decks (Russell & Graybeal, 2013). Three 75 mm x 100 mm cylinders were used for determination of CTE. Upon demolding, the cylinders along with a specimen with an embedded probe thermometer were placed in a bucket filled with saturated limewater closed with a lid and were maintained for 56 days of curing. As illustrated in Figure 5.34, four DEMEC gauge points were attached along two diametrically opposite lines of the cylinder through hard glue. By using a reference bar, the distance between the gauge points was fixed to be 100 mm. In the first cycle the measurements were taken at room temperature with the thermometer embedded in the center of the cylindrical specimen indicating a room temperature of 22.7 °C, which is hereby designated as X . Then the DEMEC strain gauge was used on the gauge points on each side of the specimens to measure the length A at temperature X . Following, the four specimens were placed in a refrigerator for five hours till they would reach thermal equilibrium. The gauge readings were recorded at intervals of 30 minutes over a five-hour period, until the two successive measurements showed a difference of less than 0.00025. After a successful reading, the temperature was also recorded with accuracy of 0.1 °C. The recorded length and temperature are designated as B and Y , respectively. Subsequently, for the second cycle, the specimens were left at ambient laboratory temperature for five hours with interval readings of 30 minutes till thermal equilibrium. The relative distance between the gauge points on each side and the temperature of the cylinder from the beginning of this cycle are designated as C and Z , respectively. Finally, the specimens were returned in the refrigerator for five hours and the same procedure as described above was followed with final temperature reading assigned to W and length D . Three CTE values were obtained using Equation 5.6. CTE1 and CTE3 correspond to the temperature transition from ambient conditions to cooling while CTE2 corresponds to the opposite transition. Table 5.14 summarizes the results

obtained from this test. The average CTE of the developed THFRGC is 10.01×10^{-6} which is very close to the CTE of steel and conventional concrete. This value is also an indicator of the Si:Al ratio of the geopolymer (Davidovits, 2020). More specifically for different formulations it is observed that CTE is proportional to the amount of molecular silicon oxide SiO_2 found inside the geopolymer's three-dimensional network.

$$CTE\ 1 = \frac{\frac{B-A}{100\ (mm)}}{Y-X},\ CTE\ 2 = \frac{\frac{C-B}{100\ (mm)}}{Z-Y},\ CTE\ 3 = \frac{\frac{D-C}{100\ (mm)}}{W-Z} \quad (5.4)$$

Table 5.14: Coefficient of Thermal Expansion Results using DEMEC mechanical strain gauge

Specimen Side:	1		2		3	
	Left	Right	Left	Right	Left	Right
<i>X</i> (°C)	22.7					
<i>A</i> (mm)	0.01	0.007	0.007	0.007	0.009	0.006
<i>Y</i> (°C)	5.3					
<i>B</i> (mm)	-0.006	-0.01	-0.006	-0.008	-0.012	-0.007
CTE 1 x 10 ⁻⁶ (/°C)	9.39	9.94	9.39	11.05	12.21	7.73
Average CTE 1 x 10 ⁻⁶ (/°C)	9.665		10.22		9.97	
<i>Z</i> (°C)	21.7					
<i>C</i> (mm)	0.009	0.008	0.008	0.006	0.008	0.006
CTE 2 x 10 ⁻⁶ (/°C)	9.14	10.98	8.54	8.54	12.2	7.93
Average CTE 2 x 10 ⁻⁶ (/°C)	10.06		8.54		10.06	
<i>W</i> (°C)	4.6					
<i>D</i> (mm)	-0.007	-0.011	-0.01	-0.013	-0.013	-0.008
CTE 3 x 10 ⁻⁶ (/°C)	9.35	11.11	10.53	11.11	12.28	8.77
Average CTE 3 x 10 ⁻⁶ (/°C)	10.23		10.82		10.52	
Average CTE x 10 ⁻⁶ (/°C)	9.99		9.86		10.19	
Average CTE x 10 ⁻⁶ (/°C) (of 3)	10.01					



Figure 5.34: Preparation of specimens for CTE test and measurement of length change with DEMEC gauge

5.5.4. Abrasion

Abrasion resistance is a physical property of hardened concrete surfaces defined as the ability of the surface to resist repeated mechanical distress. High abrasion resistance is desirable in cases of

industrial floors and construction roads. Main factors with great impact on abrasion are aggregates, porosity, compressive strength, and contact area of the concrete. The standard followed for the determination of abrasion resistance is ASTM C944 (2019). The testing apparatus comprises a drill press with a chuck that holds and rotates the abrading cutter at a speed of 200 rotations per minute bearing on the tested surface. The abrasion test setup used in this study is illustrated in Figure 5.35. Although usually the test is performed on finished specimen surface in contact with the mold, higher material loss has been observed than what is expected. This is attributed to the existence of a water film accumulated on the mold surface resulting in a thin layer of porous concrete (Husain, 2021). For this reason, sawn surfaces were used for this test. Prior to testing, the cylindrical specimen used for abrasion was cut into two pieces with a concrete saw and cleaned of all remaining dust with a damp towel. Once the sawn surfaces were dry, to ensure that the tested area would be free from dust particles, pressurized air was applied. Finally, the two specimens were weighted with a digital scale and their initial mass was recorded.

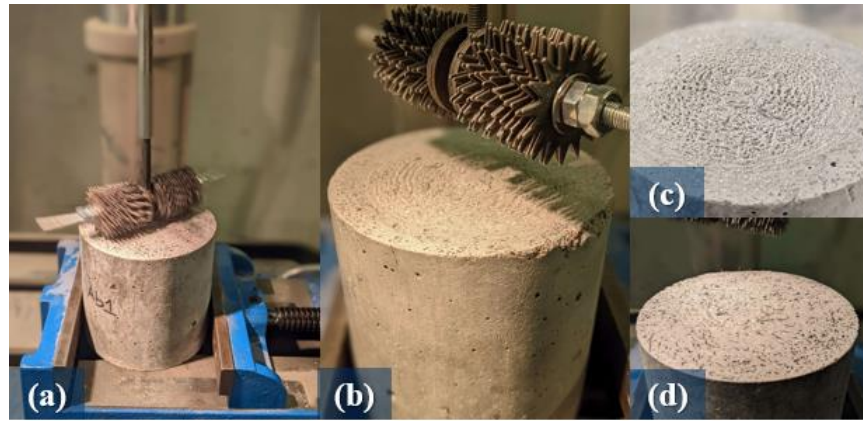


Figure 5.35: (a) Beginning of abrasion 2 minutes cycle, (b) end of abrasion cycle (c) dust accumulation due to abrasion and (d) cleaned specimen ready for the second abrasion cycle

A force of 197 ± 2 N was exerted on the clean flat sawn surface of THFRGC. As mentioned above, according to the standard the speed of the drill press is 200 revolution per minutes for a duration of 2 minutes resulting to 400 rotations. However, the minimum rotation speed of the available drill press was 260 revolutions per minute leading to modifying the abrasion time to 1.54 minutes in order to match the total rotations. Three abrasion cycles were performed on each specimen, while the masses of the specimens were recorded at the end and beginning of each cycle. Prior to the mass measurements, the abraded surfaces were wiped, and dust was removed with pressurized air. The results are summarized in Tables 5.15 and 5.16.

Table 5.15: Initial specimen weights and after abrasion cycles

Specimen	Initial Mass (g)	Before 1 st Abrasion (g)	After 1 st Abrasion (g)	Before 2 nd Abrasion (g)	After 2 nd Abrasion (g)	Before 3 rd Abrasion (g)	After 3 rd Abrasion (g)
1	1699	1698.90	1698.48	1698.50	1697.74	1697.72	1696.74
2	1700	1695.08	1694.72	1694.70	1693.94	1693.96	1692.96

Table 5.16: Summary of Abrasion Loss

Specimen	Mass Loss 1 st Abrasion (g)	Mass Loss 2 nd Abrasion (g)	Mass Loss 3 rd Abrasion (g)	Average Mass Loss (g)	Average Mass Loss (g) (of 2)
1	0.42	0.76	0.98	0.72	0.713
2	0.36	0.76	1	0.707	

The specimens demonstrated significantly high abrasion resistance compared to the abrasion resistance of a proprietary UHPFRC (Husain, 2021). The average mass loss is 40% less than that of the UHPFRC and Figure 5.36 shows the difference between the two materials. Although UHPFRC demonstrates significantly higher compressive strength and less porosity, the better abrasion performance of the developed THFRGC is attributed to metagabbro as precursor and the granite aggregates.

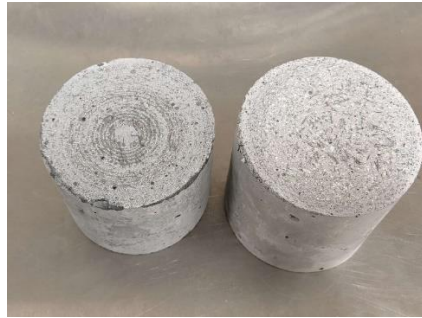


Figure 5.36: Abraded cylinders of UHPFRC (left) and THFRG (right).

5.6. Durability Properties

This section examines the behaviour of the developed THFRGC when exposed to harsh environmental conditions in order to assess its durability through a series of tests. These tests include sulphate, chloride ion penetration, and salt-scaling resistance.

5.6.1. Sulphate Resistance

Sulphate attack is a usual cause of concrete deterioration that occurs once the concrete comes in contact with water containing sulphates. Sulphates bearing compounds, most commonly sodium sulphate (Na_2SO_4) and magnesium sulphate (MgSO_4) are commonly detected in some soil types,

seawater, and wastewater treatment plants. The latter react with calcium and aluminum hydrates that may be present in geopolymer concrete to form ettringite ($\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}$), a needle like crystalline structure that can exert substantial internal stresses resulting in expansion and microcracking. A method to determine the resistance to sulphate attack is to measure the change of length over time of specimens submerged to sodium sulphate solution. The test method followed for determination of sulphate resistance of the developed material is ASTM C1012 (2018).

Four prisms with dimensions 25 mm x 25 mm x 285 mm were cast in steel molds with pre-installed gauge studs. Flow from one end was chosen as the placement method, while external vibration of the specimens with a vibrating table was implemented at the end of the casting. Then, the plain geopolymer concrete specimens were covered with a plastic sheet to prevent evaporation. The next day the specimens were demolded, and their initial measurement of length was taken using the length comparator in the same manner as described in the shrinkage test. Subsequently, the specimens were placed in a curing tank filled with a sodium sulphate solution with a concentration of 50 g/L. As prescribed by the standard, the volume of solution is proportional to the volume of the prisms. Each prism has a volume of 0.178lit and the corresponding solution volume is $4 \times 0.178 = 0.7125\text{lit}$. For a total of four prisms, 2.848lit of sodium sulphate solution were prepared by mixing distilled water with anhydrous sodium sulphate pellets. As the addition of the latter creates a precipitation of crystals, a magnetic stirrer device was used to break the precipitation and homogenize the solution (Figure 3.37). Then the specimens were placed in a curing tank filled with the prepared solution and sealed with the plastic lid till they further recording of their length at the age of 7, 28, 56 and 217 days. After each length measurement, the solution was replaced with a new one to ensure a constant replenishment of sodium sulphate.



Figure 5.37: Preparation of the sodium sulphate solution

As illustrated in Figure 5.38, the developed geopolymer concrete shows no expansion, on the contrary a slight shrinkage is observed. This is consistent with studies from the literature (Davidovits, 2020; Rangan, 2009). The high sulphate resistance of geopolymer is attributed to the different mechanism of geopolymerization. Contrary to the case of hydration in OPC, in geopolymers there are no calcium hydrates that would react with sulfuric compounds to form ettringite and thus cause expansion due to sulphates.

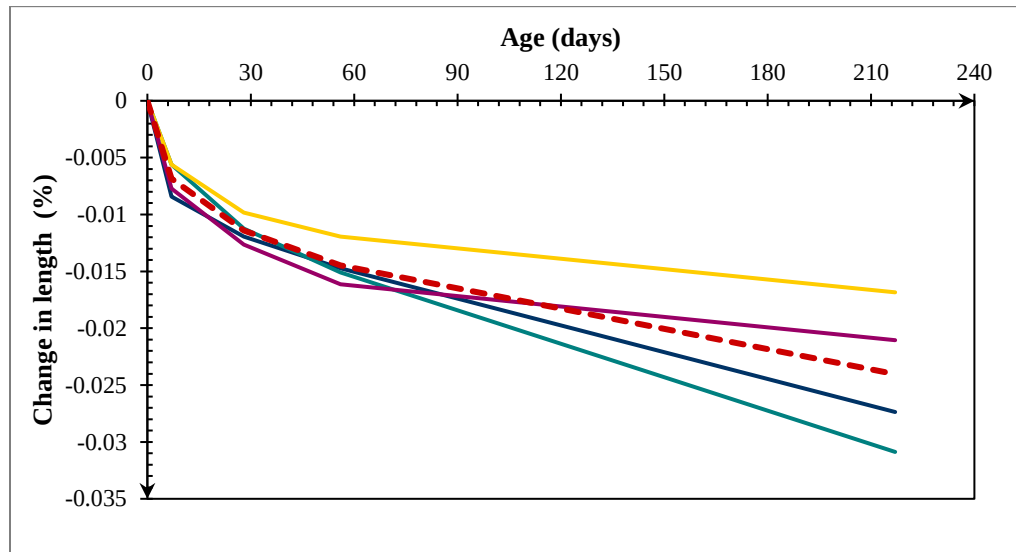


Figure 5.38: Sulphate resistance of geopolymer concrete. The dashed line represents the average (repetition of four specimen)

5.6.2. Chloride Ion Penetration Resistance

Service Life of reinforced concrete structures highly depends on the resistance of the material exposed to harsh environments. A significant form of environmental attack is chloride ingress which results to corrosion of reinforcement and ultimately to deterioration. Therefore, chloride ion penetration resistance is an important durability property that has to be determined for quality control and design. Although the process of chloride penetration is slow, international standards have been developed to assess an accelerated process of chloride ingress. In this study, the standard CSA A23.2-23C which refers to an electrical indication of concrete's ability to resist chloride ion penetration is used. The method is adaptation of ASTM C1202 (2018) and AASHTO T 277 (2022) also known as Rapid Chloride Permeability Test (RCPT), which is inaccurate as it measures ionic movement (not just chloride ions) and not permeability (Stanish et al., 1997).

One cylinder with dimensions of 100 mm x 200 mm was cast with one layer of fresh non-fiber geopolymer concrete, as the presence of the fibers could affect the results by potentially causing a short circuit in the testing apparatus. After 24 hours the specimen was demolded and stored in a bucket with saturated lime water for 56 days. On the 56th day the specimen was taken out of the curing bucket and sawn with a water-cooled concrete sawn into 3 pieces with length of 50 mm. The specimens were left to surface dry for 3 hrs. Subsequently a concrete sealer was applied to the lateral surface of each cylindrical specimen and left to dry. Next, the specimens were placed in a sealed desiccator with a vacuum pump for 3 hrs. After that period, the desiccator was filled with distilled water and the vacuum retained for an additional hour (Figure 5.39). Then, the air was released, and the specimens were left inside the desiccator to cure for extra 18 hr.



Figure 5.39: Specimens curing in desiccator filled with distilled water and vacuum.

Meanwhile, the sodium chloride and sodium hydroxide solutions were prepared. To prepare a 3% by mass sodium chloride solution, 1 lit of distilled water which weighed 994 gr., was mixed with 29.82 gr of sodium chloride. In the same manner, 1 lit of distilled water was mixed with 12 gr of sodium hydroxide pellets to produce a 0.3 N sodium hydroxide solution as prescribed by the standard.

After completion of the 18-hr curing, because of limitations in the number of cells used for the test, one specimen was fitted with a plastic spacer and rubber gaskets on the ends to ensure a tight fit into the specimen, while the rest were put into a plastic bag with 95% humidity and stored in the insulating chamber. The test cell was filled with the NaOH solution on one side and NaCl solution on the other side, as designated on the cell. Before the beginning of the test, a temperature probe was inserted in the NaCl solution space in order to record the temperature during the 6 hr

long test. For 6 hours the amount of electrical current passing through the specimen was monitored, while the power supply was 60V DC. These data are recorded in the measuring device which is controlled by a Data Monitor software. The experimental setup and instrumentation of RCPT are illustrated in Figure 5.40.

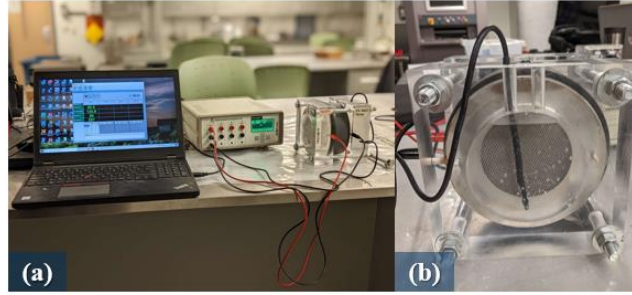


Figure 5.40: (a) User interface of Perma2 device including the measuring device, cell and software monitor (b) Side of the test cell filled with NaCl and embedded probe thermometer.

As mentioned above the tests were conducted for 6 hours at which point the control software automatically terminated the test. No temperature spikes were observed with a stable temperature between 22°C to 25°C. For specimens R1 and R2 the passing Coulombs were 995 and 983, respectively. However, for specimen R3 the test stopped prematurely due to over current, and no passed charge was recorded. The results are less than 1000 coulombs indicating very low chloride penetration as specified by the standard (Table 5.17).

Table 5.17: RCPT rating (CSA A-23.2-23C)

Charge Passed (Coulombs)	Chloride Ion Penetrability
>4000	High
2,000-4,000	Moderate
1,000-2,000	Low
100-1,000	Very Low
<100	Negligible

Although the results categorize the produced geopolymer in the low penetrability category, there are some concerns about the accuracy and suitability of this technique for geopolymer concretes. In RCPT it is assumed that the current flow passes through the sample by pore solution which acts as an electrolyte. For geopolymers it has been found that the pore solution includes other ions and is highly alkaline (Mousavinejad & Sammak, 2021). This highlights the following two limitations. Firstly, as the method measures the current passed related to all ions in the pore solution and not just chloride ions, higher values are expected, thus underestimating the resistance of the material to chloride ingress. Meanwhile, RCPT is highly dependent on the conductivity of the material

which is related to the alkaline content. For these reasons geopolymer concretes tested with RCPT in the literature show higher flow rate than OPC concretes (Mousavinejad and Sammak, 2021).

5.6.3. Freeze Thaw and Deicing Salt Scaling Resistance

Two main durability issues for concrete in cold climates are deterioration due to freeze-thaw and scaling of concrete surface due to de-icing salts. Both mechanisms induce physical deterioration that ultimately leads to cracking, spalling and corrosion of the reinforcement. To determine the resistance of the developed material in these two durability challenges, the CSA A23.2-22C (2014) test was performed on a 200 mm x 240 mm x 75 mm slab specimen. The specimen was cast in Batch 2 along with a control cylinder specimen with an embedded probe thermometer to record the temperature of the concrete at its core. The slab was cast with flow from one end and the cylinder with one layer scoop. When the initial setting time was reached, the thermometer was embedded carefully into the control specimen and held in a position that ensured its end was as close to centroid as possible. Then the specimens were covered with a damp burlap and a plastic sheet for 14 days and another 14 days in air storage. On the 27th day, the specimen was subjected to preparation for the ponding area. To create the latter, four dykes were cut from a 35 mm thick non-absorptive and waterproof plastic board and glued via silicone sealant on the perimeter of the top. Prior to the addition of the sodium chloride solution, a leak test was performed with tap water. The next day the 3 % sodium chloride solution was prepared and poured into the created pond surface area of the specimen. A schematic representation of the setup is shown in Figure 5.41.

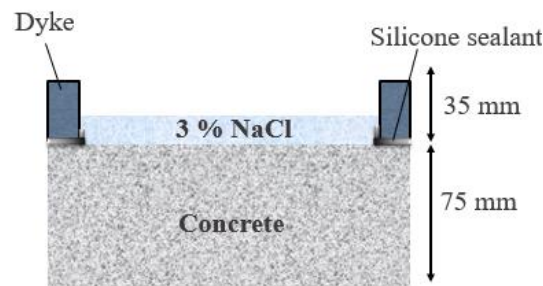


Figure 5.41: Schematic representation of test setup

As specified by the standard the depth of the solution should be 5 ± 2 mm, and a depth of 7 mm was chosen. Subsequently the specimen was covered with a plastic bag to prevent evaporation and stored in the insulating humidity chamber for 7 days with 50% RH and room temperature 22 °C. After that, the NaCl solution was replenished with a new one and then the specimen was placed in

the freezer at a temperature of -18 °C along with the control specimen for the first freeze-thaw cycle. The freezing part of the cycle lasted 16 hrs while the thawing which took place in the insulating humidity chamber, 8 hrs. In every cycle before the freeze phase, the depth of the solution in the pond was checked to be 7 mm. In case the depth dropped due to evaporation, additional distilled water was added. The scaling remnants were measured at 5, 15, 30 and 50 cycles. At end of these cycles, a new saline solution was used to rinse off the flaked pieces by tilting the specimen into a funnel. The solution containing the residue was later poured over an oven dried filter paper with known mass. The latter was then placed in the oven at 110 °C for 4 hours to dry and left to cool for an extra hour. Then the filter paper with the residue was weighted to determine the material loss of this cycle. Table 5.18 summarizes the material loss at each cycle related to the surface exposed to the solution. The cumulative material loss is 0.33 kg/m², which is less than 0.8 kg/m² which is requirement of the Ministry of Transportation of Ontario (MTO), Canada for materials used in bridge construction. Regarding the visual ratings as illustrated in Figure 5.35, there are some pop outs indicating slight to moderate scaling of the surface mortar, classifying the material in Category 2-B. The visual rating categories are listed in Table 5.19

Table 5.18: Freeze Thaw and Deicing Salt Scaling results

Number of Cycles	Specimen	Visual Rating
5	0.02	0
15	0.21	1
30	3.00	2-A
50	5.60	2-B
Total material loss (g)	8.83	-
Total (kg/m²)	0.33	-

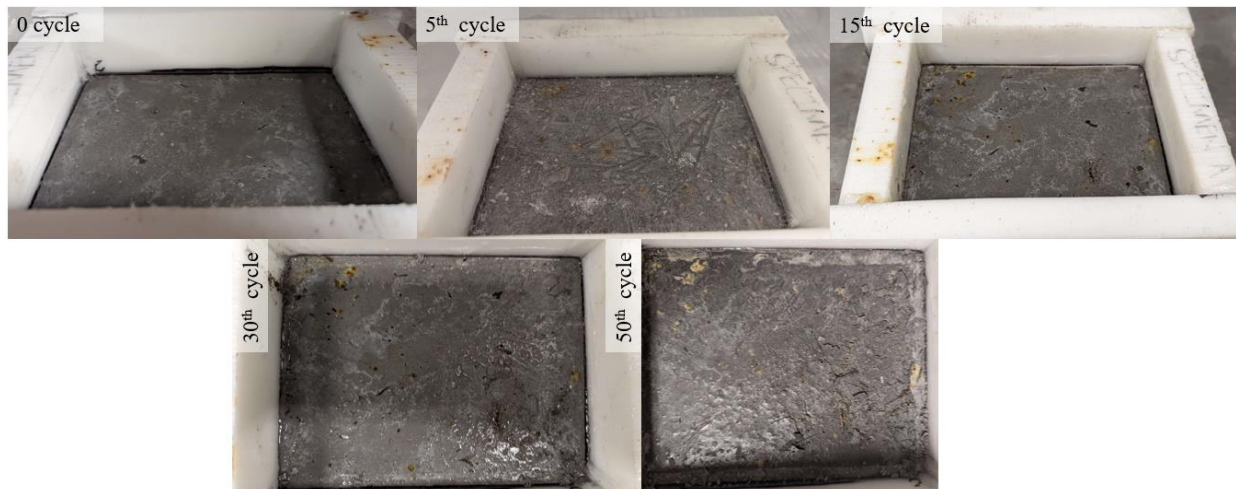


Figure 5.42: Visualization of deterioration through freeze thaw cycles and deicing salt scaling

Table 5.19: Visual rating of scaling for concrete surface (CSA A23.2-14 22C)

Category	Characteristics of Scaling Surface
0	No significant scaling observed
1	Slight scaling (3 mm depth, maximum, no coarse aggregate visible)
2-A	Slight to moderate scaling of the surface mortar (possibly a few pop outs)
2-B	Slight to moderate scaling of the surface mortar (several pop outs)
3	Moderate scaling of the surface mortar (some exposed coarse aggregate)
4	Moderate scaling of the surface mortar (significant surface of mortar loss with exposed coarse aggregate)
5	Severe scaling (coarse aggregate visible over entire surface)

5.7. Conclusions

This chapter was dedicated to a full material characterization of the developed novel THFRGC. The experimental campaign includes investigation of fresh-state, mechanical, physical and durability properties. Based on the experiments discussed here the following conclusions are made:

- The developed material is self-compacting, flowable with satisfactory setting time. To avoid early drying shrinkage cracks, the fresh THFRGC should be covered tightly so no water evaporation occurs.
- Both in tension and compression, high ductility is observed. In case of compression, due to the passive confinement of fibers, at 50% post peak the average axial deformation is 0.028, while the lateral strain is 0.038. In case of tension, the ultimate apparent tensile strain at peak tensile stress is 0.002.
- Compared to OPC concretes of the same compressive strength, geopolymer concrete exhibits lower Young's Modulus and Poisson's ratio. This is consistent with the literature and might be attributed to its different molecular structure.
- The ratio between cylindrical and cubic compressive strength is 0.7, while in OPC concrete is usually around 0.8 for medium to high strengths.
- The material exhibits tension hardening in direct tension and flexure with a hardening ratio of 1.13. In addition, multiple cracking formation is evident through DIC.
- The splitting test is not recommended for FRCs as the results are highly dependent on fiber distribution which is grossly random in case of cylinders. On the other hand, the proposed MST is a promising tests combining easy and widely available experimental setup with accurate results.

- The developed THFRGC is categorized as semi-low density concrete with an air-dry density of less than 2000 kg/m³.
- The CTE of the material is similar to that of OPC concrete.
- The only problematic properties compared to OPC FRCs are the high shrinkage and water absorption. Further research is needed to tackle these issues.
- In terms of abrasion and sulphate resistance, the material demonstrated superior properties, better than UHPFRC. The high resistance in abrasion is attributed to granite aggregates and metagabbro quarry by-product which was used as the precursor. Meanwhile, the exposure to sodium sulphate did not cause any expansion. Contrary, slight shrinkage was observed. This can be explained with the lack of calcium hydrates.
- In terms of chloride ion resistance, the material is classified in the very low penetrability range. However, the RCPT test might underestimate the resistance to chloride ion penetration in case of geopolymer concretes. This is because the latter have alkaline pore solution, and the test measures the movement of all ions and not only the chlorides.
- Finally, the developed THFRGC shows satisfying freeze thaw and deicing salt scaling resistance with mass loss under the 0.8 kg/m² limit used in bridge construction.

5.8. References

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Chapter 6: Bond of Steel Reinforcing Bars in the Developed THFRGC

6.1. Introduction

Conventional Reinforced Concrete (RC) members rely on steel reinforcement to carry tensile forces, while stresses are transferred from the concrete to the steel bars and vice versa through bond. The bond mechanism for conventional RC is illustrated in Figure 6.1(a). Application of tensile stress on the embedded bar results in a tendency of the latter to translate in the direction of the applied traction. The ribs of deformed bars interlocked with the matrix exert bearing action on the surrounding material, that may be resolved into longitudinal (bond) and radial pressures on the cover. The latter are held in equilibrium with hoop tensile stresses of the cover. Upon exceedance of tensile strength of the cover concrete longitudinal splitting propagates along the length of the bar leading to loss of bearing action of the ribs and thus debonding (loss of bond) takes place. With THFRC materials, an additional mechanism of resistance is introduced to the system via fiber reinforcement. Fibers enhance the tensile strength of concrete and sustain it to large hoop strain magnitudes in the cover, bridging the cracks developed by stress transfer while also providing passive confinement (Figure 6.1(b)).

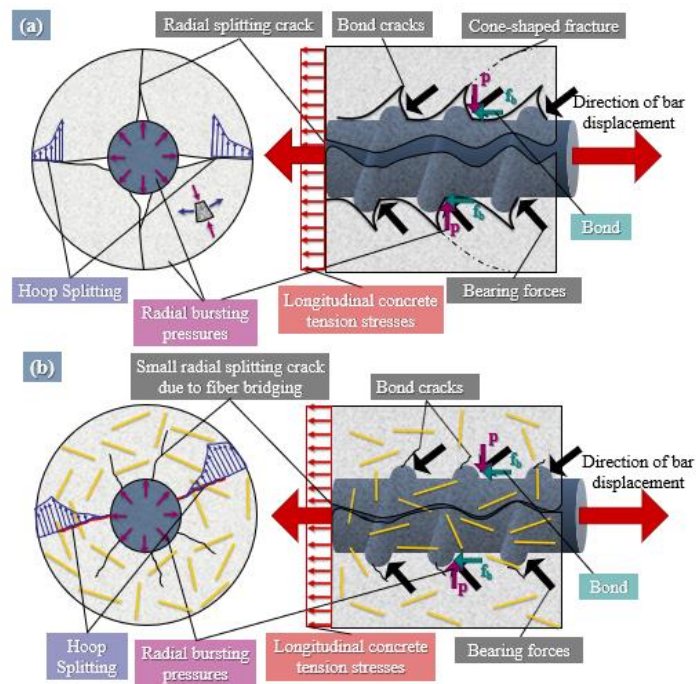


Figure 6.1: Bond mechanism in (a) conventional RC and (b) THFRC

Meanwhile, in order to advance the use of eco-friendly THFRGC, in structural applications, it is vital to comprehend the mechanics of bond of reinforcement anchored in this type of material. The study is not only important for comparison to RC or FRC made of OPC, but also aims to establish a constitutive bond-slip law for THFRGC. Taking the above mechanics into consideration it is evident that bond mechanism is strongly correlated to the tensile capacity of the cementitious matrix.

Because reinforcement development in any new concrete product is critical information in order for this new material to be deployed in structural applications, studying bond of reinforcement in the proposed THFRGC was considered essential towards the objectives of this thesis. The investigation is conducted experimentally, using Direct Tension Pullout Tests (DTPT), on account of previous findings that have illustrated that other forms of bond tests are unconservative (Georgiou et al., 2018; Tsiotsias & Pantazopoulou, 2021a). Objective of this chapter is to assess and quantify the contribution of tensile strength and post-cracking capacity of geopolymer THFRC to the bond-slip law of the embedded reinforcement. Parameter of study was the anchorage/embedment length and the concrete clear cover; the matrix consists of the developed steel fiber reinforced geopolymer concrete which was characterized in the preceding chapters. The experimental results obtained from a series of 8 specimens are further analyzed using Digital Image Correlation (DIC). Finally, a constitutive bond-slip model for bars embedded in THFRGC is established.

6.2. Parameters Affecting Bond Behaviour

It is well established from the study of bond in conventional concrete and FRC that the mode of failure and bond strength of embedded reinforcement are impacted by several parameters, such as the bar rib profile, the available embedment length, and the concrete cover thickness. The experimental setup used has been shown to have a great impact on the results producing a vast range of bond strength values depending on the kinematic restraint it imparts on the bar-concrete interface. Here a conventional ribbed M15 bar type is used for the experimental study. The influence of the development length and cover thickness are variables of the investigation.

6.2.1. Length of Anchorage

Average bond stress is obtained through the following equation:

$$f_{b,ave} = \frac{F_b}{\pi \cdot D_b \cdot L_b} \quad (6.1)$$

Where F_b is the maximum force developed on the bar, D_b is the diameter and L_b the anchorage length of the bar which comprise the lateral contact area. For short anchorage lengths the bond stress distribution along the embedment length is almost uniform particularly near failure, and therefore, the average maximum bond stress developed, $f_{b,ave}$, is a close approximation to the actual bond strength, $f_{b,max}$. For longer anchorage lengths a nonlinear distribution of bond stresses develops along the embedment length (Eleftheriou et al., 2017), and thus the difference between average maximum bond stress and actual bond strength, which is considered a local property, increases.

The governing equations of the bond problem are discussed next. A bar subjected to tensile forces eventually transfers its force through bond stresses, $f_b(x)$, to the surrounding concrete over the development length. As the bar is pulled, it tends to translate in the direction of the applied force by an amount $s(x)$, known as the bar slip. Figure 6.2 depicts the free body diagram of the bar over the entire development length, L_b . x is the coordinate of an arbitrary point along the bar axis, measured from the loaded end. Similarly, $\bar{x}$ denotes the distance from the free end of the anchorage. Slip $s(x)$ results from the accumulation of the relative length change between reinforcement and the surrounding concrete matrix and is obtained as the incremental difference between strains of the bar $\varepsilon_s(x)$ and the matrix longitudinal strains, $\varepsilon_{ct}(x)$ over an infinitesimal length dx as shown in Equation 6.2.

$$ds = -(\varepsilon_s(x) - \varepsilon_{ct}(x)) \cdot dx \quad (6.2)$$

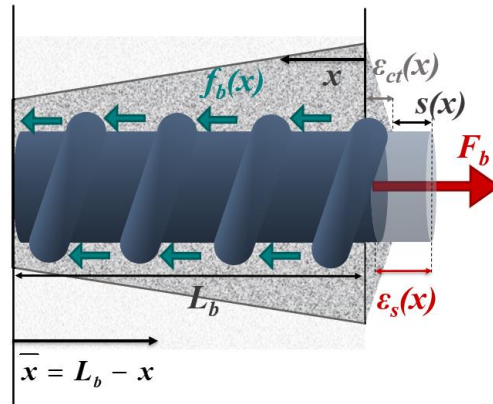


Figure 6.2: Definition of terms

Taking into consideration the assumptions that there is a linear relationship between bond stress and slip and that the reinforcement bar has not yielded yet ($f_b = k \cdot s$, where k is the stiffness of average bond-slip law), according to Eleftheriou et al., (2017), Equation 6.2 can be written as:

$$\frac{d^2 s}{dx^2} = \frac{4(\psi+1)}{D_b \cdot E_s} f_b(x), \quad \psi = \frac{A_b}{A_c} \cdot \frac{E_s}{E_c}, \quad A_c = \pi \cdot C \cdot (C + D_b) \quad (6.3)$$

In the above A_c represents the area of the concrete cover engaged in resisting the tensile forces that are transferred through bond, from bar to concrete. Here it is assumed that A_c is defined by the area of a circular ring of thickness equal to that of the cover, C , surrounding the bar. The solution of Equation (6.3) requires a relationship between bond and slip. This is usually measured from short anchorage length experiments and is considered a constitutive property of the concrete matrix. Based on results published in the literature and summarized by the fib-Model Code (fib Bulletin 65, 2010), the characteristic local bond slip law has an ascending branch that is slightly nonlinear, followed by a plateau at peak bond stress followed by a descending branch (Figure 6.2(a)). This is idealized as a trilinear envelope in Figure 6.2(b) and is used heretofore as reference for the solution of Eq. (6.3).

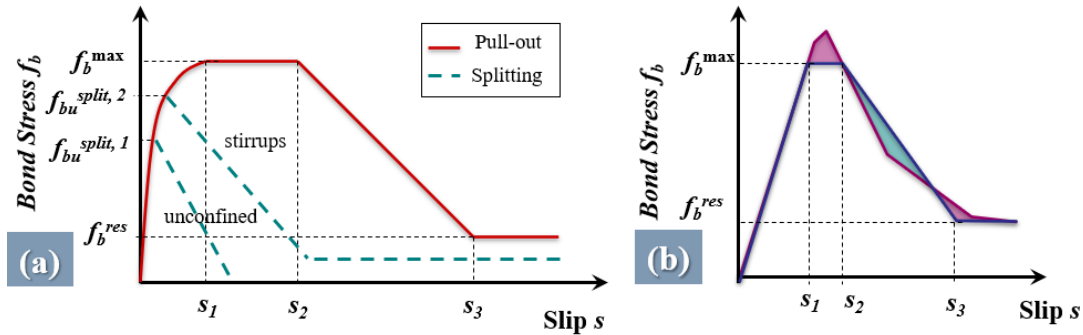


Figure 6.3: (a) Bond-Slip relationship recommended in the Model Code (b) Linearized bond stress-slip relationship (equal area principle).

When bond is in the linear ascending branch of the bond-slip relationship shown in Figure 6.2(b), so that $f_b(x) = k \cdot s(x)$ the equation may be solved exactly (Eleftheriou et al., 2017) as follows:

$$\text{Bar Strain: } \varepsilon_s(x) = \varepsilon_o \cdot \frac{(e^{-\Omega x} - e^{\Omega(x-2L_b)})}{(1 - e^{2L_b\Omega})}; \quad \Omega = \sqrt{\frac{4k(\psi+1)}{D_b \cdot E_s}} \quad (6.4)$$

$$\text{Bar Slip: } s(x) = \frac{D_b \cdot E_s \cdot \Omega}{4k} \cdot \varepsilon_o \cdot \frac{(e^{-\Omega x} + e^{\Omega(x-2L_b)})}{(1 - e^{2L_b\Omega})}$$

$$\text{Concrete Normal Strain in the Cover: } \varepsilon_{ct}(\bar{x}) = -\psi \varepsilon_o \cdot \frac{(e^{\Omega \bar{x}} - e^{-\Omega \bar{x}})}{(e^{L_b\Omega} - e^{-L_b\Omega})}$$

The next phase in this diagram (Figure 6.2(b)) corresponds to the case where bond stress is constant and equal to the strength whereas bar slip increases leading to local bond failure at the end of the plateau, s_2 . This begins at the loaded end of the bar and spreads gradually towards the free end of the bar; the length $l_p \leq L_b$ over which bond is plastified is governed by Equation 6.3, but now the solution for that segment is simpler, as follows:

$$\text{Bar Strain: } \varepsilon_s(x) = -\frac{4f_b^{\max}}{E_s D_b} x + \varepsilon_{so}, \text{ for } 0 \leq x \leq l_p \quad (6.5)$$

Where ε_{so} is the bar strain at the end of the plastified segment, beyond which the solution of Equation 6.4 presented above is valid (after replacing in the solution expression the following: where x , use $x-l_p$, and where L_b , use L_b-l_p).

The corresponding bar slip is:

$$\begin{aligned} s(x) &= \frac{2f_b^{\max}}{E_s D_b} (x-l_p)^2 - \varepsilon_{so}(x-l_p) + s_1 \rightarrow \text{for } x=0 \\ s_2 &= \frac{2f_b^{\max}}{E_s D_b} (l_{p,\max})^2 + \varepsilon_{so}(l_{p,\max}) + s_1 \end{aligned} \quad (6.6)$$

Where s_1 is the start of the plateau in the bond-slip law (end of the linear elastic ascending envelope). The maximum length $l_{p,\max}$ occurs at the onset of bar failure, at slip magnitude s_2 : if the latter is known from the idealized trilinear envelope fitted to the experiment, then $l_{p,\max}$ may be solved from the above equation. Saikali et al., (2022) illustrated the sensitivity of the above expressions by parameterizing the independent variable as a multiple of bar diameter, D_b : Thus, the anchorage length L_b and distance x from the loaded end are taken as, $q \cdot D_b$ and $r \cdot D_b$, respectively, Equation 6.4 may be rearranged as follows:

$$f_b(r \cdot D_b) = \varepsilon_o \cdot \frac{D_b \cdot E_s \cdot \Omega}{4} \cdot \frac{(e^{-\Omega \cdot r \cdot D_b} + e^{\Omega \cdot D_b (r-2q)})}{(1 - e^{-2\Omega \cdot q \cdot D_b})} \quad (6.7)$$

Selecting any specific value for parameter q , and varying parameter r so that $0 \leq r \leq q$, the shape of bond stress distribution over the available development length may be plotted. For a given value of q , and considering increasing values of $l_p = p \cdot D_b$, where $p=0$ (elastic bond), $p=1$ (i.e., over length $l_p = D_b$ measured from the loaded end, plastic bond equals the strength magnitude f_b , whereas gradually attenuating elastic bond stresses occur over $(q-p \cdot D_b)$, etc., different values of slip may be calculated for s_2 : matching this with the measured value at the onset of the post-peak branch enables determination of exactly the length over the bar where bond was plastified. From this analysis, it was surmised that bond stress distribution is not far from the average for small values of q (≤ 5), but it becomes intensely nonlinear for longer embedment lengths (large values of q). From an experimental standpoint, to obtain the coordinates of the milestone points of Figure 6.2(b) a series of tests are needed for a given bar, cover and concrete material: short embedment lengths would approximate the bond strength, f_b^{max} and the slip amount s_1 upon instantaneous failure. Longer lengths are also needed to determine the values of s_2 and s_3 ; these work by allowing redistribution of bond stresses away from the loaded end and progressively towards the anchorage. In the present study, where determination of bond strength would be the first step towards understanding the bar THFRGC interaction, the primary series of specimens tested had $5D_b$ for anchorage length following the benchmark of bond tests in the literature for bond strength characterization (fib Bulletin 72, 2014; ACI-408, 2003).

6.2.2. Concrete Cover & its Tensile Capacity

Concrete cover participates in the bond mechanism in two ways: (a) by resisting the tensile forces transferred from the bar to concrete through bond, and (b) by developing radial compressive and hoop tensile stresses to resist the pressures exerted by the ribs as they translate in the longitudinal direction, (Figure 6.1(a)). With regards to (a), longitudinal tensile stresses taken by the cover are limited by the low tensile strength and brittleness of concrete; formation of cracks orthogonal to the bar axis relieve these stresses in conventional concrete. Indeed, the crack spacing after crack-stabilization is limited by this phenomenon. However, in the case of THFRC (as well as in

THFRGC) the ability of the material to sustain tensile stresses beyond cracking affects the crack spacing leading to very closely spaced cracks prior to localization into few widened cracks.

As for (b), it is well known from experimental literature as is evident also from Equation (6.3) that larger cover thickness results to greater resistance in hoop direction and greater bursting pressures maintained by the concrete ring around the bar (fib Bulletin 72, 2014; ACI - 408, 2003; Tastani & Pantazopoulou, 2013). Cover splits when hoop tension exceeds the material tensile strength. Splitting begins from the internal surface of the cover, and it propagates outwards till it reaches the exterior surface of the member, where it is manifested by longitudinal cracks parallel to the bar. Any form of reinforcement that intercepts the crack penetration through the cover delays splitting and thus enhances bond strength. In conventional concrete this is achieved through stirrups, but in tension-hardening materials through cracking is mitigated by the action of the fibers. (Chao et al., 2009; ACI- 408, 2022). Moreover, superior interface between the cementitious matrix and the reinforcement can be achieved in case of high-performance cementitious composites. This is attributed to the density of the matrix. However, there is an upper limit to bond strength increase: for very large covers, failure mode may change from splitting to pullout as the strength of the system is eventually limited by the shear strength of the cylindrical sliding surface that is defined by the tips of the bar ribs (this is a pullout mechanism (Georgiou et al., 2018; ACI 408, 2003; Malvar, 1992; Tsiotsias, 2019).

6.2.3. Experimental Setup

The most commonly used test for determination of bond strength of reinforcement embedded in concrete is the standard pullout test (SPT). However, there are several objections to this test type and its variations, owing to systematic evidence that illustrates that boundary conditions of the test setup interfere with bond and produce artificially high values for bond strength – therefore being seriously unconservative for design.

Tsiotsias & Pantazopoulou (2021) among others reviewed the most prevalent bond setups by assembling and studying data from the literature on THFRC and performed a detailed computational investigation on the effect of test setup on bond strength using nonlinear finite element analysis. In this case the bond-slip relationship is the output of the performed analysis, as is the case during testing; the responses obtained for a given bar and identical embedment length

when pulling on it from different modelled test setups yielded very different bond-slip relationships. From among the many alternatives, the DTPT provided the most conservative estimate of bond strength as the bar specimen was subjected to pure tension and unaccounted for boundary restraints occurred. It was shown that this test setup engages exclusively the bar-to-concrete interface along with the tensile response of the cementitious matrix exactly as would occur in the tension zone of a beam, thereby establishing it as the most suitable candidate for investigating the bond behaviour of THFRCs as well as in the case of conventional concrete.

6.3. Description of Experimental Procedure

6.3.1. Investigation Parameters

A series of DTP tests were conducted in the present dissertation to investigate two important parameters affecting bond of steel reinforcement bars developed in geopolymer THFRC. The bar diameter selected for the investigation was 16 mm. Study variables were the concrete cover as a multiple of the diameter, namely $1D_b$ and $2D_b$, and the anchorage length taking on values of $5D_b$ and $10D_b$. A number of 8 tests were performed, with 2 specimens for each combination. Table 6.1 shows the combination of cover and anchorage length.

Table 6.1: Specimen code names and parameter specifications for DTP tests

Specimen ID	Bar Diameter D_b	Clear cover C	Anchorage Length L_b
G1-5-A	15M	1D _b	5D _b
G1-5-B			
G2-5-A		2D _b	
G2-5-B			
G1-10-A		1D _b	10D _b
G1-10-B			
G2-10-A		2D _b	
G2-10-B			

6.3.2. Preparation of the Formworks

Two different types of formwork were designed and used in this study to accommodate the investigation of the concrete cover thickness parameter. A descriptive arrangement of the formwork is shown in Figure 6.4, while Table 6.2 describes the annotation of the elements comprising the mold. As geopolymer concrete has strong adhesion to metal or wooden formwork even after the lubricating the surface of the mold red sheathing tape is used on the inner surfaces of the formwork for ease of demolding. Caulk was applied on top of the edges and on the perimeter of the circular

openings to prevent leakage. The free ends of the bars were clamped down on a lifting table, to keep them horizontal during casting, as there was a risk of out of plane misalignment from the mass of poured concrete. Prior to casting, all faces were lubricated using sunflower oil to ensure a smoother surface for specimen.

Table 6.2: Annotative description of Figure 6.4.

Annotation	Description	Dimensions (mm) [$2D_b/1D_b$]
A	Base plate	600 x 600 x 19
B	Front face panel	400 x 280 x 19
C	Side plates	400 x 99 x 19 / 400 x 67 x 19
D	Top/Bottom side plates	525 x 99 x 19 / 525 x 67 x 19
E	Support bar	16 x 70 + $5D_b$ / $10D_b$
F	Test bar	16 x $5D_b$ / $10D_b$
G	Connecting threaded rods	550 x 9.5
H	Wooden block for extra bar support	250 x 120 x 19
I	Aluminum Corner Brace	25.4 x 25.4 x
J	Flat Washers & Hex nuts	

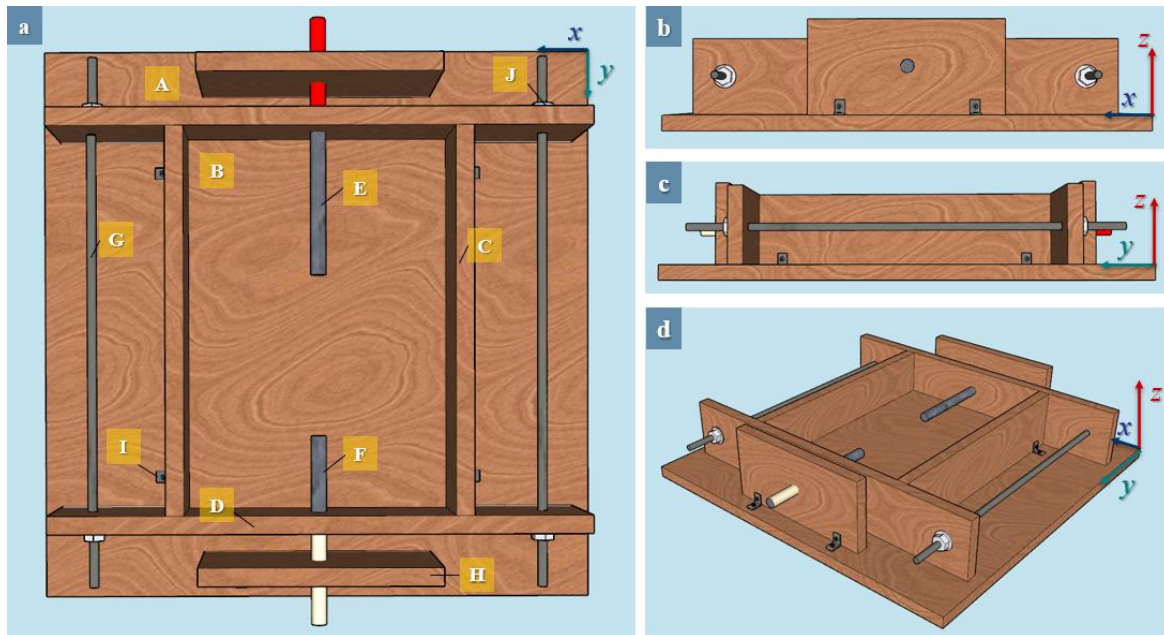


Figure 6.4: Different views of formwork design: (a) Plan view in x-y plane, (b) Side view in x-z plane, (c) Side view in y-z plane and (d) perspective view

The mix design used in this Chapter is GPC4-F described in Chapter 3 (Table 3.13) and fully characterized in terms of material behaviour in Chapter 5. Due to limitations in the mixer volume, two specimen molds were constructed for different concrete cover, resulting in 4 identical batches, namely two castings for an anchorage length of $10D_b$ and two for $5D_b$. Figure 6.5 illustrates the

two formworks used in a typical casting (clear cover of 1 and $2D_b$) along with the use of bubble level for alignment check.

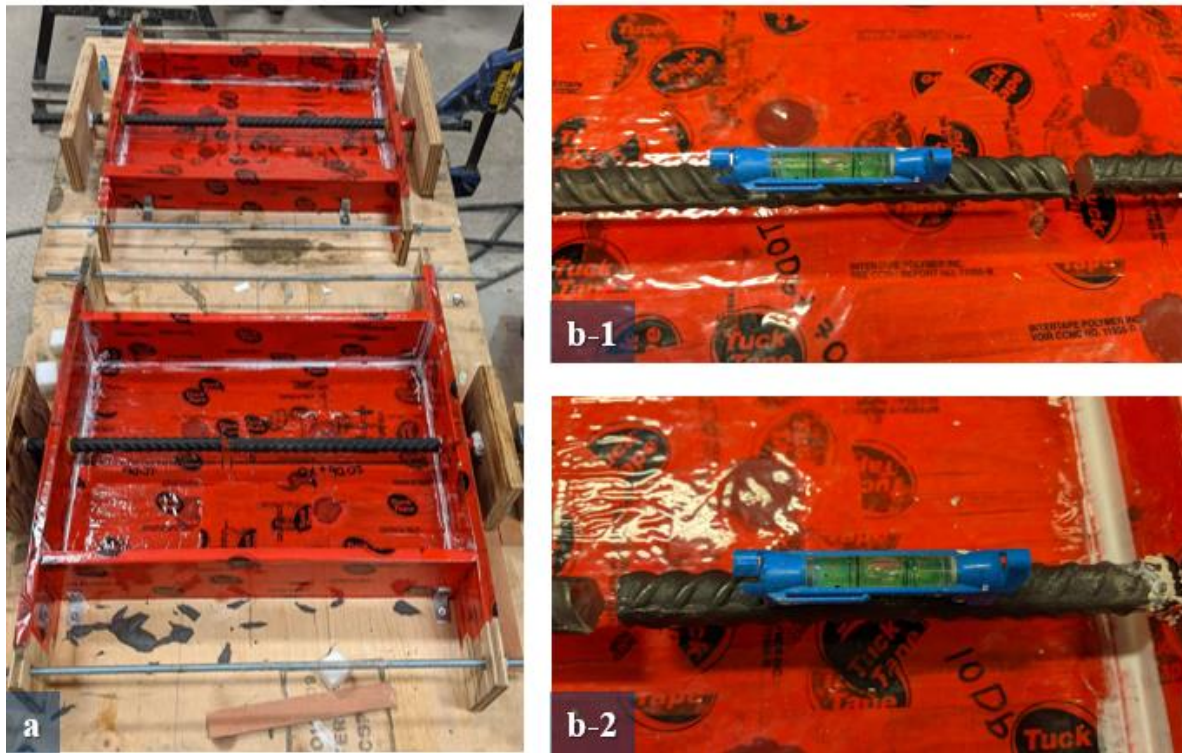


Figure 6.5: (a) Perspective view of formwork design (b) check of alignment in x-y plane ((1) support and (2) test bar)

6.3.3. Mixing, Casting and Curing of Specimens

As already mentioned before, the specimens were cast in different batches in four consecutive days. To achieve consistency in mechanical properties, proportions, mixing procedure, mixing time, casting, and curing were strictly followed and were identical in all four batches. Details on the above are provided found in Chapter 5. It was already discussed in previous chapters that the casting method is crucial to fiber distribution which has a great influence on mechanical properties. For this reason, to access the fiber contribution to the hoop tension in the cover, the casting direction was flow from one end and took place from the middle of side molds, normal to the bars as illustrated in Figure 6.6(a). Flow from one end, parallel to the reinforcement could result in unfavorable arrangement of the fibers in direction parallel to the test bar, in which case the development capacity would correspond to that of a bar embedded in plan (unreinforced) matrix

which would obscure any contribution from the tension hardening property of the material on bond strength (Figure 6.6(b)).

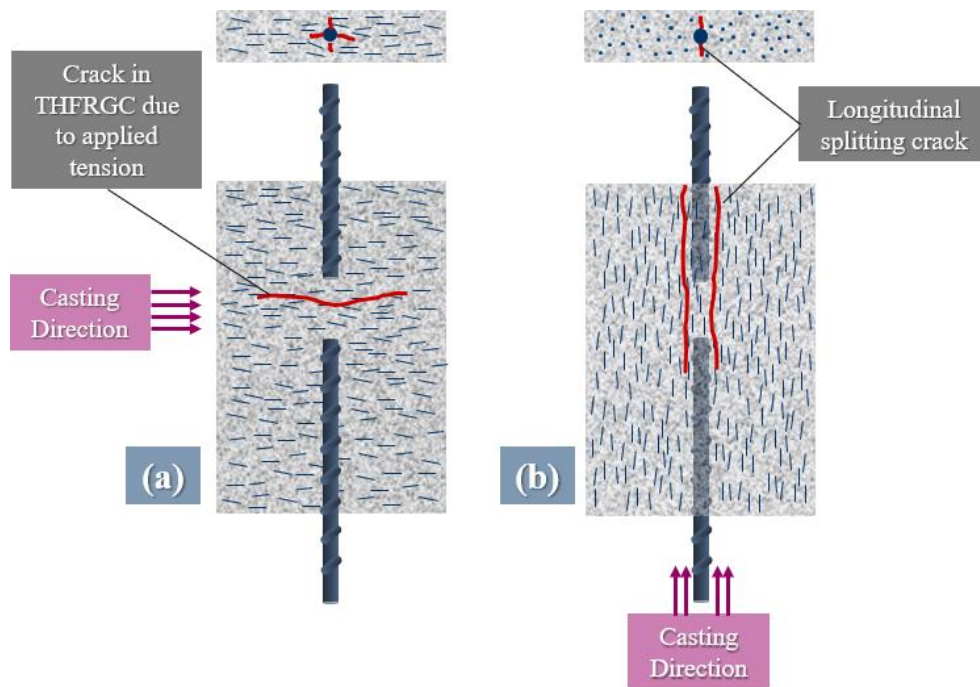


Figure 6.6: Effect of concrete placement method on failure mode (a) flow from the side, (b) flow from bottom

After casting, external vibration was applied on the specimens by slightly tapping the sides with a mallet. When a smooth finished surface was achieved, the specimens were covered with plexiglass panels and wrapped with plastic sheets for 24 hr. to avoid any evaporation and plastic shrinkage cracks. After that period, the specimens were removed from the formwork, wrapped in wet burlap sheets, placed in individual curing tanks which were further sealed with plastic sheets to prevent water evaporation (Figure 6.7). The specimens were moist cured for 28 days, while once a week a check on the moisture conditions was performed to ensure consistent curing till testing.



Figure 6.7: Moist curing of DTP specimens for 28 days

6.3.4. Material Properties

Direct tensile tests and uniaxial compressive tests were performed to obtain the stress-strain curves of the THFRGC material in which the reinforcement bars for the DTP tests were embedded. The testing procedures for the tensile and compressive tests are described in detail in Chapter 5 of this thesis. The stress-strain curves from the tests are depicted in Figures 6.8 and 6.9. The stress-strain response of the bar subjected to uniaxial tension was obtained as per ASTM A615 (Figure 6.10). Both test and support bars were Grade 400W per CSA-G30.18-M92.

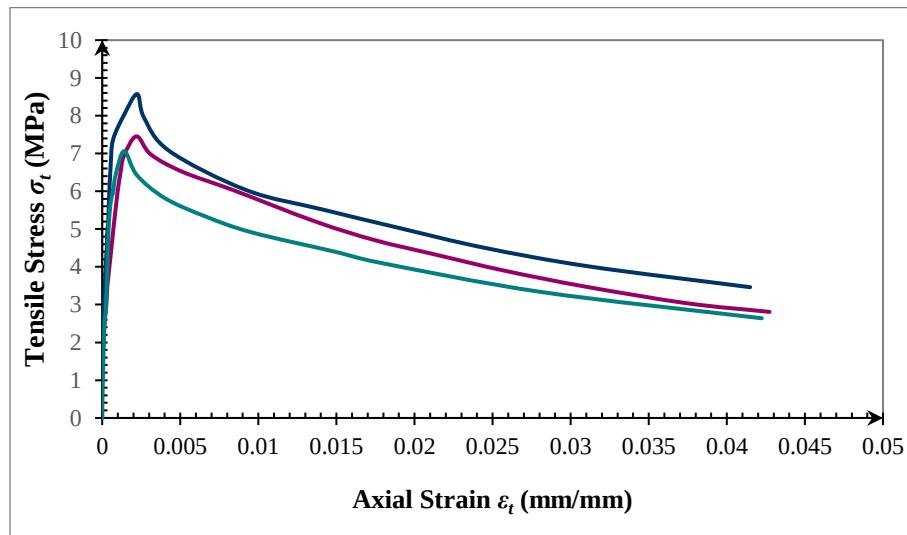


Figure 6.8: Tensile stress-strain curve of the geopolymer THFRC (repetition of three)

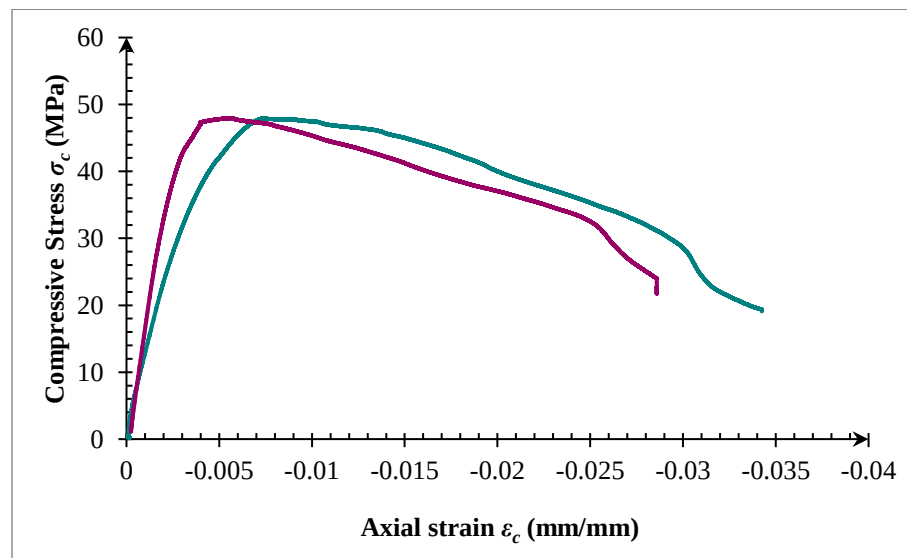


Figure 6.9: Compressive stress-strain curve of the geopolymer THFRC (repetition of two)

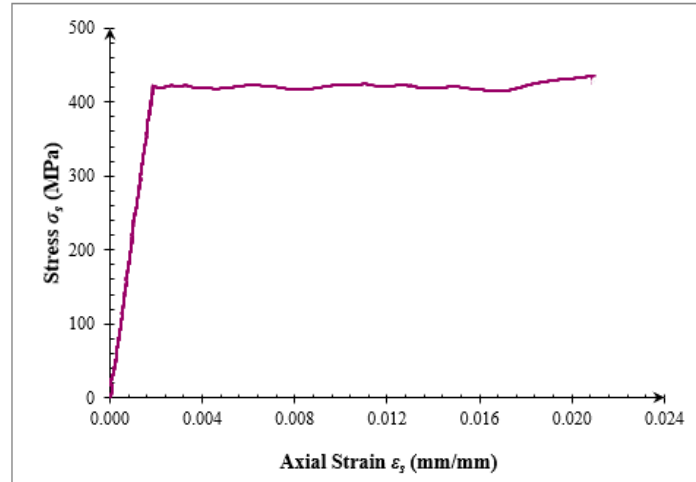


Figure 6.10: Tensile Stress-strain curve of the steel reinforcement bar used in this study

6.3.5. Specimen Preparation, Instrumentation and Testing Equipment

The specimen geometry and preparation prior to testing are illustrated in Figure 6.11. The test specimen comprises of a concrete panel with rectangular cross section. The studied bar is anchored in the specimen with an embedment length of L_b , while an auxiliary bar is placed co-axially and back-to-back with the test bar and extending on the opposite side of the panel with embedment length of $L_b + 70$ mm. In case of $5D_b$ anchorage length, the distance between the two bars that contains only fiber reinforcement is 170 mm while in case of $10D_b$ is 10 mm. For cover thickness $C = 2D_b$, Carbon Fiber Reinforced Polymer (CFRP) strips were used to prevent undesirable types of failure (Figure 6.11(b)).

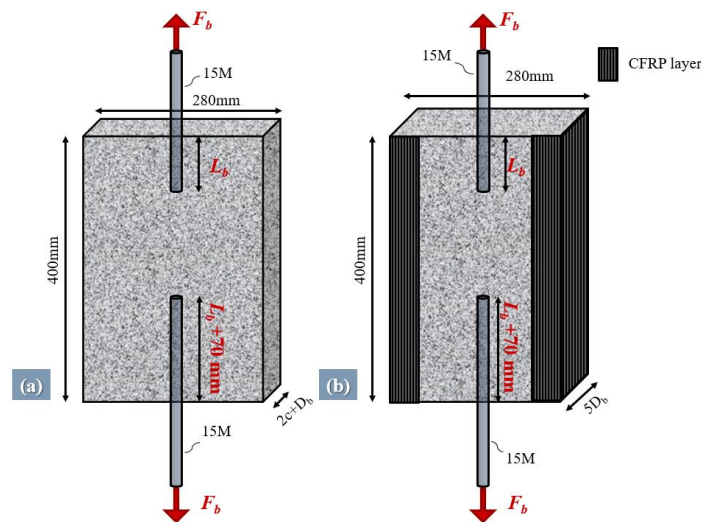


Figure 6.11: (a) Specimen geometry for DTP test (b) Specimen geometry for DTP test with additional CFRP reinforcement on the sides

Figure 6.12(a) depicts the setup in the servo-controlled testing frame where the tests were performed under displacement control. The specimens were monotonically loaded to failure at a displacement rate of 0.15 mm/min until failure, or until the load carrying capacity of the specimen was reduced in the post-peak branch to 20% of the maximum load. The instrumentation depicted in Figure 6.12(b) is further explained in this section. Finally, prior to testing, specimens were prepared for Digital Image Correlation (DIC) analysis. The front façade of the specimen was painted with a white acrylic paint, whereas the speckled pattern was achieved using black spray (Figure 6.12(a)). The software used for DIC in this thesis is the MATLAB open-source module, Ncorr (Blaber et al., 2015). The pictures were captured with a Canon DSLR camera mounted on a tripod overseeing the front façade of the specimens, while additional lighting was provided with a portable projector (Figure 6.12(c)). In addition, a timer was attached on the camera that allowed for pictures to be taken every 10 sec during the experiment.



Figure 6.12: (a) MTS Criterion test frame, (b) Back face of specimen with attached hardware components (c) Camera and lightning setup for DIC analysis

After placing the specimen on the testing frame, securing that the front façade would be normal to the camera and the proper grip length on the bars was attained, the hardware components were attached on the back face of the specimen. The hardware instrumentation is illustrated in detail in Figure 6.13 (b), (c). To measure the displacement of the test bar, a customized Teflon plate with a hole was fabricated and fastened around the bar circumference via two threaded bolts. A wooden

strip was attached through hot glue on the bottom surface of the plate, against which the moveable arm of corresponding Linear Variable Differential Transformer (LVDT) would bear. In order to record the slip, which is the displacement of the bar, related to that of concrete cover near the loaded end, another LVDT was used, which was bearing on a wooden strip glued on top of the concrete block, at 20 mm to the left of the bar. A transverse Differential Transformer (DT) was used to measure the vertical (splitting) crack width, bearing on an according aligned metallic L-bracket. Finally, the LVDTs and DT were attached on an aluminum prismatic piece, hot-glued on the concrete in direction parallel to the anchorage length.

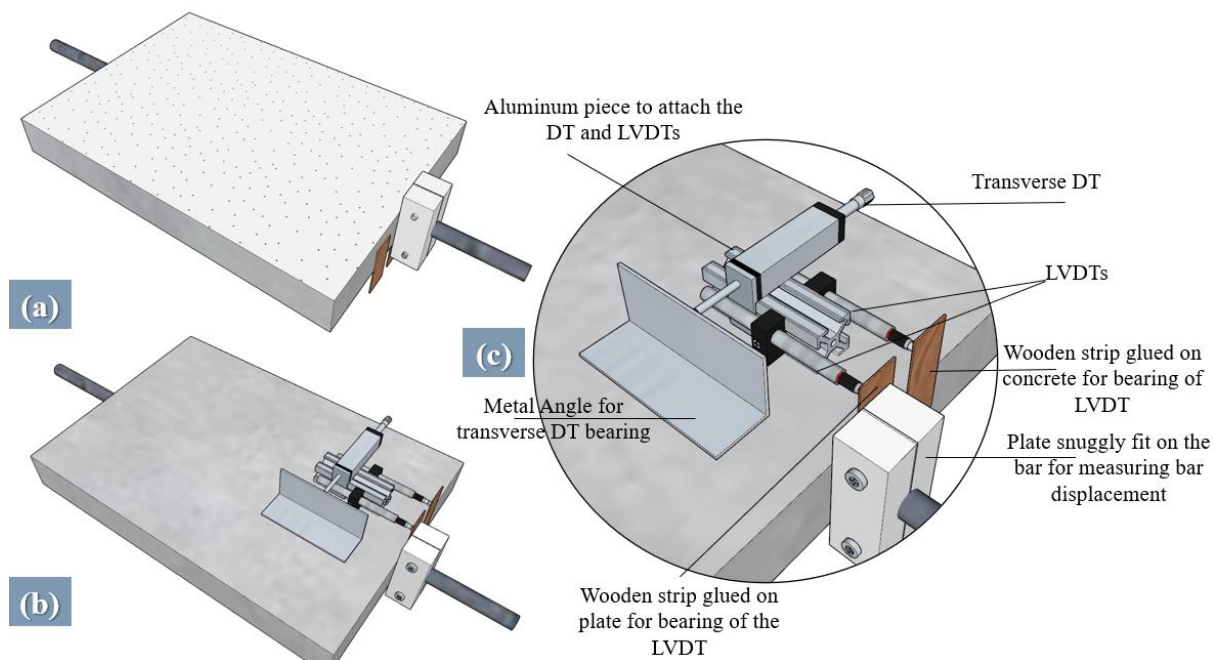


Figure 6.13: Schematic representation of (a) Front façade prepared for DIC (b) Back face of specimen with attached instrumentation (c) Detail of the hardware components

6.4. Experimental Results from the DTP Tests

6.4.1. Modes of Failure

As already discussed above, one common type of bond failure for deformed steel reinforcement bars embedded in plain concrete is cover splitting. Longitudinal cracks originating from the bar's end reach the boundaries of the concrete surface and in case of inadequate confinement (transverse reinforcement or fibers) lead to splitting of the specimen. As the bar translates with respect to concrete in the direction of the applied pullout force, bursting pressure is exerted by the ribs, capable to split the concrete. Characteristic feature of splitting is the formation of crack plains in

planes radial to the axis of the bar. Thanks to the fibers, dense matrix, and strain hardening behaviour of the studied geopolymer concrete, multiple crack plains (i.e., not a single splitting crack) occurred at failure of the specimens.

For specimens with small cover thickness (G1-5 and G1-10), regardless the embedment length, a combination of splitting and tension failure below the end of the bar (in the central part of the block between the two co-axial bars) occurred (Figure 6.14). More specifically, the bar pulled out from the concrete panel, while the latter despite being cracked, remained in one piece. Initially a splitting crack was formed along the length of the bar. Almost simultaneously, a transverse crack located close to the unloaded end initiated, indicating transfer of stresses from the reinforcement to the THFRGC in normal tension.

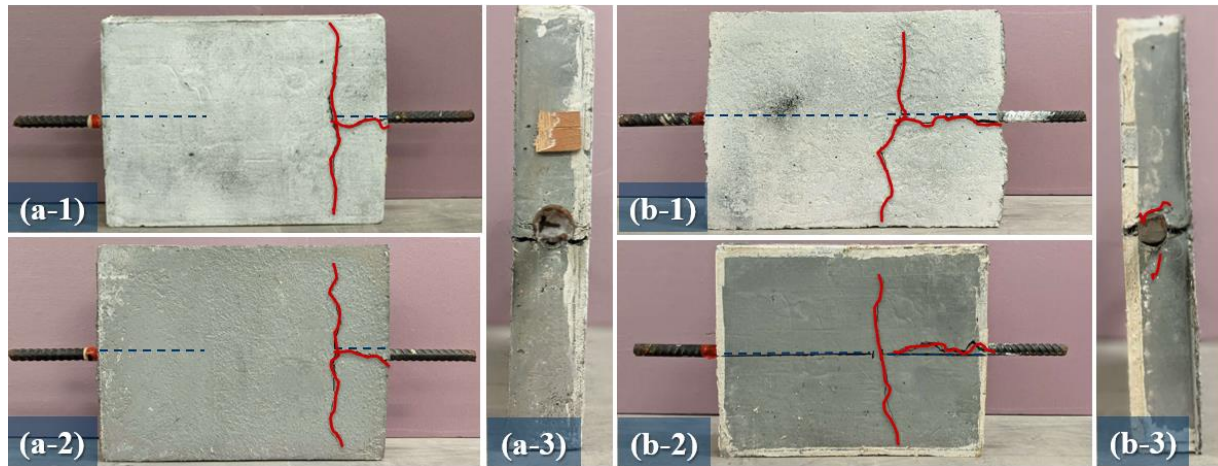


Figure 6.14: Failure of specimens (a) G1-5 and (b) G1-10. (The red lines represent the cracks and the blue dashed lines the embedment length).

For specimens with wider cover in case of reduced anchorage lengths (G2-5), the failure was again a combination of splitting and matrix tension in the central region between the test and support bars. However, the splitting cracks were narrower and did not penetrate along the whole length of anchorage as illustrated in Figure 6.15. Transverse tension cracks were more prominent, whereas for G2-5-B, multiple diagonal cracks are also formed initiating from the main transverse tension crack near the bar's end towards the loaded end. The inclined cracks are formed in an angle of 58° , which can be used to determine the angle between principal tensile and compressive stresses generated in the concrete ring due to the radial bursting pressures.

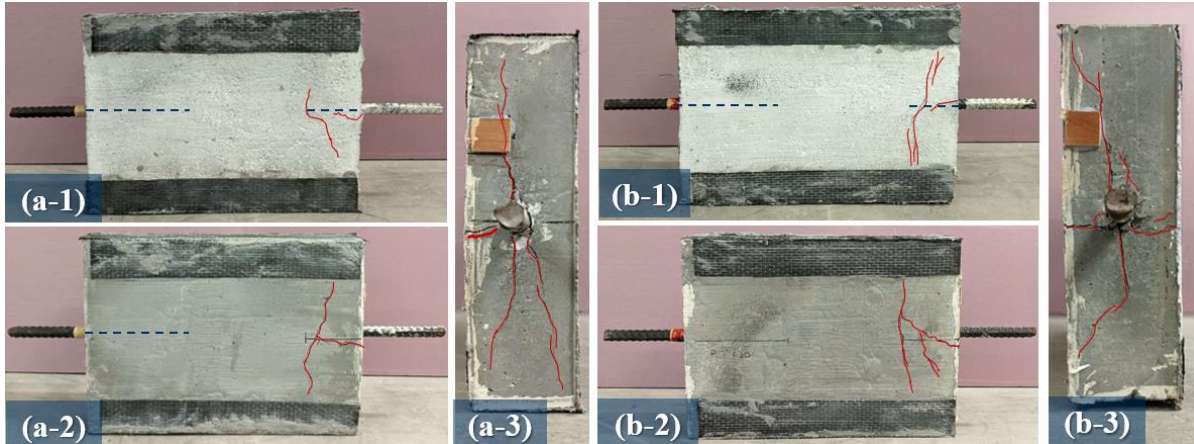


Figure 6.15: Failure of specimens (a) G2-5-A and (b) G2-5-B. (The red lines represent the cracks and the blue dashed lines the embedment length).

Finally in case of increased cover and longer anchorage (G2-10), bond failure did not happen. Instead, the load surpassed 80 kN, which corresponds to the load yielding the bar. After this point the reinforcement entered the hardening range and in this stage tension failure occurred in the central region between test and support bars. In case of G2-10-A, after yielding of the bar a narrow and short splitting crack emanated from the loaded end, whereas later in the experiment, a horizontal crack near the end of the anchorage formed from the one side of the specimen. The failure was identified as tensile failure of the matrix, while the load dropped abruptly to 20% of the maximum and thus the experiment was terminated. To avoid this type of failure and to be able to get the full response, all the other specimens with clear cover of $2D_b$ were externally reinforced using CFRP sheets on the sides to prevent the premature failure of the central zone in tension. Specimen G2-10-B, after yielding of the bar, in the hardening zone, failed by forming splitting crack and tension failure in central zone, with the latter to be more prominent.

6.4.2. Load-Slip Curves and Average Bond Strength

Figures 6.16 and 6.17 show the load-slip curves of the specimens with $5D_b$ and $10D_b$ embedment length, respectively. As already defined above, slip is the relative translation of the bar, calculated as the difference between the accumulated length change of the bar from that of surrounding concrete matrix over the same distance (=embedment length). This can be interpreted as the difference between the LVDTs that recorded the vertical translation of the bar and the concrete at the loaded end. However, due to the applied tensile load on the bar, elongation of the free portion of the latter has to be accounted for and subtracted from the LVDT reading accordingly. For cases

where the bar attained elastic response, namely did not exceed its yielding strength, the elongation in the free part of the bar, (ΔL_{bar}), is calculated as follows:

$$\Delta L_{bar} = \frac{\sigma_s}{E_s} \cdot GL_{bar} = \frac{F_b}{E_s \cdot A_b} \cdot GL_{bar} \quad (6.8)$$

In Equation (6.8) σ_s is the stress developed on the bar due to the applied load P , E_s is the Modulus of Elasticity of steel bar, A_b is the bar cross-sectional area and GL_{bar} is the gauge length between the point on the platform which is fastened around the bar and the point where the embedment starts. For the cases in which the bars surpassed their yielding point, the elongation is calculated differently. More specifically, after yielding for the plateau and hardening stage, the corresponding strain of the bar from the stress-strain curve of the material is multiplied with the aforementioned gauge length. In case where tension failure occurs in the concrete block (in the central region between test and support bar) prior to tensile rupture of the test bar, then the test bar's elongation follows a descending branch with a slope with magnitude equal to the initial modulus of elasticity of the bar having a negative sign.

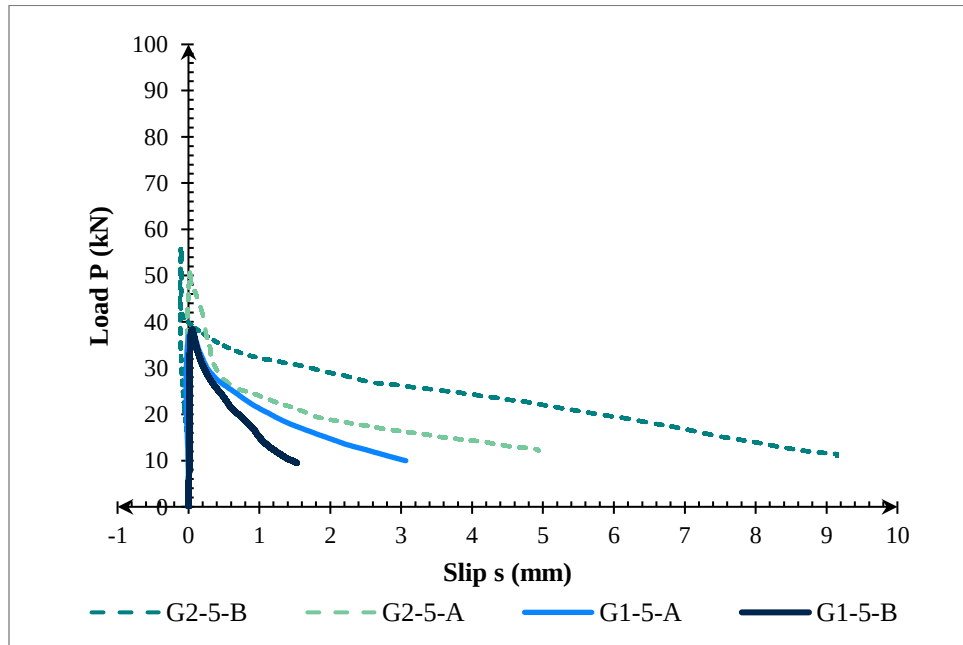


Figure 6.16: Load-slip curves for DTP specimens with G1-5 and G2-5 specimens

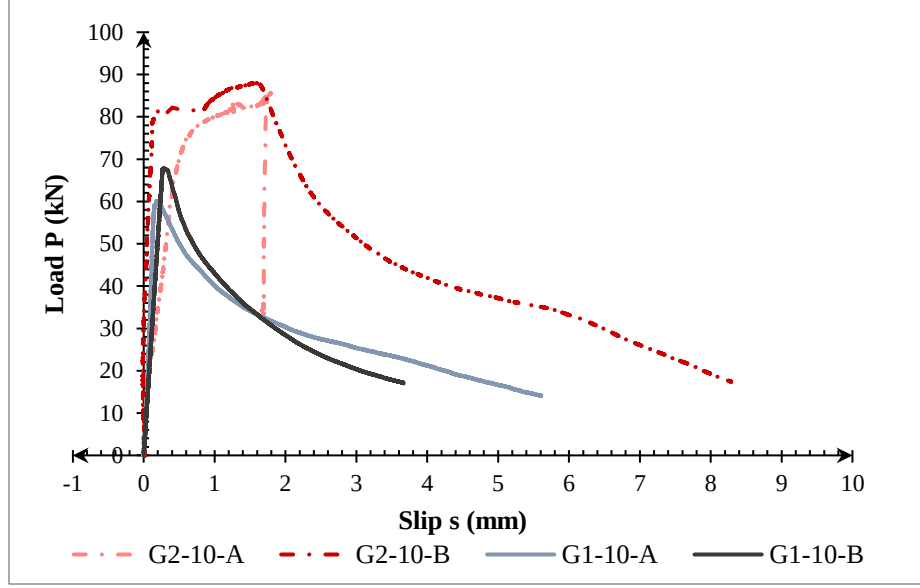


Figure 6.17: Load -slip curves for DTP specimens with G1-10 and G2-10 specimens

Figure 6.18 shows the average bond stress calculated for all specimens by dividing the applied load F_b , with the contact perimeter of the bar as shown by Equation 6.9:

$$f_b \approx f_{b,ave} = \frac{F_b}{\pi \cdot D_b \cdot L_b} \quad (6.9)$$

The highest average bond stress is observed for the $2D_b$ clear concrete cover and $5D_b$ embedment length. For the same cover, increasing the embedment length yielded lower average bond stress, which is explained by the nonlinearity of the bond stresses in longer embedment lengths, while in case of $2D_b$ clear concrete cover and $10D_b$ embedment length, the reinforcement yielded prior to the occurrence of bond failure. The experimental average values are plotted in Fig. 6.18. The highest maximum average stress estimates (obtained for $5D_b$ embedment) are considered as nearest to the bond strength values – 9.5 MPa for a cover $C = 1D_b$, and 13.3 MPa for a cover $C=2D_b$. Considering the average compressive strength $f_c=50$ MPa, the above values correspond to $f_{b,1Db}=1.34\sqrt{f_c}$ and $f_{b,2Db}=1.88\sqrt{f_c}$ respectively. It is noted that these values are within the same range as the bond strengths estimated from $5D_b$ embedment DTP tests for UHPFRC by Tsiotsias & Pantazopoulou, (2021b) and from beam tests by Saikali et al., (2022).

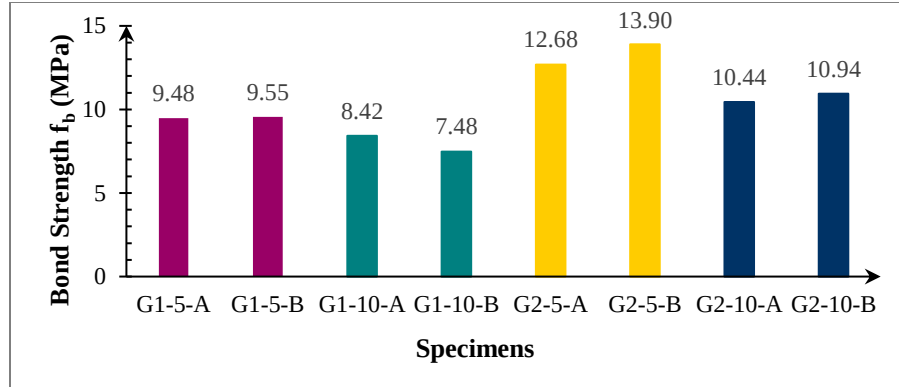


Figure 6.18: Average Bond Strength for all DTP specimens

6.4.3. Load-Splitting Crack Width Curves

Figures 6.19 and 6.20 illustrate the load versus longitudinal crack opening curves for the two different embedment lengths, as obtained from the transverse DT. Dashed curves represent the cases with cover $C=2D_b$. Specimen G1-5-A is missing from Figure 6.19 due to an experimental error regarding the placement of the transverse DT. The location of the DT is in the vicinity of the loaded end of the bar. As it is observed from both graphs, the displacement in the horizontal direction is more prominent in the post-peak range, and it is greater in case of specimens G1-10. This is expected as the reduced cover promotes the development of splitting crack, and the longer embedment enables redistribution of bond stresses and therefore larger amount of reinforcement slip and crack opening attained. However, in case of G2-10A, the longitudinal crack opening is insignificant, as the bar failed outside the embedment, in tension.

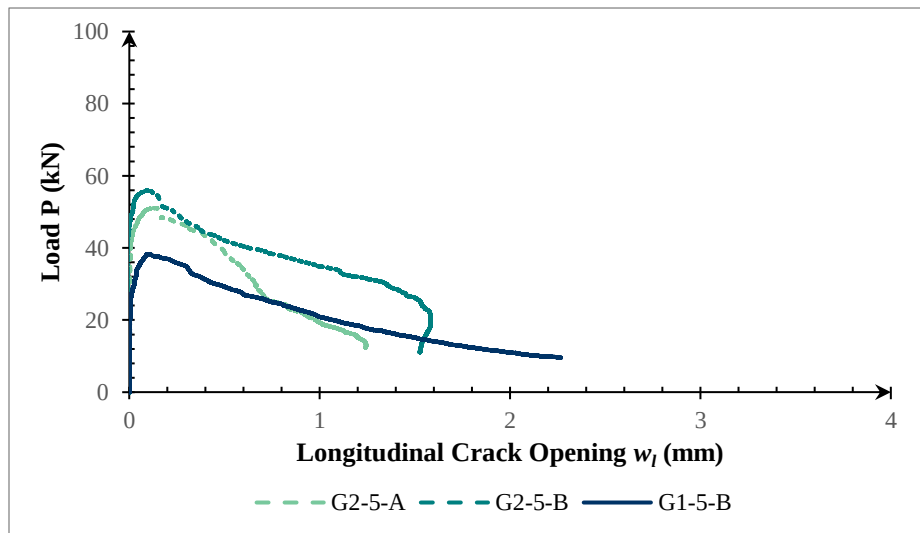


Figure 6.19: Load-splitting crack width for DTP specimens with G1-5 and G2-5 specimens

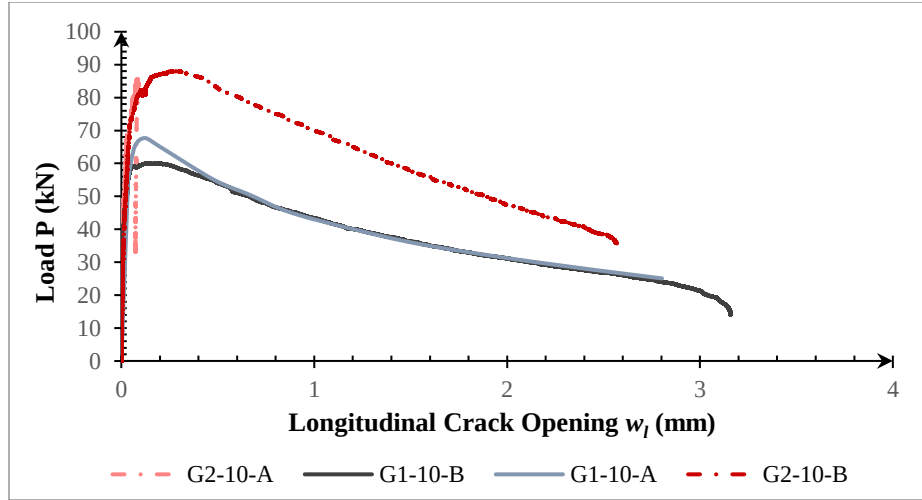


Figure 6.20: Load-splitting crack width for DTP specimens with G1-1- and G2-10 specimens

6.4.4. Digital Image Correlation Analysis

All specimens were further analyzed using Digital Image Correlation. Figures 6.22-6.29 illustrate the surface strains in horizontal and vertical direction for specimens G2-10-B, G1-10-B, G1-5-B and G2-5B. In each figure P1 and P6 are 40% of the peak load, in the ascending and descending branches, respectively, with P4 being the ultimate load. Then P3 is between P2 and P4. Finally, P2 and P5 are 80% of the peak load, in the ascending and descending branch, respectively. The points are illustrated in Figure 6.23.

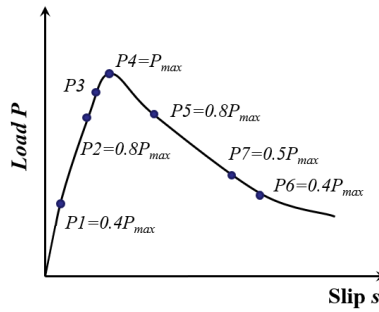


Figure 6.21: Characteristic Points of Load-Slip curve for DIC strain field plots

It is observed that in the horizontal direction, where the splitting crack occurs, before the maximum (P1-P3) there is some strain concentration in concrete in the proximity of the loaded end of the supporting bar. The latter is more evident in case of increased embedment length. Regarding the surface strain field in the vertical direction, where the pullout cracks can be observed, specifically for the increased embedment length, the first strain concentrations are observed in the loaded end of the support bar, but when the load approaches its maximum value, the localization moves closer

to the studied bar and the pullout crack is formed. In addition, multiple pullout microcracks are evident in that cases. Finally, as expected, the increased cover is effective in hindering the development of both splitting and transverse tension crack in the central part of the concrete block.

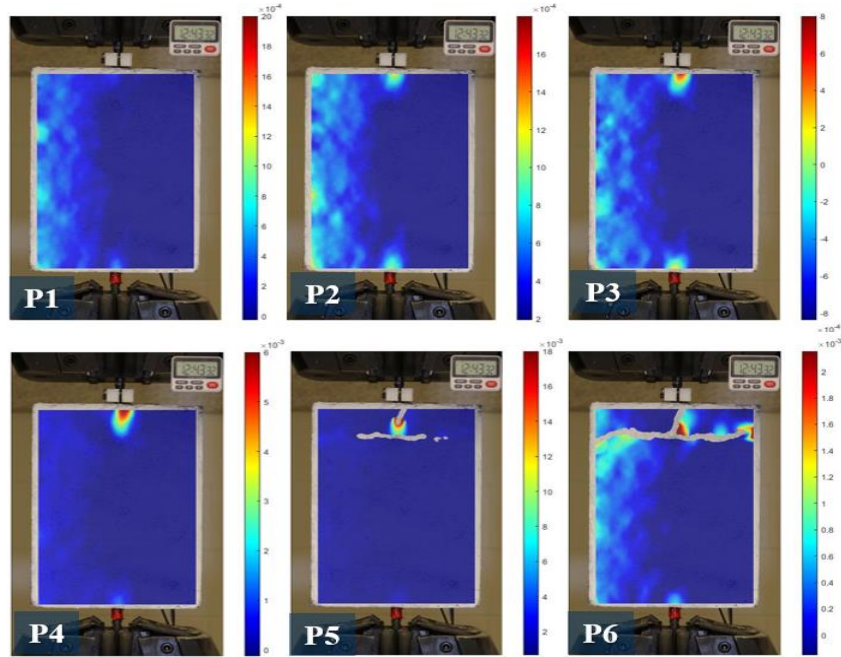


Figure 6.22: Surface strains ε_{xx} for specimen G1-5-A

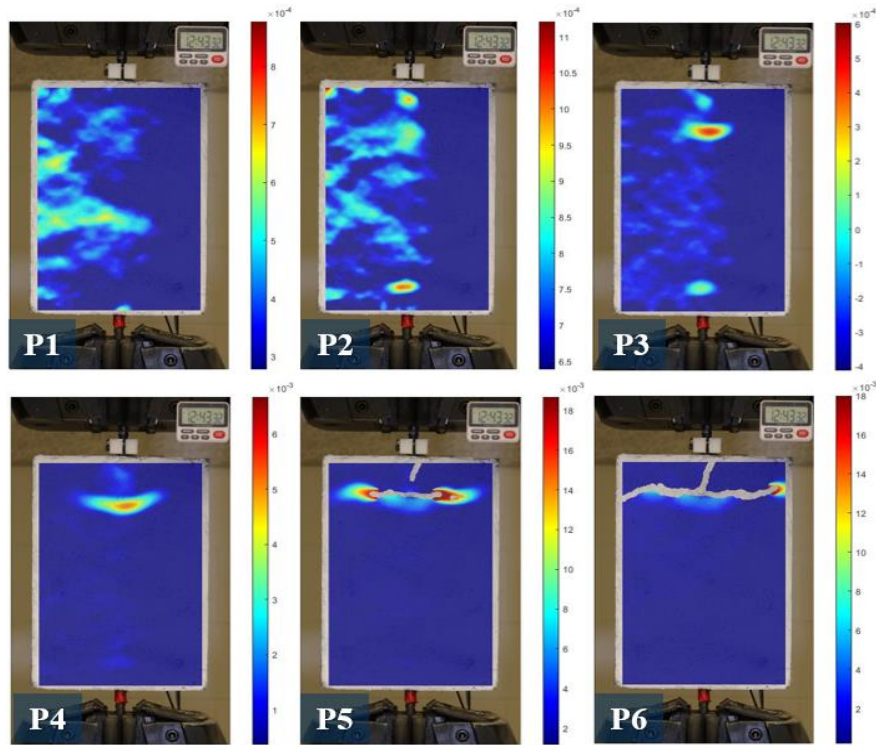


Figure 6.23: Surface strains ε_{yy} for specimen G1-5-A

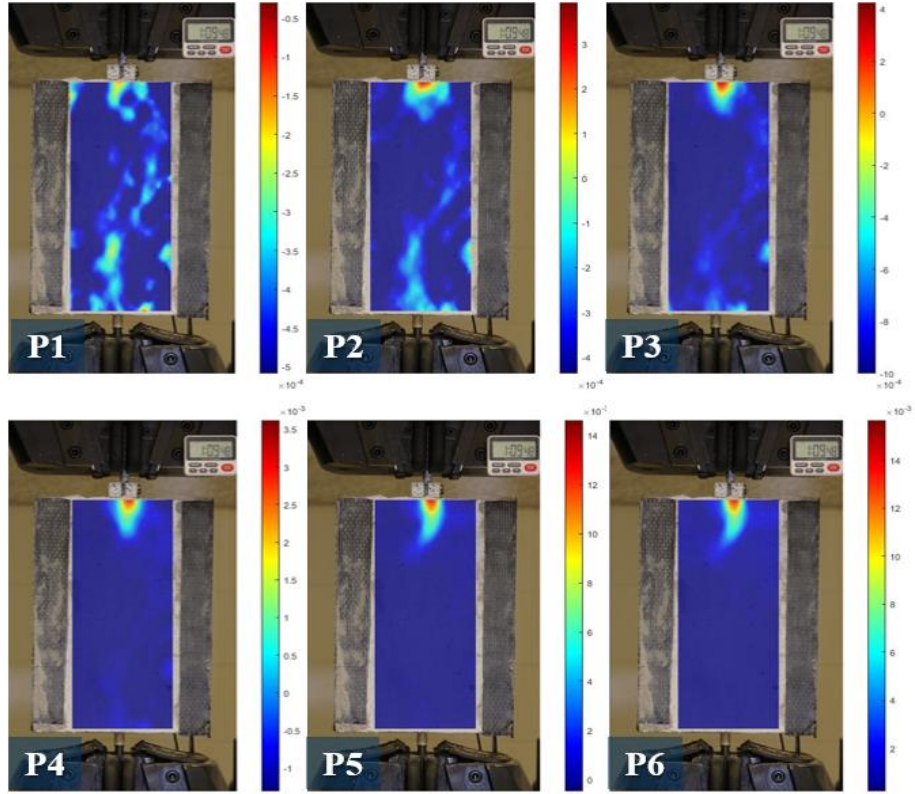


Figure 6.24: Surface strains ϵ_{xx} for specimen G2-5-A

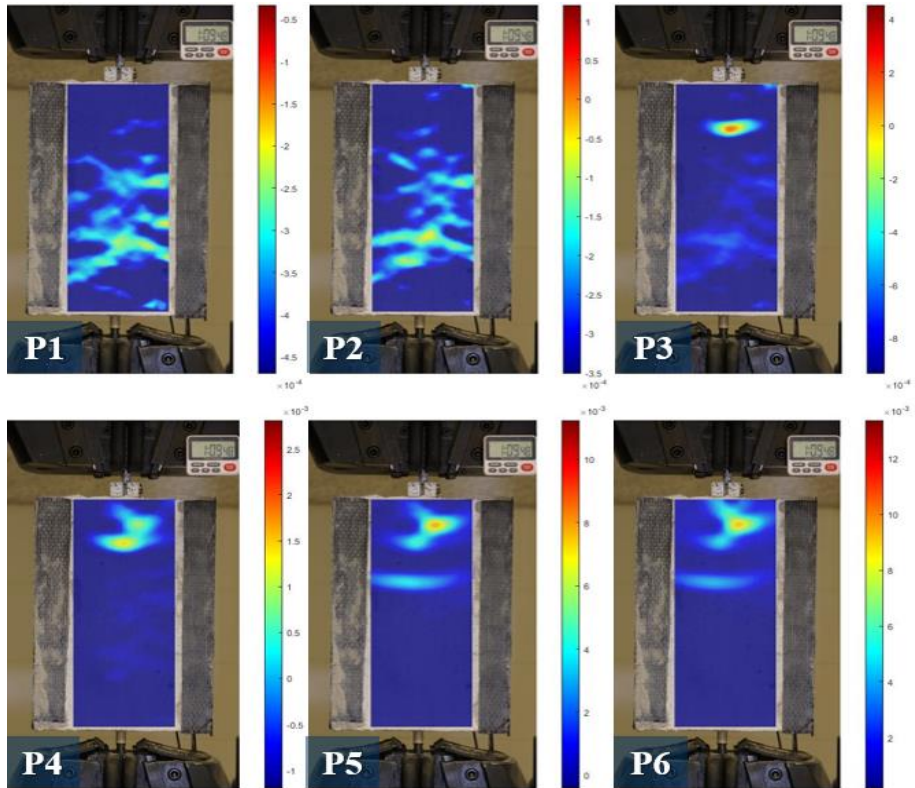


Figure 6.25: Surface strains ϵ_{yy} for specimen G2-5-A

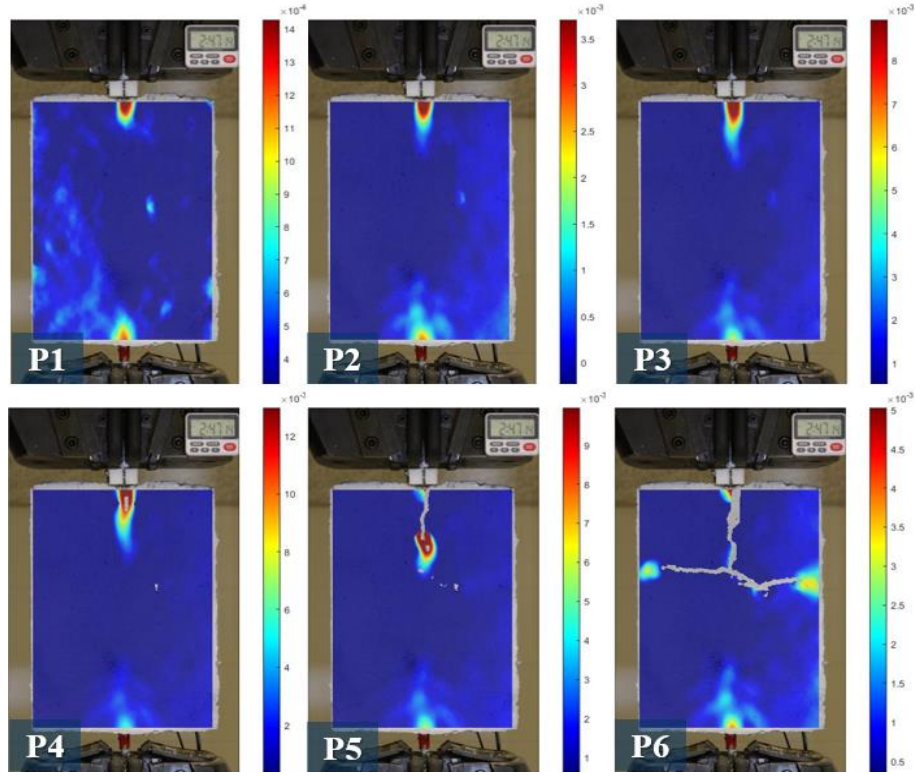


Figure 6.26: Surface strains ϵ_{xx} for specimen G1-10-A

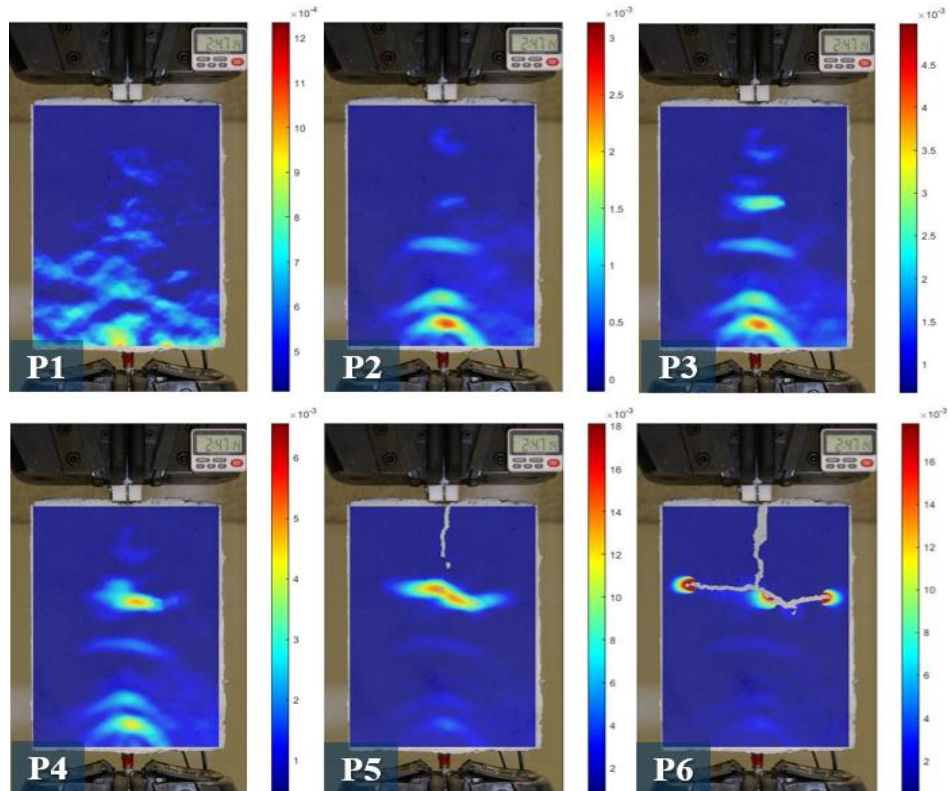


Figure 6.27: Surface strains ϵ_{yy} for specimen G1-10-A

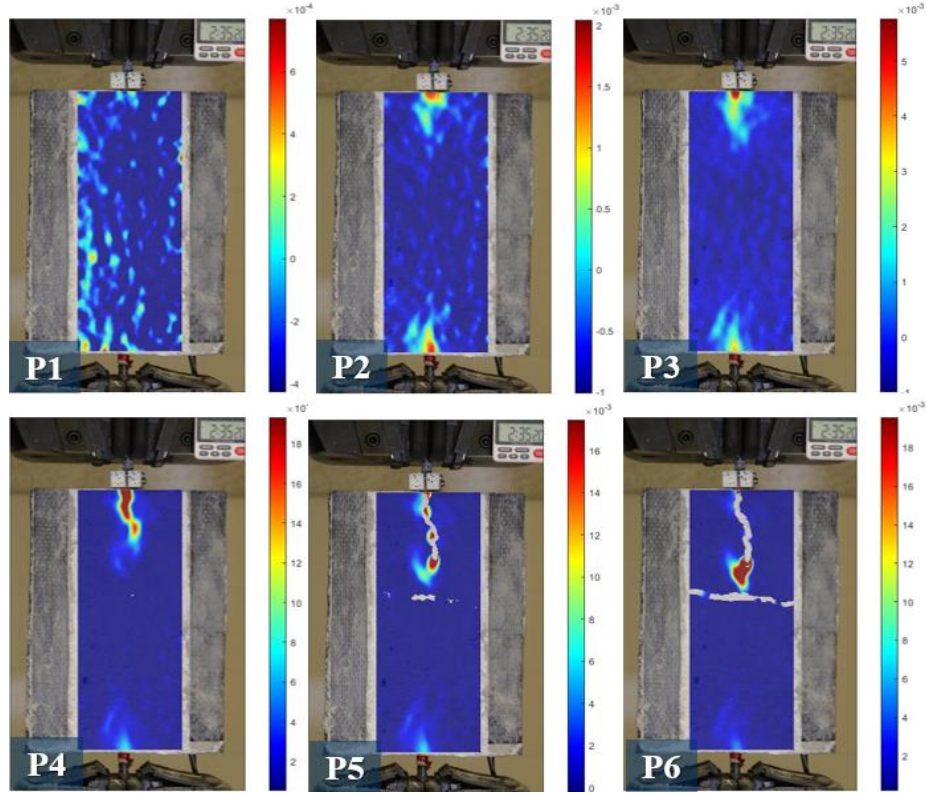


Figure 6.28: Surface strains ε_{xx} for specimen G2-10-B

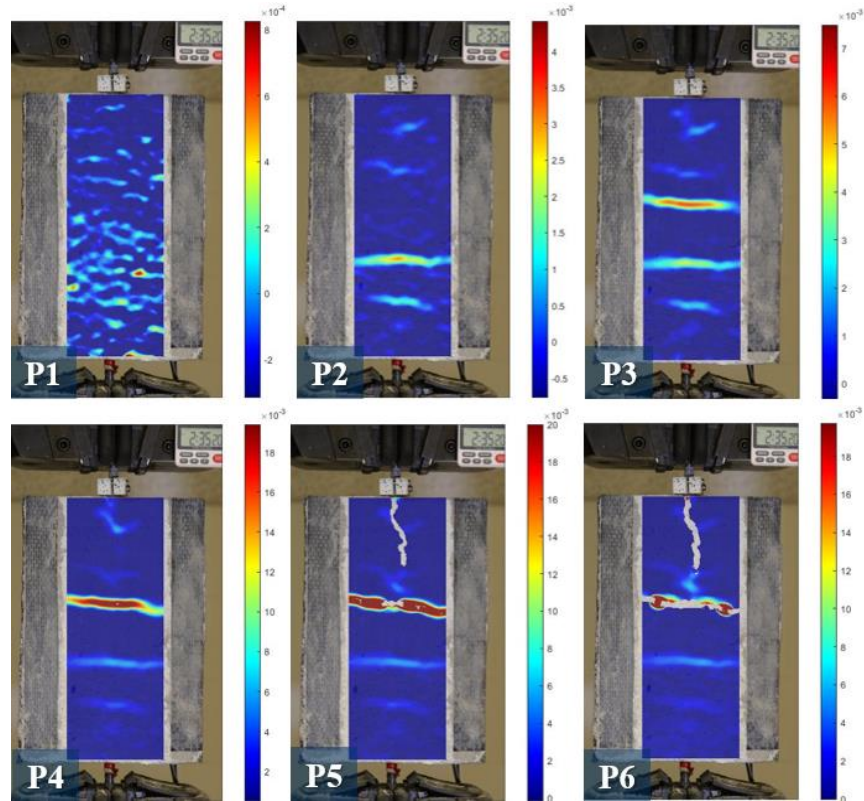


Figure 6.29: Surface strains ε_{yy} for specimen G2-10-B

DIC was further employed to determine the strain profile along the length of the bar and along the transverse tension crack. This was performed using the “Ncorr_post_CSTool” (Ncorr,) an open source DIC post-processing tool designed to extract and display strains in a cross-section. The cross sections used to obtain these strain profiles are illustrated in Figure 6.30. For the splitting crack, the averaging band width was set to be equal to the bar diameter (15 mm), while for the pullout crack, it was set at 13 mm, which is equal to the length of a steel fiber. The strain profiles were determined at three load stages, i.e., at 80% of the ultimate pre-peak, at the ultimate, and at 50% at post peak (Figure 6.21).

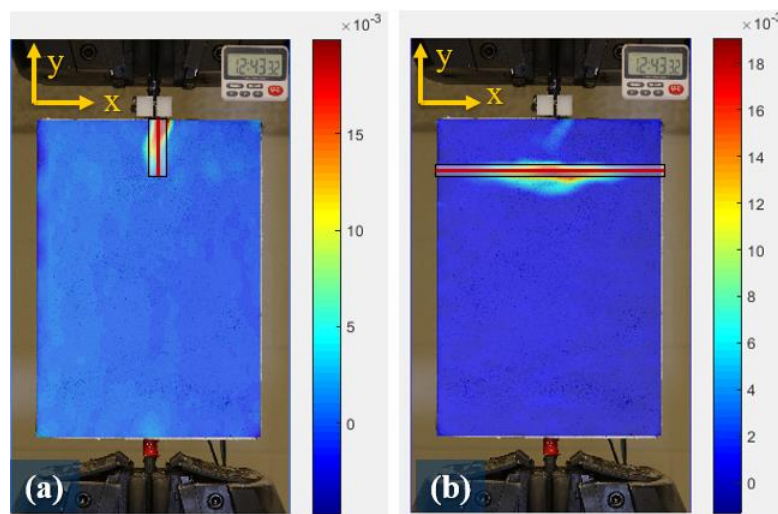


Figure 6.30: Cross sections for strain profiles (a) along the length of the bar, (b) transverse to the bar, at the location of the expected tension crack

Figures 6.31-6.34 show the strain distribution of concrete along the bar and along the expected transverse crack for the four different cases examined. In Fig. 6-31 it is observed that concrete is engaged near the loaded end while the load is before peak. Then the strains at the loaded end diminish indicating that the bar has debonded from the concrete at the peak and strain penetration has occurred over a length of at least 16mm, pushing the bar development towards the end of the anchorage. In the lower set of graphs, the x axis is oriented in the transverse direction, and the tensile strains in the direction of the bar axis are depicted in red (along the y axis): note that there is an almost symmetric distribution of normal strains (still compressive in the ascending branch), which become tensile at the peak and post-peak, signifying the opening of the transverse crack below the free end of the bar, and being maximum at the central axis of the specimen, while attenuating with transverse distance from the bar.

It is noted however, that for thicker covers strain penetration does not occur; the concrete cover is engaged throughout the test; in the case of longer anchorage and thin cover the strain penetration reaches 48 mm ($3D_b$) at peak load, whereas in the case of the thick cover and long embedment strain penetration propagates even further (to $4D_b$) but beyond bar yielding, the whole system unloads since localization occurs outside the specimen (in the free part of the loaded bar).

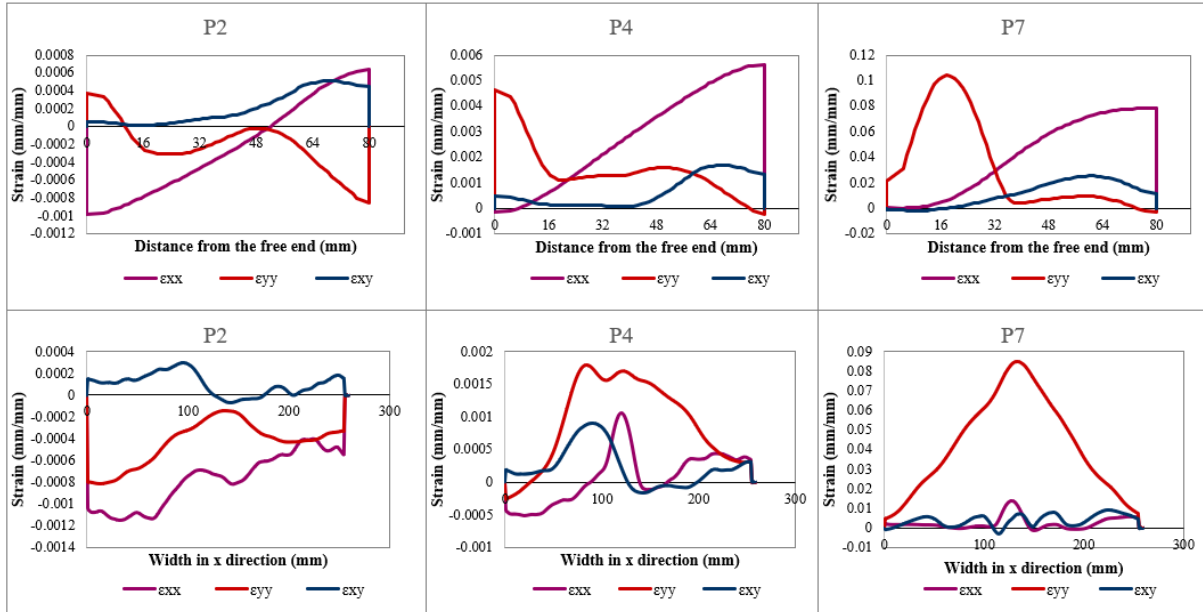


Figure 6.31: Strain profiles at different load stages along the length of the bar (first row) and along the length of the transverse crack (second row) for specimen G1-5-B

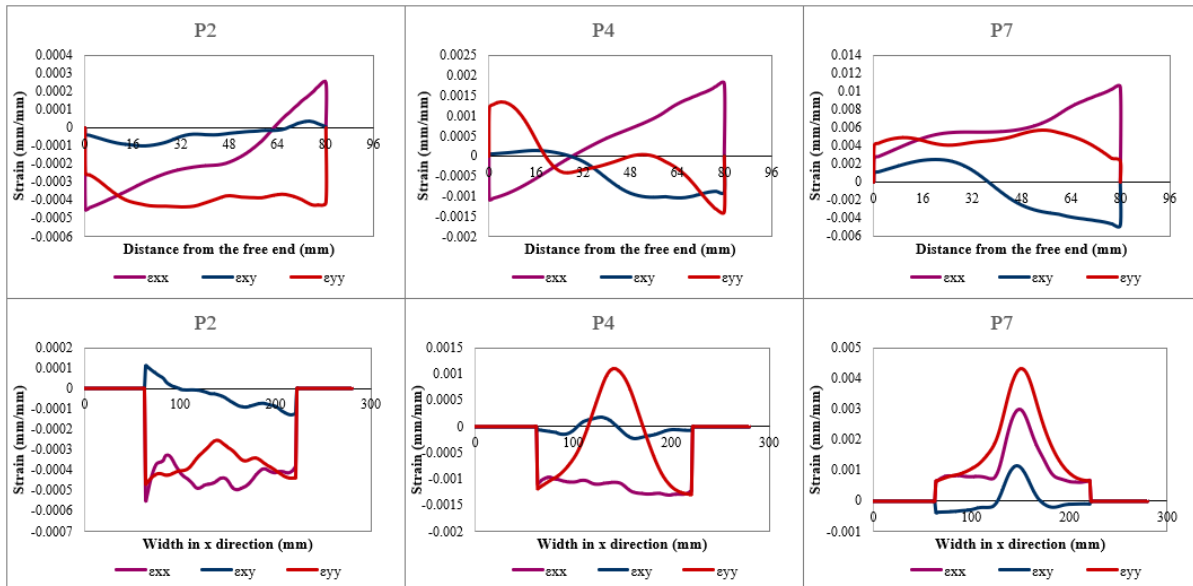


Figure 6.32: Strain profiles at different load stages along the length of the bar (first row) and along the length of the pullout crack (second row) for specimen G2-5-A

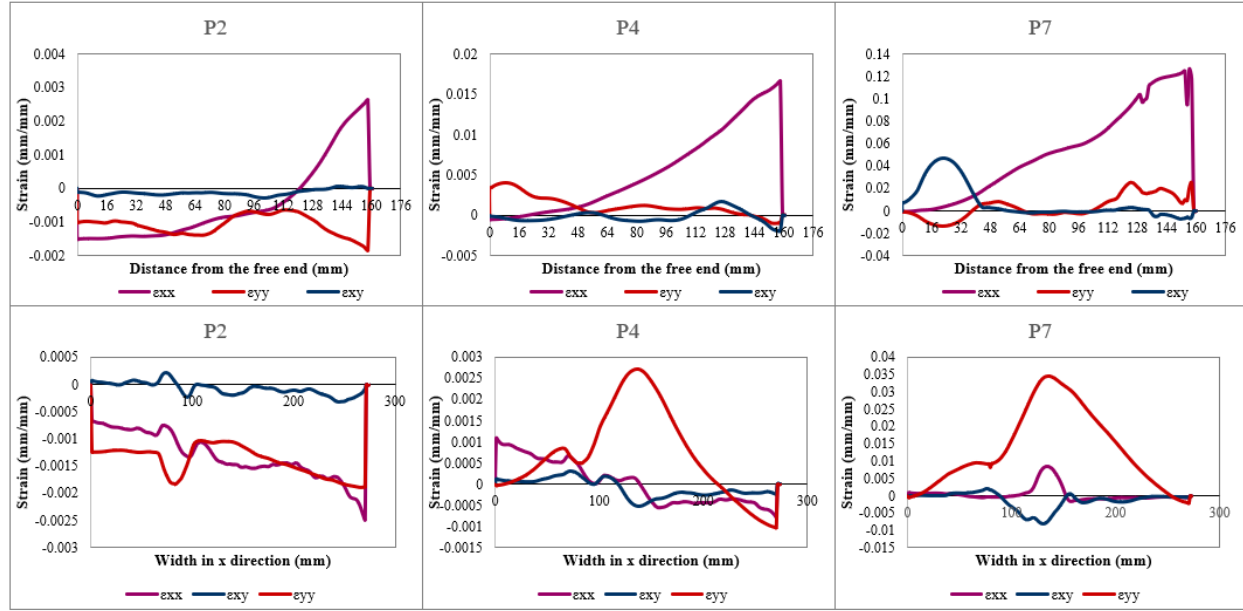


Figure 6.33: Strain profiles at different load stages along the length of the bar (first row) and along the length of the pullout crack (second row) for specimen G1-10-A

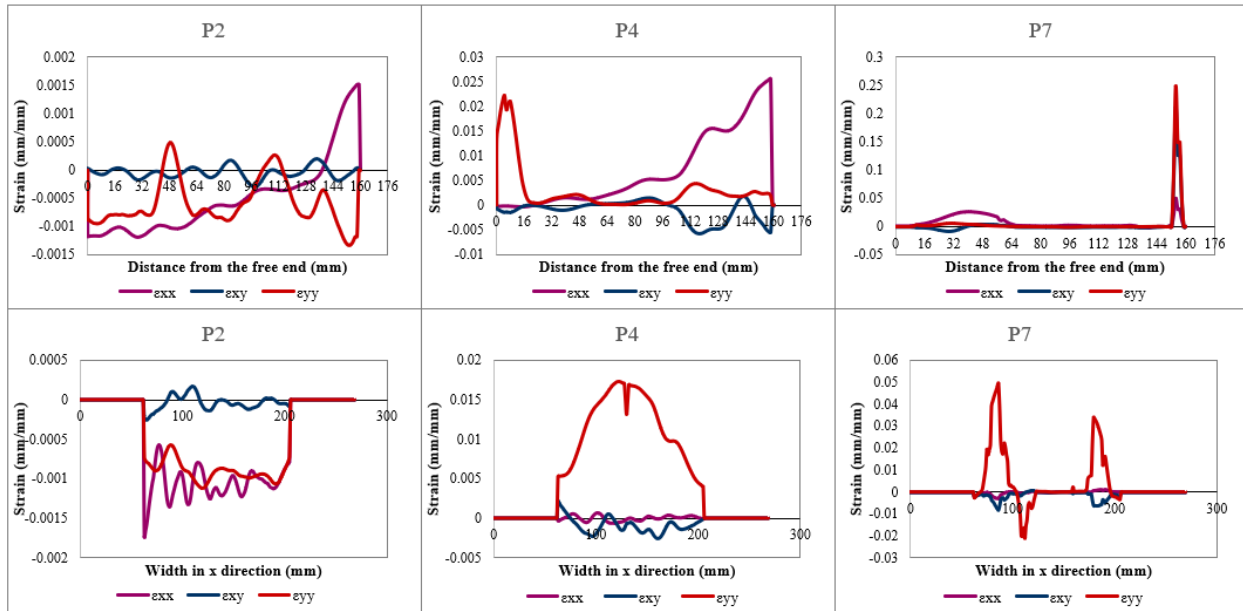


Figure 6.34: Strain profiles at different load stages along the length of the bar (first row) and along the length of the pullout crack (second row) for specimen G2-10-B

6.5. Conclusions

The experimental study presented in this chapter documents the bond behavior of deformed bars embedded in geopolymer THFRC matrix and anchored in a tensile stress field. In a DTP test bond occurs in the most unfavorable conditions, where the surrounding cover is also subjected to

longitudinal tension. The parameters studied were the anchorage length of the bar as well as the concrete cover thickness. The failure mode in all cases with exception to double cover and embedment length occurred by splitting along the bar and some partial strain penetration; in the case of the thicker cover, an embedment length of $10D_b$ was sufficient to yield the bar. Bond strength was estimated from the tests with shorter anchorage lengths ($5D_b$) - which are consistent with the literature on bond (fib Bulletin 65, 2010; ACI-408, 2003; RILEM, 1982; RILEM/CEB/FIB, 1978); values obtained were, $1.34\sqrt{f_c}$ for a cover of $1D_b$ and $1.88\sqrt{f_c}$ for a cover of $2D_b$. Slip s_l (end of the linear range of bond) was estimated at first attainment of peak load sustained as 0.05 mm.

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Chapter 7: Strength of Geopolymer THFRC Under Biaxial Stress States

7.1. Introduction

The superior tensile behaviour of THFRC in general is linked to enhanced shear capacity, which is of great significance for structural design. Although there are shear design provisions for conventional reinforced concrete in codes and standards, the so-called concrete contribution term which accounts for the strength of the material in diagonal tension might be highly conservative. Differences from conventional concrete exist on account of the presence of fibers (i.e., the internal confinement of the material and the sustained tensile strength up to high levels of tensile strain), and the lack of coarse aggregates which are associated with aggregate interlock.

As in the case of bond, pre-requisite for implementation of geopolymer THFRCs in structural design is evidence regarding their behaviour under general states of stress in structural members which invariably include some shear action. The experimental determination of response under general states of stress is possible by direct shear/push-off tests and by flexural tests. In this chapter, a series of push-off specimens with varying angle of the angle of interface plane were tested. The results of these tests are later used to develop a Mohr-Coulomb type failure criterion, and a more general biaxial failure envelope. Digital Image Correlation (DIC) is further used to analyze the state of failure on the specimens tested.

7.2. Description of the experimental procedure

7.2.1. Investigation Parameter-Varying Angle of Shear Plane

Four different types of push-off specimens were designed with studied parameter the angle of shear plane, so as to vary the state of stress. By varying the angle different combinations of shear and normal stress at the plane failure occur, which will further be used to derive the failure envelope of the THFRGC. The values of the angle considered ranged from 0 to 63°, while the sliding plane was set to be between 8400-8900 mm². For each angle value, 2 specimens are fabricated and tested. The geometry of the four different types are given in Figure 7.1, while Table 7.1 shows the labels corresponding to each specimen. For complete definition of the failure envelope the data obtained

earlier from material characterization under uniaxial compression and uniaxial tension will also be considered.

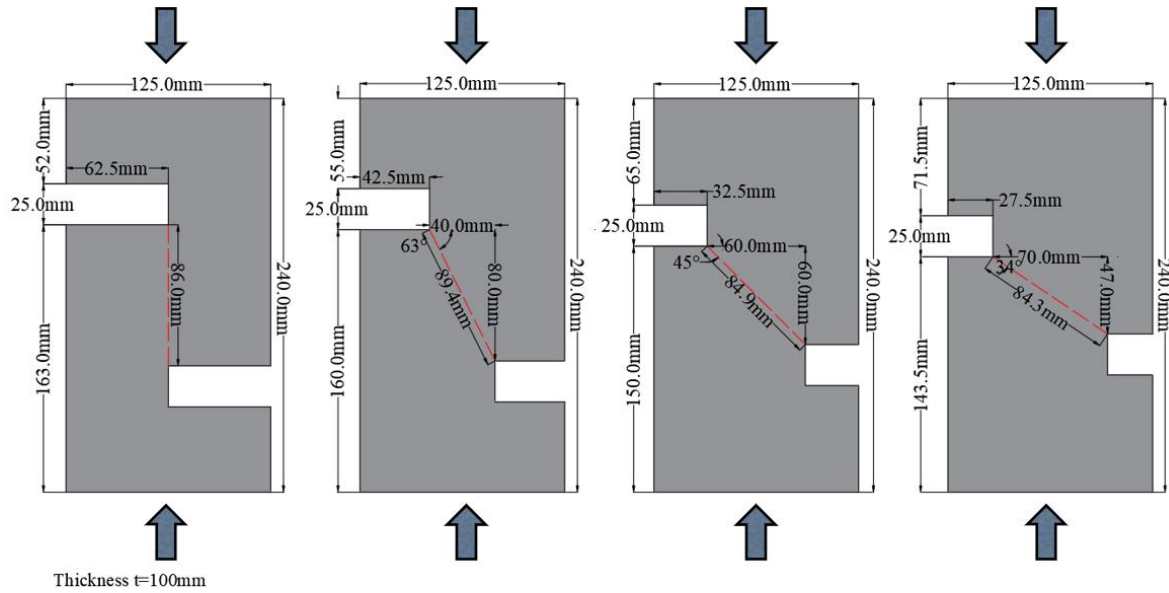


Figure 7.1: Geometry of push-off specimens

Table 7.1: Specimen code names and parameter specifications for push-off tests

Specimen ID	Varying Angle κ (°)	Stress-State
PS-A	90	Pure Shear
PS-B		
CS63-A	63	Compression Shear
CS63-B		
CS45-A	45	Compression Shear
CS45-B		
CS34-A	34	Compression Shear
CS34-B		

7.2.2. Preparation of the Experiments

The mix design used in this Chapter is GPC4-F described in Chapter 3 (Table 3.13) and fully characterized in terms of material behaviour in Chapter 5. For all the eight specimens, the same type of wooden formwork was used and was only modified through the use of inserts to create the slits shown in Figure 7.1 based on the studied angle. All molds had the same external dimensions and were subsequently adjusted using rigid foam where needed, to adjust the width of the concrete block. The inserts were 3-D printed plastic parts of different dimensions and are also attached on the molds with hot glue. To achieve a smooth demolding red sheathing tape was used on the inner surfaces of the formwork, the inserts, and the rigid foam pieces. A descriptive arrangement of the formwork is shown in Figure 7.2, while Table 7.2 describes the annotation of the elements

comprising the mold. Prior to casting, all faces were lubricated using sunflower oil to ensure a smoother surface for the specimen.

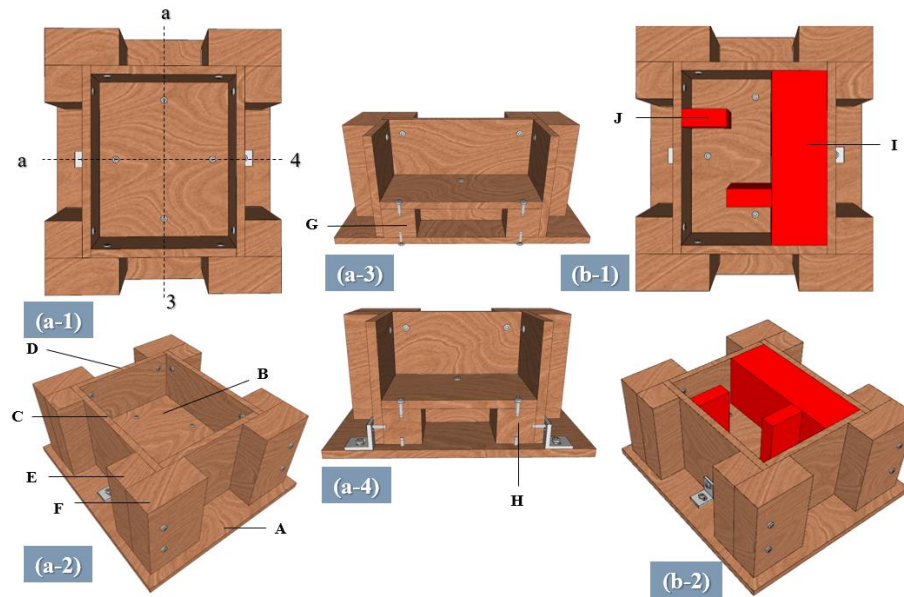


Figure 7.2: Different views of formwork design: (a-1) Plan view (a-2) perspective view, (a-3) side view in a-a section, (a-4) side view in a-a, (b-1) Plan view of modified formwork, (b-2) perspective view of modified formwork

Table 7.2: Annotative description of Push-Off Mold parts shown in Figure 7.2

Annotation	Description	Dimensions (mm) [0/63/45/34]
A	Base plate	360 x 360 x 11.9
B	Front face panel	240 x 200 x 11.9
C	Side plates	263.8 x 162.7 x 11.9
D	Top/Bottom side plates	200 x 162.7 x 11.9
E	Square Support Pieces	50.8 x 50.8 x 162.7
F	Rectangular Support Pieces	50.8 x 101.6 x 162.7
G	Base Support Square Pieces 1	50.8 x 50.8 x 138.4
H	Base Support Square Pieces 2	50.8 x 50.8 x 200
I	Rigid Insulation Foam	75 x 240 x 100
J	Inserts	(62.5/42.5/32.5/27.5) x 25 x 100

Due to limitations in the mixer volume, two specimen molds were constructed for a different study angle, resulting in 4 pairs of identical specimens. Figure 7.3 illustrates an example of the finished formwork used in a typical casting.



Figure 7.3: Formworks for specimens CS63 and CS34

7.2.3. Mixing, Casting and Curing of Specimens

On account of capacity limitations of the mixer, the specimens were cast in different batches in four consecutive days. For maximum consistency, proportions, mixing procedure, mixing time, casting, and curing were strictly followed to be identical in all four batches. The mix design and procedure is described in detail in Chapters 4 and 5. As the placement method has proven to have a great impact on mechanical properties, particularly positive when fiber alignment is achieved, casting with flow from one end was employed for the push-off specimens. Half of the specimens were cast with flow from one side whereas the rest were cast with flow from the bottom as illustrated in Figure 7.4. After casting external vibration was applied by placing the specimens on a vibrating table for 30 seconds at mild vibration levels. Then the specimens were covered with plexiglass sheets and an external plastic sheet to prevent evaporation. After 24hr, the specimens were demolded, wrapped in wet burlap sheets, placed in curing tanks which were further sealed with plastic sheets to prevent water evaporation. The specimens were moist cured for 28 days, while once a week a check on the moisture conditions was performed to ensure consistent curing till testing

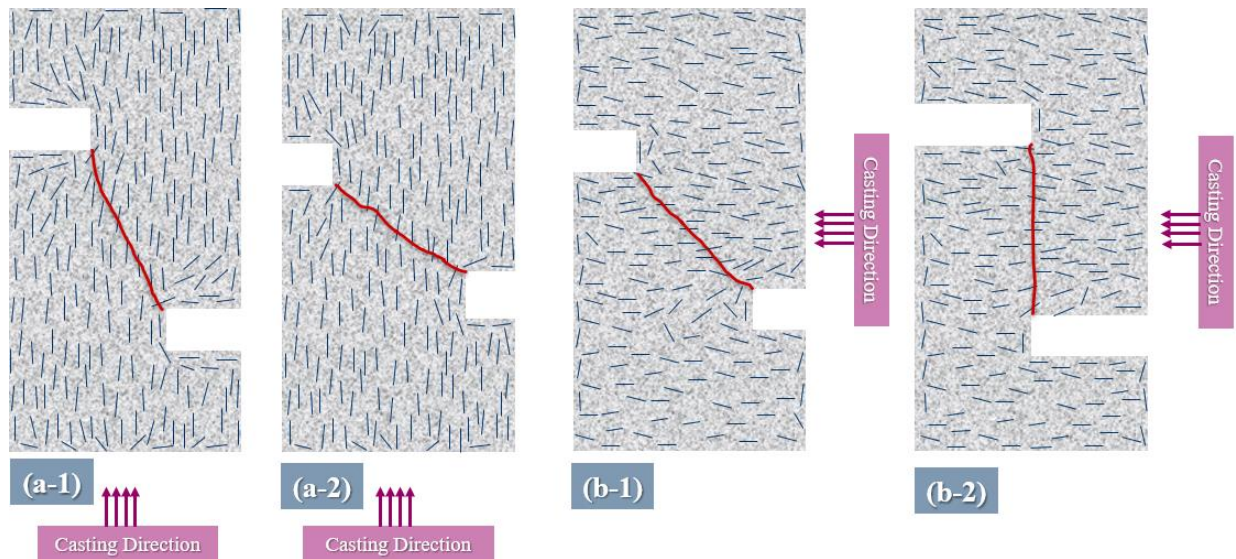


Figure 7.4: Fiber Distribution based on the placement methods used in this study along with the anticipated interface failure (a) flow from bottom OW1 (a-1: CS63, a-2 CS34), (b) flow from the side OW2 (b-1: CS45, b-2 PS)

7.2.4. Specimen Preparation, Instrumentation and Testing Equipment

Prior to testing, since no embedded steel reinforcement was used, to avoid failure due to tension/flexure in regions outside of the anticipated failure plane, the specimens were externally

reinforced using Carbon Fiber Reinforced Polymer (CFRP) sheets. The Material Properties of the CFRPs are listed in Chapter 5. The CFRP sheets were cut in ribbons of the required size with extra length in each side to account for adequate anchorage. Then in order to achieve a strengthening pattern, the sheets were glued on the specimen over designated areas as illustrated in Figure 7.5. For CS34 and CS45, if the transverse FRP was placed in the vicinity of the inserts, the anchorage would interfere with the anticipated failure plane. For this reason, the transverse FRPs are moved a few centimeters away from the edges of the recesses. For optimum bonding of the CFRP reinforcement, the specimens were left to dry for 24 hr. Then the front face of the specimen was painted white and black spray paint was used on top of the white background to create the speckle pattern required for DIC Analysis. Finally in order to ensure flat, smooth, and leveled loading surfaces, a layer of hydrostone plaster was applied on the top and bottom loading surfaces.



Figure 7.5: Push-off specimens wrapped with CFRPs

Before placing the specimen on the testing frame, the hardware required to support the instrumentation were attached on the sides and back face of the specimen. The instrumentation is shown in detail in Figure 7.6. Two Differential Transformers (DT) were used to record the vertical displacement of each side. The vertical displacement of the sliding plane is defined as the difference between these DT measurements. Since the base of the frame is moving, the DTs were fixed on magnetic base DT holders which were attached on the upper part of the frame which doesn't move. Two metal angles were glued at midheight of the sides in order to bear on the DTs. Finally, to record the sliding displacement projected in the horizontal axis x , a Linear Variable Differential Transformer (LVDT) was mounted on an aluminum prismatic piece, hot glued on the concrete parallel to the vertical axis of the specimen, while a metallic L bracket is glued on the other end of the LVDT for bearing. More details about the hardware components are shown in Table 7.3.

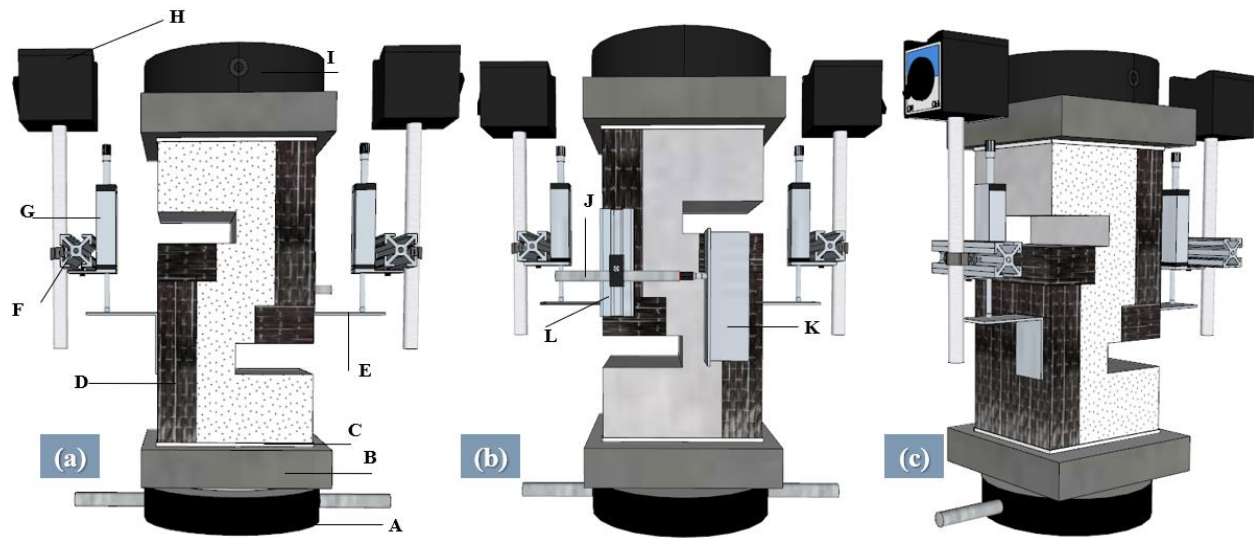


Figure 7.6: Schematic representation of experimental set-up: (a) front view; (b) back view and (c) perspective view

Table 7.3: Annotative description of experimental hardware components shown in Figure 7.6

Annotation	Description
A	Spherical Hinge Base
B	Loading Plate
C	Hydrostone Plaster Layer
D	CFRP sheet
E	L Bracket for DT
F	H-aluminum piece for mounting DT
G	DT
H	Magnetic Base Holder
I	Fix Compression Platen
J	LVDT
K	L Bracket for LVDT
L	H-aluminum piece for mounting LVDT

Figure 7.7 depicts the setup in the compression testing frame where the tests were performed under displacement controlled loading. The specimens were monotonically loaded to failure at a displacement rate of 0.15 mm/min until failure, or until the load carrying capacity of the specimen was reduced in the post-peak branch to 20% of the maximum attained load.

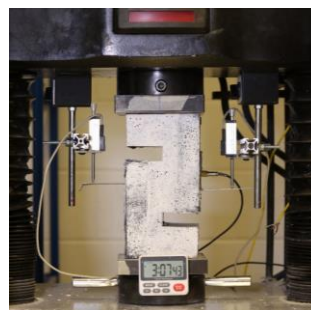


Figure 7.7: Experimental setup for Push-Off Tests

7.3. Experimental Results from the Push-Off Tests and Discussion

7.3.1. Modes of Failure

- *Pure Shear (PS)*

For pure shear specimens, before ultimate load was reached, multiple cracking formed around the shear plane, initiating from the corners of the recesses. Then the anticipated shear crack prevailed along the shear plane. After localization, compressive cracks started to emanate at the top and bottom ends of the main shear crack, while further in the descending branch some tensile cracks were created in the proximity of the support of the formed cantilever parts as shown in Figure 7.8.

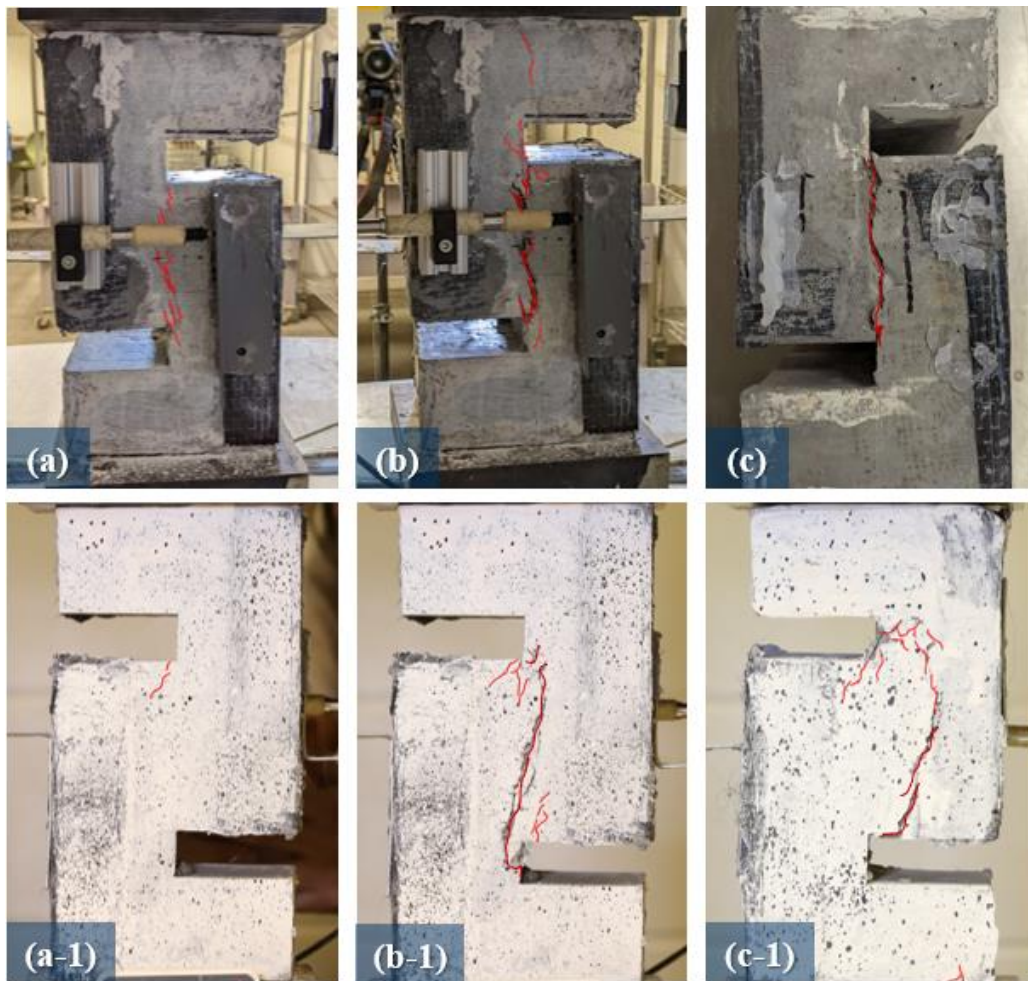


Figure 7.8: Crack formation (a) Initiation of Cracking on back face of PS-A (a-1: Front Side), (b) Failure Crack on back face of PS-A (b-1: Front Side), (c) Failure Crack on back face of PS-B (c-1: Front Side)

- *Compression Shear with $\kappa=63^\circ$*

Regarding CS63 specimens as illustrated in Figure 7.9, although tensile cracks on the fixed end of the formed cantilever above and below the recesses were created first, the failure along the sliding plane between the armpits of the two recesses dominated, while multiple cracks propagated through the localized shear crack. Compared to PS, CS63 specimens had wider crack openings and withstood higher deformation overall.

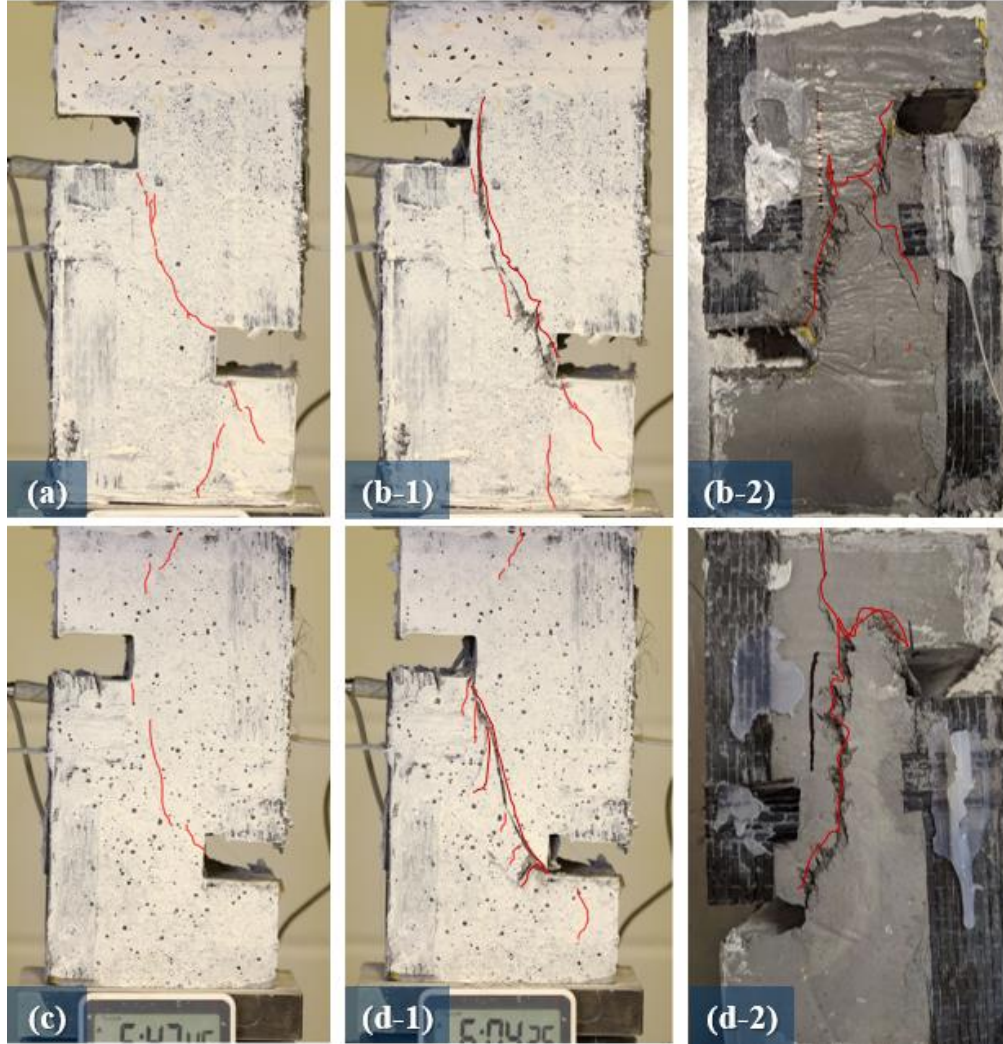


Figure 7.9: Crack formation. (a) Initiation of Cracking on CS63-A, (b-1) Failure Crack on CS63-A, (b-2) Back face of CS63-A, (c) Initiation of Cracking on CS63-B, (d-1) Failure Crack on CS63-B, and (d-2) Back face of CS63-B

- *Compression Shear with $\kappa=45^\circ$*

As the angle of the sliding plane is reduced, the failure becomes more compression-dominated (Figure 7.10). In case of CS45 specimens, higher loads and deformations were reached, whereas

the failure occurred as anticipated in the plane defined by the end recesses. The multiple shear cracks were also accompanied by multiple compression cracks. Finally, delamination of FRP and concrete spalling were evident.

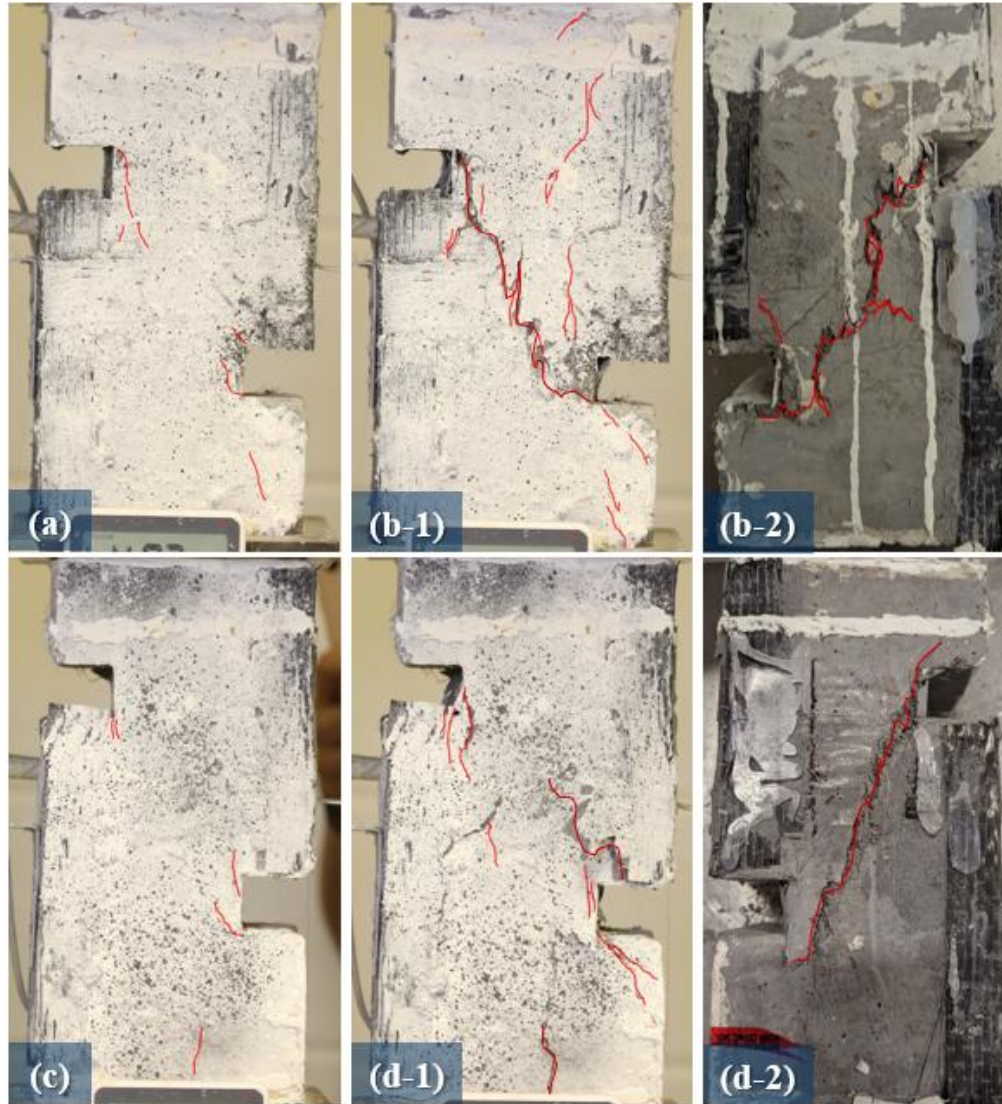


Figure 7.10: Crack formation. (a) Initiation of Cracking on CS45-A, (b-1) Failure Crack on CS45-A, (b-2) Back face of CS45-A, (c) Initiation of Cracking on CS45-B, (d-1) Failure Crack on CS45-B and (d-2) Back face of CS45-B

- *Compression Shear with $\kappa=34^\circ$*

For this case, one of the two specimens showed the anticipated failure in the plane of sliding defined by the recesses, developing multiple compressive cracks, whereas the second showed mainly compression and tensile cracks propagating from the support of the formed cantilever parts (Figure 7.11). The concentration of the compression cracks on the top of the specimen could be attributed to the casting method. It has been observed in previous chapters that flow from one end

(from the bottom) can create plane of weakness towards the other end where fewer fibers are found. Also, owing to the method of casting, the fibers were aligned parallel to the specimen's axis and thus they did not provide sufficient confinement in the ends by arresting compressive cracks which would run parallel to the fibers in that case. Other phenomena observed include delamination of FRPs, concrete spalling and excessive deformation, while failure was followed by a loud cracking sound.

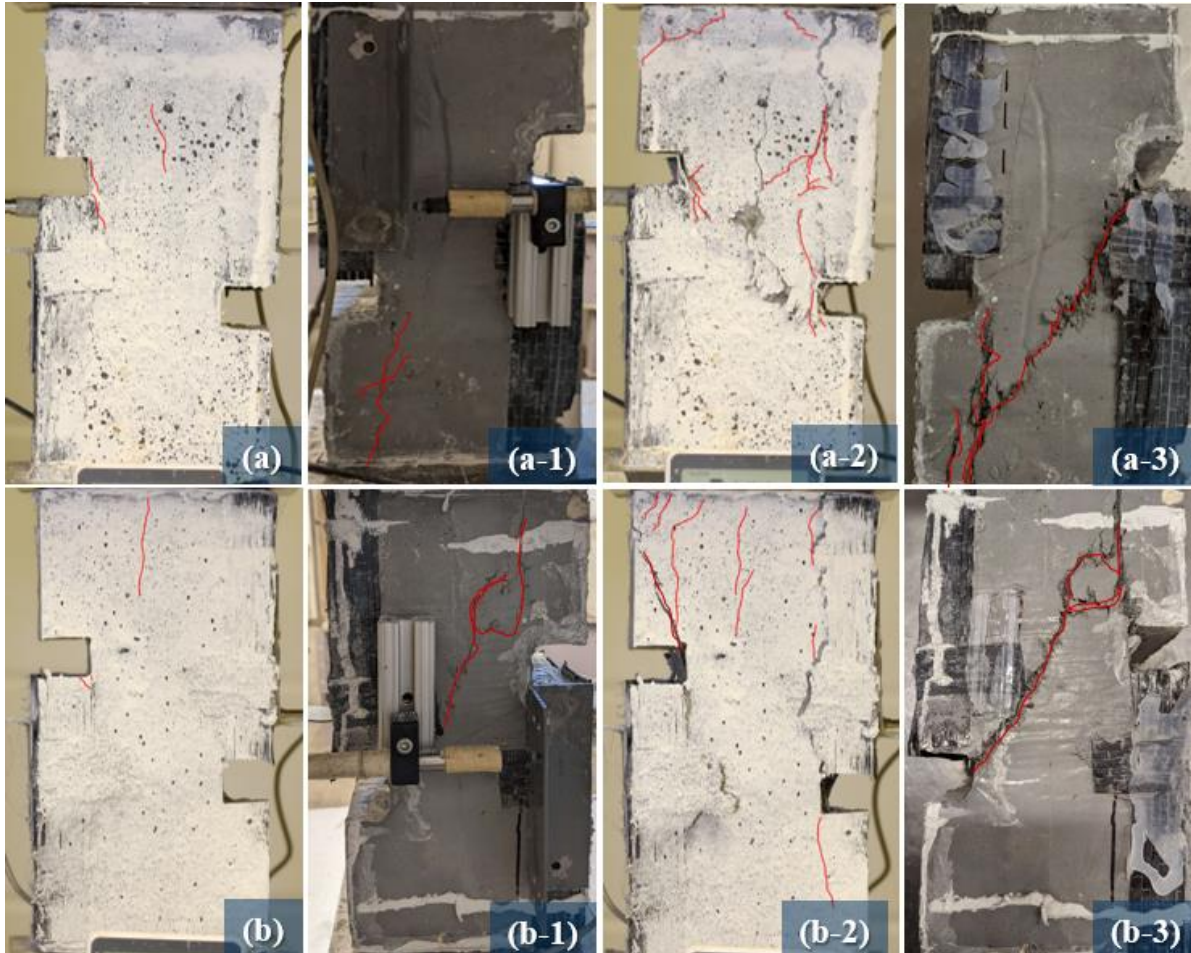


Figure 7.11: Crack formation: (a) Initiation of Cracking on CS34-A, (a-1) Back face of CS34-A at initiation of cracking, (a-2) Failure Crack on CS34-A, (a-3) Back face of CS34-A at failure; (b) Initiation of Cracking on CS34-B, (b-1) Back face of CS34-B at initiation of cracking, (b-2) Failure Crack on CS34-B, and (b-3) Back face of CS34-B at failure

7.3.2. Force-Deformation Behaviour

The force-deformation (lateral and vertical) response curves of the specimens with varying interface angle are plotted in Figures 7.12-7.15. All specimens exhibited softening prior to ultimate load. The onset of the descending branch is associated with crack localization. In this stage, the

main crack widens, and the fibers pull out slowly. It is evident from the plots that the decrease of the angle of the interface is correlated to an increase in the stiffness prior to localization. It is also observed that the ultimate load increases with the decrease in the angle of the sliding plane on account of the contribution of compressive stresses imparted on that plane by the angle of failure, except for the case CS34. This is further evidence on the significance of casting method, as in the case of CS34 where the fibers are expected to be oriented parallel to the axis of the specimen and thus not able to arrest compressive cracks.

- *Pure Shear (PS)*

For PS specimens, the maximum average load was 96 kN with the corresponding vertical and lateral displacement of the shear plane being 0.83 mm and 0.57 mm, respectively. The end of the elastic range is distinct in the force-horizontal displacement curve and corresponds to an average value of 55 kN which is 57% of the peak load and 0.05 mm. This cardinal point represents the apparent first cracking and is also evident in the force-vertical deformation curves as the change of slope at the end of the elastic portion. The post-peak capacity is 27% of the maximum load, namely 26kN.

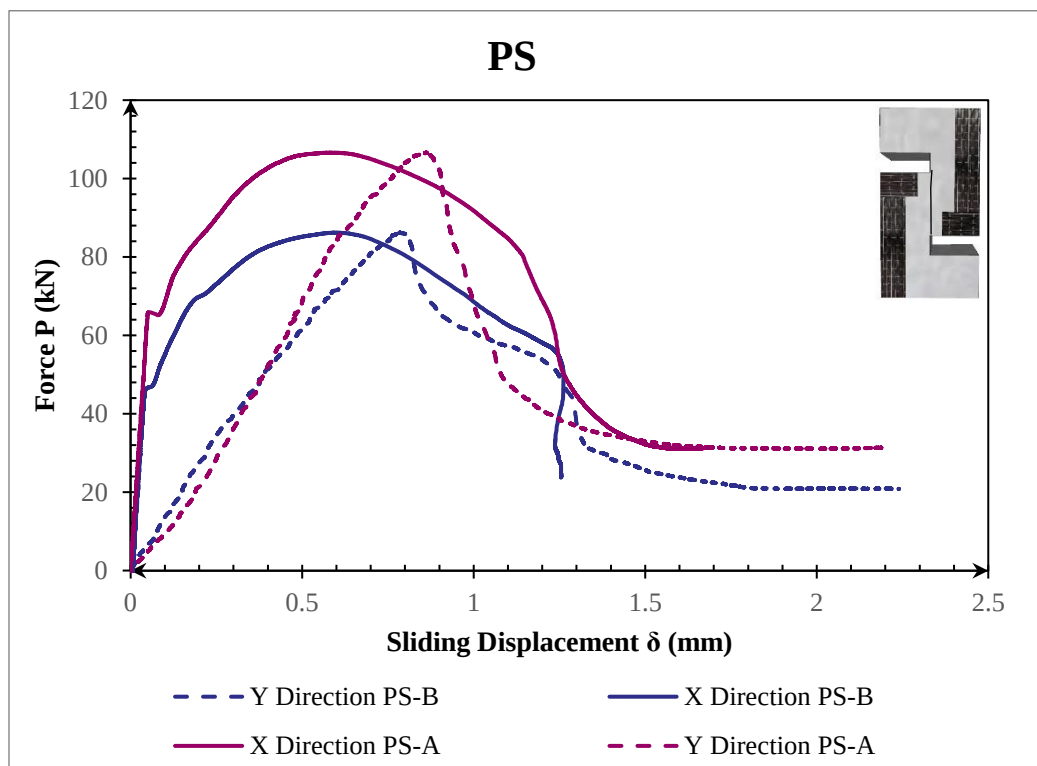


Figure 7.12: Load- horizontal/vertical deformation curves of PS push-off specimens

- *Compression Shear with $\kappa=63^\circ$*

In case of CS63 the ultimate average load was 182 kN with the corresponding vertical and lateral displacement of the shear plane to be 1.77 mm and 0.7 mm, respectively. The end of the elastic behaviour is not as distinct in the diagram as in the case of PS. Nevertheless, first cracking corresponds to 77 kN (which is 42% of the maximum load) and 0.042 mm lateral displacement. The post-peak capacity is 40% of the maximum load, namely 74kN. It is evident that the contribution of compression compared to PS is significant for not only attaining higher ultimate load but also higher post-peak capacity in terms of load and deformation.

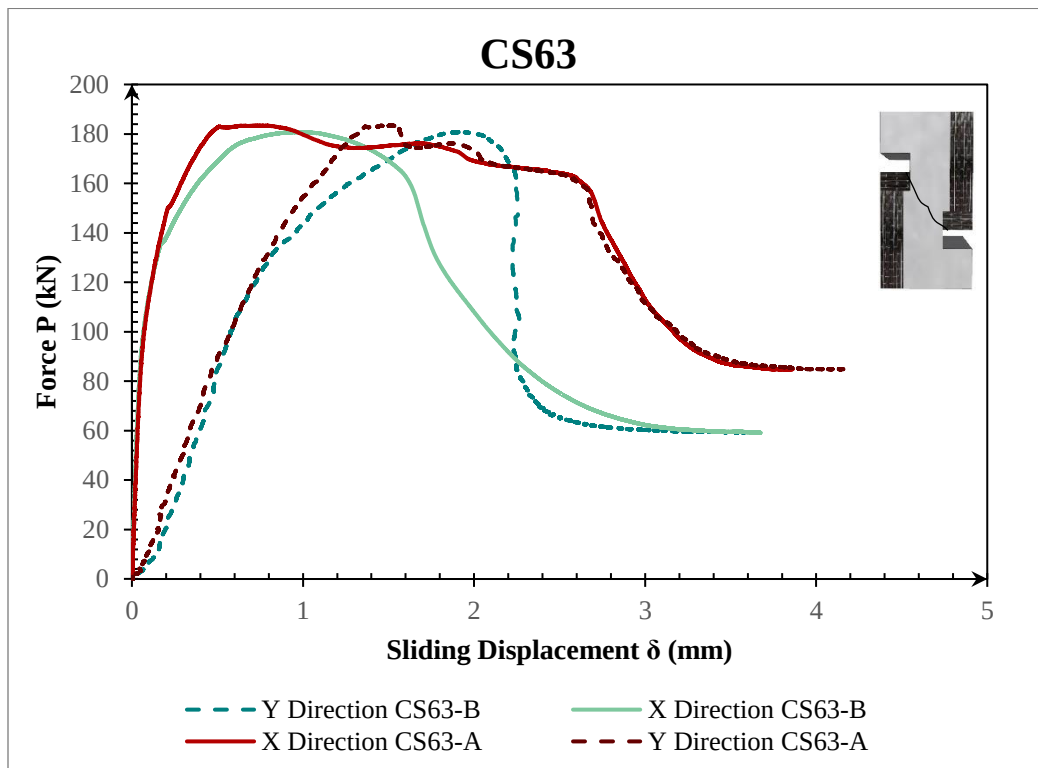


Figure 7.13: Load- horizontal/vertical deformation curves of CS63 push-off specimens

- *Compression Shear with $\kappa=45^\circ$*

For a shear plane angle of 45° , where the contribution of shear is equal to the contribution of compression, the peak average load was 368 kN with the corresponding vertical and lateral displacement of the shear plane to be 1.37 mm and 0.45 mm, respectively. Similar to CS63, the first cracking is not clearly visible in the response curve, but with a closer look it corresponds to 195 kN (which is 53% of the ultimate load) and 0.072 mm lateral displacement. The post-peak capacity is 31% of the maximum load, namely 115kN. It is evident that the contribution of

compression compared to PS is significant for not only attaining higher ultimate load but also higher post-peak capacity in terms of load and deformation.

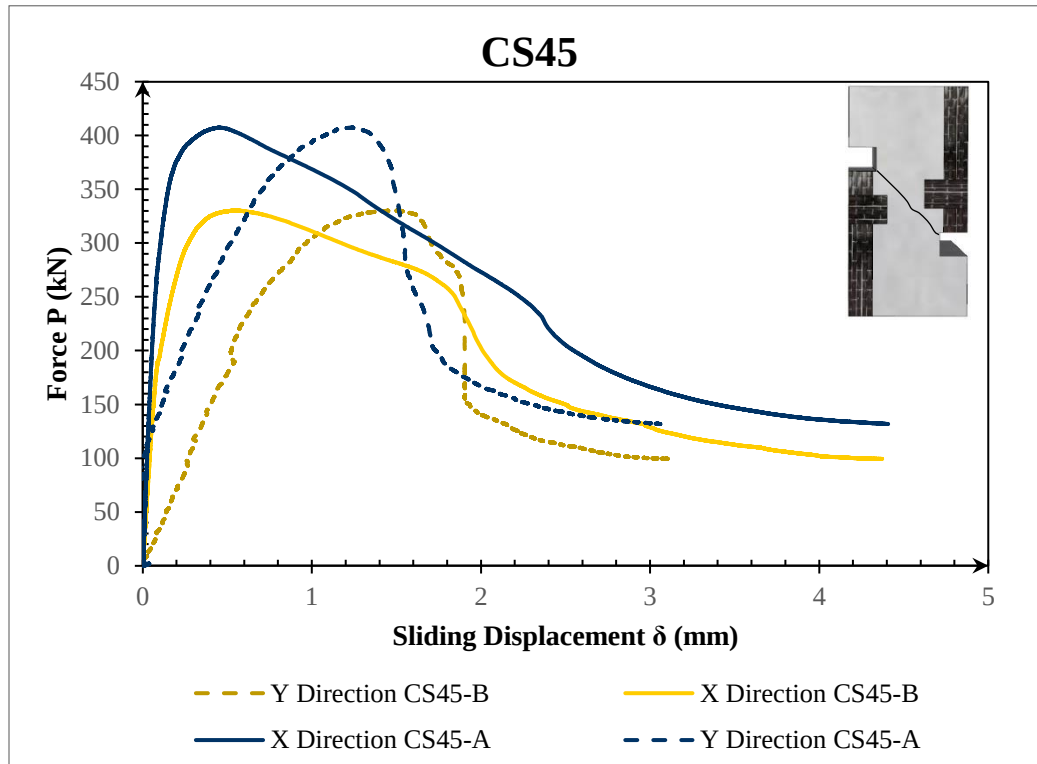


Figure 7.14: Load- horizontal/vertical deformation curves of CS45 push-off specimens

- *Compression Shear with $\kappa=34^\circ$*

Finally, the case of CS34 was expected to demonstrate the highest load capacity, due to higher contribution of compression. However, because of the non-preferential fiber alignment induced by the parallel to the axis of the specimen casting, CS34 push-off specimens did not yield the highest load. Namely, the average ultimate load was 338.5 kN with corresponding vertical and lateral displacement of the shear plane of 1.32 mm and 0.91 mm, respectively. The first cracking occurred at 210 kN (62% of maximum load) and 0.09 mm lateral displacement. Meanwhile, the residual load capacity corresponded to 108 kN which was 32% of the maximum load. Finally, it is evident that the CS34 specimens withstood excessive lateral displacement in the post-peak branch.

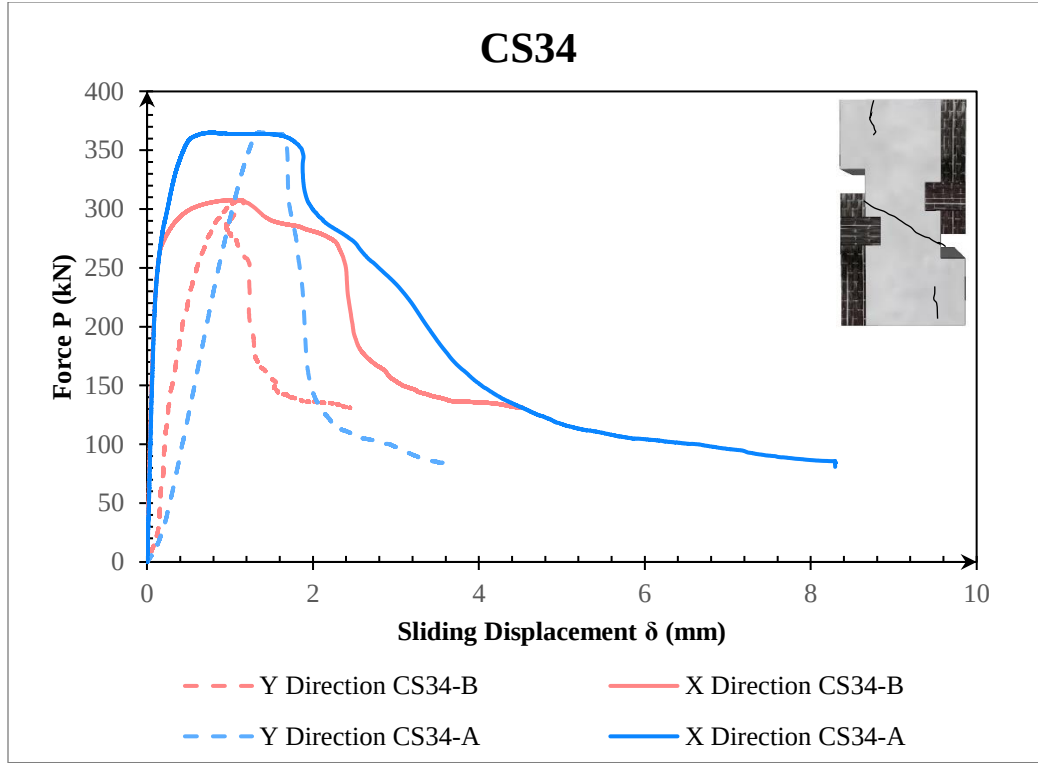


Figure 7.15: Load- horizontal/vertical deformation curves of CS34 push-off specimens

7.3.3. Analysis of Stresses Acting on the Sliding Plane

In order to obtain the biaxial failure envelope of THFRGC, the above results are further analyzed. A frictional failure model is the simplest method to relate the load components acting on the sliding plane- here these are further resolved to normal (σ_n) and parallel (τ) stresses by dividing the load components with the area of the inclined sliding plane. Although there is no internal transverse reinforcement to contribute to shear strength, the internal confinement, q_{lat} , exerted by the fibers should be taken into consideration. The confining pressure generated by the fibers has been demonstrated experimentally to have the magnitude (but of opposite direction) of a uniaxial tensile strength, f_t , of THFRC (Georgiou & Pantazopoulou, 2016). However, taking into consideration the wall effects and the preferential fiber alignment observed in thin direct tension specimens, the effectiveness of confinement should be reduced for cases where wall effects diminish, and the specimen thickness is such as to encourage a 3-D fiber orientation even partially. Since the orientation factor for specimens with cross section 50 mm x 25.4 mm and fiber length of 13 mm according to the theoretical expression presented in Chapter 4 is $\alpha = 0.722$ and the tensile strength was 7.7 MPa, for push off specimens with a height 100 mm, $\alpha = 0.562$ and thus it is assumed that 6

MPa should be used for in plane passive confinement. The calculations of normal and shear stresses for the two different casting methods employed are shown in Figure 7.16, whereas Table 7.4 summarizes these calculations for each case. The dimensions of the shear plane are not as designed, but as measured in each failed specimen. The component of the intrinsic confining pressure ($q_{lat}=f_t$) normal to the sliding plane provides extra frictional resistance and thus shear strength τ . However, the component of q_{lat} that is parallel to the sliding plane for casting direction from bottom to top ought to be neglected when it is adding to shear strength as it compresses the fibers.

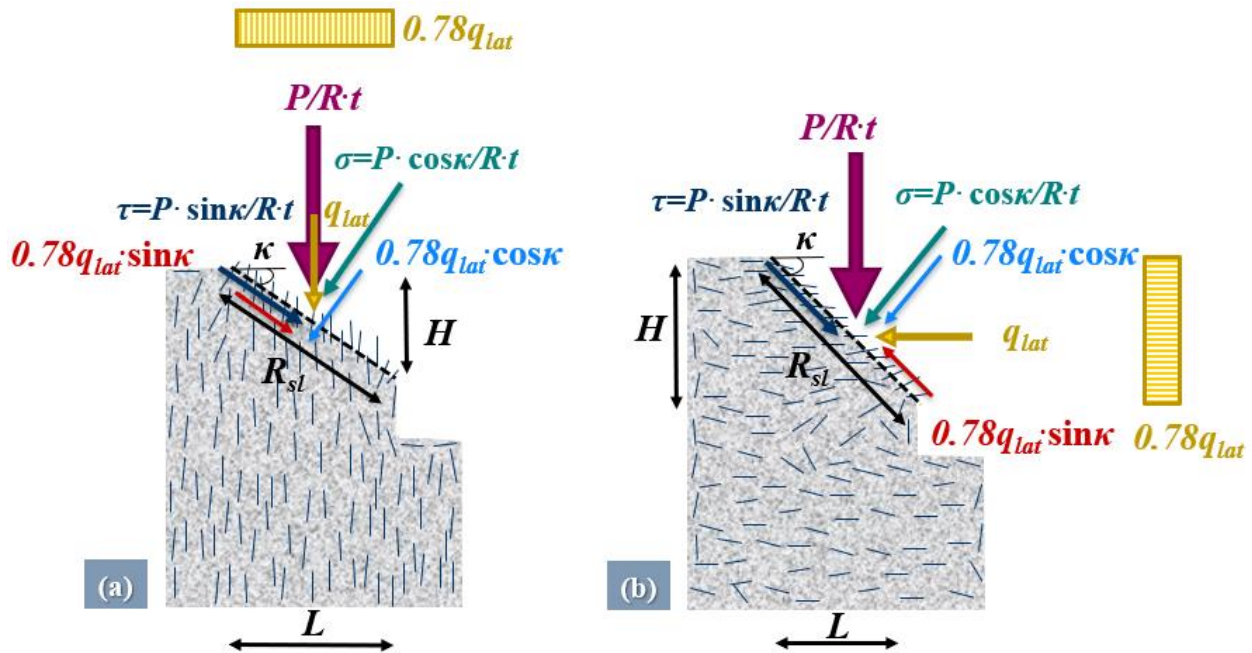


Figure 7.16: Normal and shear stress along the sliding plane for (a) parallel to the y axis and (b) normal to the y axis fiber distribution taking into consideration the lateral confining pressure exerted by the fibers

Table 7.4: Tangential and Normal stresses of push-off specimens

Specimen ID	Ultimate Load (kN)	H (mm)	L (mm)	κ (°)	R_{sl} (mm)	τ (MPa)	σ (MPa)
PS-A	106.61	86	0	90	86	12.4	6.0
PS-B	86.21	84	0	90	84	10.26	6.0
CS63-A	183.36	78.2	39	63.5	87.39	18.78	12.04
CS63-B	180.72	96.91	39.46	67.8	104	16.09	8.83
CS45-A	407.36	115.28	57.85	63.65	128	25.86	19.5
CS45-B	330.39	120.93	47.71	68.5	130	21.45	14.9
CS34-A	365	87	64	53.65	108	27.22	23.59
CS34-B	307.49	114	69.7	58.6	133.6	19.65	15.12

The results from Table 7.4 are further used to draw a Mohr-Coulomb failure envelope as illustrated in Figure 7.17. The vertical axis represents the shear stresses τ developed along the shear plane

while the horizontal represents the normal stresses taking into consideration the confining pressure. The failure surface has the features of a frictional material. The failure surface corresponds to Mohr-Coulomb condition with the following equation:

$$\tau = c + \sigma \tan \varphi \quad (7.1)$$

The linear function intersects the normal stress axis at the value of f_t (in the tension side of the normal stress axis) and the shear stress axis at the value of cohesion c with tangent equal to φ which is the internal friction angle. For the experimental results these constants are $c=6.88$ MPa and $\varphi=42.41^\circ$.

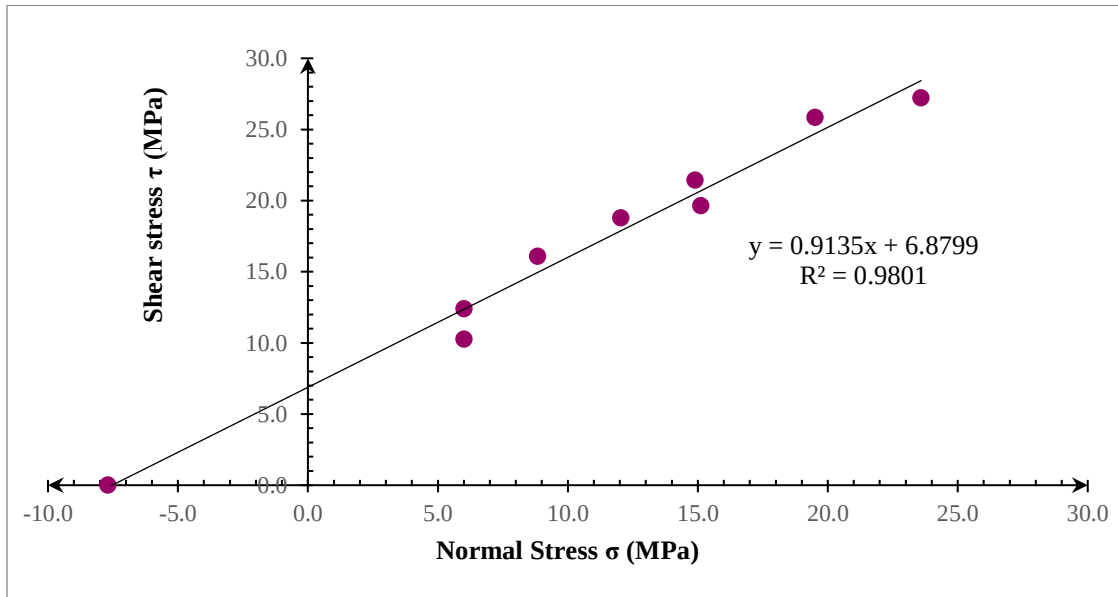


Figure 7.17: Shear and normal stresses from push-off specimens and Mohr-Coulomb failure criterion

7.3.4. Three-Parameter Failure Criterion

Concrete strength in real life where multiple stress states may occur simultaneously depends on the combination of the three principal stresses, σ_1 , σ_2 , σ_3 . In this context pure stress states such as uniaxial compression, tension, or pure shear are points on the failure envelope drawn in the 3-D system of principal stresses. Many mathematical models have been proposed to represent the failure surface thus defined in the 3-D space of stress. Mohr-Coulomb envelope that has been discussed above is a simple two parameter failure model but lacks sophistication as it does not take into consideration the intermediate stress and is linear. For this reason, a more complex three-parameter model shall be used to define the failure envelope of THFRGC in the 3-D space. In this

section the Bresler-Pister Criterion is chosen (Bresler & Pister, 1958). This is a commonly accepted failure model for concrete that utilizes octahedral stresses but the influence of third invariant (J_3) is neglected to limit the number of calibrated parameters that would need to be obtained from material mechanical tests. The function of the failure criterion is given by Equation (7.2) (Chen, 2007).

$$\tau_{oct} = a + b \frac{\sigma_{oct}}{|f_c|} + c \left(\frac{\sigma_{oct}}{|f_c|} \right)^2 \quad (7.2)$$

In the above equation parameters, a , b , and c are established through curve fitting of the available experimental data. The octahedral normal and shear stresses along with the first and second stress invariants are given by Equations (7.3) and (7.4)

$$\sigma_{oct} = \frac{1}{3} I_1, \quad I_1 = \sigma_1 + \sigma_2 + \sigma_3 \quad (7.3)$$

$$\tau_{oct} = \sqrt{\frac{2}{3} J_2}, \quad J_2 = \frac{1}{6} \left[(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2 \right] \quad (7.4)$$

Since the specimen thickness does not allow the assumption of plane stress, σ_z was assumed to be equal to the out of plane passive confinement provided by the fibers as a fraction of the tensile strength. For simplicity is taken as $\sigma_z = 1.7$ MPa (the rest 22 % of the tensile strength of 7.7 MPa as the 78% of the tensile strength is the lateral confinement). The failure surface according to Bresler-Pister Criterion obtained by using the experimental data from all the push off specimens (PO), direct tension (UT) and compression (CC) along with their casting type (OW for flow from one way and R for random) is shown in Figure 7.18. The experimental data are normalized by the compressive strength of the material and fitted with the black parabolic line of the following form:

$$\tau_{oct} = 0.145 - 1.696 \frac{\sigma_{oct}}{|f_c|} - 1.062 \left(\frac{\sigma_{oct}}{|f_c|} \right)^2 \quad (7.5)$$

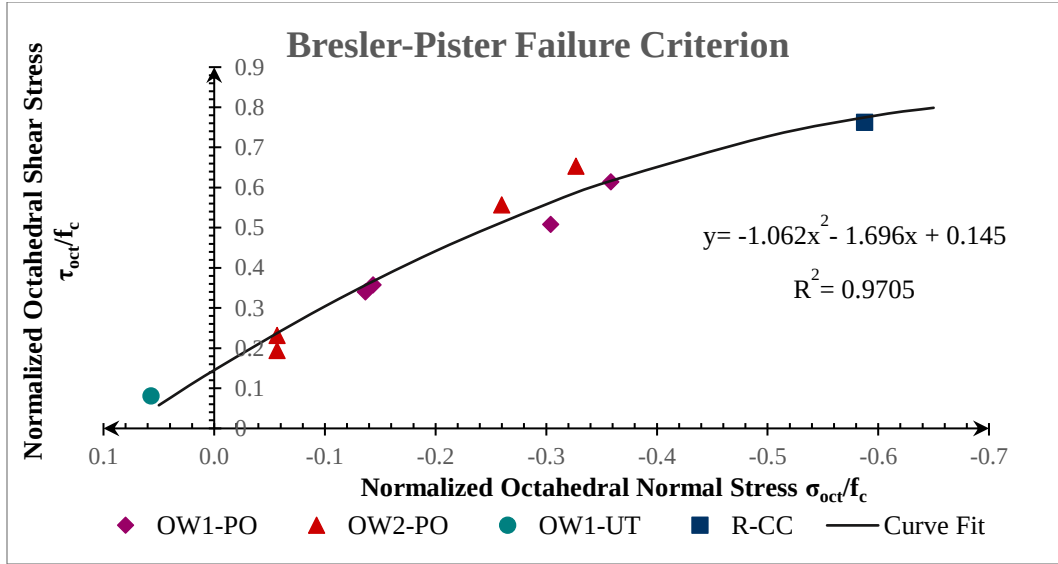


Figure 7.18: Bresler-Pister failure criterion from experimental data for THFRGC

7.3.5. Digital Image Correlation Analysis

All push-off specimens were further analyzed using Digital Image Correlation. The raw data were recorded every 0.1 sec (10 Hz) while the photos for DIC were captured every 10 sec. For every specimen, 14-20 pictures were selected for DIC Analysis to minimize computational time. The pictures selected correspond to specific points of the response curve in order to reproduce the complete behaviour (Figure 7.19). Out of those photos, 6 are used to show the surface normal and shear strains for 4 specimens, 1 of each type. Figures 7.20-7.23 illustrate the normal and shear strains at different stress states for each case.

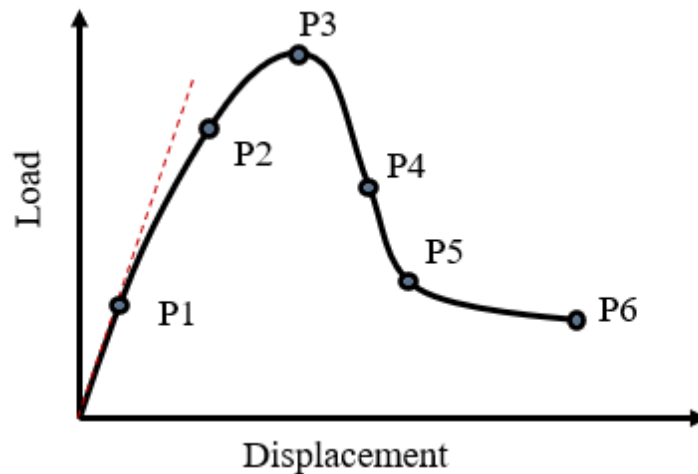


Figure 7.19: Selection of characteristic points of shear resistance curve for DIC strains

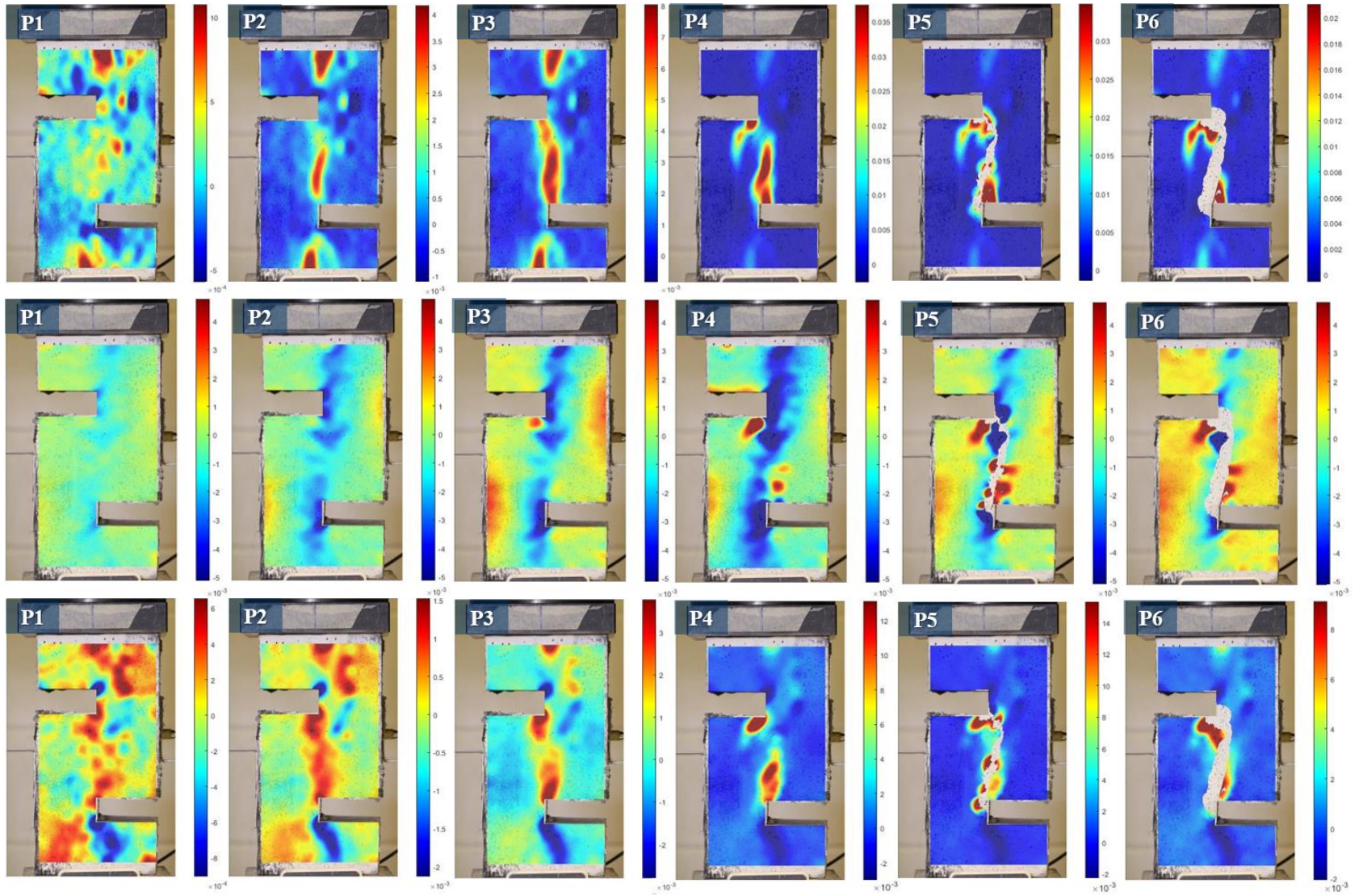


Figure 7.20: Strains ϵ_{xx} (first row), ϵ_{yy} (second row) and τ_{xy} (third row) for PS-A specimen

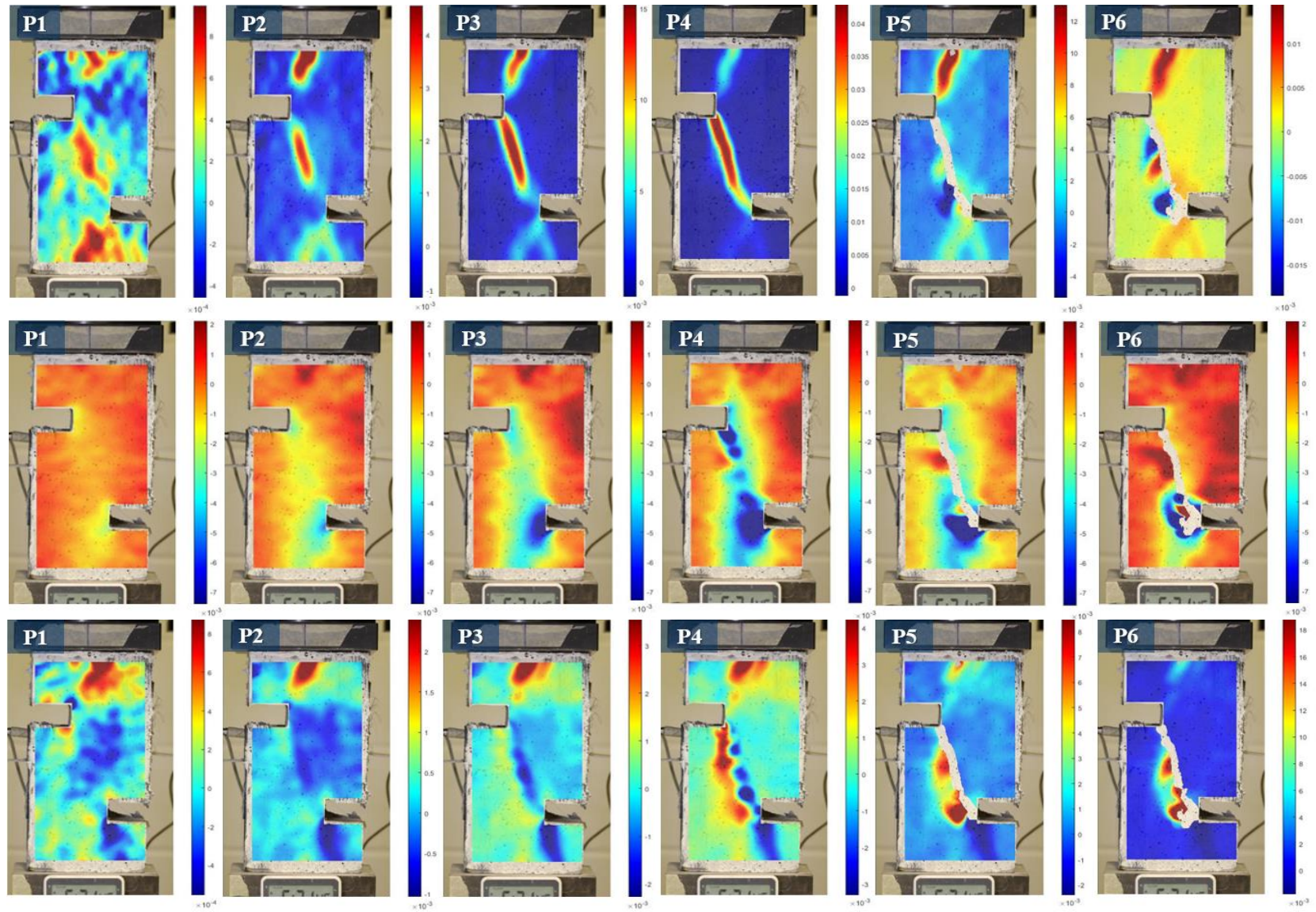


Figure 7.21: Strains ϵ_{xx} (first row), ϵ_{yy} (second row) and τ_{xy} (third row) for CS63-B specimen

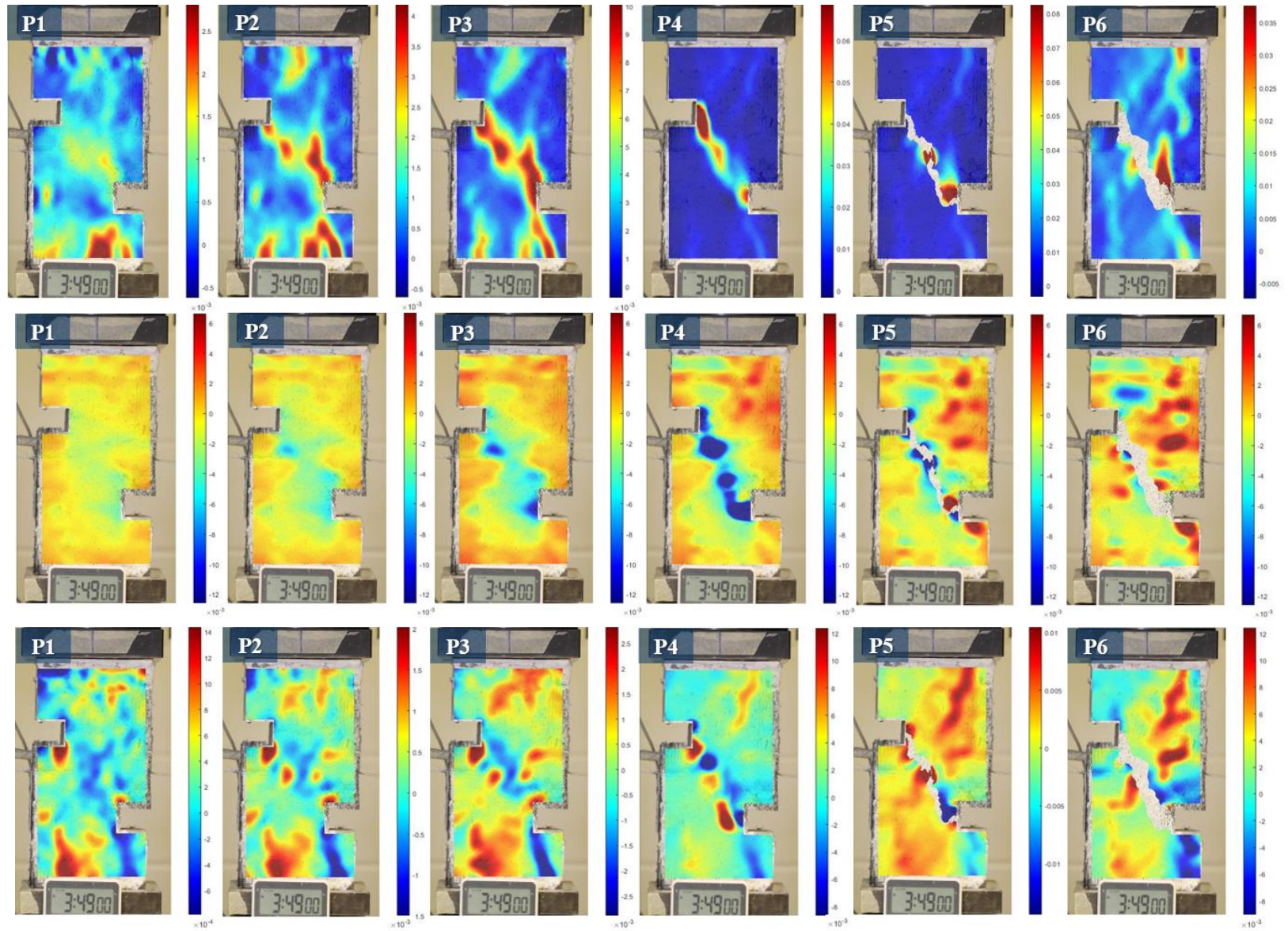


Figure 7.22: Strains ϵ_{xx} (first row), ϵ_{yy} (second row) and τ_{xy} (third row) for CS45-A specimen

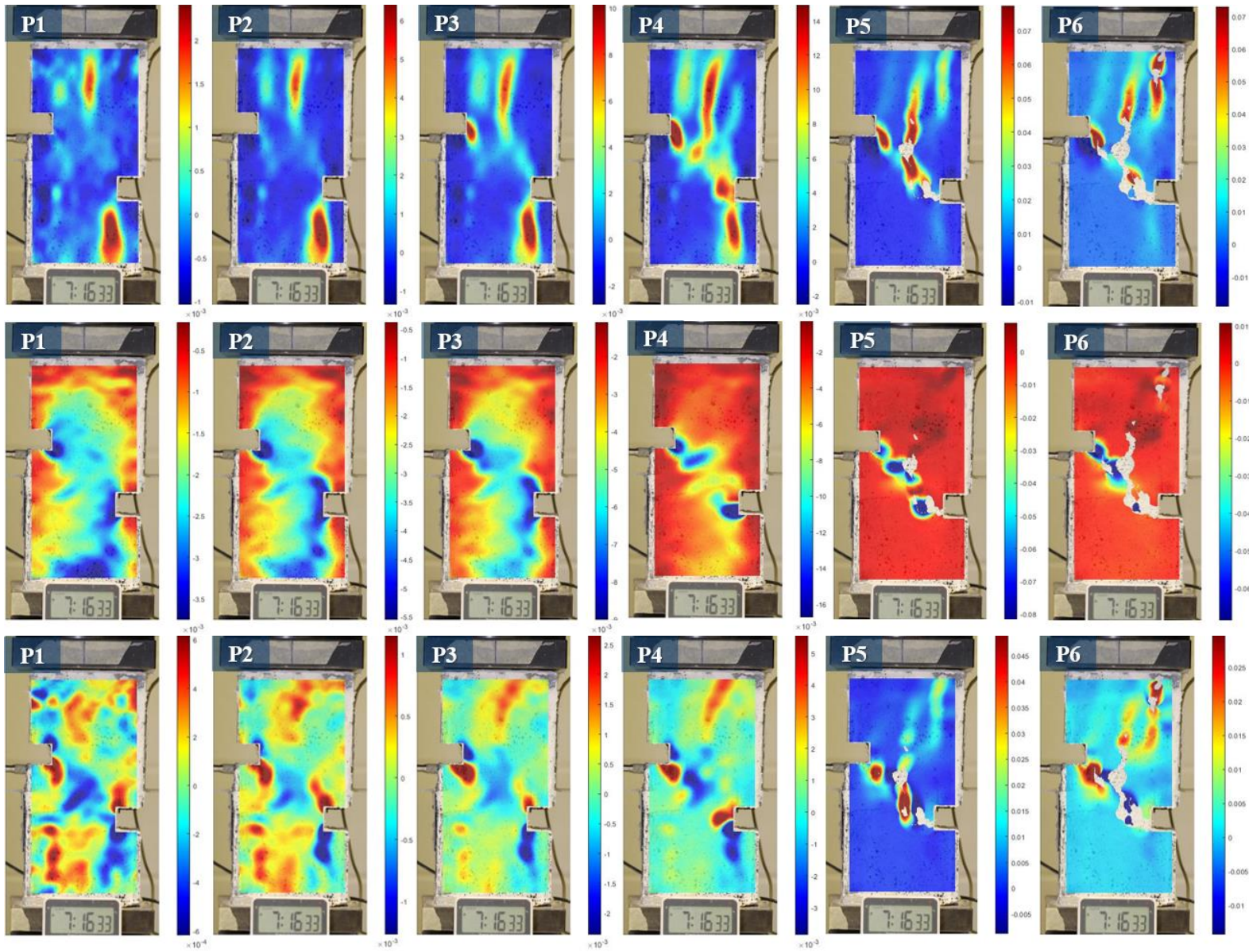


Figure 7.23: Strains ϵ_{xx} (first row), ϵ_{yy} (second row) and τ_{xy} (third row) for CS34-A specimen

It is observed that when the angle of the shear plane increases, the first apparent cracking (P1) is mainly compressive, as it is illustrated in the DIC images. After the loss of linearity and before the ultimate load, multiple cracking in forms of diagonal compressive and tensile cracks in the sliding plane is evident with localization to be manifested at ultimate load. The localization can be realized in DIC by the formation of areas around the crack with red color corresponding to high strains while the rest of the specimen is blue indicating the unloading. At the softening branch the localization is clearer except in case of CS34 specimens where new compressive cracks are formed. At P5 which corresponds to the residual capacity, minor concrete spalling occurs resulting to a disturbance on the speckle pattern. For this reason, DIC images for P5 and P6 have discontinued strain maps, as the areas where concrete cover was lost have excessive deformation and might affect the results if not excluded from the calculations.

Furthermore, virtual extensometers are established through DIC in order to record the slip for each case and compare it with the slip obtained through DTs. The locations of the virtual gauges are shown in Figure 7.24, while in Table 7.5 the gauge lengths of these for each case are given. The slip is calculated as the square root of sum of squares in the x and y directions.

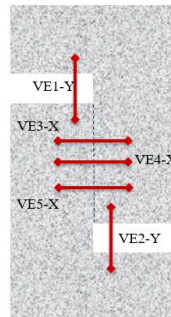


Figure 7.24: Virtual Extensometer positions using DIC.

Table 7.5: Gauge Length of Virtual Extensometer depending on the specimen type

Specimen ID	Gauge Length (mm)				
	VE1-Y	VE2-Y	VE3-X	VE4-X	VE5-X
PS	40	40	45	45	45
CS63	35	35	40	40	40
CS45	40	40	60	60	60
CS34	35	35	75	75	75

As illustrated in Figure 7.25, there is a difference in the slip measurement from the results of DIC analysis and the experimental hardware. The only case where the difference is not so prominent is the case of pure shear (PS) where the slip is measured only by the DTs mounted on the sides and

not the LVDT mounted on the back side. This further proves that the difference is due to the fact that the speckles monitored for DIC were on a different face than the mounted LVDT.

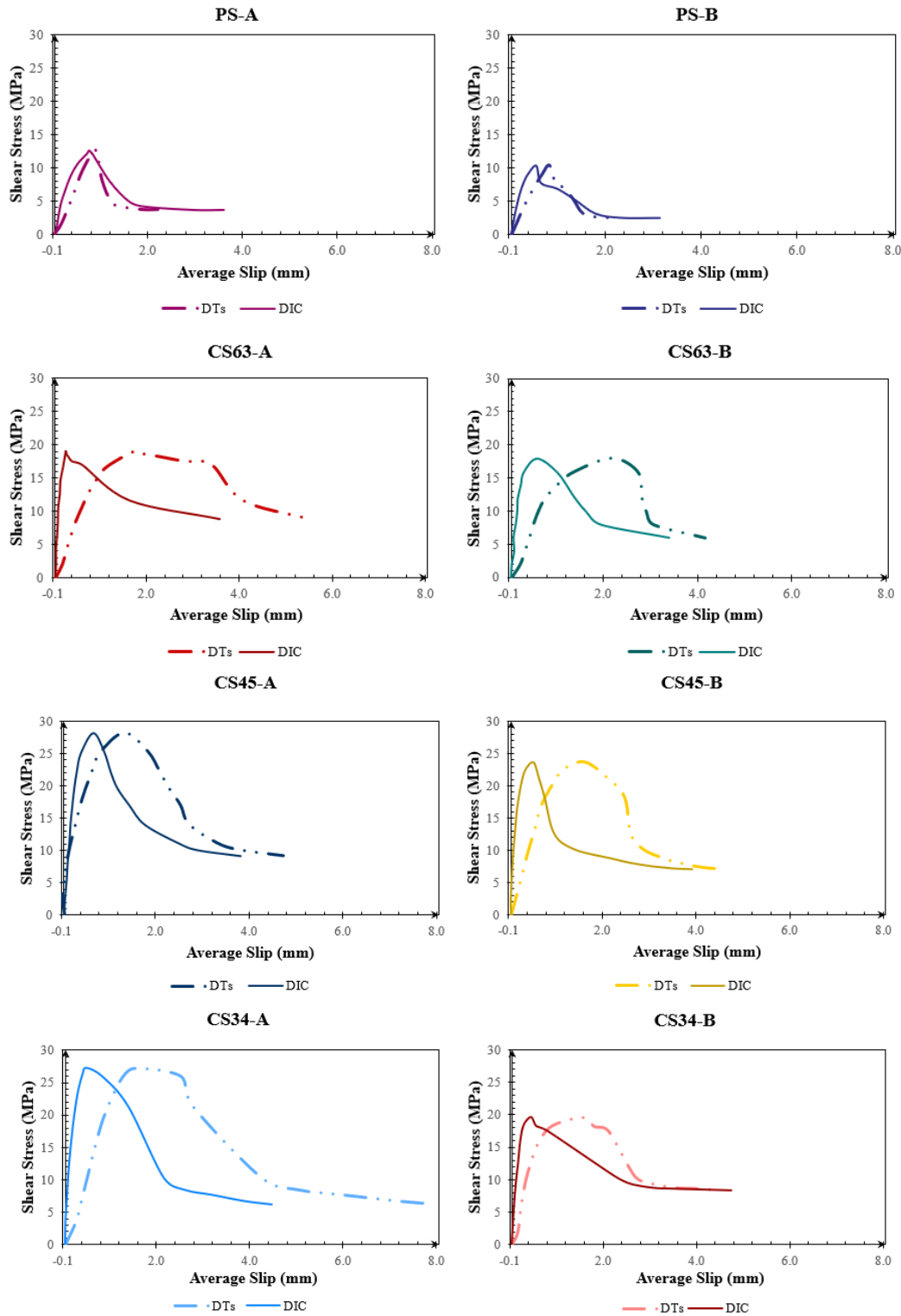


Figure 7.25: Shear Stress-Slip law for push off specimens using DTs and DIC

7.4. Conclusions

This chapter investigated the interface shear performance of geopolymer THFRC with varying shear plane angle and without reinforcing steel to cross the intended sliding plane. Eight push-off specimens with angle of shear plane varying from $90-34^{\circ}$ were tested, and their results were further analyzed and used to observe the failure modes, load-deformation behaviour, shear stress-slip law and behaviour of failure plane under combined stress states (normal compression and shear and pure shear). In addition to that and taking into consideration the tensile and compressive strength of the material as recorded from previous chapters, the push-off test results were used to derive the experimental Mohr-Coulomb envelope, and the Bresler-Pister three parameter failure criterion which can be combined with other components of a damage plasticity approach for non-linear analysis of tension hardening geopolymer composites. It was found that the presence of the steel fibers enhances the shear strength of the geopolymer concrete by acting as a partial replacement of transverse reinforcement while fibers also provide passive internal confinement through the mass of the material with magnitude equal to, 0.78 of the tensile strength approximately (owing to the prominent one dimensionality of the fibers due to casting from one end).

7.5. References

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Chapter 8: Non-Linear Finite Element Analysis of Tensile Properties of THFRC⁹

8.1. Introduction

Beyond the improved understanding of the overall material behaviour of Tension Hardening concrete-like which may be attained experimentally, the difficulties and the uncertainties of conducting inverse analyses and also the evaluation of the contribution of the material to the various mechanisms of resistance require the utilization of Non-Linear Finite Element Analysis (NLFEA). With proper calibration and validation, NLFEA has proven to be an excellent tool not only for design of new and assessment of existing structures, but also for evaluation of the parametric sensitivities of the material constitutive models to various states of stress. In this regard, the combination of experimental and computational investigation is an important vehicle in the effort to characterize and verify the tensile properties of THFRC (the term here places the emphasis on the mechanical property of hardening in tension and in this context, it is meant to include also THFRGC). In this Chapter, the available inverse analysis procedures adopted in codes for tensile characterization of THFRC are reviewed and are subsequently correlated with two different advanced nonlinear finite element models that have been adapted to THFRC, with benchmarking reference to experimental results obtained from standard flexural prism tests.

8.2. Literature Review

8.2.1. Non-Linear Finite Element Analysis (NLFEA) of THFRC

The Finite Element Analysis (FEA) method is implemented to complement and validate the experimental results, especially when in depth comprehension of the material behaviour is needed. Besides the suitable representation of the boundary conditions, convergence criteria and the relevance of the analysis results are highly dependent on the constitutive law used to simulate the material behaviour. For this reason, most of the commercial computational softwares are not equipped with a material model especially designed for THFRC and calibration of existing models intended for conventional concrete models is needed for an accurate simulation. Some researchers

⁹ Part of this chapter has been used in the Author's publication with her supervisor, titled: "Comparative Evaluation of Nonlinear F.E.A. Inverse Analysis of Tensile Properties of UHPFRC" Proceedings of fib Symposium 2021 Concrete Structures: New Trends for Eco-Efficiency and Performance

have conducted computational modelling of THFRC implementing the Concrete Damage Plasticity (CDP) model in ABAQUS, but the accuracy of calibration with the four-point bending tests is still to be understood. Shafieifar et al. (2017) investigated the flexural capacity of THFRC beams, while FHWA researchers analysed the structural performance of THPFRC using the same constitutive model (Graybeal, 2010). A combination of CDP with element deletion strategies has been used for the computational evaluation of THFRC fracture by Zhang et al. (2021).

Several studies have also been conducted using the ATENA software. Paschalis et al. (2018) implemented the SBETA constitutive model to study the performance of THFRC. However, the model lacks ability to simulate the tension stiffening and the tension softening post-peak behaviour. In this regard, the user-defined fracture-plastic model, also available in ATENA has been used as an alternative. Valikhani et al. (2020) investigated numerically the normal strength concrete to THFRC bond strength using the aforementioned model while Tsitsias (2019) studied the bond strength of THFRC under direct tension pull-out implementing user defined fracture-plastic models.

Mezquida-Alcaraz et al. (2021) used the DIANA FEA platform to validate the tensile response of THFRC derived from simplified inverse analysis on four-point bending tests. They used a combination of discrete cracking model as interface behaviour based on total deformation theory and a smeared cracking approach based on a fixed total strain crack model according to the experimental crack width. Naeimi & Moustafa (2020) using the same model by implementing user defined stress strain curves in tension and compression investigated the structural and seismic behaviour of THFRC bridge piers.

Wilson & O'Flaherty, (2021) utilized the Microplane model developed by Bažant & Gambarova (1984) available in ANSYS to simulate the flexural behaviour of THFRC beams. The model calculated the elastic response with success, although the softening branch after the peak strength was not reproduced. Chen & Pei (2017) investigated the flexural response of THFRC with different types of steel fibres in ANSYS. They utilized a combination of a constitutive model proposed by Yongsheng et al. (2007) that takes into consideration the fibre content, damage, and strain rate and utilizes the William-Warnke five parameter failure criterion (William & Warnke, 1975). Finally, Yin et al. (2017) implemented the Karagozian and Case (Malvar et al., 1997) concrete damage

model available in LS-DYNA to numerically assess the material behaviour of THFRC. It is generally used to perform explicit dynamic FEA but can be manipulated through implicit controls to fit a static loading condition. The model was successful in estimating accurately the peak load and the ascending branch, but the post-peak branch did not agree well with the experimental results.

All the aforementioned FEA studies examined the material behaviour utilizing a single constitutive model based on different experimental results. In essence, it is difficult to decide based on comparison of the reported results, which constitutive model is more suitable to predict and validate the material response of THFRC. This research aims to contribute towards that, by examining two different constitutive models from different Software to validate and vet the tensile response of THFRC derived from simplified inverse analysis on experimental results from four-point bending tests. The two developed models are further compared with a third simulation using a different constitutive damage plasticity model the Concrete Damage Plasticity Model (CDP) in ABAQUS software found in Ralli et al. (2021)

8.3. THFRC Non-Linear Finite Element Analysis

The compression law was obtained from uniaxial compression of cylinders and prisms (Figure 8.1(a)). Un-notched four point bending tests were conducted on specimens having dimensions 100 (mm) x 100 (mm) x 370 (mm) and a clear span of 300 mm according to CSA A23.2 Annex U (2019). (Figure 8.1 (b), (c), and (d)). The closed form simplified inverse analysis by López et al. (2016) adopted by CSA S6 Annex 8 (2018) was used to obtain the tensile constitutive law of THFRC, while the Both sets of experimental results were taken from Husain (2021). The experimentally obtained stress-strain in tension and compression curves are illustrated in Figure 8.2.

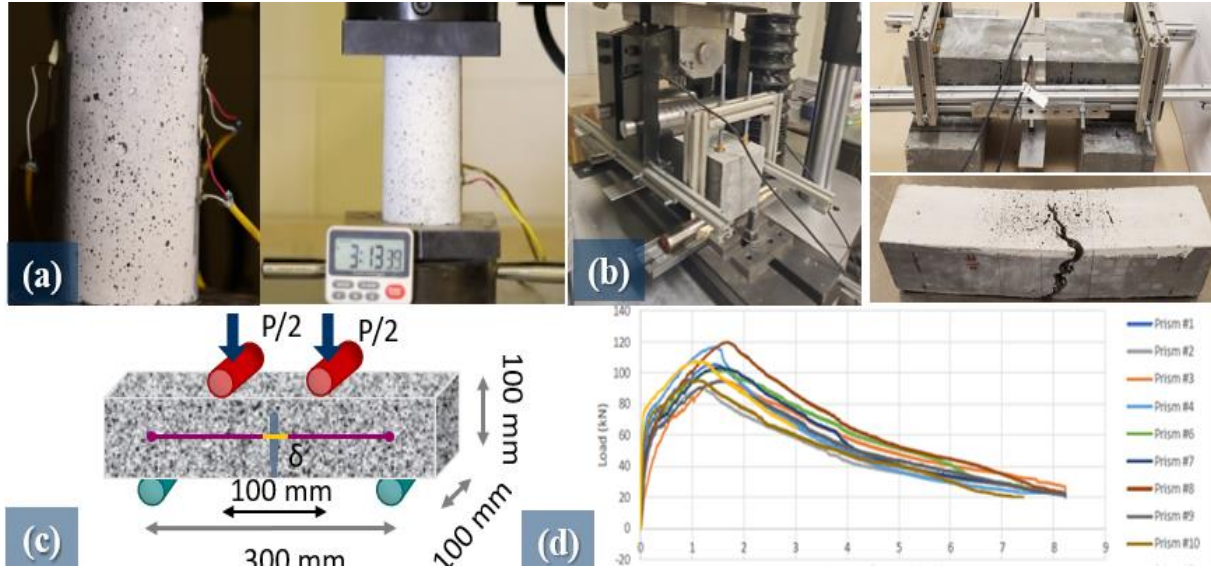


Figure 8.1: (a) Cylinder subjected to uniaxial compression, (b) experimental setup and fractured specimen from the bending test, (c) geometry and schematic representation of bending test and (d) resistance curve of flexural tests used for inverse analysis

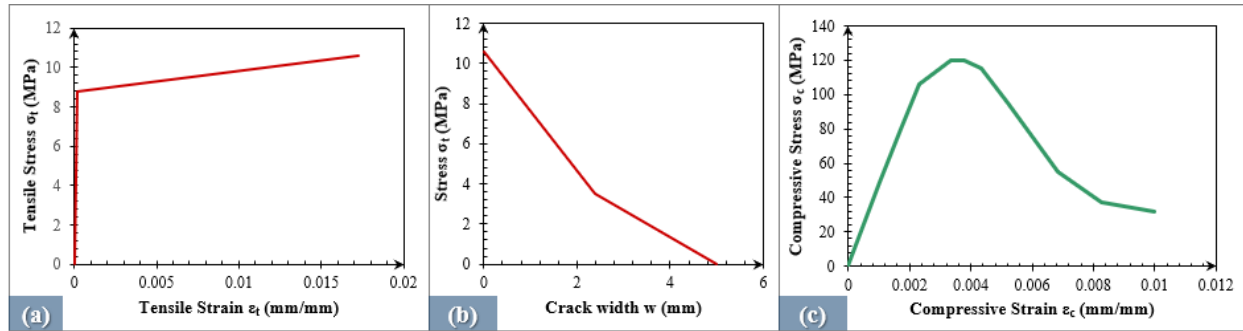


Figure 8.2: (a, b): Linearized tensile stress-strain law obtained from simplified inverse analysis; (c): Compressive stress-strain law

After obtaining the resistance curve from the experimental results the simplified inverse analysis was implemented as shown in Figure 8.3. Results of this analysis are to be benchmarked by the finite element inverse analysis and will provide an independent check of the dependability of the Code-adopted inverse analysis method.

According to the recommended procedure, the characteristic points of the load-deflection curve include the end of linearity (P_o, δ_o) that determines the initial slope of the resistance curve; Points 1 and 2 represent 75% and 40% of the initial slope, respectively. Point 3 corresponds to 97% of the peak load and Point 4 is on the resistance curve at a load equal to 80% of point 3. Using the aforementioned points, cracking strength ultimate tensile strength and their corresponding strain values are determined in order to establish a linearized tensile stress-strain. Basic assumptions of

the method are linear elastic behaviour in compression, a bilinear stress-strain tensile law until the peak and a bilinear stress-crack opening behavior in the post-peak softening range. The method takes into consideration the location of the dominant crack which prevails after localization and also covers the post-cracking behaviour. As mentioned previously, the method is only valid for the case of third point loading, whereas the load at Point 3 should exceed the load at Point 2. The latter restriction limits applicability of the approach to concretes with a limited range of resilience – for example it has been shown that the method is not directly applicable to prisms comprising Engineered Cementitious Composites (Pantazopoulou et al., 2019).

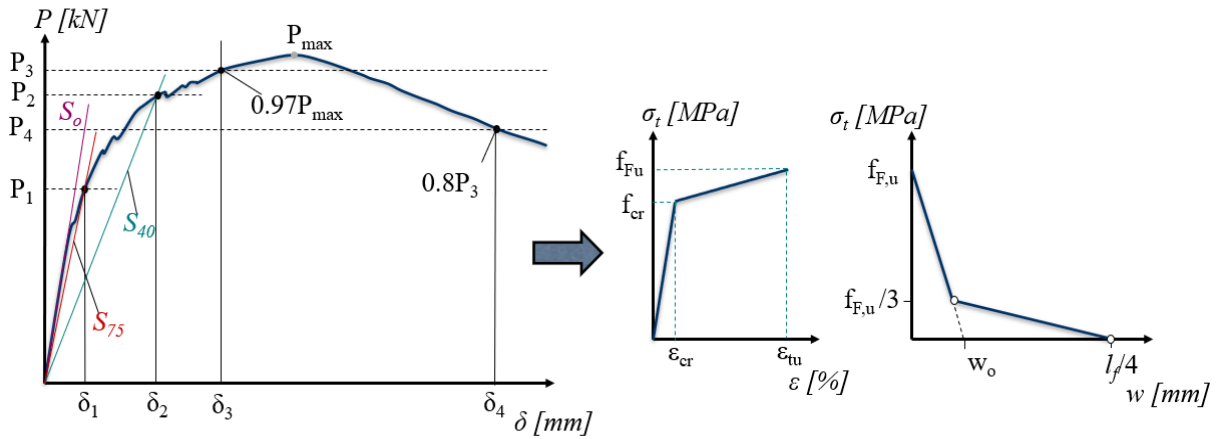


Figure 8.3: Characteristic points of inverse analysis and linearized tensile stress-strain laws (modified from Lopez et al., 2016)

- *Geometry of FEA Models*

To further validate the reliability of the simplified inverse analysis, beside the simulation of the 100 (mm) x 100 (mm) x 370 (mm) (4PBT) specimens specified by CSA, the thinner prisms having 50 mm depth by 100 mm width by 400 mm span (AFNOR) have been also studied (AFNOR NF EN 13670/CN, 2013). The 3-D NLFEA was conducted using two different softwares with different damage plasticity models. One of the models used an explicit dynamic formulation whereas the other did not account explicitly for the inertial effects developed during the post-peak precipitous failure. To save computational time, symmetry was exploited, and one quarter of the beam was simulated as shown in Figure 8.4.

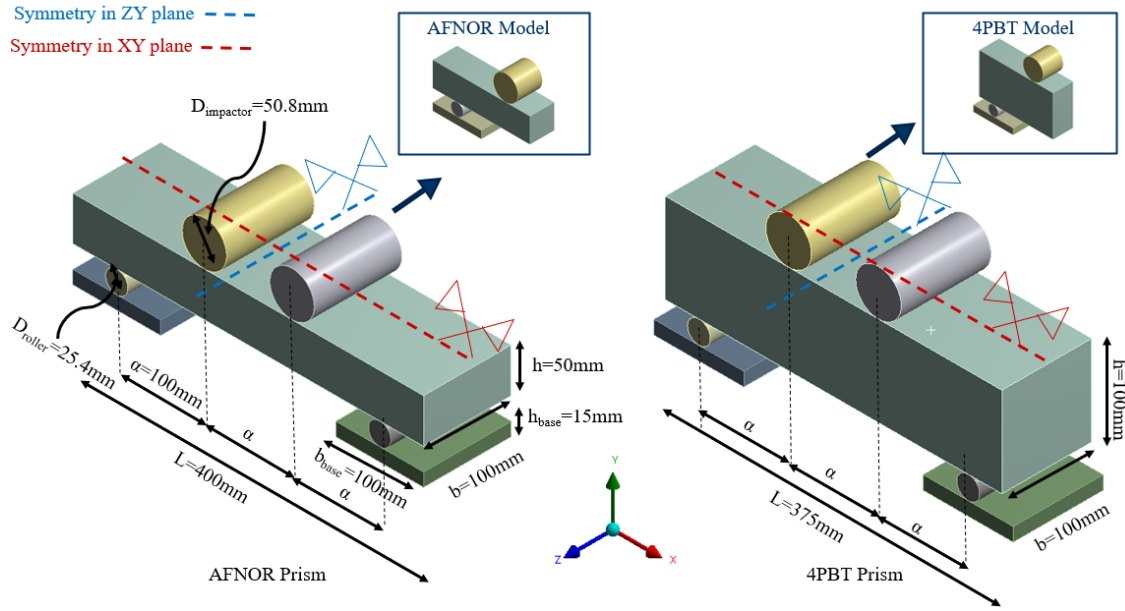


Figure 8.4: Geometry of the standard prisms along with the experimental hardware and their modeled parts

8.3.1. Static Structural Implicit Analysis Using ATENA 3D

ATENA 3D Engineering platform was chosen for this analysis on account of the available advanced concrete models. The model uses an implicit solver considering the studied simulation as a static phenomenon.

- *Constitutive Model for THFRC: Fracture-Plasticity Model*

Three-dimensional constitutive material models for concrete in combination with plasticity and fracture models are available in the ATENA software. Fracture is modelled considering an orthotropic smeared crack model based on the Rankine tensile criterion (Rankine, 1857), while Menetrey & Willam, (1995) hardening/softening plasticity model is implemented to simulate the crushing of concrete with a three-parameter failure surface. The variant of fracture-plastic models CC3DNonLinCementitious2 (CC3DNLC2) along with a rotated crack model is implemented in this study. It takes into consideration various constitutive laws such as tensile and post-cracking softening behaviour via fracture energy, compression behaviour and the softening effect of lateral tensile strain on the compression capacity (Figure 8.5). The parameters of CC3DNLC2 used in this study are found in Table 8.1.

Regarding the compression curve, the ascending branch consists of a linear part till f_{c0} and then a nonlinear segment with a parabolic shape till f_c . Then the descending curve is represented with a

linear decrease till plastic displacement w_d which corresponds to an apparent compressive stress-free strain ε_d . The compressive strength reduction in the cracking state is dominated by the parameter r_c and is based on the Modified Compression Field Theory (MCFT) by Vecchio F.J. & Collins, (1986). According to the latter, the compressive strength should decrease when transversal tensile strain increases.

For modeling the constitutive law in tension, a linear ascending branch was employed up to the tensile strength followed by an exponential softening. The crack opening displacement for the model is derived from the aforementioned inverse analysis. More specifically, the crack width at complete release of stress post peak w_c is based on the fracture energy of the concrete G_f which is the area under the tensile stress vs. crack width opening diagram.

Finally, with regards to shear behaviour, this simulation did not consider aggregate interlock, while the crack shear stiffness factor that relates to tensile and shear stiffness of the crack was set to default based on the Model Code 90 (CEB Bulletin No. 213/214, 1993).

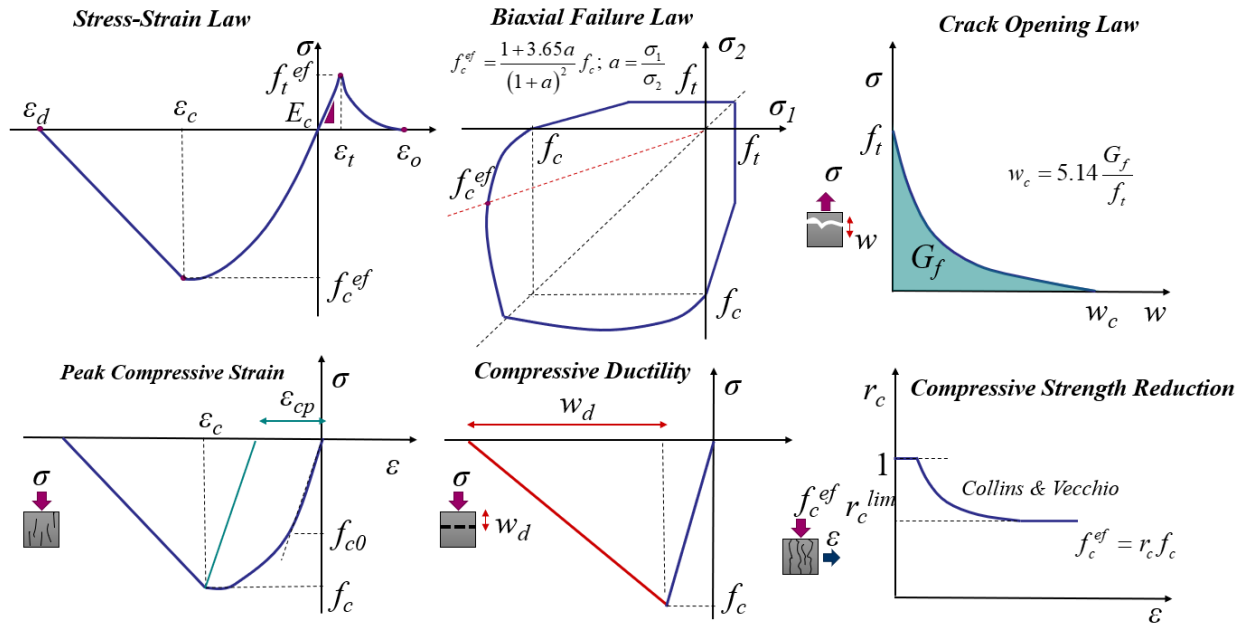


Figure 8.5: Constitutive Laws of Concrete for CC3DNLC2 model in ATENA

- *Constitutive Model for Experimental Setup Parts*

The structural steel experimental hardware (base, loading and support shafts) is also simulated. The constitutive model used for these parts is the material prototype CC3DBiLinearSteelVonMises

(CC3DBLSVM). The mechanical properties of the material are listed in Table 8.1, while Figure 8.6 shows the constitutive law of the material used for modeling structural steel in ATENA.

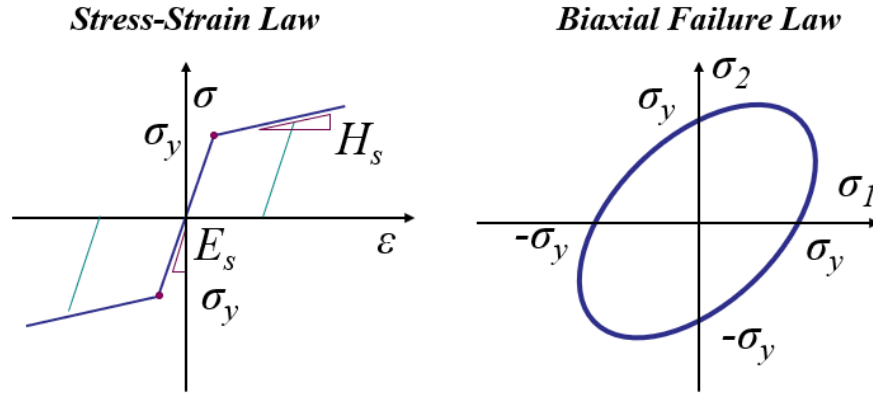


Figure 8.6: Constitutive law of Structural Steel for ATENA.

Table 8.1: Material Properties of constitutive models used in the implicit FE ATENA model

Parameter	Material	Model	Symbol	Value	Unit
Modulus of Elasticity	THFRC	CC3DNLC2	E_c	48000	MPa
	Structural Steel	CC3DBLSVM	E_s	200000	MPa
Poisson's Ratio	THFRC	CC3DNLC2	ν_c	0.2	-
			ν_s	0.3	-
Hardening Modulus	Structural Steel	CC3DBLSVM	H_s	10000	MPa
Yield Strength			σ_y	550	MPa
Tensile Strength	THFRC	CC3DNLC2	f_t^{ef}	10.57	MPa
Plastic Strain			ε_{cp}	0.00127	mm/m
Onset of Crushing			f_{c0}	48	MPa
Critical Compressive Displacement			w_d	0.08	mm
Compressive Strength Reduction			r_c	1	-
Compressive Strength			f_c	120	MPa
Fracture Energy			G_f	21360	J/m ²

- *Loading and Boundary Conditions*

The FEA model was loaded under displacement-controlled steps, where a displacement incremental step of 0.1 mm was applied on the steel loading roller for 100 steps, resulting in a total displacement of 10 mm. Symmetry was employed to save computational time as previously mentioned and illustrated in Figure 8.4. The metal base on which the roller support is bearing is simulated as a pin with restraints in Y and X direction. The support shaft is allowed to rotate about its axis and more information about the simulation of the roller support are found in the contact

conditions section of this model. The boundary conditions used in ATENA model regarding symmetry and external supports are shown in Figure 8.7.

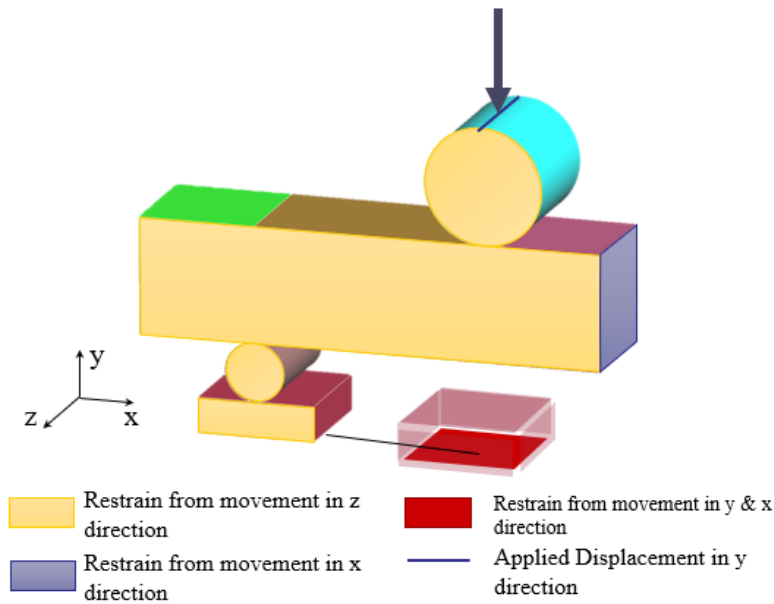


Figure 8.7: Loading & boundary conditions of the implicit FE model in ATENA

- *Contact Conditions*

In order to simulate more accurately the support conditions of the prisms and the interaction between the bodies in contact, the concrete prism was modeled using three 3-D macro elements as illustrated in Figure 8.8. The partition was made in a manner that accurately represents the ability of rollers to slide across the surfaces in contact. The connection between the macro-elements is affected through the master-slave option, where one surface in contact is considered master and the other slave. To ensure that the macro-elements together simulate the concrete beam as a continuous and homogeneous body, a fixity connection with restraints in X and Y direction is applied between them. Figure 8.8 also shows all the contact conditions applied on the respective surfaces, while the labels of the legend are explained in Table 8.3. The roller action for the shafts is simulated as restraint in Y direction, while movement in X direction is free. In case of contact between the top surface of the concrete beam and the cylindrical face of the loading roller, the body moving down to form a contact is the loading shaft and thus its surface is set as slave. Similarly, for contact between the bottom face of the beam and the support roller, the beam is the body moving down to form a contact and for this reason, this face is set to be the slave.

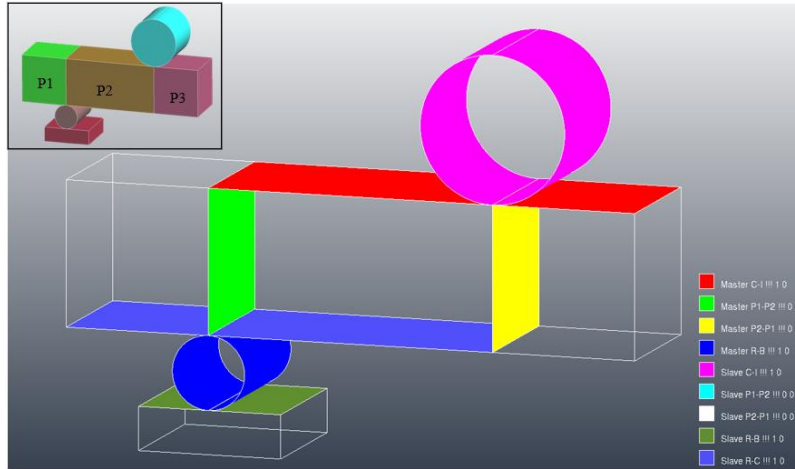


Figure 8.8: Contact conditions and 3D Macro-elements.

Table 8.2: Details of contact surfaces in the ATENA implicit FE model

Contact Name	Master	Slave	Restriction in Y	Restriction in X
C-I	Concrete (P2+P3)	Impactor	Yes	No
P1-P2	Concrete P1	Concrete P2	Yes	Yes
P2-P3	Concrete P2	Concrete P3	Yes	Yes
R-C	Roller	Concrete (P1+P2)	Yes	No
R-B	Roller	Base	Yes	No

- *Mesh Type*

The THFRC prisms were modeled using 3-D solid brick elements with eight nodes (hexahedral). The mesh is uniform with an element size of 10 mm. For the steel shafts, tetrahedral elements were used with axially symmetric discretization, while the steel plate has tetrahedral elements with an element size of 5 mm. The mesh of the two models is shown in Figure 8.9.

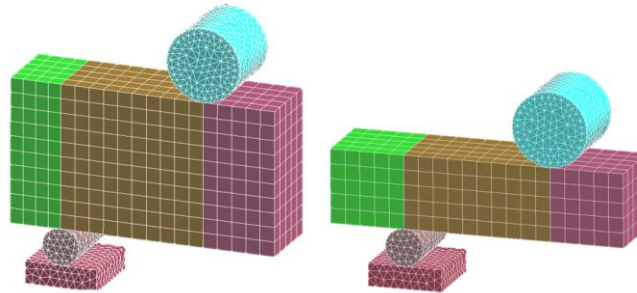


Figure 8.9: Mesh representation of the implicit FE models in ATENA

- *Solution Parameters*

The solution algorithm used in the NL-FEA in ATENA was the Newton Raphson method (Figure 8.10) with 100 loading increments and a multiplier of 10, meaning 0.1 mm were applied in each

step. Furthermore, the line search method is employed. The later decreases the work done to calculate the out of balance internal forces on displacement increments (Červenka., 2020).

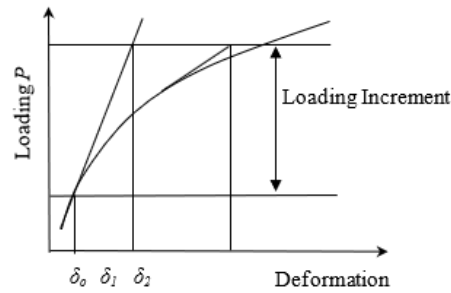


Figure 8.10: Full Newton Raphson method

For both specimens and FEA models the same size of mesh was used (10 mm) and the same type of finite elements (hexahedral). The friction between the concrete specimen and the steel shafts used for loading and supports was set equal to 0.5, while the friction between the support roller and the steel base was set to 0.25. Finally, the loading was applied using displacement control.

8.3.2. Quasi-Static Explicit Dynamics Analysis Using AUTODYN/ANSYS

In FEA, there are implicit and explicit solvers that use different methods to determine the underlying equations. Based on the application studied, there are models where the implicit solver is more suitable and models where explicit solvers perform best. This mainly depends on the nonlinearity of the system and the duration over time. As nonlinearity increases and time period decreases, the model/application becomes dynamic and is more accurately studied with explicit time integration. However, there is an overlap in the “Quasi-Static” application area, where both methods can be used to solve the problem. This is beneficial when the limitations of the implicit solver are reached, and explicit can be used as an alternate. Limitations usually include non-convergence due to nonlinearities that include large deformations and frictional contacts, and steep post-peak strength loss. In this sense, for explicit time integration there is no inversion of the stiffness matrix, no checks for convergence as the equations are uncoupled while all nonlinearities are included in the internal force vector (ANSYS, 2022). However, in order to perform accurately quasi-static analysis with explicit dynamics, some extra steps are needed. More specifically, through mass and time scaling, the inertia forces are controlled, and thus the kinetic energy is kept to minimum so there is no change in the physics of the simulation. In this section, along with the constitutive model, boundary conditions and mesh type, these steps are also described.

- *Constitutive Model for THFRC: RHT Model*

The second concrete model that was selected to be studied is the RHT (Riedel Hiermaier Thoma), i.e., an advanced plasticity model for brittle materials developed by Riedel (2000). As illustrated in Figure 8.11(a), it is inspired by Chen's work (W. F. Chen, 1982) on fully triaxial stress-based failure surfaces (Riedel, 2009). The model is available in ANSYS-AUTODYN and LS-DYNA and is usually used for simulating impact loading. Instead, in this study, quasi-static analysis was implemented. The procedure followed to ensure a quasi-static analysis is described in the following sections. Pressure hardening, strain hardening and softening, and third invariant dependence are some of the robust features of the model. In this section, a detailed description of the model is presented and calibrated based on the known experimental data.

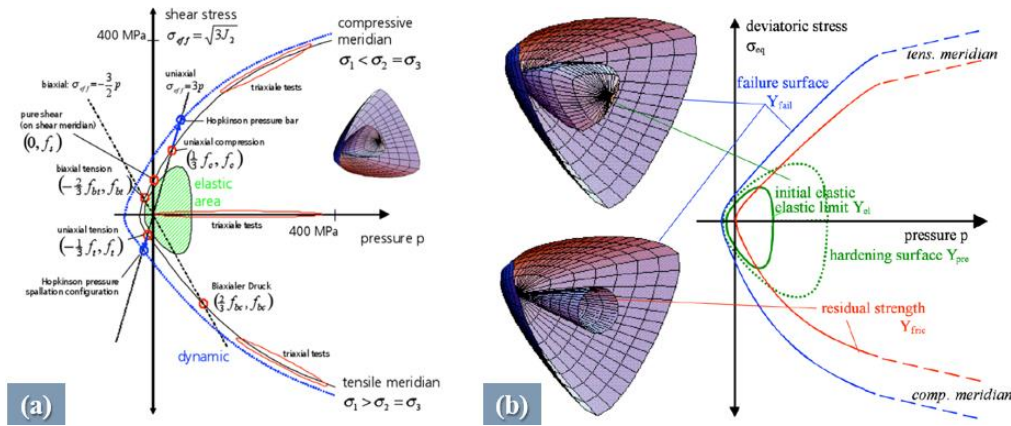


Figure 8.11: (a) Elastic and failure surfaces based on Chen's concept, (b) the three RHT limit surfaces of elasticity, ultimate strength and post failure shear resistance depending on pressure, strain rate, triaxial stresses, controlled by porous compaction, hardening and damage evolution (Riedel, 2009).

As illustrated in Figure 8.11(b), RHT model consists of three strength surfaces, namely the elastic limit surface $Y_{elastic}$, the failure surface, $Y_{failure}$ and the residual surface $Y_{residual}$. All three of them are needed for a proper simulation of the hardening and softening behaviour of the material. Firstly, for stresses in the elastic range, the elastic limit surface $Y_{elastic}$ prevails, whereas as stresses increase nonlinearity is introduced via strain hardening till the failure surface $Y_{failure}$ is reached (Hu et al., 2016). The mathematical description of the elastic limit surface is given in Equation (8.1).

$$Y_{elastic} = Y_{failure} \times F_{elastic} \times F_{cap} \quad (8.1)$$

The above equation shows that the elastic limit surface is a result of scaling down the failure surface by introducing a scaling factor $F_{elastic}$ and a parabolic cap function F_{cap} . The latter is used

to guarantee consistency between inelastic volumetric and deviatoric stresses (Riedel et al., 2010). The scaling factor $F_{elastic}$ is the ratio between elastic or cracking strength to the ultimate strength for tension and compression. Initiation of inelastic behaviour of THFRC under tension is different than compression. As observed in literature but also from the experimental data used in this study, inelasticity starts at 80% and 40 % of the respective strengths, under tension and compression, respectively.

The strain hardening in the model is defined by the pre-peak loading surface $Y_{pre-peak}$. As illustrated in Figure 8.12, it is obtained via interpolating between the elastic limit surface and the failure surface. The pre-peak surface is described mathematically in Equation (8.2) where ϵ_{pl} is the plastic strain before ultimate strength, while $\epsilon_{pl,pre-softening}$ is the total plastic strain determined by the secant modulus defined between the elastic limit surface and the failure surface.

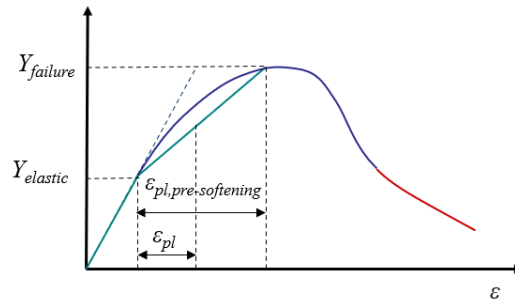


Figure 8.12: Bilinear Strain hardening in RHT model (modified from ANSYS, 2022)

$$Y_{pre-peak} = Y_{elastic} + \frac{\epsilon_{pl}}{\epsilon_{pl,pre-softening}} (Y_{failure} - Y_{elastic}) \quad (8.2)$$

The failure surface $Y_{failure}$ which must pass through the point of uniaxial compression at a coordinate of (1/3, 1) as illustrated in Figure 8.11a is expressed mathematically as a function of the compression meridian $Y_{TXC}(p)$ where p the hydrostatic pressure, Lode angle θ and strain rate $\dot{\epsilon}$:

$$Y_{failure} = Y_{TXC}(p) \times R_3(\theta) \times F_{rate}(\dot{\epsilon}) \quad (8.3)$$

The strain rate effect is expressed through the hardening factor $F_{rate}(\dot{\epsilon})$ which is also called Dynamic Increase Factor (DIF). The hardening factor is different in tension and compression. Equation (8.3) describes DIF as a function of the quasi-static strain rate $\dot{\epsilon}_0$ and the strain rate

exponents in tension δ and compression a . In this study, DIF was kept in its default value and since there were no dynamic effects, further investigation in that property was not deemed necessary.

$$F_{rate}(\dot{\epsilon}) = \begin{cases} \left(\frac{\dot{\epsilon}}{\dot{\epsilon}_o}\right)^a & \text{for } p > \frac{f_c}{3} \text{ with } \dot{\epsilon}_o = 30 \times 10^{-6} s^{-1} \text{ compression} \\ \left(\frac{\dot{\epsilon}}{\dot{\epsilon}_o}\right)^{\delta} & \text{for } p > \frac{f_c}{3} \text{ with } \dot{\epsilon}_o = 3 \times 10^{-6} s^{-1} \text{ tension} \end{cases} \quad (8.4)$$

The compressive meridian is further described by Equation 8-5.

$$Y_{Txc}(p) = A \cdot \left(\frac{p}{f_c} - \frac{p_{spall}}{f_c} \right)^N \quad (8.5)$$

In Equation (8.5), p/f_c is the hydrostatic pressure normalized with respect to the compressive strength while p_{spall}/f_c is the normalized spalling/tensile strength. A and N are strength parameters of the compressive meridian, which might be determined through triaxial compression tests.

The Lode angle function $R_3(\theta)$ represents the relationship between the failure strength of concrete, the second and third invariant of the deviatoric stress tensor and hydrostatic pressure and is described in Equation (8.6), Q_2 is the portion of the equivalent strength r_t on the tensile meridian to the strength of triaxial compression r_c . As observed from Equation 8-6, the Lode angle depends on Q_o which is the ratio of tensile and compressive strength and B_Q which defines the rate at which the fracture surface undergoes a brittle to ductile transition. As illustrated in Figure 8.13, the deviatoric section at low pressures transitions from a triangular shape (brittle nature) to a circular at high pressures (ductile nature) (Chen, 1982).

$$R_3(\theta) = \frac{2(1-Q_2^2)\cos\theta + (2Q_2-1) \cdot \sqrt{4(1-Q_2^2)\cos^2\theta + 5Q_2^2 - 4Q_2}}{4(1-Q_2^2)\cos^2\theta + (1-2Q_2)^2} \quad (8.6)$$

$$Q_2 = \frac{r_t}{r_c} = Q_o + B_Q p^*, \quad 0.51 \leq Q_2 \leq 1.0$$

$$\theta = \frac{1}{3} \cos^{-1} \left(\frac{3\sqrt{3}J_3}{2J_2^{1/2}} \right), \quad 0 \leq \theta \leq \frac{\pi}{3}$$

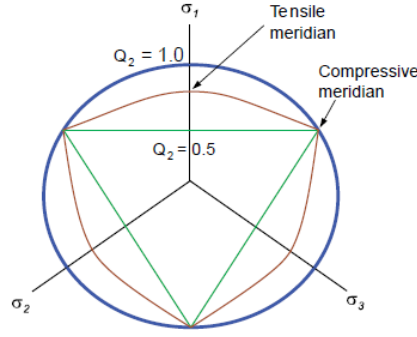


Figure 8.13: Typical deviatoric cross section of strength surfaces and third invariant dependence in RHT model (ANSYS, 2022)

After initiation of failure, the RHT model also describes mathematically the residual strength of crushed concrete by introducing an independent residual strength surface $Y_{residual}$ with Equation (8.7). With residual strength surface, a certain level of shear strength is due to friction between the crushed particles. B and M_1 are dimensionless constants that can be determined through curve fitting of the experimental data. Generally lower values of B will only reduce the residual strength and accelerate the failure of the material.

$$Y_{residual} = \min \left\{ B \cdot \left(\frac{p}{f_c} \right)^{M_1}, Y_{TNC} \cdot SFMAX \right\} \quad (8.7)$$

The second term in the above equation is used as a limit to the maximum residual shear strength to be a fraction of the current fracture strength (SFMAX).

As discussed above, the RHT model provides through interpolation a hardening phase between the elastic and the failure surface. In a similar manner, there is a softening phase which describes the gradual loss of load carrying capacity at post peak. This is called post failure surface $Y_{fracture}$ and can be obtained through linear interpolation between the failure surface $Y_{failure}$ and the residual surface $Y_{residual}$ and incorporation of a damage factor D by means of Equation (8.8).

$$Y_{fracture} = Y_{failure} + D \times (Y_{failure} - Y_{residual}) \quad (8.8)$$

The damage is assumed to be accumulated on account of inelastic deviatoric straining (shear induced cracking) according to Equation (8.9), where d_{ep} is the plastic strain increment, D_1 & D_2 shape parameters and ϵ_{fmin} the minimum failure strain which can be $\epsilon_{t,fmin}$ for tensile failure and

$\varepsilon_{c,fmin}$ for compressive failure. However, it has been observed that the default values for the damage parameters in the RHT model are problematic in some problems, and there are studies available in the literature recommending modifications (Hu et al., 2016).

$$D = \begin{cases} \int_0^{\varepsilon_p} \frac{d\varepsilon_p}{D_1 \left(\frac{p}{f_c} - \frac{p_{spall}}{f_c} \right)^{D_2}}, & \text{for } D_1 \left(\frac{p}{f_c} - \frac{p_{spall}}{f_c} \right)^{D_2} > \varepsilon_{fmin} \\ \int_0^{\varepsilon_p} \frac{d\varepsilon_p}{\varepsilon_{fmin}}, & \text{for } D_1 \left(\frac{p}{f_c} - \frac{p_{spall}}{f_c} \right)^{D_2} \leq \varepsilon_{fmin} \end{cases} \quad (8-9)$$

Damage accumulation may have two effects in the model: strain softening and reduction in shear stiffness. The current shear modulus G is mathematically expressed through the expression:

$$G = (1 - D)G_{elastic} + DG_{residual} \quad (8.10)$$

In addition to the RHT model, a crack softening failure criterion in a form of a linear softening law is employed to model the tensile softening behaviour with consideration of the fracture energy. After peak stress σ_t , the law follows a constant strength degradation rate with respect to the cracking strain. Therefore, the softening law parameters defined in the model is the ultimate tensile strength σ_t and the fracture energy G_f . During the cracking, the discrete crack width w obtained from the inverse analysis is smeared out over a certain length, which in FE is equal to the characteristic length L_{ch} . The calculation of the characteristic length depends on the element type and is discussed in future sections. Since the fracture energy is known experimentally along with the ultimate tensile strength, the following equation is used to determine the tensile failure strain $\varepsilon_{t,fmin}$:

$$\varepsilon_{t,fmin} = \frac{2G_f}{\sigma_t \cdot L_{ch}} \quad (8.11)$$

Finally, the shear modulus G and bulk modulus K_{bulk} were defined using Young's Modulus E_c and Poisson's ratio ν as follows:

$$G = \frac{E_c}{2(1+\nu)}, \quad K_{bulk} = \frac{E_c}{3(1-2\nu)} \quad (8.12)$$

As obtained through the uniaxial compression experimental data and already defined in the implicit model the Young's Modulus is $E_c=48$ GPa and Poisson's ratio is $\nu=0.2$.

For this study, no modifications were performed in constants and parameters other than strength ratios and the minimum tensile strain to failure obtained using Equation (8.11). Table 8.3 presents the default parameters of RHT, and the ones used in the present study.

Table 8.3: Material parameters of RHT model for THPFRC

Parameter	Symbol	Default Values for C140	Value used in this model	Unit
Density	ρ_c	2520	2520	kg/m ³
Use Cap on Elastic Surface		Yes		
Compressive Strength	f_c	140	120	MPa
Tensile Strength ratio	f_t/f_c	0.1	0.088	-
Shear Strength ratio	f_s/f_c	0.18	0.25	-
Intact Failure Surface Constant	A	1.6	1.6	-
Intact Failure Surface Exponent	N	0.61	0.61	-
Tension/Compression Meridian Ratio	$Q_{2.0}$	0.06805	0.06805	-
Brittle to Ductile Transition	B_Q	0.0105	0.0105	-
Hardening Slope	$G_{el}/(G_{el}-G_{pl})$	2.0	2.0	-
Elastic Tensile Strength ratio	f_{cr}/f_t	0.7	0.8278	-
Elastic Compressive Strength ratio	$f_{c,0}/f_c$	0.53	0.4	-
Fracture Strength Constant	B	1.6	1.6	-
Fracture Strain Exponent	M_1	0.61	0.61	-
Maximum Fracture Strength Ratio	$SFMAX$	1E+20	1E+20	-
Damage Constant 1	D_1	0.04	0.04	-
Damage Constant 2	D_2	1	1	-
Minimum Tensile Strain to Failure	$\varepsilon_{t,min}$	0.01	0.3	%
Residual Shear Modulus Fraction		0.13	0.13	-
Bulk Modulus	K_c	35270	26667	MPa
Shear Modulus	G	35270	20000	MPa
Maximum Principal Tensile Stress	σ_t	-	10.57	MPa
Fracture Energy	G_f	-	21360	J/m ²

- *Constitutive Model for Experimental Setup Parts*

As in case of the implicit model, the material model of structural steel was used for the simulation of the experimental setup parts. The material properties of Structural Steel in ANSYS are given in Table 8.4.

Table 8.4: Material Properties of Structural Steel used for setup parts in ANSYS

Parameters	Symbol	Value	Unit
Density	ρ_s	7,850	Kg/m ³
Young's Modulus	E_s	210,000	MPa
Poisson's Ratio	ν_s	0.3	-
Bulk Modulus	K_s	166,667	MPa
Shear Modulus	G_s	76,923	MPa

- *Loading and Boundary Conditions*

The loading and boundary conditions employed in the explicit dynamics analysis were the same with those previously explained in the implicit model. However, there were some differences due to the introduction of nonlinear contact conditions which are described in detail in the next section. More specifically, the displacement of 10 mm was applied on the cylindrical face of the impactor, while the cylindrical face of the roller support was restricted in moving in the Y direction. Figure 8.14 illustrates the loading and boundary conditions for the explicit dynamics FEA model.

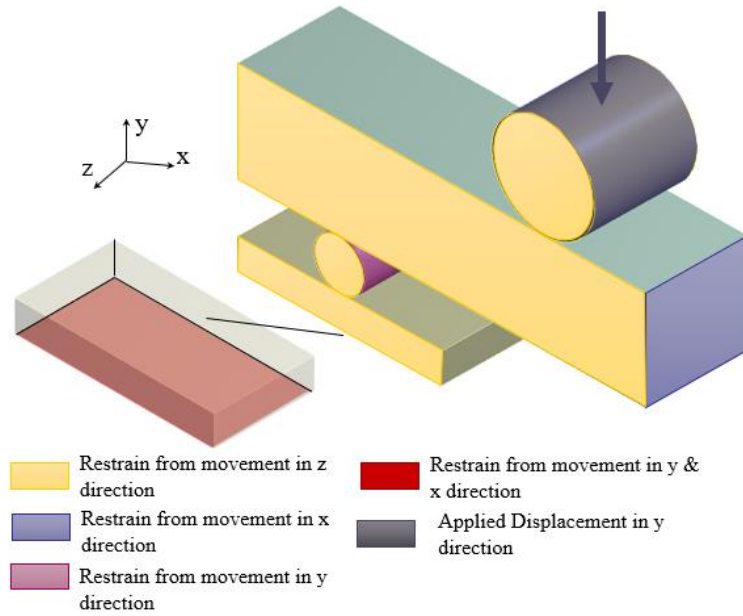


Figure 8.14: Loading and boundary conditions of explicit FE model in ANSYS

- *Contact Conditions*

An advantage of the explicit method over the implicit is the ability to simulate and solve without convergence issues nonlinear contact models, especially sliding or moving. This is attributed to the automatic contact detection at any point of loading. To simulate accurately the contact of the flexural prisms with the impactor and the support roller but also the contact between the roller and the steel base, frictional conditions were used (Figure 8.15). Frictional body interaction is a symmetric sliding contact and is based on the Coulomb type of friction between bodies ($F = \mu R$). The frictional contact is determined through the friction coefficient μ which is given in the following Equation (8.13) (ANSYS, 2022).

$$\mu = \mu_d + (\mu_s - \mu_d) e^{-\beta v} \quad (8.13)$$

In the above equation, μ_d is the dynamic friction coefficient, μ_s is the static friction coefficient, β is the exponential decay coefficient and v the relative sliding velocity at the point of contact. In this study, for simplicity, it is assumed that the dynamic friction coefficient is equal to the static coefficient of friction. For contact between steel roller and steel base, the friction coefficient is 0.2, while for concrete and steel is 0.5.

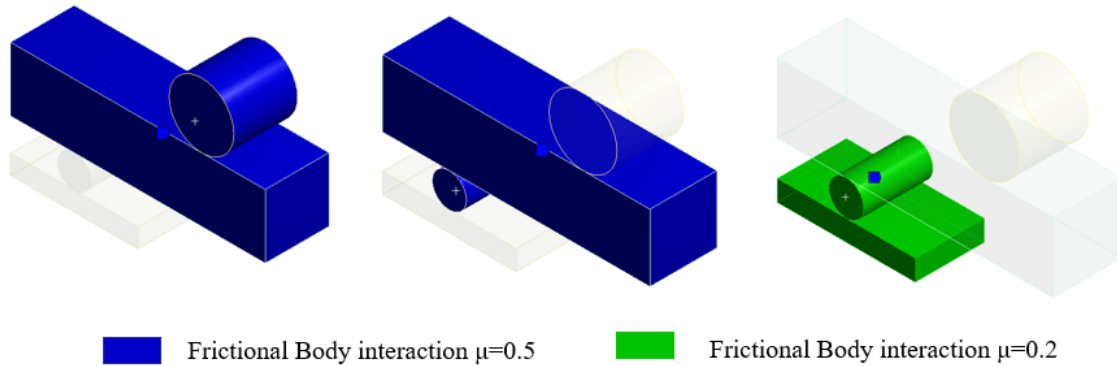


Figure 8.15: Frictional body interactions of the explicit FE model in ANSYS

- *Mesh Type*

Generally, in explicit dynamics a uniform mesh is advised across a body. This is because the time step is controlled by the smallest mesh element throughout the model, as it is explained in a future section. Due to this dependency, the element size difference might skew the results more significantly than in the implicit solver (ANSYS, 2022). Additionally, hexahedral elements are the most suitable type for this simulation. For this reason, only hexahedral elements were used for all the bodies. For the concrete beam, a mesh size of 10 mm was employed, while for the steel parts it was taken equal to 5 mm. The impactor was discretized in an axially symmetric manner. Figure 8.16 illustrates the mesh generated in the explicit FEA model.

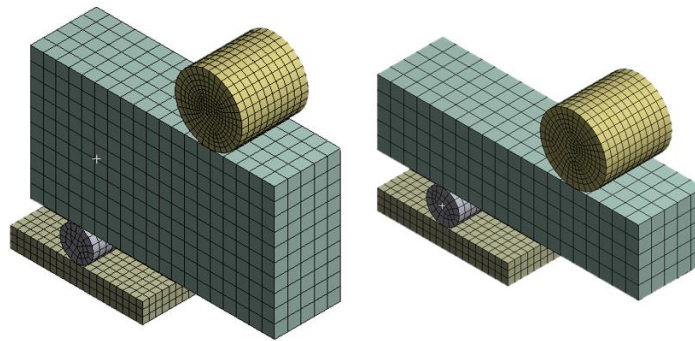


Figure 8.16: Generated mesh for explicit FEA models in ANSYS

- *Solution Parameters: Time Scaling, Mass Scaling and Wave Propagation*

Explicit Dynamics FE simulations determine the dynamics response of a structure due to impact or rapidly changing time-dependent loads. As a result, the momentum exchange between moving bodies and inertial effects are significant. However, as mentioned above, if the inertia forces are not controlled, the high velocity can change the physics of the model (ANSYS, 2022). Contrary to the implicit method, whose accuracy does not depend on the size of the time step, the explicit method has a time-size requirement. For this reason, the time-step size of the solver has to be determined accordingly. More specifically if the time step is not small enough, stability issues may occur. To avoid the latter, a limit is imposed on the time-step size so that a stress wave (disturbance) is unable to travel further than the smallest element characteristic length in single time step (ANSYS, 2022). This criterion which is related to time integration in the explicit solver is described by the Courant-Friedrichs-Lewy (CFL) condition, as shown in Equation (8.14):

$$\Delta t \leq f \cdot \left[\frac{L_{ch}}{C_{sound}} \right]_{\min} \quad (8.14)$$

In CFL condition, Δt is the time increment, f is the stability timestep factor, which is set to 0.9 by default, C_{sound} the local material sound speed or wave speed of the material, and L_{ch} is the characteristic length of an element. The latter can be obtained according to the type of the element. In this study hexahedral elements were used, and the calculation of the characteristic length is the volume of the element divided by the square of the longest diagonal of the zone and scaled by $\sqrt{2/3}$. For a 10 mm hexahedral element the characteristic length was calculated as 2.72 mm.

When the material's sound speed wave C is not available, as in the present case, the Young's Modulus of the material E and its density ρ , can be used in order to obtain an approximation. Material's sound speed corresponds to the primary elastic wave (longitudinal), and as longitudinal waves travel faster than transverse, they can be used to control the time-step size. The material's sound speed is calculated based on the following Equation (8.15).

$$C_{sound} = \sqrt{\frac{E}{\rho}} \quad (8.15)$$

Since there are two materials in this simulation, there are two different sound speed waves and taking into consideration the different mesh size between the beam and the steel parts, there are two different time steps. Both time step sizes were examined and resulted in very similar responses. However, the time step size obtained using the concrete sound speed wave is used as it was computationally less expensive. The CFL minimum time step is 0.56 μsec with an end time of 1 sec.

Finally, on account of using a relatively large-scale analysis of a bending test that also includes the experimental hardware, another feature of explicit analysis was employed to expedite the computational procedure. This feature is mass scaling, a method in simulation to reduce run time for large scale explicit analysis. As shown above in the CFL condition, the maximum timestep that can be used in the explicit solver is inversely proportional to the sound speed of the material (ANSYS, 2022). Therefore, it is directly proportional to the square root of the mass of material in an element, as described in the following Equation (8.16).

$$\Delta t \propto C_{\text{sound}} = \frac{1}{\sqrt{C_{ii}/\rho}} = \sqrt{\frac{m}{V_e C_{ii}}} \quad (8.16)$$

In Equation (8.16), C_{ii} is the material stiffness, m the material mass and V the element volume. With mass scaling, the mass of the smallest elements of the model are artificially increased leading to increasing the maximum allowable stability and timestep and ultimately decreasing the computational time. The change in mass results to change in inertial properties that are known to have a significant effect on the explicit solution. For generally uniform mesh, this occurs in a small portion of the model and not to all the elements, so the global accuracy remains the same (ANSYS, 2022).

8.4. Analytical Results & Discussion

Figure 8.17 illustrates the analytical results of the resistance curves obtained from the different NLFEM models in comparison with the representative (average) experimental curve for the two different prism types. In case of the 4PBT prism, the analytical results agree with the experimental values. This is expected, since the inverse analysis procedure from which the tension stress-strain response has been extracted, was based on this group of specimens. However, in the case of

AFNOR prisms, discrepancies are observed regarding the post-peak softening branch. This could be an indicator that the constitutive law in tension obtained from inverse analysis on the 4PBT prisms is not an intrinsic property of the material since it fails to reproduce the independent test result (AFNOR) and further calibration is called for. This is also evident in Figure 6, where the longitudinal stress-strain response at the onset of crack localization obtained at the critical point of the prism from the NLFEM is compared to the stress strain curve obtained experimentally through inverse analysis of the 4PBT and input in the analysis of both 4PBT and the AFNOR specimens. It is noted that both explicit approaches (CDP and RHT) estimate an earlier localization which suggests that the hardening rate may have been overestimated in the inverse analysis. Figure 8.18(left) zooms into the strain-hardening zone; it is observed that both the CDP and the CC3DNLC2 models illustrate a mild-post cracking softening, which underscores a weakness of the inverse analysis result: In deriving the relationships for calculation of the milestone points of the tension response, all strain energy is attributed to tension fracture; however it appears that other sinks of strain energy exist in the actual structure, such as those occurring under the loading shafts and above the supporting rollers. Contrary to the plane sections – remaining plane assumption, that underlies the inverse analysis derivation, it is clear from the FEA results that vast non-uniformity of strains occurs across the width of the section, so that only a single point reaches and undergoes tension fracture as per the provided input, whereas all other points with a slightly lower strain magnitude generally unload instead of following the provided post-peak branch of the tension stress-strain law. Indeed, the obtained curves are overwhelmed with the post-peak response which is nevertheless of inferior importance and validity as it depends heavily on the rate of the dynamic analysis conducted in the post-peak range.

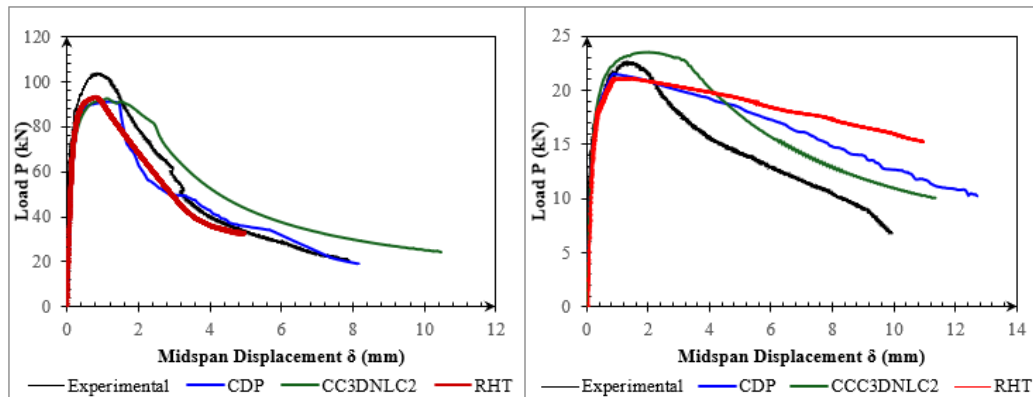


Figure 8.17: Comparison between analytical and experimental results for 4PBT (left) and AFNOR specimens (right)

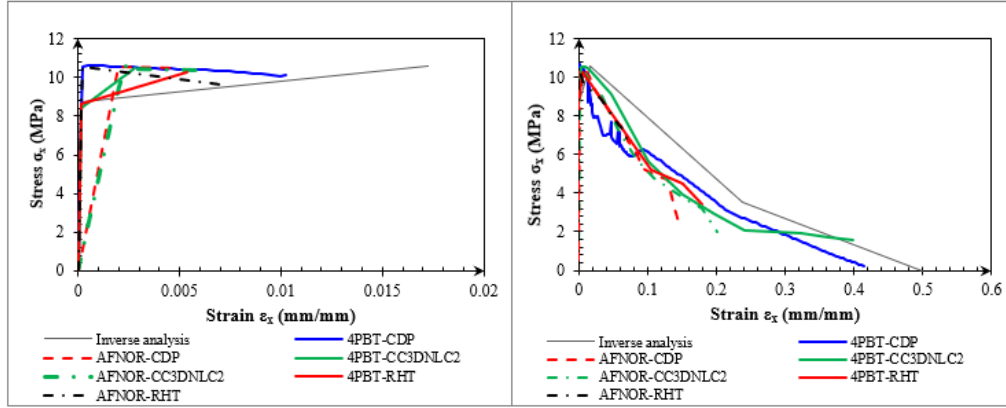


Figure 8.18: Comparison between numerically obtained stress-strain law in tension and inverse analysis

Figures 8.19 and 8.20 compare the strain responses and crack widths between the 4PBT and the AFNOR specimens. Each plot presents 3×3 snapshots. The first column of plots in the figure corresponds to the response at peak load, the second corresponds to a post-peak point at a load equal to 50% of peak, and the last column corresponds to the end point of the analysis. In each plot, the first row of plots presents the longitudinal strain distribution obtained from the RHT analysis; the second presents the distribution of the principal plastic strains obtained from the CDP (the model implemented in ABAQUS), whereas the last row presents the estimated crack widths from the CC3DNLC2 model. ATENA software is also equipped with the capability of graphical representation of cracks. In this way, besides the localized crack, microcracking with width greater than $1\mu\text{m}$ is also evident (see through black straight lines in Fig. 8.19). The comparison is meant to illustrate the effective difference between models in estimating the position of localization, as well as the effect of the specimen form on the tendency for crack localization.

Note that whereas at peak load the plastic deformation is rather distributed in all cases considered, the extent is different in each of the three software outputs; the difference is accentuated in the 4PBT paradigm, whereas the three models are consistent to each other in the case of the AFNOR specimen. In the post-peak range based on the experimental evidence the multi-crack phenomenon which characterizes the strain hardening range of the response of THFRC is attenuated and a single crack dominates; in the 4PBT tests it is often noticed that the dominating localized crack occurs near the location of the loading shaft. This is consistent with the observations from the RHT and the CC3DNLC2 models. Instead, the CDP model captures a broad process zone in the 50% of post-peak load as well as in the end of the test: both of these response snapshots do not jive well with the experimental evidence. Similarly, the position of localization is captured best (with

regards to the experimental observation) in the case of the CC3DNLC2 model, where the crack localizes near the loading shaft, but still within the constant moment zone of the 4PBT setup, whereas all other cracks close.

The length of the localization region that was effectively obtained from the analysis generally surpassed the characteristic length that was input and was generally different between the models considered. From among the three models, the CC3DNLC2 illustrated cracking by flaking (narrowly spaced, horizontal cracks) in the compression zone, a feature that was actually observed in the tests.

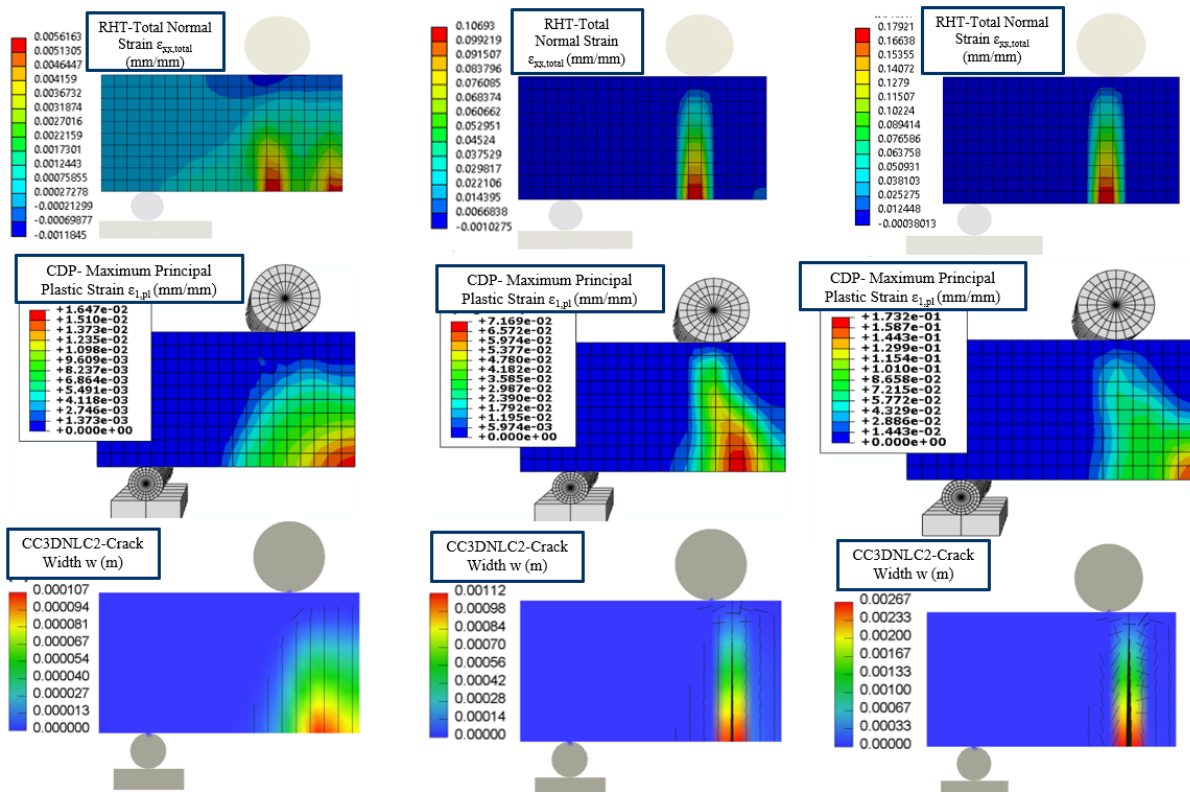


Figure 8.19: Crack pattern in 4PBT specimen for the three different concrete damage plasticity models at: Peak Load (left), 50% of Peak Load at softening (middle) and at the end of the resistance curve (right)

The differences between the three modelling approaches are attenuated and the responses become compatible with the experimental evidence in all three models when considering the AFNOR specimen performance. It is observed that AFNOR specimens demonstrate a more desirable crack pattern with a greater process zone, attributed to its more favourable shear span to aspect ratio. In the cases of RHT and CDP models the crack localizes in the midspan whereas in case of CC3DNLC2 model, the crack localizes in the area of constant moment but closer to the shaft. In

all three models multi-cracking in the process zone is visible, a finding that agrees with the experimental results. Localization is strongly evident in the case of the CC3DNLC2 model, with crack-widths in the dominant crack in the range of 1.4 mm, as compared with 2.7 mm in the case of the 4PBT. Strain values in the CDP and the RTH model results are in-agreement in the onset of localization as well as in the end of the response curve. Stark differences are observed in the intermediate range of the post-peak branch for both, since the exact geometry of that part of the curve depends on the details of the dynamic analysis conducted in the two explicit schemes.

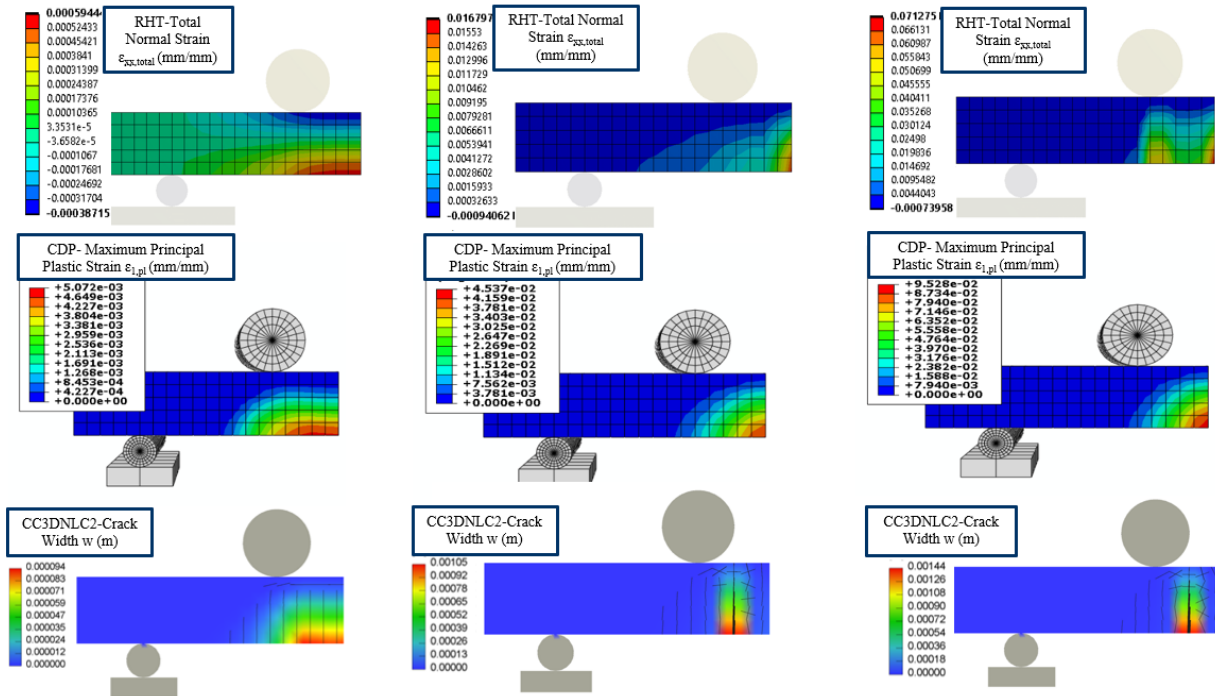


Figure 8.20: Crack pattern in AFNOR specimen for the three different concrete damage plasticity models at: Peak Load (left), 50% of Peak Load at softening (middle) and at the end of the resistance curve (right)

8.5. Conclusions

This chapter investigated the validity of inverse analysis approaches used in extracting the tension stress-strain response of THFRC materials. In this regard two different nonlinear finite element analysis models were used to simulate the response of two standard flexural tests that are currently accepted in order to conduct quality control tests for the characterization of the THFRC materials. These are the so-called 4PBT, which is currently used to extract the tensile properties of THFRC, as well as the so-called AFNOR test which has improved aspect ratio designed to attenuate the effect of shear on the response. Two prominent plasticity models were calibrated to model THFRC

materials throughout the range of strain-hardening and strain softening response. Although macroscopically the models were able to calibrate the response curve of the reference test specimens, differences in terms of the location of the dominant crack widths, energy dissipation, spread of inelasticity and tensile stress – strain responses were found between the three models, with convergence improving in the case of the more slender, flexural tests with the AFNOR specimen form. Importantly, it was concluded that the constitutive law in tension extracted from inverse analysis on the prisms specified by CSA is not an intrinsic property, and therefore an improved test method would be required to promote understanding of the constitutive behavior of THFRC in tension.

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Chapter 9: Tensile Characterization of THFRCs & Proposed Modified Splitting Test¹⁰

Two characteristics of THFRCs are the enhanced tensile strength and ductility due to the addition of steel fibers which demonstrate good bond but a relatively compliant pullout-slip interaction with the dense matrix. These are of utmost significance in the development of structures with controlled service cracking, high ductility under extreme events and significant durability. With the available sustained tensile strength up to large tensile strain levels, new opportunities for novel structural design with THFRC become feasible. Shear, which is a diagonal tension failure mechanism, bond and anchorage which are supported by the hoop tensile strength of the cover, confinement that precludes compression softening, torsion and crack control could be easily determined once the tensile strength and strain capacity are known. Nowadays, there is intensive ongoing research towards the development of structural design procedures for THFRC, on condition that consensus may be reached in the meantime, regarding a vetted test procedure to find through material characterization testing, the tensile stress – strain law of the material.

Relevant studies regarding a dependable test of the tensile properties of THFRC are of particular interest in Canada, where there is significant growth in the use of this class of materials in bridge engineering on account of the increased durability requirements related to the harsh winter exposure. Therefore, extensive use in design of THFRC rides on the thorough understanding and characterization of the constitutive behaviour of the material, especially in tension, so that the associated material constants may be established as intrinsic properties.

9.1. *Tensile Characterization Techniques*

9.1.1. Direct Tension Tests

The experimental characterization of the tensile strength of THFRC is mainly based on procedures in codes and standards used for fiber reinforced concrete (FRC). Uniaxial tests are currently offered as the possible alternative to flexural prisms. In fact, direct tension tests are considered most conservative; a counterargument to using direct tension tests both for characterization and

¹⁰ Part of this chapter has been used in the Author's publication with her supervisor, titled: "Correlation Between Tensile Strength & Modulus of Rupture for Ultra-High-Performance Concrete (UHPFRC)" Proceedings of the 2019 National ABC Conference including Automation, Service Life and UHPC

for quality control is that this type of test may be challenging to conduct. The specimen is usually a prism cast or saw-cut from a bigger structural member subjected to uniaxial tension. The response is captured through recorded characteristics such as distribution, spacing and width of cracks during the test and the stress-strain response can be obtained. However, the variation in geometry, boundary conditions and effect of size of the specimens on fiber orientation hinders the establishment of a standard test and prevents correlation between different studies. Figure 9.1 illustrates some of the direct tension tests proposed by codes and researchers. In North America, (AASHTO T 397, 2022) and CSA S6 - Annex 8 (CSA S6, 2025) have adopted a test developed by Graybeal & Baby, (2019). The test comprises of a constant squared cross-section prism with fixed ends subjected to direct tension. An advantage of the procedure is that due to its relatively large cross section (50 mm x 50 mm), fiber distribution has lesser effect the mechanical properties as prominently as in cases of thinner specimens, although in this case the requirement placed by the current Annex U of CSA A23.1:19/CSA A23.2:19, (2019) whereby the specimen size ought to be at least 5 times the fiber length is violated (most steel fibers used are at least 13 mm long, to the minimum dimension ought to have been at least 65 mm to satisfy that requirement). However, at the same time to achieve the 2-inch thickness (50 mm) which is considered minimal so as to ensure some objectivity in fiber distribution, the test requires special gripping device and cannot be performed in most laboratories with the available grips in universal testing frames due to the thickness restraint, without interventions in the specimen geometry. This is not an issue in case of SIA 2052, (2016) standard which proposed a direct tension test on a dog-bone shaped specimen with same conditions but with half the thickness (25 mm). For determination of tensile strength, (RILEM TC 162-F, 2003) uses a cored specimen with a circumferential notch at its mid-height. The cylinder is glued to the press platens by means of an epoxy resin and instrumented with six LVDTs. Besides these standards, many researchers have developed their own direct tension test, with most adopting a dog-bone shaped specimen with varying geometry and gripping action (Georgiou & Pantazopoulou, 2017; Mechtcherine et al., 2011; Wille et al., 2014).

The mechanisms of the established tests are either transfer of forces through shear or bearing action. In the first case, steel/aluminum plates are glued on the specimen ends or CFRP layers wrapping the gripping sides to eliminate friction from being directly applied to the concrete, to prevent local failure due to stress concentrations, and to encourage the formation of the critical cracking plane within the gauge length. Alternatively gripping can engage bearing action instead

of interfacial shear (Georgiou & Pantazopoulou, 2017). The advantage is the elimination of the thickness restraint imposed by many Universal Testing Machines as already mentioned.

Another challenge for the direct tension tests is in the boundary conditions as they have great influence, especially after the formation of a first crack which could be owing to non-uniformity of fibers; onset of a crack from one side of the specimen automatically generates eccentricity (Figure 9.2) even in perfectly aligned specimens, in either plane of action. Restraints to free rotation also affect the recorded response: For example, Benson & Karihaloo (2005), Wille et al. (2014), and many other researchers reported the influence of fixed ends versus rotating ends on the measured properties.

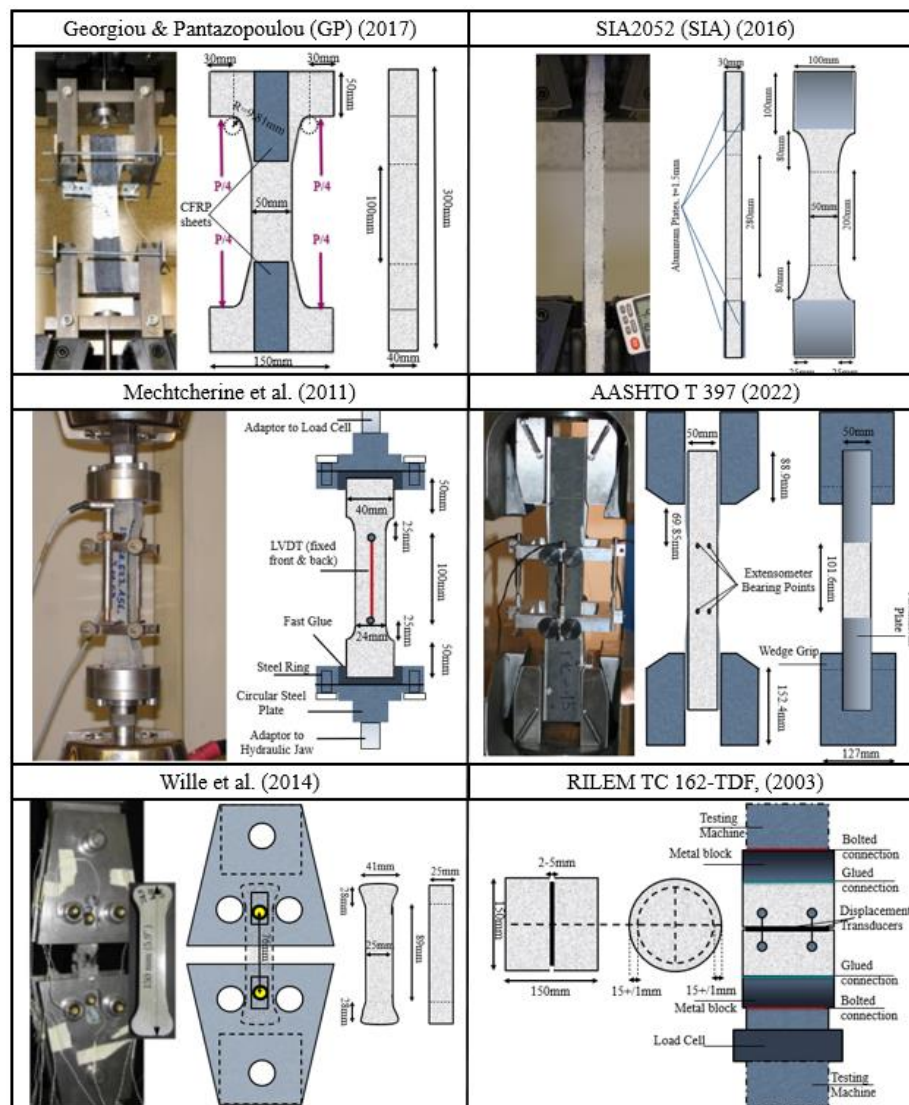


Figure 9.1: Test Setups for Direct Tension Testing of UHPFRC (extended from Pantazopoulou et al., 2019)

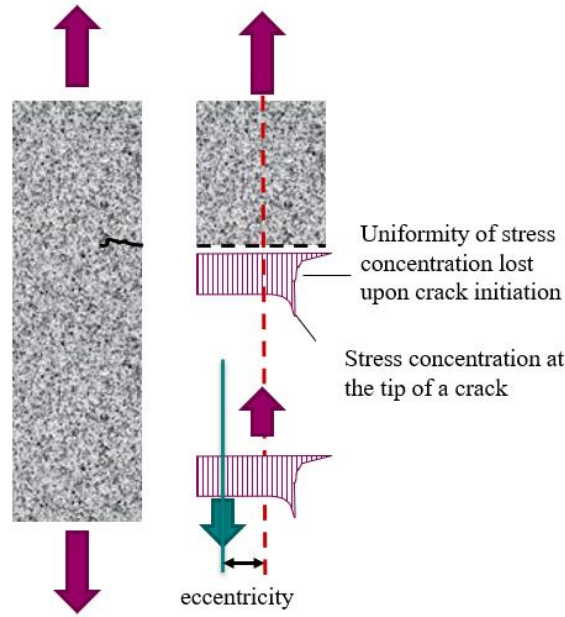


Figure 9.2: Eccentricity in direct tension specimen owing to the crack initiation leads to precipitous failure (Ralli & Pantazopoulou, 2019)

9.1.2. Modulus of Rupture Tests and Inverse Analysis

The use of flexural tests for experimental characterization of the tensile behavior of THFRC as a concept has been extended from the notched beam tests that are often used for assessing the tensile strength of conventional concrete (modulus of rupture tests). Because the material is strain-hardening, notches could not be used, as the static system of the three-point bending test in a notched beam does not allow for spread of plasticity. To enable the development of multiple cracking, which is the underlying source of the macroscopic strain-hardening characteristic of the tension response of THFRC, four-point bending tests became standard practice; in the central constant moment region multiple cracking occurs leading to significant length change of the test specimen. Depending on the support and loading conditions, the required length change can be partially restrained leading to overshooting of the load carried and the corresponding tensile strength that could be obtained from inverse analysis (Ralli and Pantazopoulou, 2019). For this reason, roller supports are provided in both ends of the specimen rather than the conventional pin-roller that is often used for beam testing (AFNOR NF EN 13670/CN, 2013). Rollers are also used for load application in the central span; the placement is symmetric, however, some closed form expressions that have been derived for inverse analysis of the flexural tests for data reduction and extraction of the tensile stress-strain response may be tied to specific assumptions and geometric

details that do not lend them applicable to all types of four-point bending tests (e.g. López et al., 2016) Canadian (CSA A23.1:19/CSA A23.2:19, 2019; CSA S6, 2018), Japanese (JSCE, 2006), French (AFNOR NF EN 13670/CN, 2013) and Swiss (SIA 2052, 2016) Standards all include bending tests for assessing the tensile properties tests especially for strain hardening UHPFRC. In fact, the above inverse analysis method has been adopted by several of these codes: it has been benchmarked to the so-called third point bending test, whereby the intermediate symmetric loads are equidistanted with the end supports (a distance of $L/3$ between support rollers and loading rollers, where L is the span length). The adopted test setup in the CSA S6-Annex 8 (2019) refers to a beam specimen with square cross section a , where the distance between supports is equal to 3 times the section dimension a . Clearly, this setup has a strong shear component to it, as the shear span aspect ratio is equal to 1. An improved version that is still compatible to the inverse analysis model described above is the so-called AFNOR specimen (AFNOR NF EN 13670/CN, 2013) where the cross-sectional height is $0.5a$, so that the shear span aspect ratio is improved to 2.

The intrinsic difficulties and uncertainties of the testing methods necessitated the adoption of flexural tests by several codes and standards; but the dependency on the inverse analysis procedure to extract the properties reduces the attractiveness of the flexural tests as outlined in the preceding, because there is evidence of systematic overestimation of the cracking and tensile strengths at least as compared to the direct tension companion specimens. Alternative back-calculation approaches are available in the literature, without being fraught with the underlying assumptions that lead to this overestimation or the geometric restrictions of the test setup (Georgiou & Pantazopoulou, 2016; Mobasher et al., 2014); however, the combined uncertainty that has been introduced by the use of shear-dominated specimens as well as the sensitivity of the flexural test to the hardware details (shaft diameter and degree of restraint provided by shafts rotating about stationary axles which is an industry standard in many test setups of prominent servocontrolled equipment available in most labs today), has undermined the confidence in the flexural tests and particularly in the characterization of properties obtained through inverse analysis methods.

9.1.3. Indirect Tensile Tests

Other alternatives for indirect evaluation of the tensile characteristics are possible, provided the response is dominated by clearly identifiable, pure tensile failure. As in the case of flexural tests, the motivation here is to come up with a test setup that uses the available equipment and hardware

that most labs have, without requiring special gripping devices as is the case with the direct tension test. Variations of such alternatives are the well-known Brazilian splitting test whereby a cylindrical specimen is subjected to compressive load, along two diametrically opposed generating lines of the cylinder (Figure 9.3(a)). Uniform transverse tensile stress state is generated almost lengthwise and height wise in the diametric plane that is parallel to the applied loads (Figure 9.3(b), (c)). However, for THFRC materials the response becomes complicated after diametric cracking of the cylinder, because of the ductility in the slitting plane; thus, significant load bearing may occur after cracking, where complex stress states may develop (Fig. 9.3(d)) without being possible to identify and separate the clear contribution of post-cracking pure tensile resistance of the material in the obtained response.

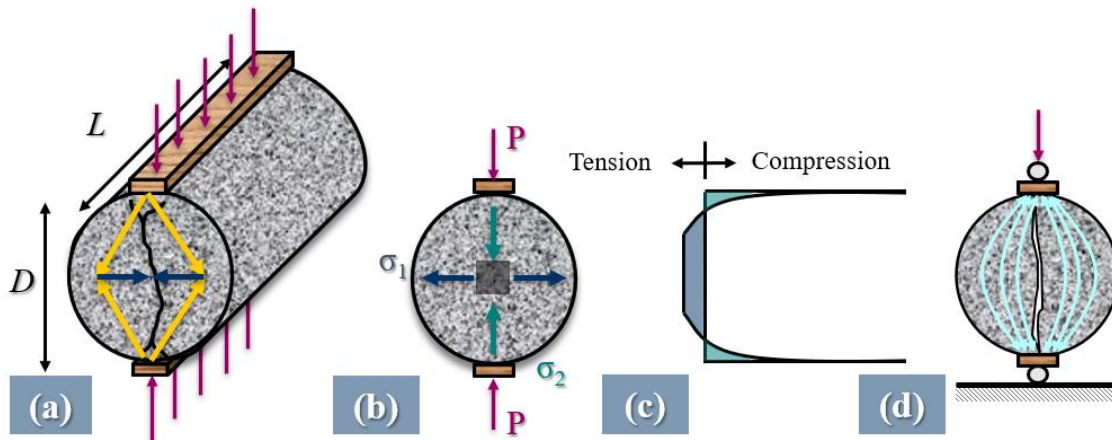


Figure 9.3: Splitting test (a) Strut & Tie Mechanism, (b) biaxial stress state (c) stress profile along the diametric plane and (d) complex stress state after cracking

Another test originally proposed by Linsbauer & Tschegg, (1986) and later further developed and modified by Brühwiler & Wittmann (1990) is the Wedge Splitting Test (WST). As illustrated in Figure 9.4, the test comprises a notched cube (could be also a cylinder) bearing on a linear support fixed on steel plate, while rollers attached to loading devices are sitting on top of the specimen. Finally, the setup includes a stiff steel frame consisting of a pair of wedges fixed at the upper plate between the rollers. Crack propagation occurs in the vertical direction and the specimen splits into two halves. The horizontal component of the force (P_h) on the rollers is defined as splitting force (F_s) and may be calculated with respect to the wedge angle α and the applied load (vertical component (P_v)) according with Equation (9.1):

$$F_s = P_h = (P_v/2) / \tan \alpha \quad (9.1)$$

A COD gauge clip measures the crack opening allowing for determination of fracture energy and stress-crack opening relationship (Löfgren et al., 2004). However, a variability in the number of fibres across the fracture plane with the notch, affects the reliability of the test due to high dispersion in experimental results.

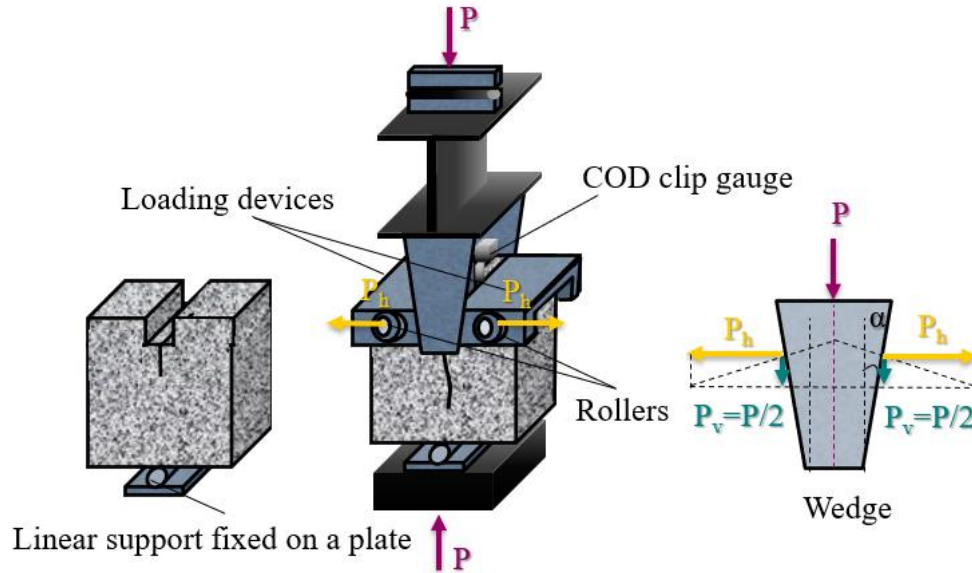


Figure 9.4: WST principle, configuration, and static system (modified from Brühwiler and Wittmann, 19990)

Following the same philosophy, di Prisco et al. (2013) proposed the Double Edge Wedge Splitting Test (DEWST). Although the test was developed for FRC, it has been implemented for characterization of tensile response of THFRC (Io Monte & Ferrara, 2020). As illustrated in Figure 9.5, the approach comprises of a double notched panel (100 mm x 100 mm x 25 mm) with cylindrical wedges sitting on the two triangular grooves at a 45° angle and compressive load applied through them. Due to its geometry, the trajectory of principal compressive stresses diverges from the fracture plane (ligament), while a pure uniaxial transverse tensile stress state is generated along the same fracture plane from the wedging action. The intensity of the latter can be obtained through equilibrium. At this point it should be noted that the contact between rollers and groove edges could exert friction through a tangential force and thus affect the transverse tensile load. For this reason, special care is needed to minimize the friction effects. An apparent feature of DEWST is the spatial disengagement between tensile and compressive stresses. Moreover, the latter is beneficial especially in case of THFRC as it prevents the generation of plastic deformations to occur in the small area under the loading device (see Fig. 9.3(c)) (ACI 544.9R-17, 2017) . The resulting transverse splitting force F_{sp} due to the applied compressive load (P) and with respect to

the friction coefficient (μ) and the inclination angle of the wedge groove ($\theta=45^\circ$) can be obtained using Equation (9.2):

$$F_{sp} = P \frac{\cos \theta - \mu \sin \theta}{\sin \theta + \mu \cos \theta} \quad (9.2)$$

Gluing brass platens to the groove edges and lubricating the contact surface with graphite is found to be the optimum preparation to minimize friction, as the friction coefficient was reduced to $\mu=0.06$ and thus the splitting force to $F_{sp}=0.89P$ (ACI 544.9R-17, 2017).

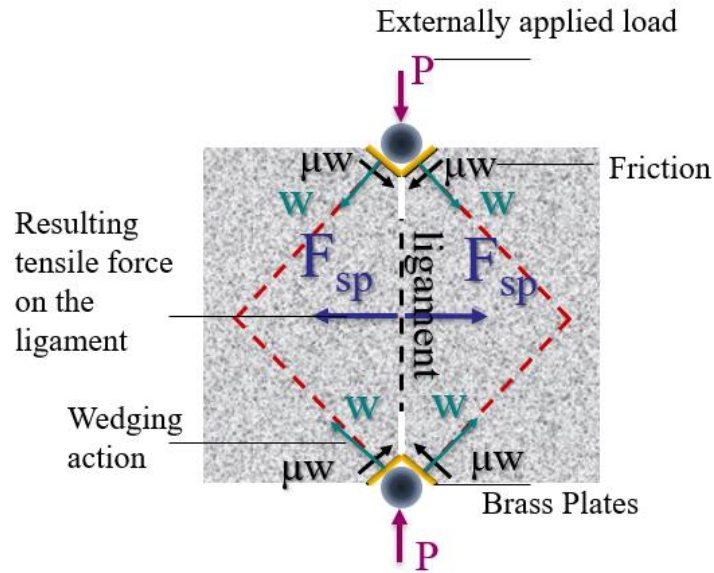


Figure 9.5: Geometry and force diagram in DEWST specimen

The Double Punch Test (DPT) or Barcelona test is another approach developed by Molins et al. (2009) for indirect tensile characterization of FRC, but recently adopted for UHPFRC, too (Tuladhar & Chao, 2019). The test comprises a cylindrical FRC specimen with height equal to its diameter that is subjected to compression through two steel punches, placed on top and bottom surfaces of the cylinder along its axis as illustrated in Figure 9.6(a). The punch diameter is usually one quarter of the cylinder's diameter and height. Tensile stresses are generated over diametric planes due to the applied compression. The failure pattern includes the formation of semi-cylindrical wedges beneath each punch, separated by vertical and radial cracks (Figure 9.6(b)). During the test, the vertical and total circumferential opening displacement are measured, by means of LVDTs and a circumferential extensometer at midheight, respectively (ACI 544.9R, 2017). Tuladhar & Chao (2019) obtained the average crack width by dividing the total measured

crack width by the number of cracks (N) which was determined by visual inspection upon completion of the test. The tensile strength can be obtained through plasticity analysis (Tuladhar & Chao, 2019). Equation (9.3) calculates the tensile strength with respect to the applied load and the height (h) and diameter (D) of the cylinder and the diameter of the punches (a):

$$f_t = \frac{0.75P}{\pi(1.2hD - a^2)} \quad (9.3)$$

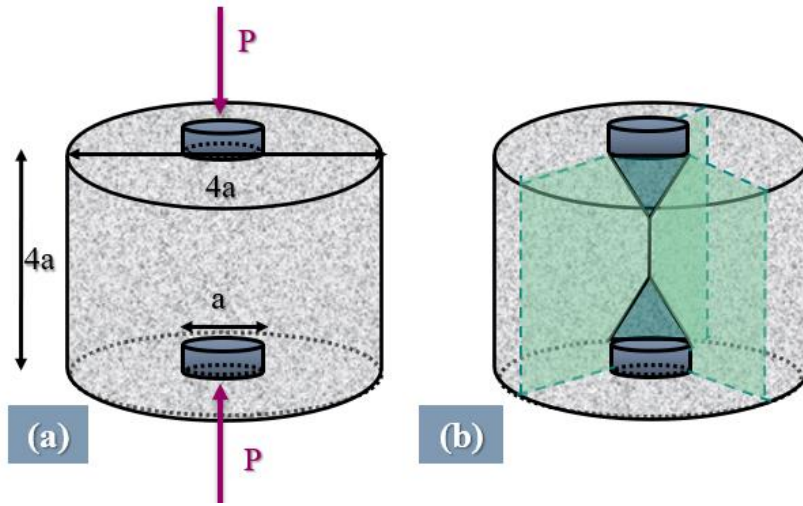


Figure 9.6: (a) Double Punch Test layout (adopted from Molis et al., 2009) (b) Failure surfaces and formation of semicylindrical wedges

The main benefits of this test are: (i) simple geometry which can be either cast or cored, (ii) simple setup and testing procedure utilizing a widely available equipment (standard, cylinder compression testing frame) and (iii) lower standard deviation in the data, compared to bending and DTT. The latter is due to the fact that the multiple crack planes reveal large areas of the specimen crossed by fibers; in this manner results are averaged thereby dwarfing the influence of fiber distribution on any single critical crack plane, which is the case in the standard tests (Tuladhar & Chao, 2019). However, no information about the effect of fiber orientation on the material behaviour may be obtained from DPT particularly in cored specimens.

9.2. Proposed Modified Splitting Test

9.2.1. Background

It was demonstrated in the preceding review that characterization of the tensile behaviour of THFRC remains a conundrum in research community with implications on both the Testing and

the UHPFRC industry, as this step is essential for the introduction of any such material in construction. Alternative tests that combine the simplicity of splitting test with elimination of stress distribution uncertainties are needed, so as to facilitate quality control testing by private testing facilities who may not have access to UTMs with special gripping hardware; indeed, most of these testing centers rely mostly on compression testing machines. In this section, a series of new models to evaluate easily and accurately the tensile response of THFRC are presented. All of the approaches are based on the strut-tie model, the same model used for the conventional splitting test. The differentiation lies on modifying the specimen's geometry by removing parts of the cylindrical disk in order to form the compression struts that transfer the load to the tie, which would be a well-defined central gauge region with constant cross-section that develops a pure tensile stress state. The load is applied through stainless-steel rollers that lie between a notch on the specimen and the loading steel plate. All contacts between hardware and specimen are assumed to be frictionless and care ought to be taken in the final implementation of the prevailing design to ensure the validity of this assumption.

9.2.2. Design Approaches

In order to investigate the suitability of the strut and tie model for tensile characterization and how to optimize the design of the specimen and experimental setup, eight different designs were simulated in Nonlinear Finite Element Analysis using the material models that were formulated in Chapter 8. The specimen designs are illustrated in Figure 9.7. The first two designs are alternatives to the DEWST, where instead of a rectangular slab, the specimen is a cylinder with triangular grooves with 45° angle on diametrically opposed edges. The difference between the designs of DEWS and DEWS-M is the absence of the ligaments on the vicinity of the grooves. The rest of designs are based on clearly isolating a central chord that will undergo pure tension when the specimens is subjected to splitting. More specifically in designs with MS in their label, the loading rollers are sitting on 45° triangular grooves placed on diametrically opposing lines. The differences between those designs are in the shape of the recess parts, the cross section of the central cord and the width of the formed neck under the grooves. Design MS2 is a modification of a test proposed by Resan et al. (2020) for normal concrete. Finally, for STF, the wedges are bearing on the grooves of the specimen.

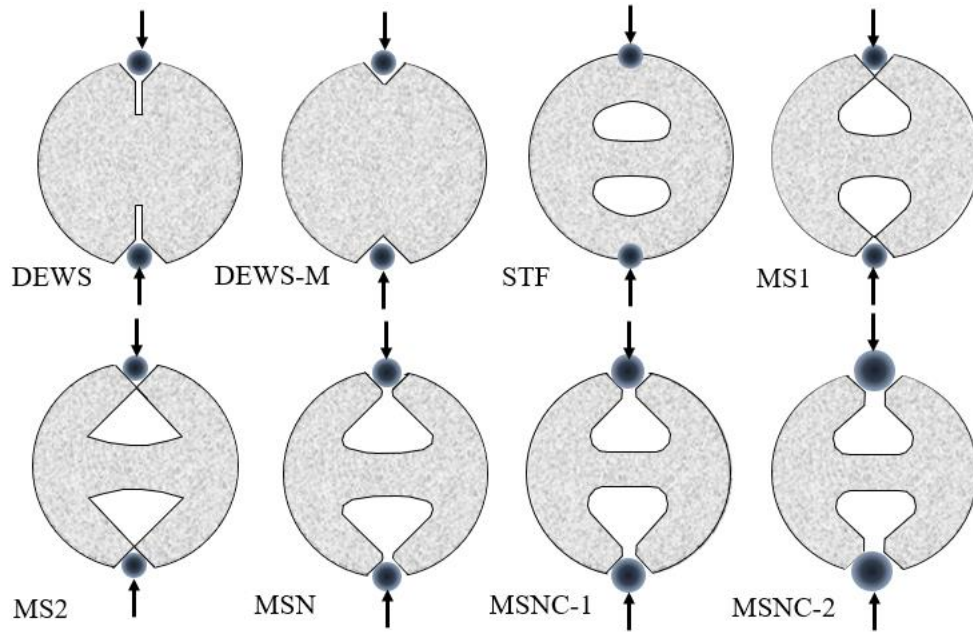


Figure 9.7: Different Designs considered for Modified Splitting Test

As discussed above, the designs of MS category were based on the formation of a mechanism of strut and tie. Due to the 45° angle that is created in the strut and tie, the tensile force R exerted from the central chord is equal to the applied load P . The statics of this mechanism are illustrated in Figure 9.8.

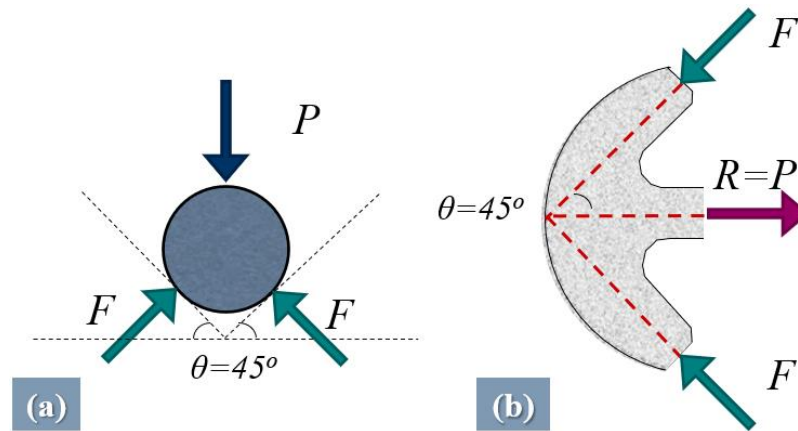


Figure 9.8: Free Body Diagrams of (a) roller and (b) specimen for MS category.

From the Free Body Diagram illustrated in Figure 9.8(a) and considering equilibrium in the y axis, the component F of the load P is given by Eq. (9.4):

$$P = 2F \sin \theta \quad (9.4)$$

Then, by establishing equilibrium in the x axis in Figure 9.8(b), the resultant force R is obtained from Eq. (9.5):

$$R = 2F \cos \theta \quad (9.5)$$

For $\theta=45^\circ$, the applied load P is equal to the resultant force R in the central chord. The above is true in case of a frictionless support. For frictional contact between the specimen and the roller, friction may be taken into consideration by using Equation (9.2).

9.2.3. Computational Validation

All the above design approaches were simulated in a Non-Linear Finite Element Model in order to assess which one is more appropriate for implementation and further experimental investigation. The simulation was performed in an explicit dynamics' solver in ANSYS software using the RHT concrete constitutive model adapted to THFRC in Chapter 8. Conducting quasi-static analysis with RHT has been explained thoroughly in Chapter 8. The comparison between the various designs is made based on the ratio between the applied load P, the exerted tensile force F and the failure plane location. It is important that the P:F ratio has to be as close to 1 as possible and also that the failure plane to occur within an area of constant cross-section.

- *Geometry of FE Models*

In all cases symmetry was exploited in all axes and therefore, one eighth of the designed forms was simulated. The roller was also simulated in order to investigate the contact conditions and examine the effect of support type. An example of the simulated geometry is shown in Figure 9.9.

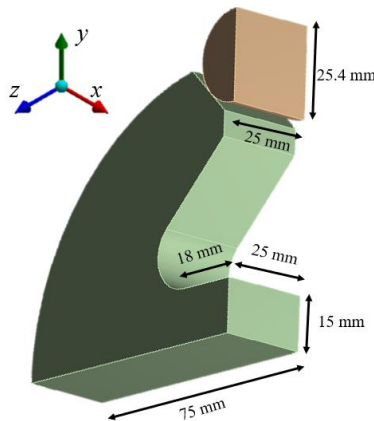


Figure 9.9: Simulated geometry of MSNC-1

- *Constitutive Material Model for UHPFRC and Steel Support Roller*

The constitutive behaviour of THFRC was simulated using the explicit solver of ANSYS, AUTODYN (the RHT model is available in this explicit algorithm), while the structural steel roller was simulated using a bilinear model for structural steel. Table 9.1 and 9.2 summarize the material properties of the constitutive models used for THFRC and steel.

Table 9.1: Material Properties of RHT model for UHPFRC

Parameter	Symbol	Value	Unit
Density	ρ	2520	Kg/m ³
Use Cap on Elastic Surface		Yes	
Compressive Strength	f_c	120	MPa
Tensile Strength ratio	f_t/f_c	0.088	-
Shear Strength ratio	f_s/f_c	0.25	-
Intact Failure Surface Constant	A	1.6	-
Intact Failure Surface Exponent	N	0.61	-
Tension/Compression Meridian Ratio	$Q_{2.0}$	0.06805	-
Brittle to Ductile Transition	B_Q	0.0105	-
Hardening Slope	$G_{el}/(G_{el}-G_{pl})$	2.0	-
Elastic Tensile Strength ratio	f_{cr}/f_t	0.8278	-
Elastic Compressive Strength ratio	$f_{c,0}/f_c$	0.4	-
Fracture Strength Constant	B	1.6	-
Fracture Strain Exponent	M_1	0.61	-
Maximum Fracture Strength Ratio	$SFMAX$	1E+20	-
Damage Constant 1	D_1	0.04	-
Damage Constant 2	D_2	1	-
Minimum Tensile Strain to Failure	$\varepsilon_{t,min}$	0.3	%
Residual Shear Modulus Fraction		0.13	-
Bulk Modulus	K	26667	MPa
Shear Modulus	G	20000	MPa
Maximum Principal Tensile Stress	σ_t	10.57	MPa
Fracture Energy	G_f	21360	J/m ²

Table 9.2: Material Properties of Structural Steel Roller

Parameters	Symbol	Value	Unit
Density	ρ	7,850	Kg/m ³
Young's Modulus	E_s	210,000	MPa
Poisson's Ratio	ν_s	0.3	-
Bulk Modulus	K_s	166,667	MPa
Shear Modulus	G_s	76,923	MPa

- *Loading, Boundary Conditions, Body Interactions and Mesh*

As mentioned previously symmetry in xy, xz, and zy planes was considered: it was implemented by means of restrains in the corresponding axis normal to the plane of symmetry. The loading in form of displacement (2 mm at end time of 0.1 sec.) was applied on the roller as illustrated in Figure 9.10. Mass and time scaling were applied as discussed in Chapter 8.

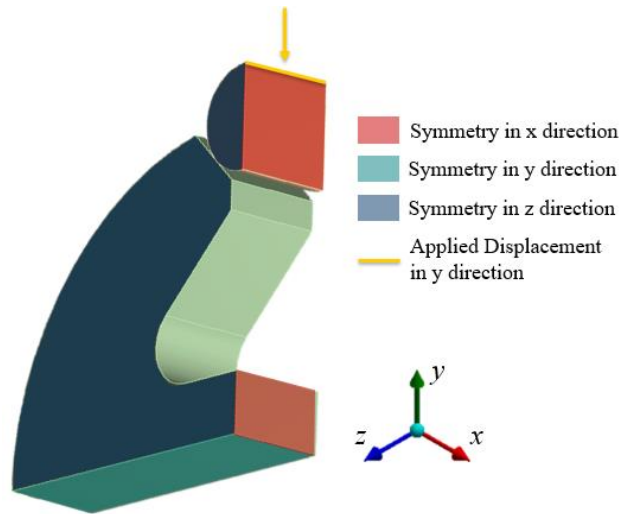


Figure 9.10: Loading and boundary conditions for FE model

The interaction of the impactor roller with the specimen is also simulated in order to study the effect of support conditions and contact between specimen and experimental setup. In this section, for all cases a frictionless rolling contact was simulated. The mesh comprised 3 mm hexahedral elements; these were selected as being most suitable for the explicit solver.

- *Analytical Results*

Figures 9.11-9.18 illustrate the results of the analyses of the 8 different designs through contour plots of stress and strain. Using this representation, it is observed that especially plastic strains predict the failure plane, while they also provide more insight in the actual stress distribution which could not be obtained experimentally. More specifically, the first row of every figure shows the maximum and minimum principal stresses with their corresponding strains at the peak load, whereas the second row shows the corresponding results in the post peak range, specifically at the end of loading. According to those, designs DEWS and DEWS-M are rejected as their stress distribution is fraught with issues similar to the conventional splitting test, and thus they are undesirable.

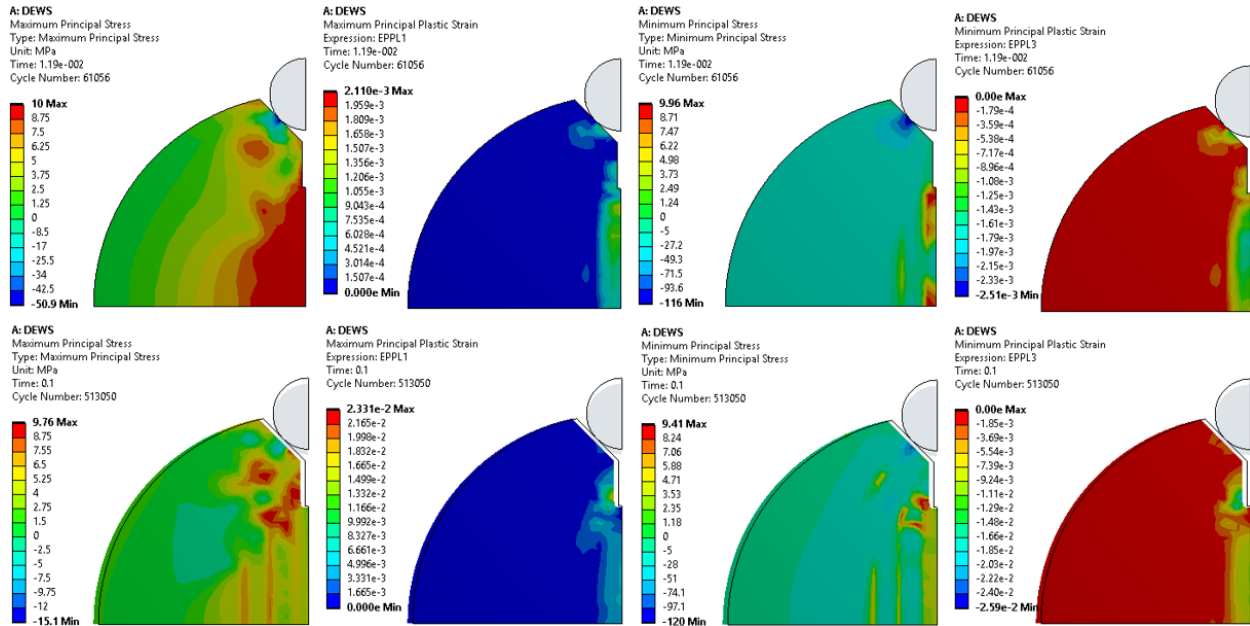


Figure 9.11: Maximum and Minimum Principal Stresses and plastic Strains at peak load and end step for DEWS design

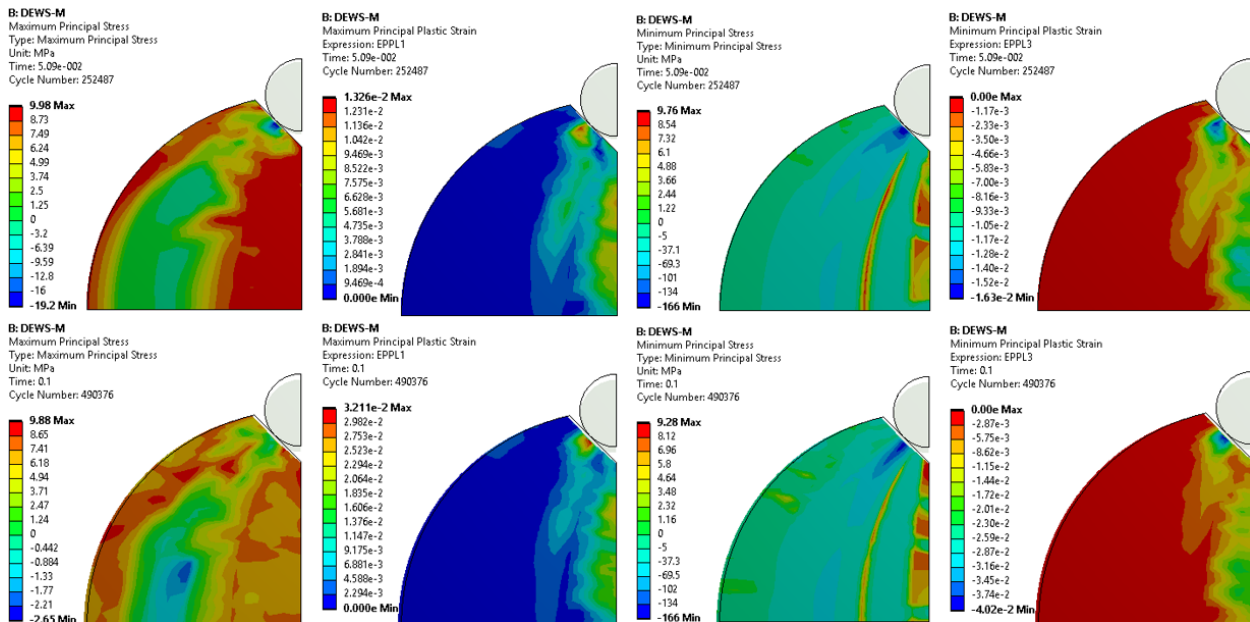


Figure 9.12: Maximum and Minimum Principal Stresses and plastic Strains at peak load and end step for DEWS-M design

Although design STF has a chord that is supposed to be isolated to act in pure tension, the failure plane is found to occur under the impactor. This type of failure is unsuitable as it occurs outside the range of interest, and it is highly influenced by the boundary conditions at roller support. The latter was avoided in design MS1. In this case the failure plane occurs in the chord but is closer to

the arc. However, the design is not optimum as it would be preferred that the central chord ought to have a constant cross-sectional area over a sufficient gauge length.

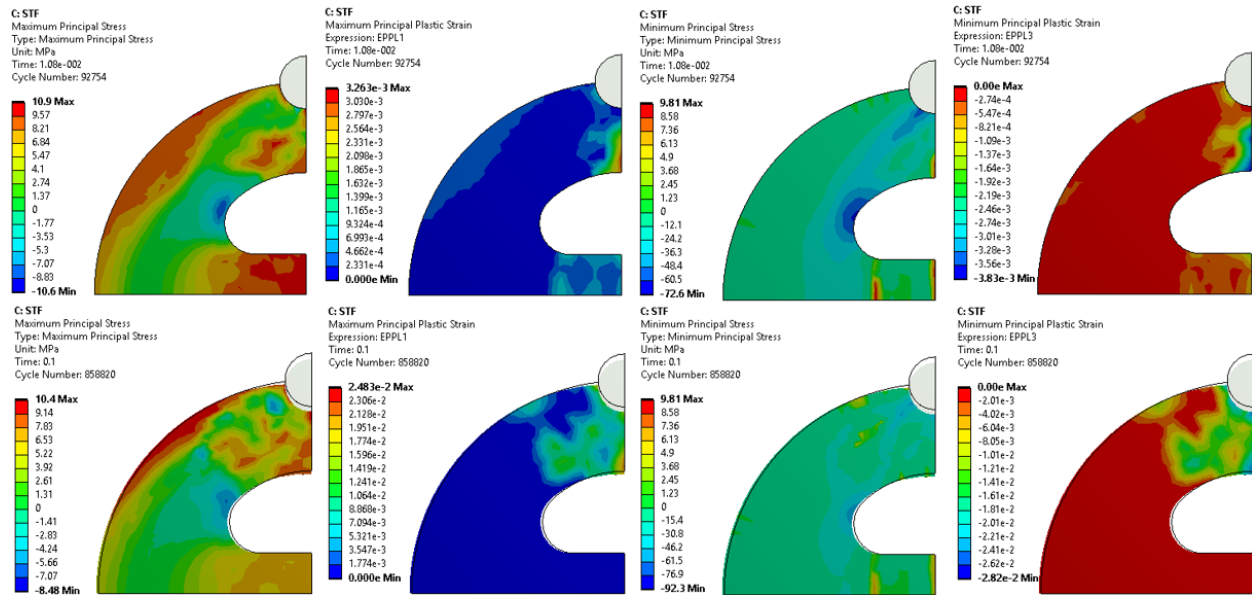


Figure 9.13: Maximum and Minimum Principal Stresses and plastic Strains at peak load and end step for STF design

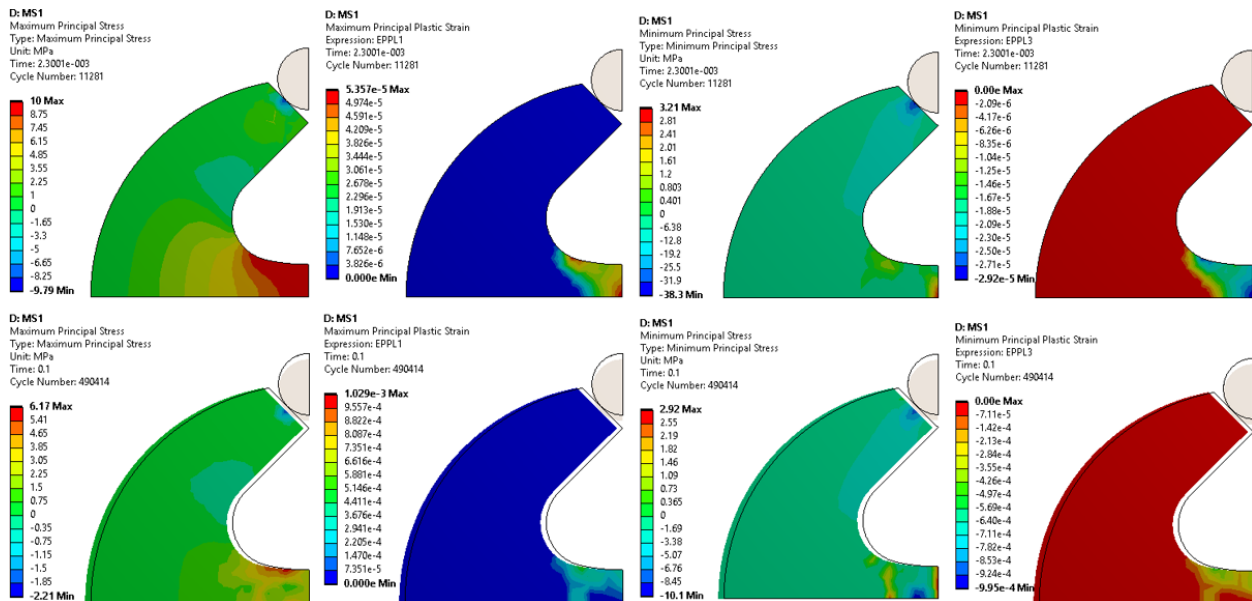


Figure 9.14: Maximum and Minimum Principal Stresses and plastic Strains at peak load and end step for MS1 design

Designs MS2 and MSN have increased gauge length but still no constant cross-section. As illustrated in Figures 9.15 and 9.16 the stress distribution and crack pattern are pretty similar to

MS1 with the case of MSN to have additional complications owing to some stress concentrations around the impactor.

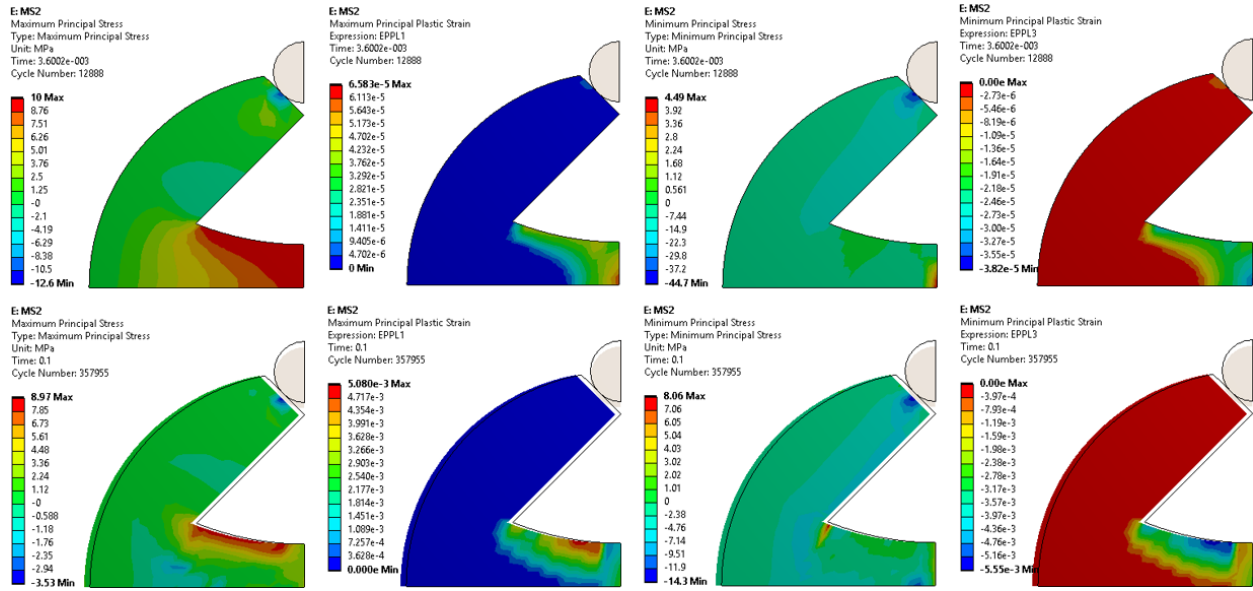


Figure 9.15: Maximum and Minimum Principal Stresses and plastic Strains at peak load and end step for MS2 design

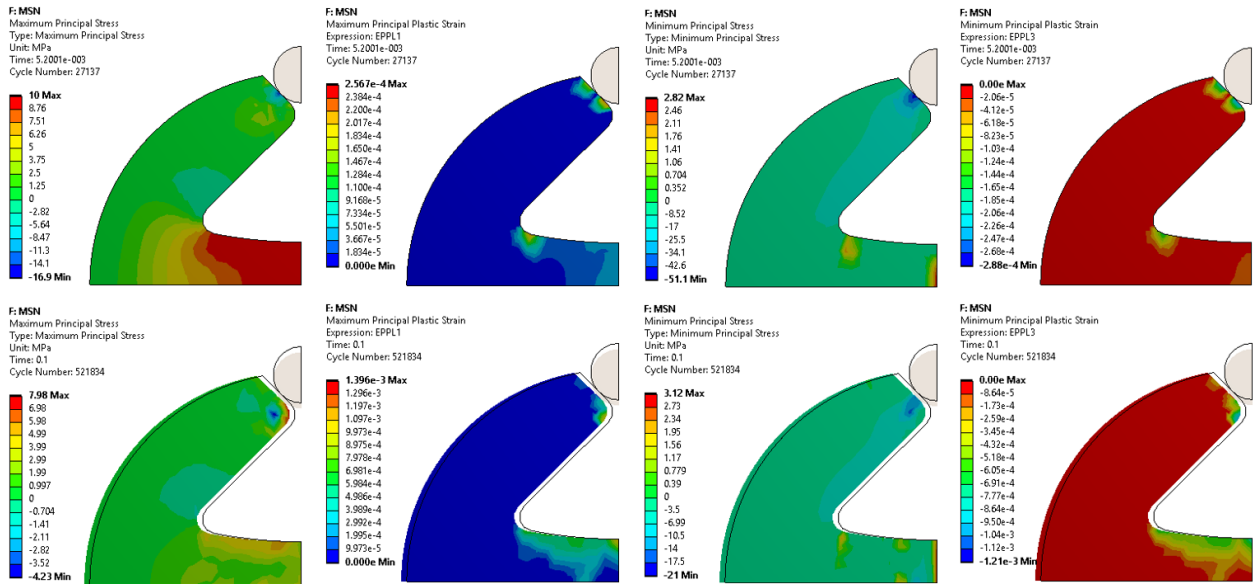


Figure 9.16: Maximum and Minimum Principal Stresses and plastic Strains at peak load and end step for MSN design

The concentration of stresses around the impactor diminishes in cases of MSNC-1 and MSNC-2. The extra advantage is the existence of constant cross-section over a sufficient gauge length. Although the cracking initiates in the vicinity of the arc, in the end it localizes in the midspan of the gauge length for MSNC-1. For case MSNC-2, due to widening of the neck under the impactor,

a larger diameter roller is used. The two designs have similar response and will be used both in the following section for experimental validation.

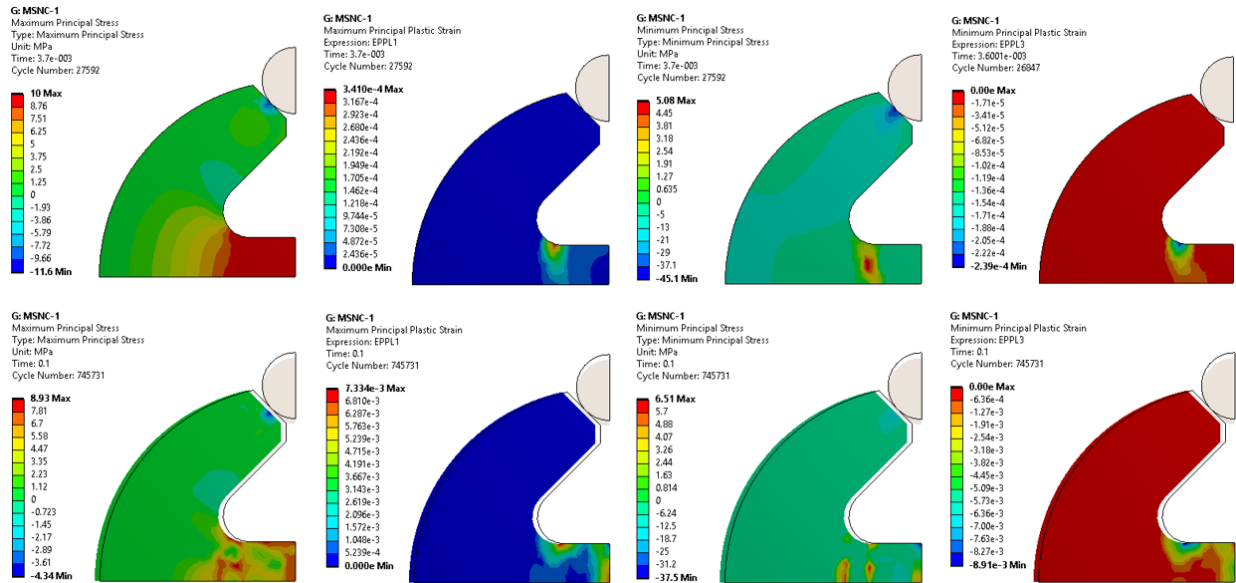


Figure 9.17: Maximum and Minimum Principal Stresses and plastic Strains at peak load and end step for MSNC-1 design

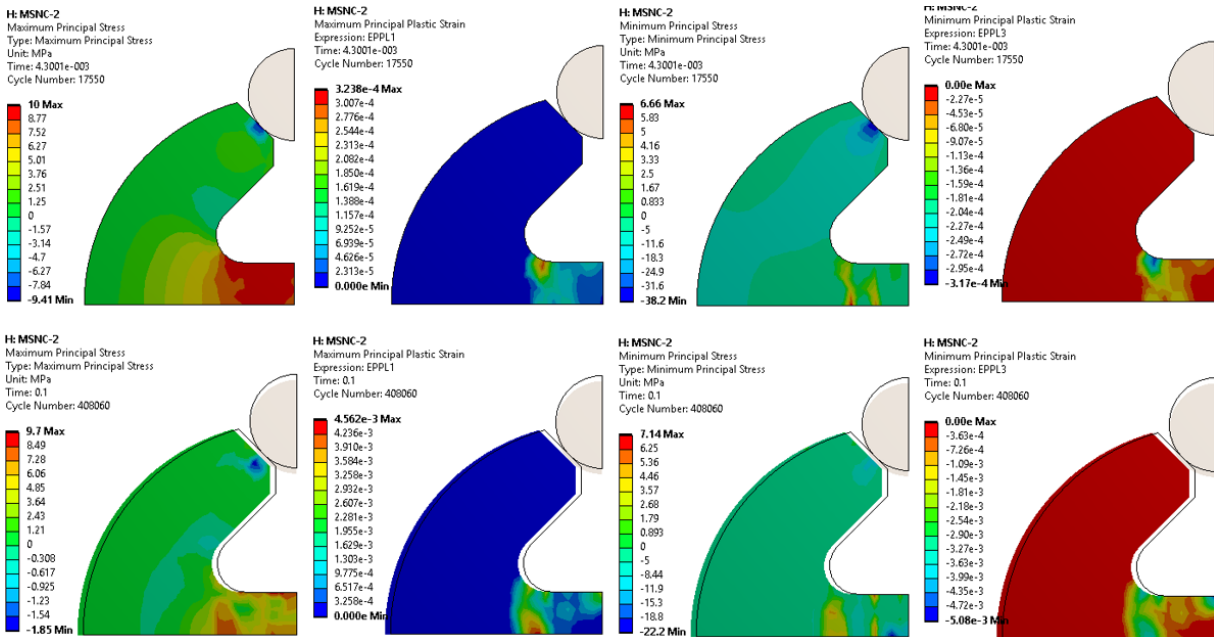


Figure 9.18: Maximum and Minimum Principal Stresses and plastic Strains at peak load and end step for MSNC-2 design

Finally, the ratio between the applied load P and the resultant normal force at the isolated central chord, R , is used to assess the suitability of the available designs. Table 9.3 summarizes the results of the models at peak load and at the end of loading. All designs have a ratio of around 1

and keep this ratio is maintained at the same value except for the case of STF, where there is also a significant resultant force developed under the impactor. The closest to 1 ratio is found in MSNC designs. Based on this information and the stress and strain distribution presented above, MSNC designs are kept as the most suitable option in the development of a novel experimental test setup for tension characterization of THFRC.

Table 9.3: Ratio of peak load to resultant internal force for the different designs depicted in Figure 9.7

Design	P/R at peak load	P/R at post peak
DEWS	0.97	1.05
DEWS-M	1.12	1.09
STF	2.64	2.8
MS1	1.06	0.94
MS2	1.04	1.01
MSN	1.06	1.07
MSNC-1	1.03	1.05
MSNC-2	1.03	0.96

9.2.4. Experimental Validation

The experimental validation was performed through testing of a total of 34 specimens. Two different proprietary UHPFRC mixes were used for this experimental campaign, referred to as FD and DE, respectively (both characterized as THFRC materials). The numeral following this code name refers to the batch number. FD batches use 20 mm long fibers with a diameter of 0.2 mm, while DE batches use short fibers with a diameter of 0.2 mm and a length of 13 mm. The details of the batches are summarized in Table 9.4. All specimens were cast with flow from one end, parallel to the central chord and demolded 24 hrs after casting and cured in water till the age of testing, namely 28 days. In order to perform a practical test, direct contact of concrete grooving area to the steel roller was chosen. Prior to testing the grooving area was smoothened with sandpaper to minimize friction. For the last batch DE3, brass plates were used in the contact area for comparison with the other cases.

Table 9.4: Batch details for experimental validation of the proposed indirect tensile test

Batch	Label	Design	Full Scale	Half Scale	No. Specimens
FD1	FD1B	MSNC-1	Yes	-	3
FD2	FD2B	MSNC-1	Yes		3
	FD2M	MSNC-1	-	Yes	3
DE1	DE1M	MSNC-2	-	Yes	6
DE2	DE2M1	MSNC-1	-	Yes	3
	DE2M2	MSNC-2	-	Yes	6
DE3	DE3M	MSNC-2	-	Yes	10

- *Formwork Design and Preparation*

For the formwork design, different materials and techniques were used. For Batch FD1, only full-scale specimens of 300 mm diameter were cast. The mold consisted of a hand-cut form tube fixed on a square birch plywood base using hot glue. The inserts were made of rigid insulation foam. In the early attempts, they were drawn on a rigid insulation foam of 1 in. thickness (Figure 9.19(a)), then the pieces were cut and glued together to the desired thickness (Figure 9.19(b)). Subsequently they were hot glued down on the tube form and base (Figure 9.19(c)). Figure 9.20 illustrates a schematic representation of the form of FD1B specimens with dimensions. The challenge of this formwork is that the rigid foam was difficult to cut in this geometry resulting in some imperfections especially around the roller support. Additionally, form tubes of that standard size had usually some variations in their actual diameter affecting the geometry of specimen.

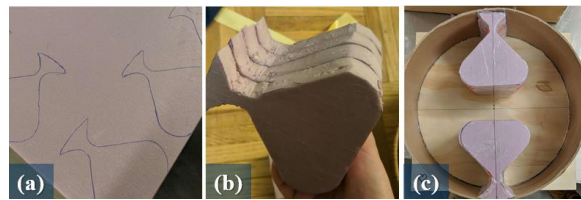


Figure 9.19: Preparation of formwork for FD1B specimens

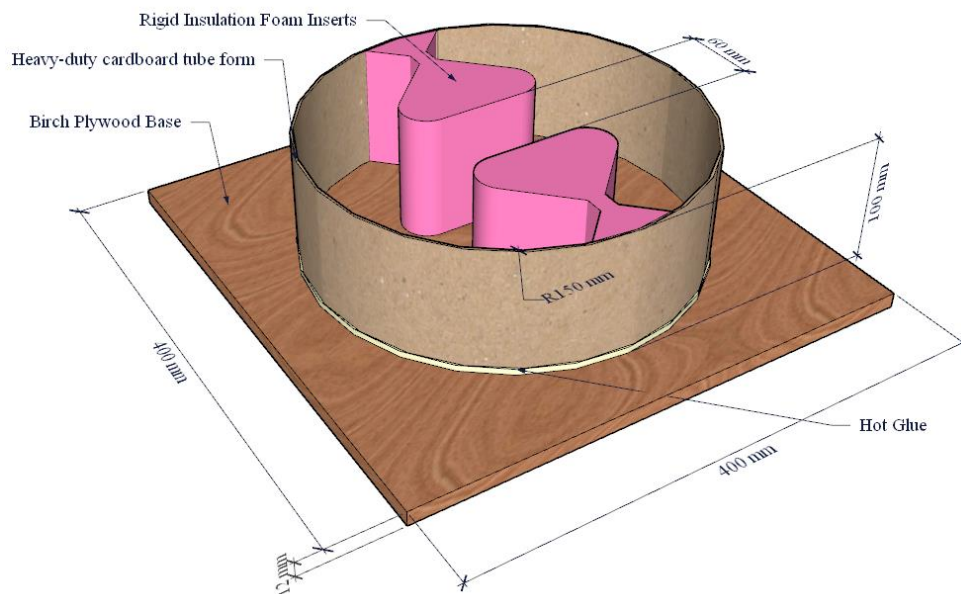


Figure 9.20: Formwork design for FD1B

In Batch FD2, a different approach was followed for formwork preparation, namely the inserts resulted from casting liquid foam into a negative form. Nine plexiglass sheets with the shape of

MSNC-1 specimen and thickness of 11 mm were laser cut and glued together and wrapped with a sheathing tape to create the negative form. Then, they were placed inside the form tube which was fixed on the wooden base. Prior to preparation of the liquid foam, the faces of the plexiglass sheets left exposed were covered with a releasing agent (Figure 9.21(a)). Then, the two-part liquid urethane was mixed and poured in the form till midheight (Figure 9.21(b)). After 15 minutes, the foam expanded reaching the full height and left for 20 minutes curing (Figure 9.21(c)). Finally, the plexiglass sheet was removed, leaving a similar configuration with the previous formwork design (Figure 9.21(d)). These inserts had smoother surfaces and fewer discrepancies in their dimensions, compared to the hand-cut insulation foams. For this batch, half scale specimens (FD2M) were also manufactured with the formwork preparation to be similar to the larger scale. The only difference is that instead of form tube and wooden base, standard plastic cylinder molds with a diameter of 150 mm were used to minimize geometrical flaws imparted by the exterior mold of the specimen.

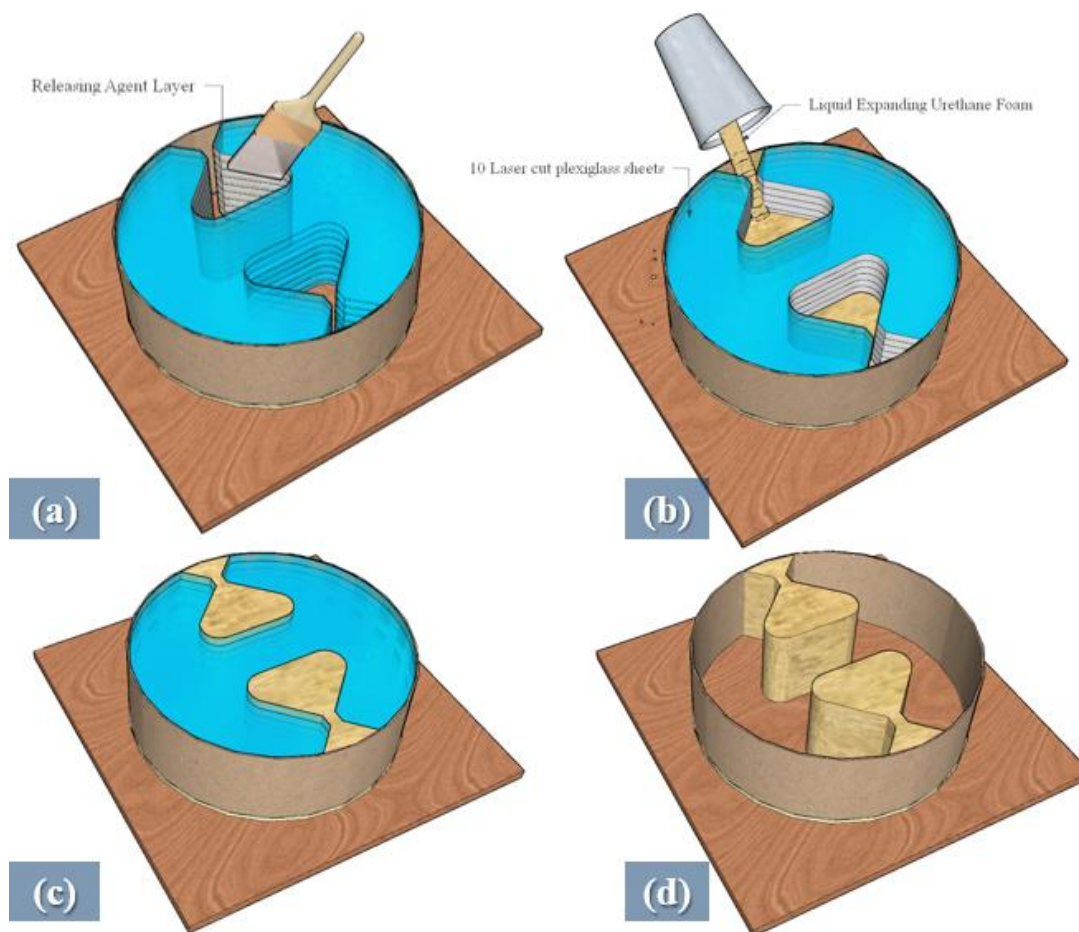


Figure 9.21: Formwork preparation for FD2B

After comparing the full and the half scale specimens it was found that they give consistent results with each other and there is no evidence of a size effect. For this reason, henceforth only half scale specimens were cast (Batches DE1 and DE2). Instead of inserts made of liquid expanding urethane, for these castings 3D printed plastic inserts were manufactured and hot glued on the bottom of the cylinders (Figure 9.22). This technique proved to be the most convenient and less time consuming of all three, while no discrepancies were found in the dimensions of the specimens.

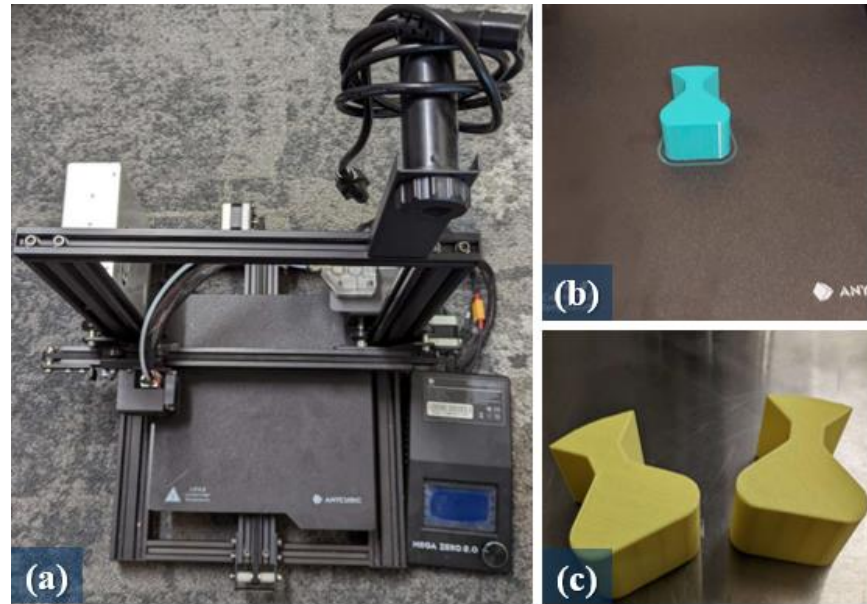


Figure 9.22: (a) 3D printer (b) printing of an insert (c) final 3D printed inserts

- *Batch FD1- Preliminary Testing*

For this batch, three large scale specimens FD1B were cast. The mix was a commercially available UHPFRC and was poured from one end, parallel to the central chord with constant cross-sectional area. Till the age of testing which was the 28th day, the specimens were left in a water tank to cure. Prior to testing, strain gauges were installed on the top and bottom face at midspan of the central cord in order to record any undesirable bending or unsymmetrical loading (Figure 9.23). On the grooving area (contact area with rollers), a layer of plaster was applied to facilitate smooth contact. This was done to eliminate very rough surfaces with imperfections resulting from difficult demolding. The final step of preparation was to paint the front face white and to speckle it with black spray in order to create a pattern suitable for DIC analysis. The specimens were tested under compression in the servo-hydraulic Universal Testing Machine under compression (UTM). The load was applied in a form of displacement and with a rate of 0.15 mm/ min through two steel

rollers sitting on the diametrically opposed grooving areas. A schematic representation of the test setup is illustrated in Figure 9.23. Finally, in order to perform DIC analysis a camera was set up in front of the specimen to capture pictures every 10 sec. DIC analysis was used to obtain the transverse displacement of the central chord through virtual extensometers and the principal strain distributions in the specimens. Three virtual extensometers with a gauge length of 70 mm were employed through DIC with a distance of 20 mm from each other as illustrated in Figure 9.23.

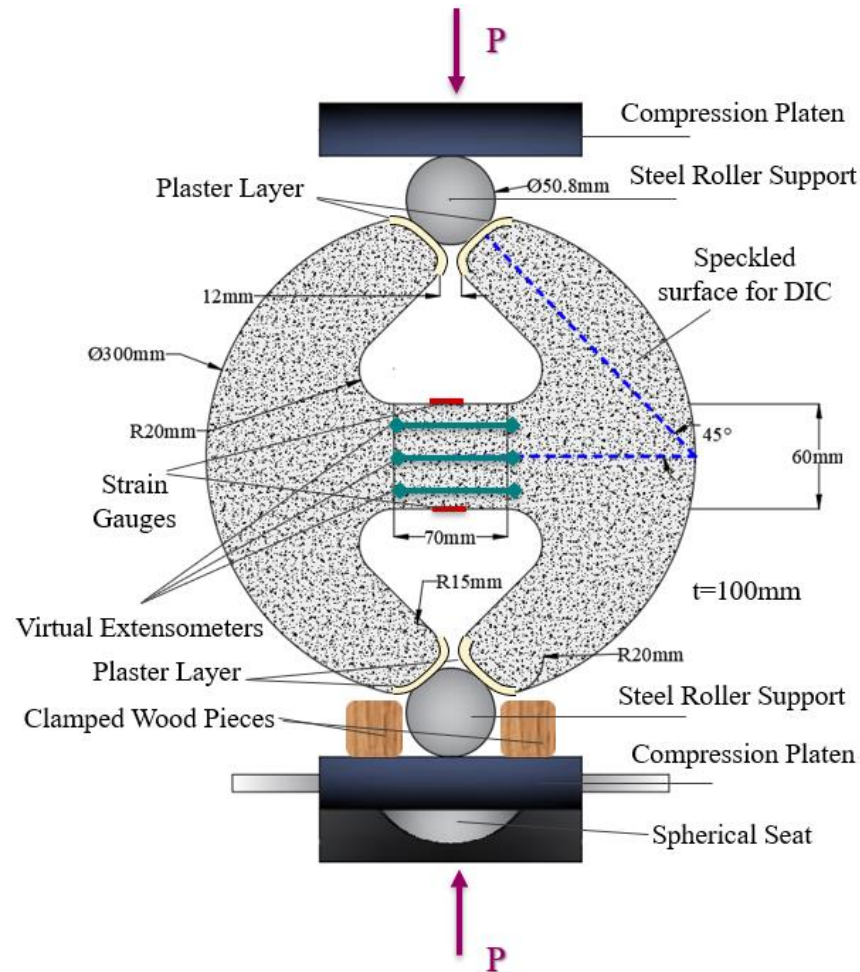


Figure 9.23: Schematic representation of test setup for FD1B specimens

The tensile stress-strain curves for the three specimens are shown in Figure 9.24, while the cracking and ultimate strengths are listed in Table 9.5. The ultimate strength can be easily obtained through the graph, while for cracking strength the principal strain distribution contour plots from DIC are used. More specifically, the cracking load corresponds to the plot where the localization of the crack occurs.

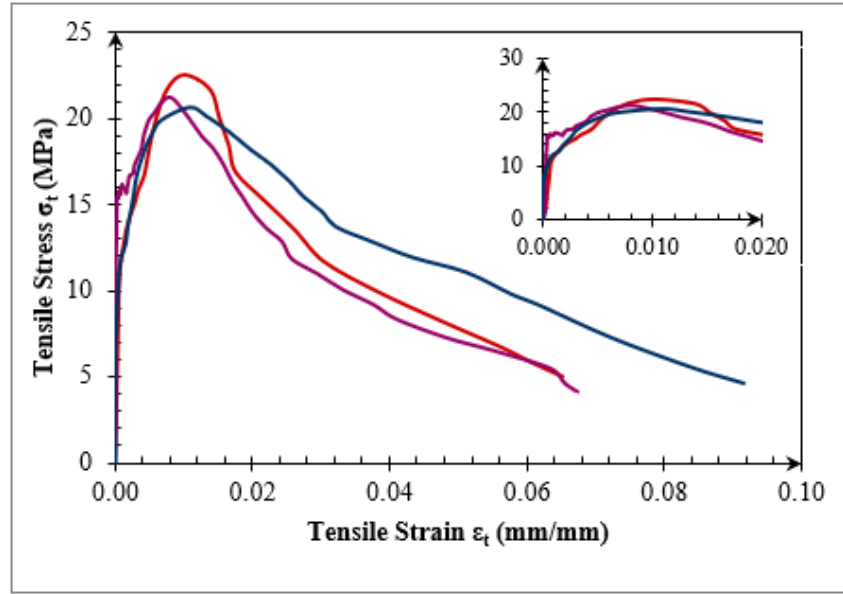


Figure 9.24: Tensile Stress- horizontal displacement curves for FD1B specimens (repetition of three specimens; insert zooms into the range in the ascending envelop up to the onset of degradation)

Table 9.5: Tensile Properties extracted from FD1B specimens

Specimen	f_{cr} (MPa)	ϵ_{cr}	f_{tu} (MPa)	ϵ_{tu}
1	12.7	0.001128	21.25	0.008332
2	8.2	0.00078	20.87	0.01083
3	11.75	0.00071	22.54	0.01021
Average	10.83	0.000872	21.53	0.0097

As mentioned above, to extract the transverse deformation of the specimens, DIC Analysis was performed. For every specimen the 10% of the total pictures taken were selected for DIC Analysis to minimize the computational time. The pictures selected correspond to specific points of the response curve in order to capture the complete behaviour. Out of those photos, 6 are used to show the surface principal strains. P4 is the ultimate load corresponding to the ultimate tensile strength. P3 is the load corresponding to the first cracking tensile strength. P2 and P5 correspond to 0.8 of P4, while P1 and P6 represent 0.4 of the maximum load. P1-P3 are in the ascending branch of the resistance curve, while P5 and P6 are post-peak. Figure 9.25 illustrates the maximum principal strain at different loads. In the elastic region (P1), the central chord is in tension while as the load values approach to cracking, some strain concentrations are observed in the proximity of the fillets on both ends of the central chord. The concentrations become more prominent at cracking load, where multitasking is also observed, a first indicator of strain hardening behaviour. At ultimate load, the localization has started and progresses with crack propagation, which along with the high strain concentrations around crack band are represented with a white line of increasing width.

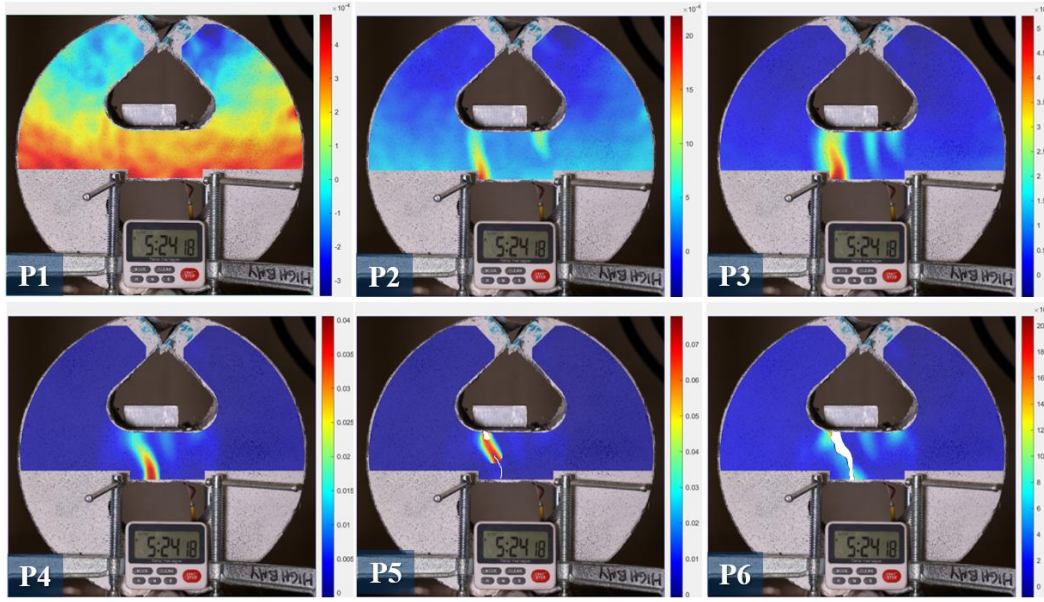


Figure 9.25: Maximum Principal Surface Strains for specimen FD1B

Finally, Figure 9.26 illustrates the fractured specimens. As it may be seen, the crack patterns for specimens FD1B1 and FD1B2 are similar, while for specimen FD1B3 cracking is different across the depth. This is an indication of out of plane bending which can also be explained with the considerably higher load attained compared to the other two specimens.

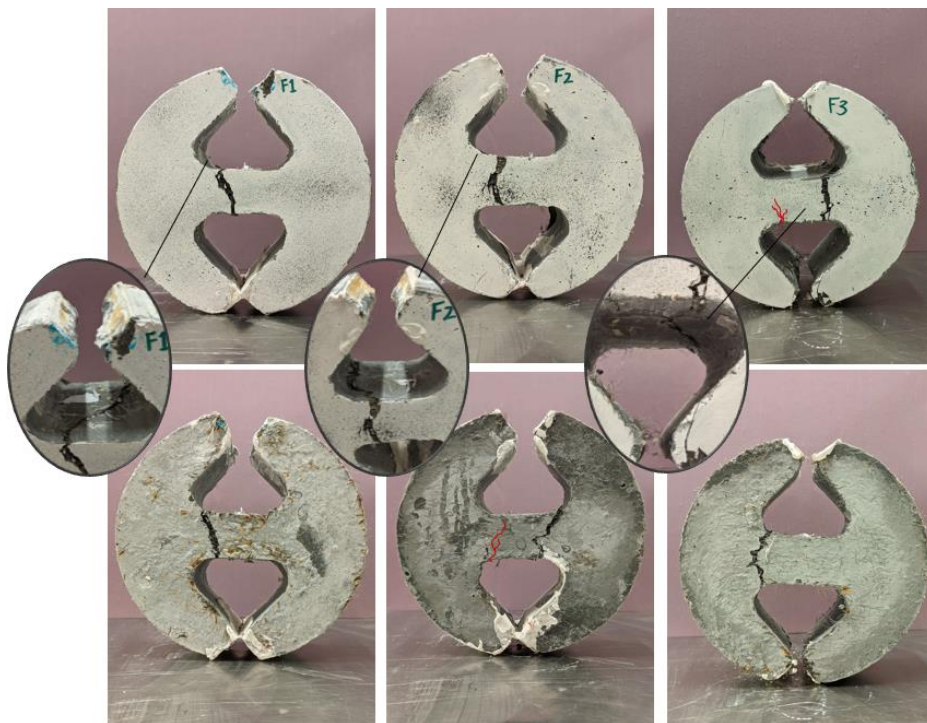


Figure 9.26: Fractured specimens for FD1B

- *Batch FD2- Size Effect*

For batch FD2, the size effect was studied through casting and testing full FD2B and half scale FD2M specimens. The experimental setup for both types of specimens is illustrated in Figure 9.27.

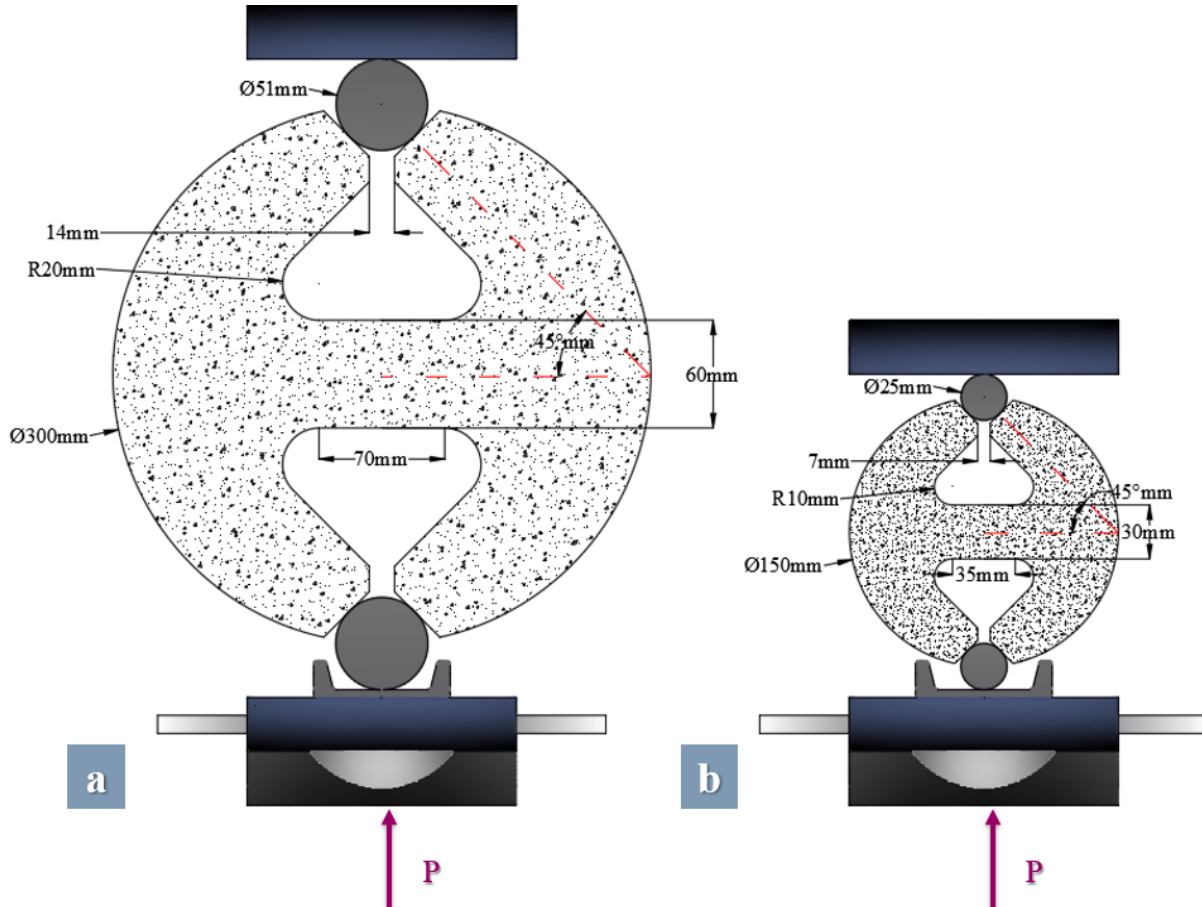


Figure 9.27: Schematic representation of test setup for FD2 (a) full scale specimens FD2B and (b) half scale specimens FD2M

Similar to FD1, the horizontal displacement was obtained via Virtual Extensometers which are enabled by DIC analysis. For the half scale specimens, the gauge length was set to 55 mm. The tensile stress-strain responses for full and half scale specimens are shown in Figures 9.28 and 9.29. All three full scale specimens had a similar cracking and ultimate strength. However, FD2B1 demonstrates higher modulus of toughness compared to the other two full scale specimens. For the half scale specimens, specimen FD2M3 failed prematurely due to torsion, thus the considerably low strength. It is observed that all specimens except FD2M3 demonstrate similar cracking and ultimate strength with no noticeable size effect. Table 9.6 summarizes the results of this batch (FD2M3 has been excluded from the averaging as an outlier).

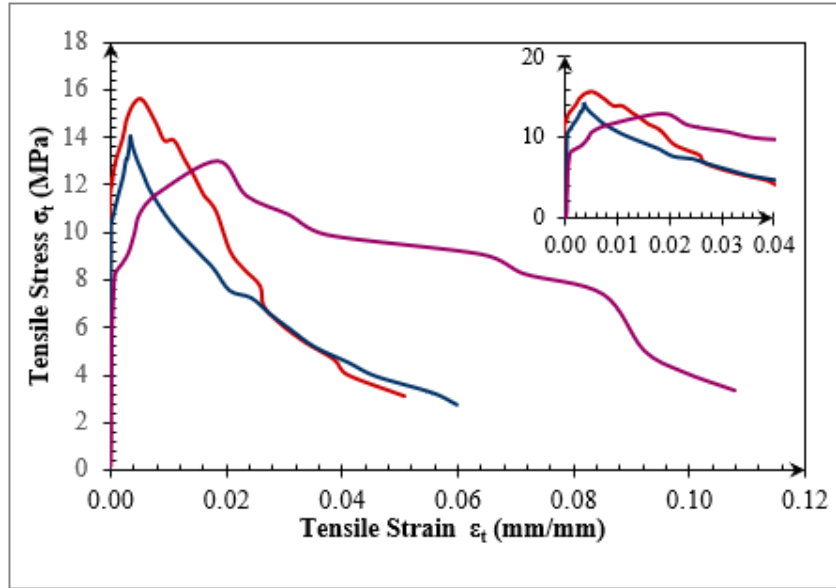


Figure 9.28: Tensile Stress-Strain Curve for FD2B specimens (repetition of three specimens)

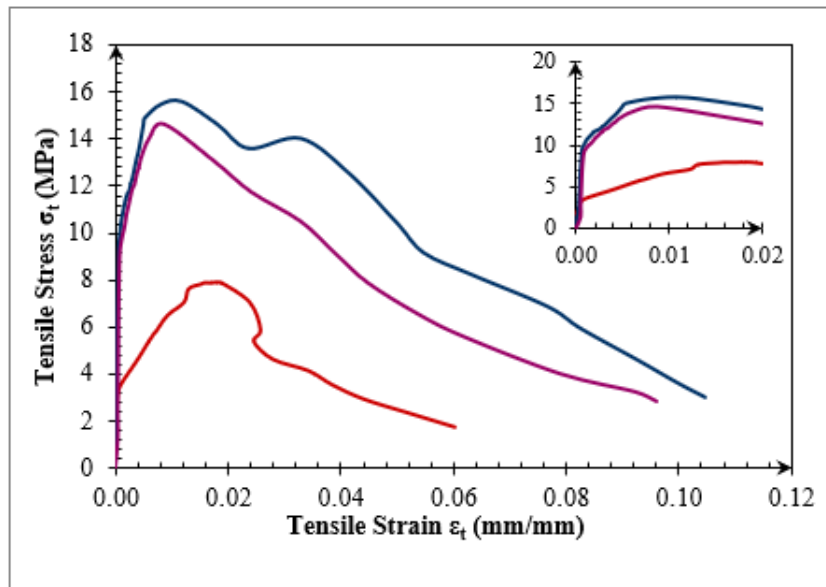


Figure 9.29: Tensile Stress-Strain Curve for FD2M specimens (repetition of three specimens)

Table 9.6: Tensile Properties extracted from FD2 specimens

Specimen	f_{cr} (MPa)	ϵ_{cr}	f_{tu} (MPa)	ϵ_{tu}
FD2B1	8.26	0.00036	13.0	0.01861
FD2B2	10.41	0.00024	14.06	0.0035
FD2B3	11.77	0.00016	16.37	0.00547
Average	10.15	0.000253	14.48	0.00919
FD2M1	9.25	0.000858	14.65	0.0086
FD2M2	11.44	0.001815	15.63	0.01185
FD2M3	2.98	0.0004	7.96	0.02
Average	10.345	0.001365	15.14	0.01022

The surface principal strains for a full and a half scale specimen at the same characteristic points of the curve described for the previous batch are shown in Figures 9.30 and 9.31. For the full-scale specimen FD2B1, there was slight bending in the transverse direction at low strains (P1) which might be attributed to the adjustments of the rollers in the elastic region. As the test progressed, this behaviour diminished, and cracking (P3) appeared prior to 80 % of the peak load (P4) in a form of increased concentrated strains at the vicinity of the right fillet. The localization became more prominent especially after 80% of the ultimate load with the crack width to increased from 0.6 mm at ultimate stress to 6 mm at 60% drop of the ultimate load. A white area represents the crack band along with high strain concentrations. For half scale specimens, there were some stress concentrations around the grooving area, in the elastic region (P1) which can be attributed to flaws that resulted during demolding. At the cracking load (P3), higher strains were concentrated on the central chord region, which is desirable leading to localization of cracking within the gauge length (P4). As in previous cases the crack initiated in the vicinity of the fillet. The width of the crack increased in the post peak, while some concentrations were observed near the neck formed under the grooving areas. This could explain the slight increase of load in the post peak region.

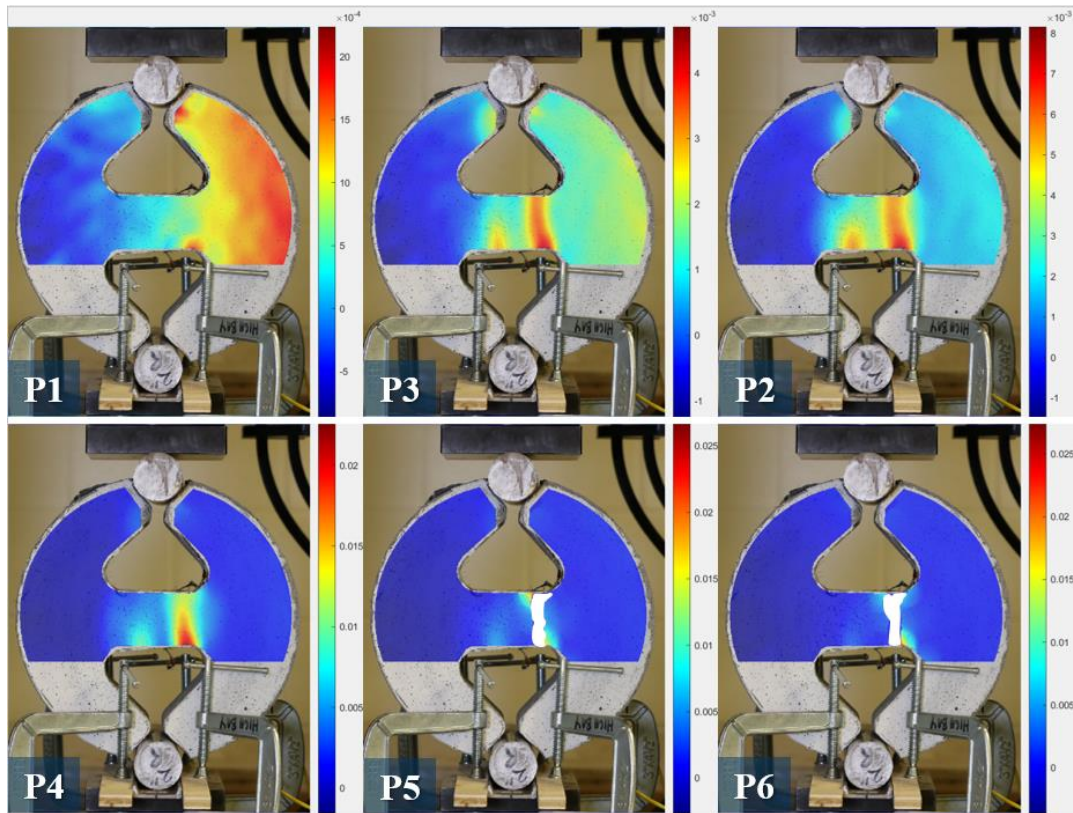


Figure 9.30: Maximum Principal Surface Strains for specimen FD2B1

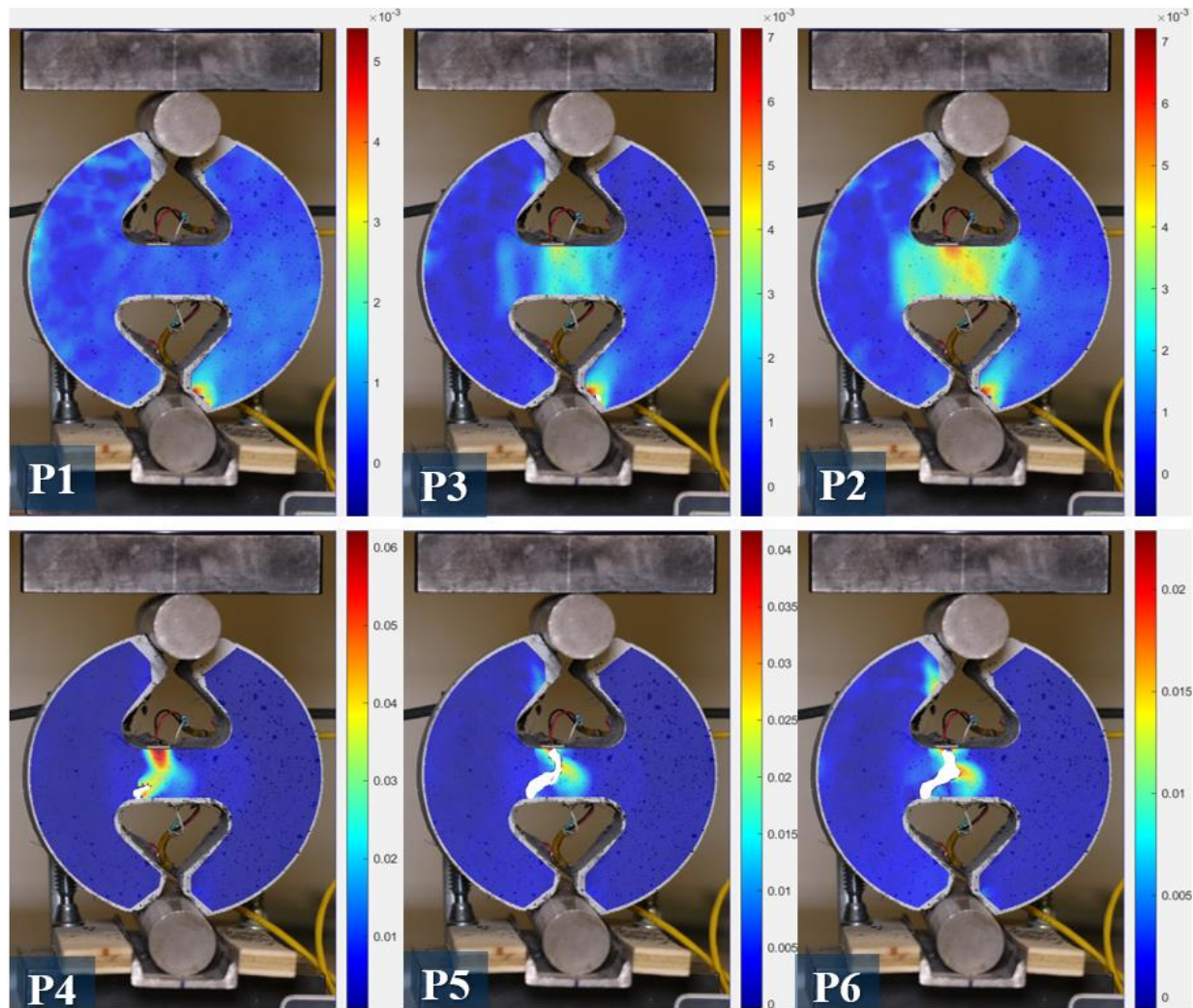


Figure 9.31: Maximum Principal Surface Strains for specimen FD2M2

- *Batch DE1- Effect of Supports*

In batch DE1, the effect of different support conditions on half-scale specimens was investigated. Namely, a pin- pin (-P) and a roller- roller (-R) support were studied. For the roller- roller support, a cylinder with increased diameter was used and the angle was changed from 45 to 50°. The bottom roller was placed inside a C channel to limit the horizontal translation. The latter was mounted on the bottom compression platen with glue. In case of an angle $\theta=50^\circ$, the ratio between the applied load and resultant force is no longer 1, but 1.19. This means that the resultant stress has to be divided by 1.19. Figures 9.32 and 9.33 show the experimental setup for the two different conditions. For these specimens an LVDT was mounted on the back side with a gauge length of 55 mm, in order to record the horizontal displacement of the central chord. The front face was also painted in a speckle pattern to facilitate DIC analysis.

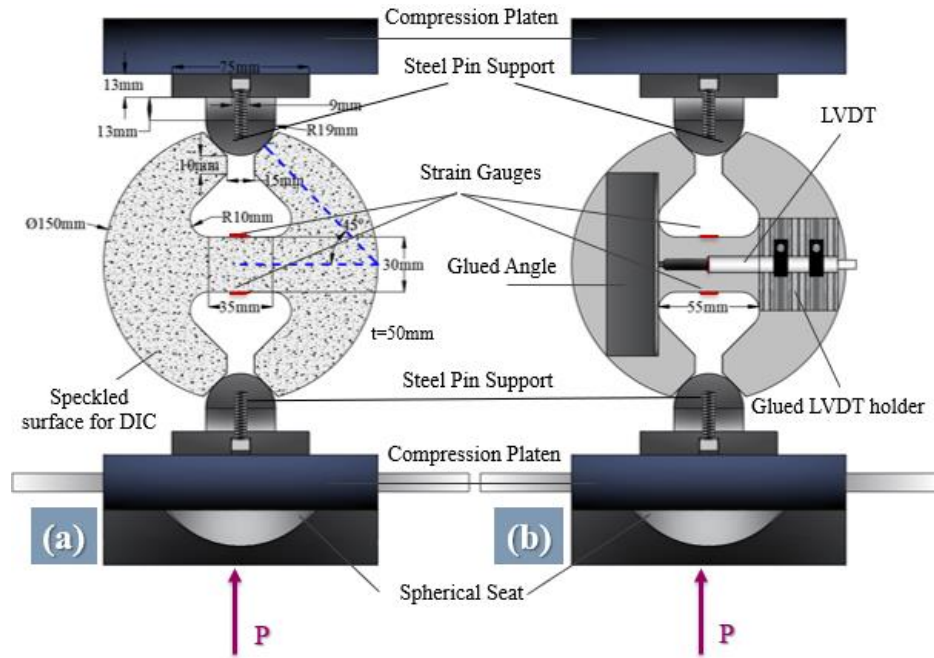


Figure 9.32: Experimental setup for DE1M-P specimens

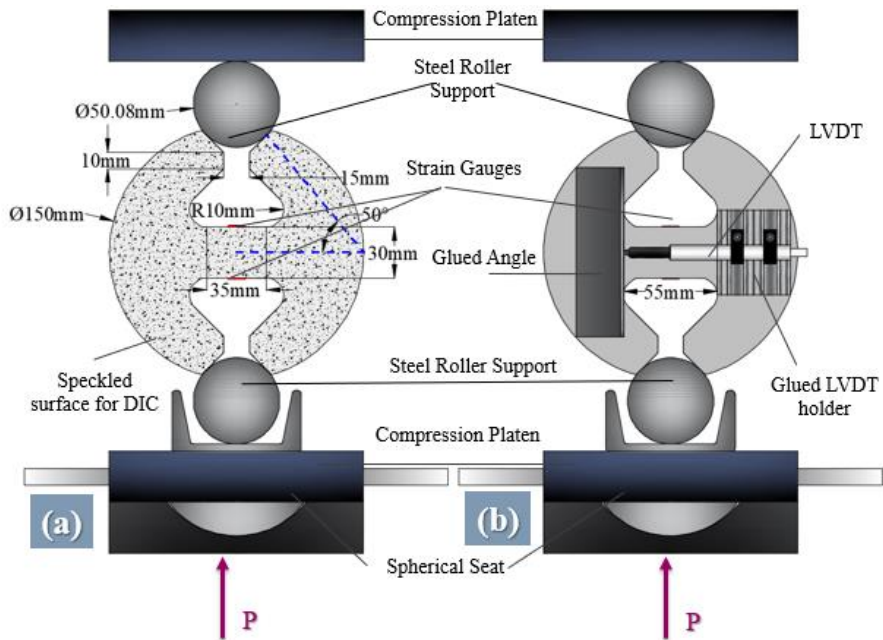


Figure 9.33: Experimental setup for DE1M-R specimens

Figures 9.34 and 9.35 illustrate the tensile stress-strain response of DE1M-P and DE1M-R specimens. Specimens supported with pins, show higher load capacity than those with rollers. This might be attributed to the redundancy that is added to the system in case of a pin-pin support. Furthermore, the stress-strain laws for DE1M-P specimens show less discrepancy compared to DE1M-R, as all three attained similar resistance and toughness. A distinct characteristic is that for

DE1M-P specimens the curves demonstrate a prominent jagged-lines pattern especially in the post peak range, which was accompanied by an intermittent sound during the experiment similar to cracking. For DE1M-P3, this pattern was also observed in the ascending branch and was linked to premature compression failure of the concrete near the supports, which led the test to end earlier than the other two. Table 9.7 summarizes the tensile properties of this batch.

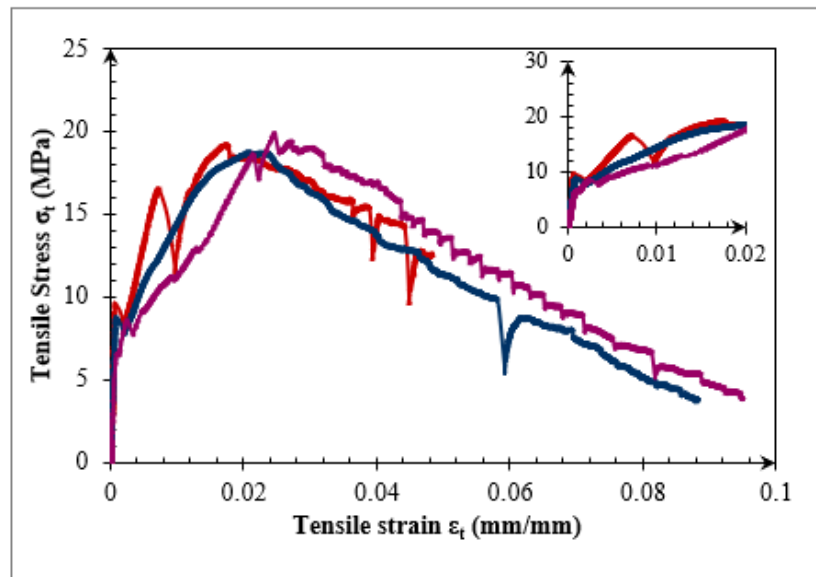


Figure 9.34: Tensile Stress-Strain Curve for DE1M-P specimens. The insert zooms into the ascending branch (repetition of three specimens)

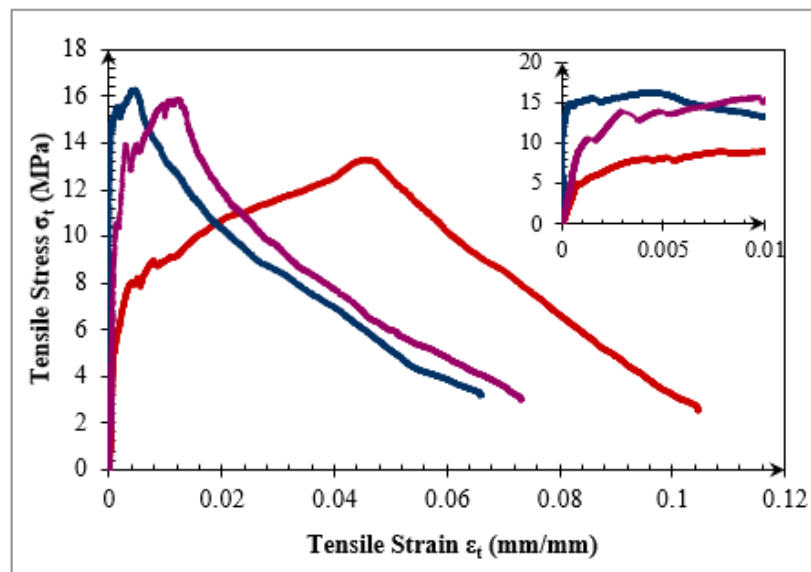


Figure 9.35: Tensile Stress-Strain Curve for DE1M-R specimens. The insert zooms into the ascending branch (repetition of three specimens)

The above results were investigated further using DIC Analysis. The surface maximum principal strain distribution at different loads for DE1M-R2 and DE1M-P1 is illustrated in Figures 9.36 and 9.37, respectively. In case of DE1M-R2 the use of rollers with increased diameter not only increased the contact angle with the grooving area by 5° , but also it seemed to create higher stress concentrations at the grooving area in the elastic region, which were diminished after localization. For the double pin supported specimen, till the maximum load, the central chord experienced primarily compression, which is clearer from the minimum strain distribution in Figure 9.39. This undesirable stress-state could be correlated with the higher resistance loads observed in case of pin supported specimens. At ultimate load there was a tensile crack on the left side of the fillet, while the right side experienced high compressive strains. At 80% residual strength (P5), the dominant tensile crack changed position from left to right side, while at 60% drop of load, the crack on the left side propagated further. This multiple cracking observed, was in accordance with the jagged pattern post peak for the DE1M-P specimens. From these analyses, it is evident that double pin supports create a redundant behaviour with more complicated stress-state, which is undesirable.

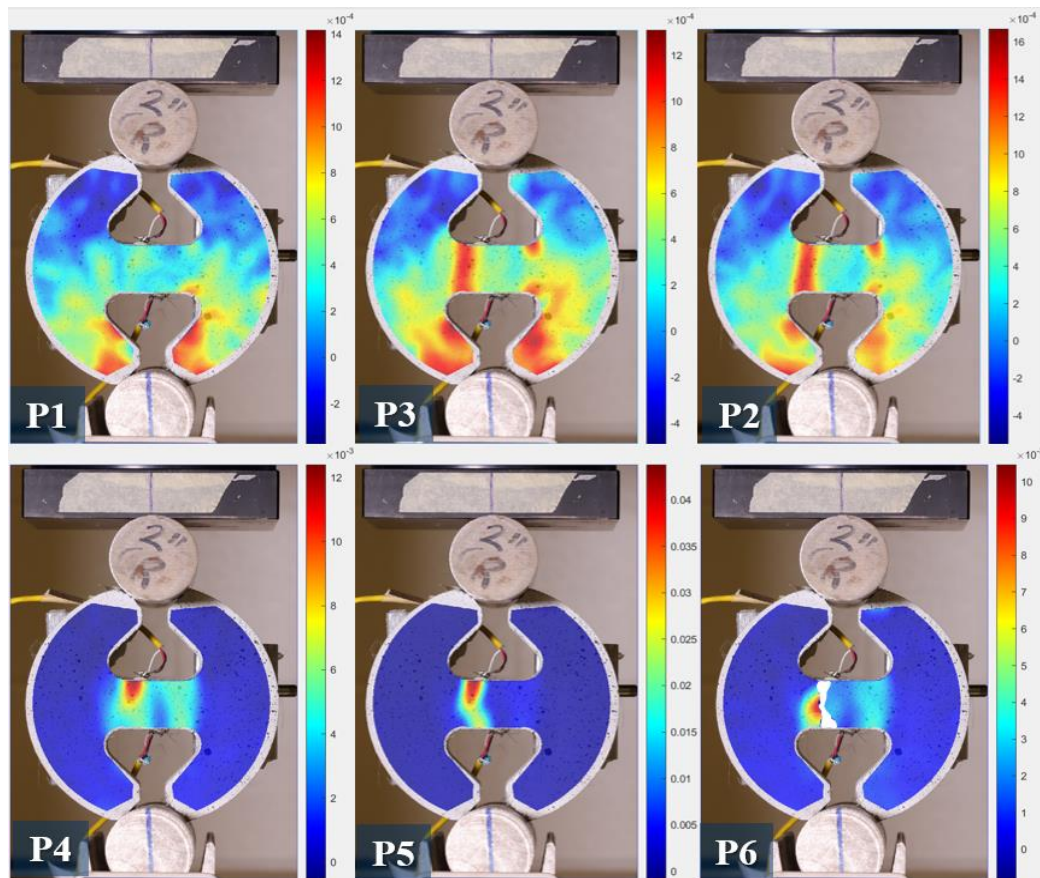


Figure 9.36: Maximum Principal Surface Strains for specimen DE1M-R2

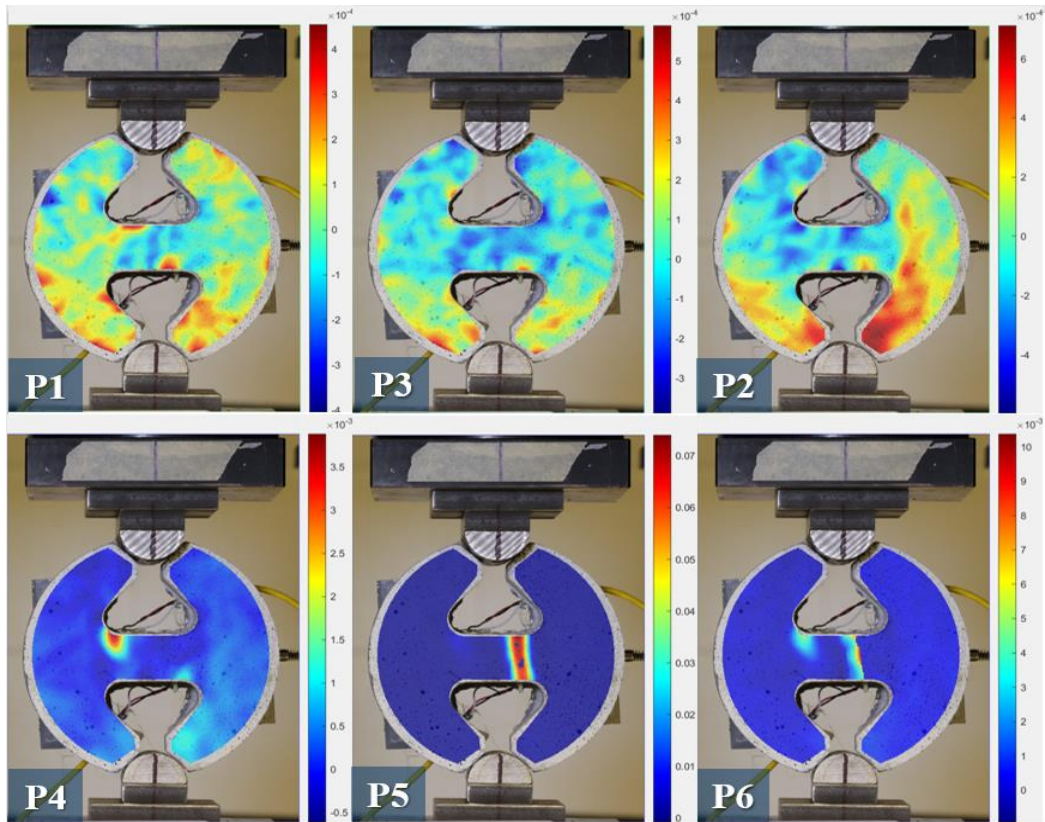


Figure 9.37: Maximum Principal Surface Strains for specimen DE1M-P1

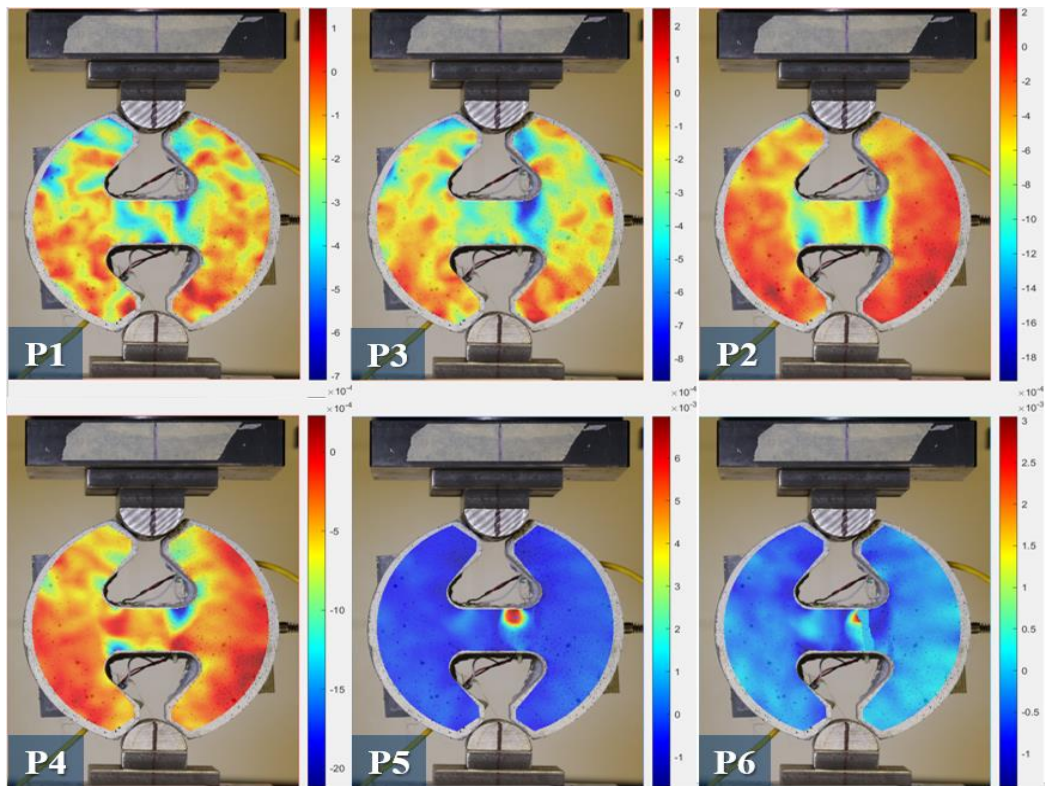


Figure 9.38: Minimum Principal Surface Strains for specimen DE1M-P1

- *Batch DE2-Effect of Supports and Geometry*

Due to discrepancies between the supports and geometry of the specimens tested in Batch DE1, further investigation of the effect of the supports was performed. All the specimens were half scale. More specifically MSNC-2 specimens were tested with pin supports as illustrated in Figure 9.32 (DE2M-P specimens), and roller supports with a diameter of 1.5 in. to ensure the 45° angle that was required for a force ratio = 1 (DE2M-R specimens). It is worth noting here that the pin supports were further cleaned and filed with sandpaper before using them to ensure a smoother surface compared to the previous set of experiments. Finally, MSNC-1 specimens were also tested with roller supports of diameter of 1.0 inch with the same configuration shown in Figure 9.27(b) (DE2M-S specimens). The transverse displacement of all specimens was measured by means of an LVDT mounted on the back side at a gauge length of 55 mm.

Figure 9.39 shows the response of DE2M-P specimens. A high tensile stress is observed for this case of specimens, despite the pre-treatment of the pin support cylindrical surface which was done in order to eliminate friction. This was considered conclusive evidence that the high tensile resistance observed when using pins could be attributed to the redundancy of the system. DE2M-P3 specimen was chosen as the average specimen to illustrate the strain distribution obtained by DIC with the same methodology explained above. Figure 9.40 shows the maximum principal surface strains developed at different characteristic loads. As it may be seen again, first cracking (P3) occurs prior to 80% of the peak load ($P2=0.8P4$) indicating a strongly hardening behaviour. The distribution in the elastic range is generally uniform with exception to some concentrations close to the grooving area. In the plastic region before the ultimate load, higher strains were concentrated in the central chord, but at ultimate load the crack localized in the vicinity of the fillet. This is consistent with previous tests and FEA. Two explanations are linked to that crack location: (a) the discontinuities in the geometry of the cross section creates stress concentrations and (b) the flow from one end despite providing fiber alignment in the central chord, creates an asymmetrical fiber distribution near the fillet of the opposite side of casting point – this was reported in Chapter 5 where model concrete experiments using the proposed form were conducted. In the descending branch, stress concentrations create local failure around the grooving area which is also explained by the jagged line of the branch.

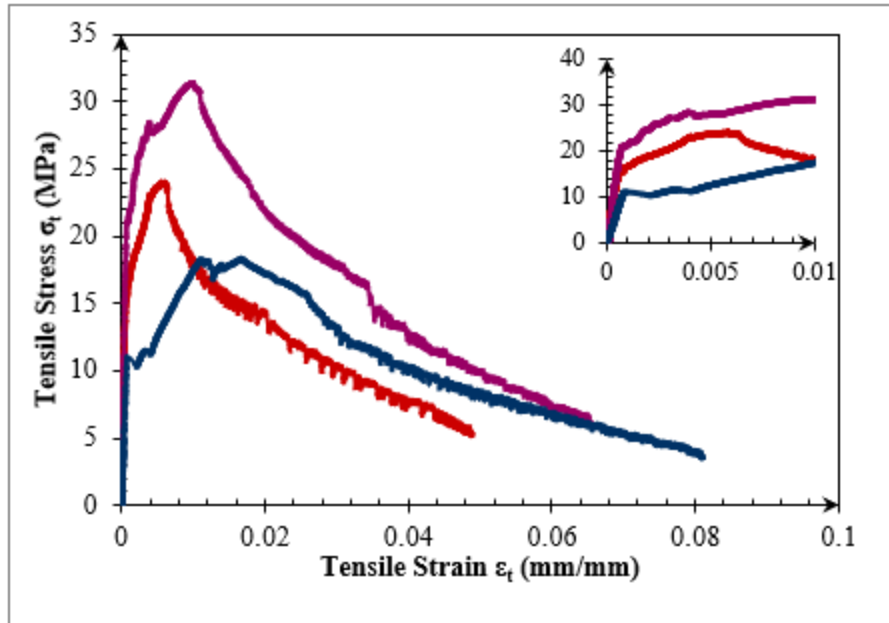


Figure 9.39: Tensile Stress-Strain Curve for DE2M-P specimens. The insert zooms into the ascending branch to max. strain of 0.01 (repetition of three specimens)

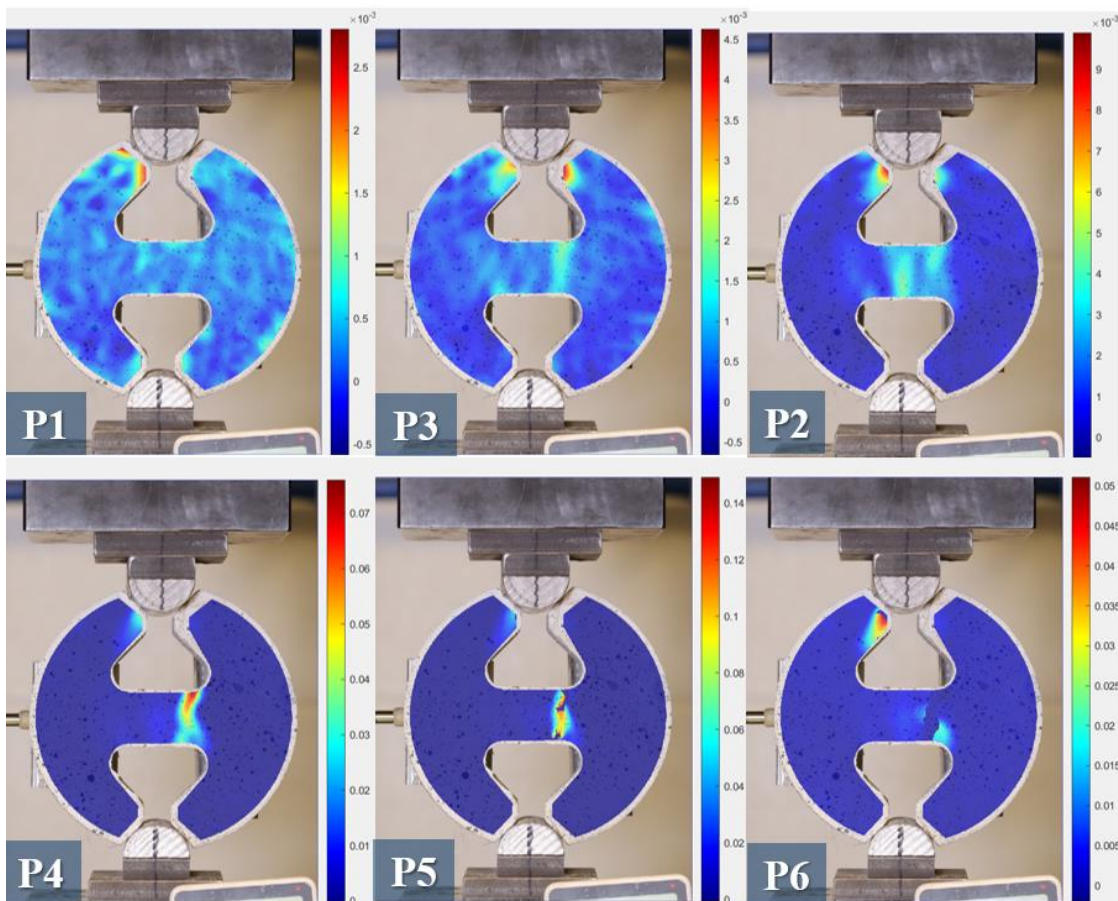


Figure 9.40: Minimum Principal Surface Strains for specimen DE1M-P1

DE2M-R specimens demonstrated lower tensile resistance as seen in Figure 9.41. Although all three specimens attained similar strengths, specimen DE2M-R2 experienced out of plane bending. As it observed in the enlarged portion of the graph before the ultimate load in Figure 9.41, the three specimens have the same stiffness and similar first cracking strength.

For this group of specimens, specimen DE2M-R3 and DEM-R2 were further analyzed with DIC to study the strain distribution at different load. For both cases, the maximum and minimum principal strains are illustrated in Figures 9.42-9.44. For DE2M-R2 the localization of compressive strains in the central chord before the ultimate load in combination the crack forming first in the back side and then propagating to the front suggest an out of plane bending failure. Regarding specimen DE2M-R3, a similar response was observed with the central region to experience compression in the horizontal direction and after the maximum load, localized tension cracks. The reason for this behaviour could be linked to a twist of the C channel despite that is glued on the bottom platen with the spherical hinge.

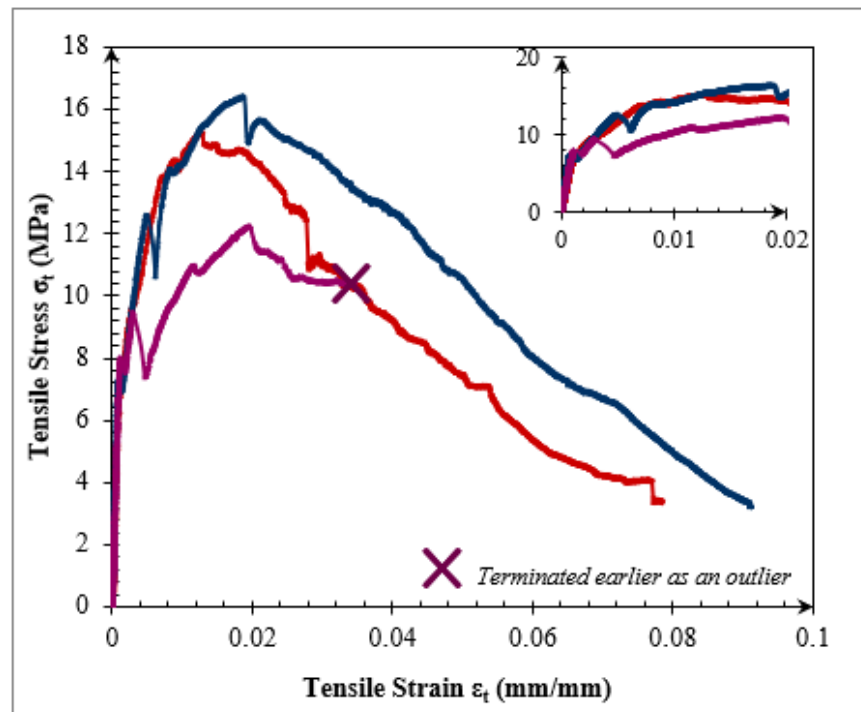


Figure 9.41: Tensile Stress-Strain Curve for DE2M-R specimens. The insert zooms into the ascending branch (repetition of three specimens)

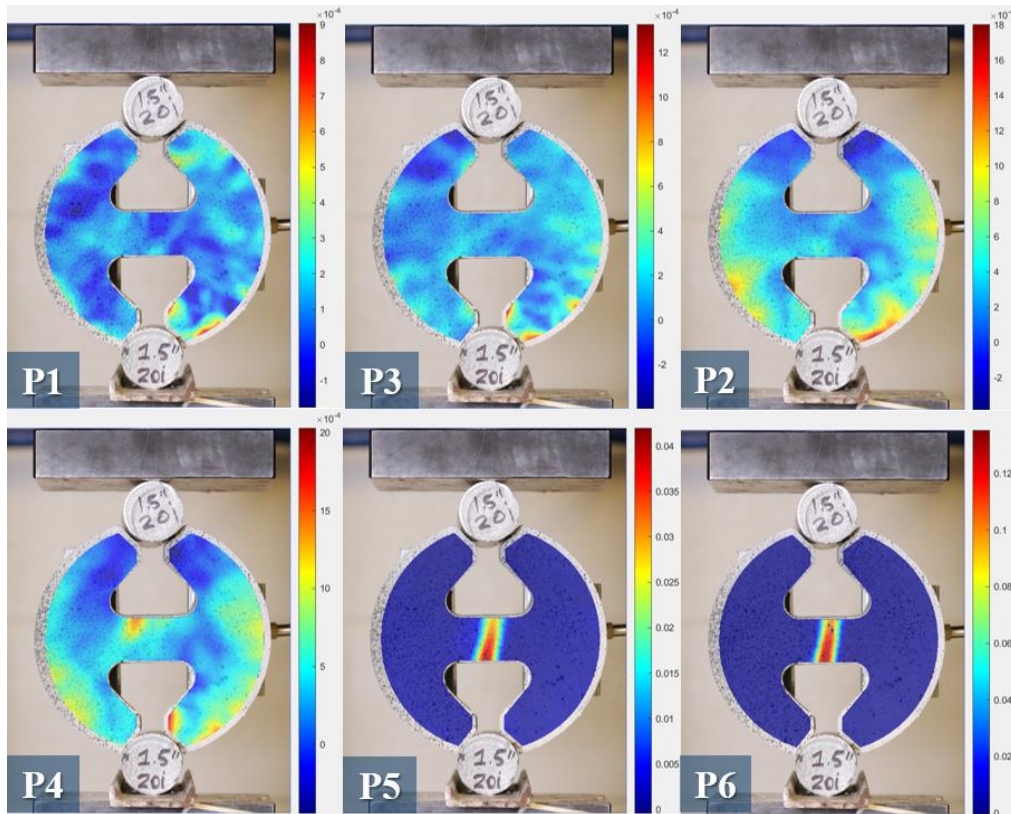


Figure 9.42: Maximum Principal Surface Strains for specimen DE2M-R2

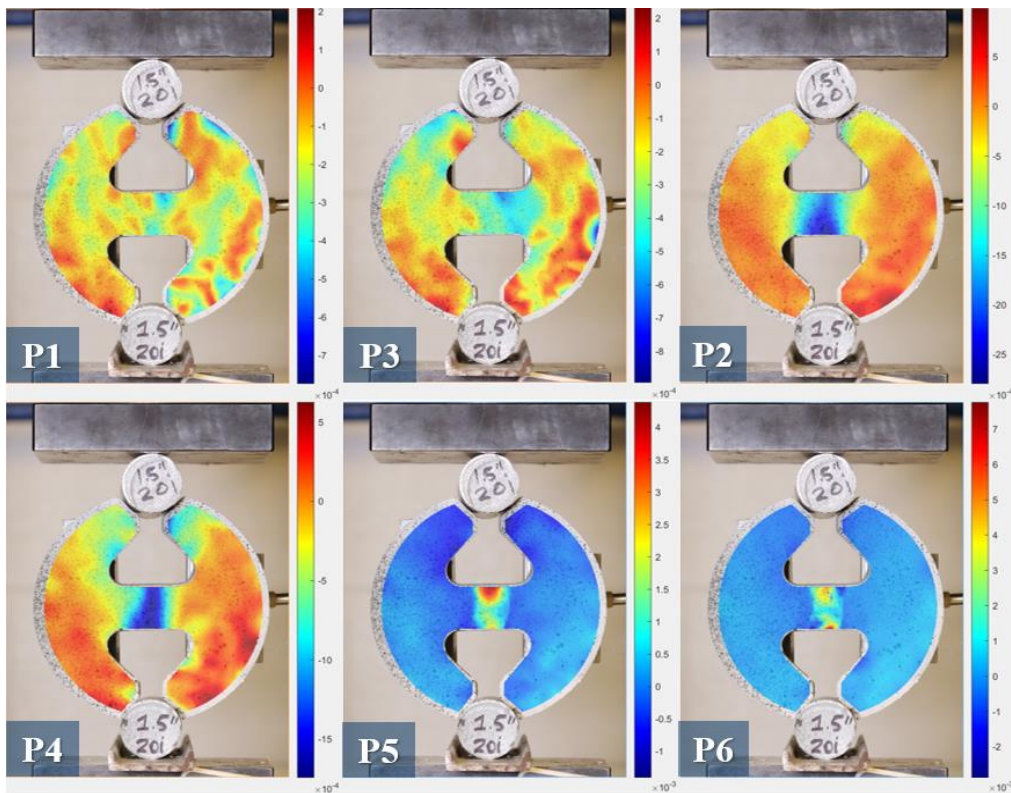


Figure 9.43: Minimum Principal Surface Strains for specimen DE2M-R2

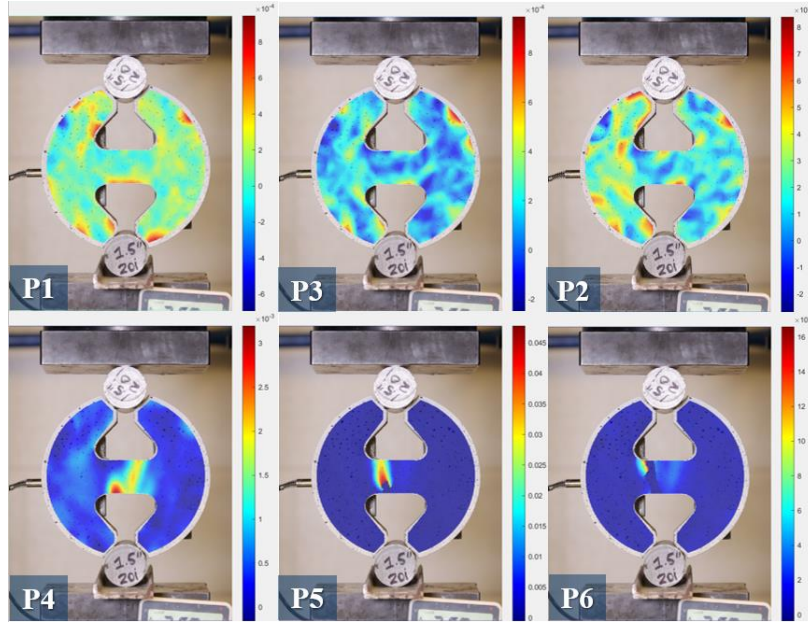


Figure 9.44: Maximum Principal Surface Strains for specimen DE2M-R3

Finally, Figure 9.45 illustrates the response of the DE2M-S specimens. This group exhibited the lowest tensile stresses, while the shape of the curves indicated that there was some torsion and/or out of plane bending. This was also seen in the failure pattern of DEM-S2 illustrated in Figure 9.46. The front face of the specimen did not show any major cracks, while the back face had a crack in the central chord region. The out of plane bending was more prominent regarding the axis of the specimen from the top view in Figure 9.46(c).

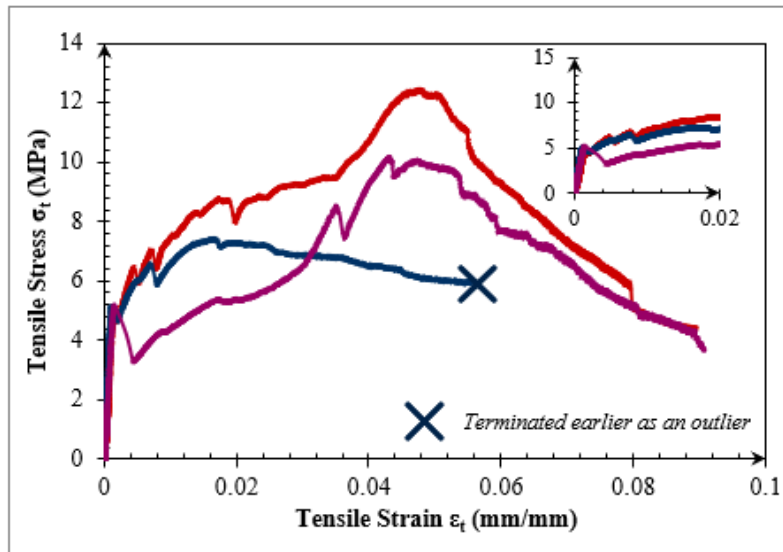


Figure 9.45: Tensile Stress-Strain Curve for DE2M-S specimens. The insert zooms into the ascending branch. (repetition of three specimens)



Figure 9.46: Fractured specimen due to out of plane bending (a) front view, (b) back view and (c) side view

Specimen DE2M-S3 is further studied via DIC analysis. Strain distribution in horizontal/transverse direction (ϵ_{xx}) at different loads are shown in Figure 9.47. At the early stages of the test around cracking, torsion was induced in the bottom half of the specimen due to adjustment of the roller and slight movement of the C channel. This phenomenon caused a compressive strain in the lower part of the central chord till 80% of the peak load in the ascending branch. Then at maximum load (P4), the top part of the central chord towards the right fillet experienced tension with onset of localization of tensile cracks being evident. At the descending branch the rest of the specimen unloaded, and the propagation of the crack continued.

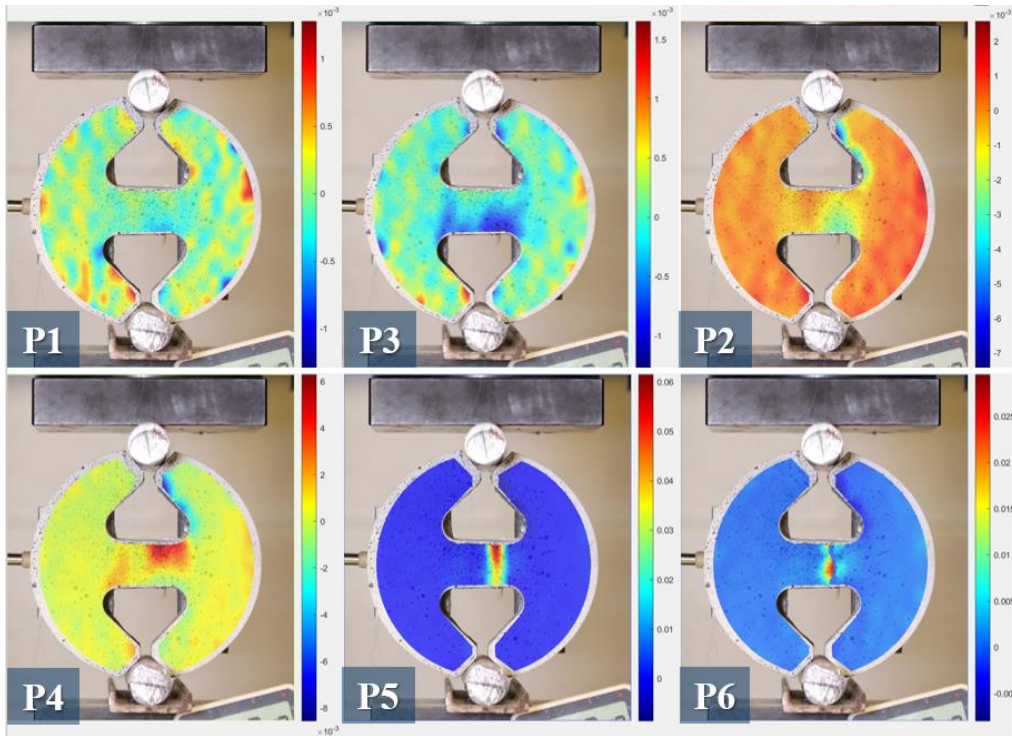


Figure 9.47: Transverse Surface Strains ϵ_{xx} for specimen DE2M-S3

- *Batch DE3-Effect of Friction and Testing Jig*

To prevent the unfavorable rotation of the rollers and the spherical hinge, a different testing jig, which is part of the conventional splitting test instrumentation was employed. Two lubricated smooth cylindrical shafts with diameter of 1.5 in. acted as support and loading rollers. The minimized friction was ensured with the placement of brass plates on the grooving area, while the instrumentation was the same as previous tests. Figure 9.48 illustrates the setup used for batch DE3.

The tensile stress-strain behaviour of the batch DE3 specimens is illustrated in Figure 9.48. The tensile strain is obtained as the average of the two faces: the front from virtual extensometers via DIC analysis and the back from LVDT. Virtual extensometers and LVDT had the same gauge length, namely 50 mm. The response is generally consistent with well-defined first apparent cracking, hardening and ductile post-peak branch. Compared to previous batches, the minimization of friction along with the new jig improved the response and consistency of results. The tensile properties of the specimens along with their average values and standard deviation (SD) are summarized in Table 9.7. The italic font of some specimens in Table 9.7 indicates unfavourable failure and have been excluded from calculation of average properties. Specimens T8 and T3 failed at the vicinity of the gauge length and thus are discarded. Meanwhile T9 and T10 failed with out of plane bending and were discarded, too.

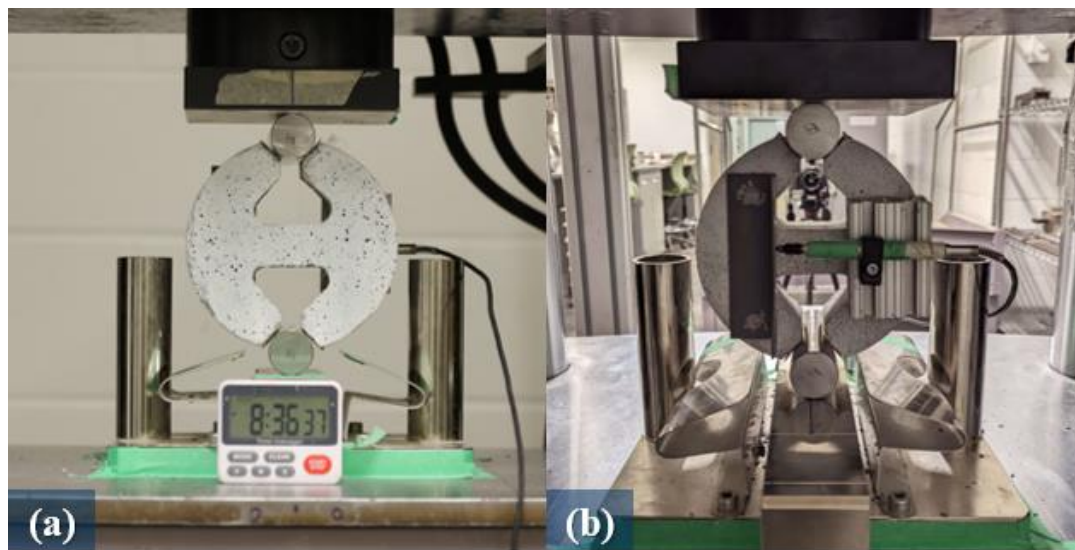


Figure 9.48: Experimental setup used for batch DE3; (a) front face of specimen with speckled pattern for DIC, (b) back face of specimen with LVDT for measurement of transverse elongation

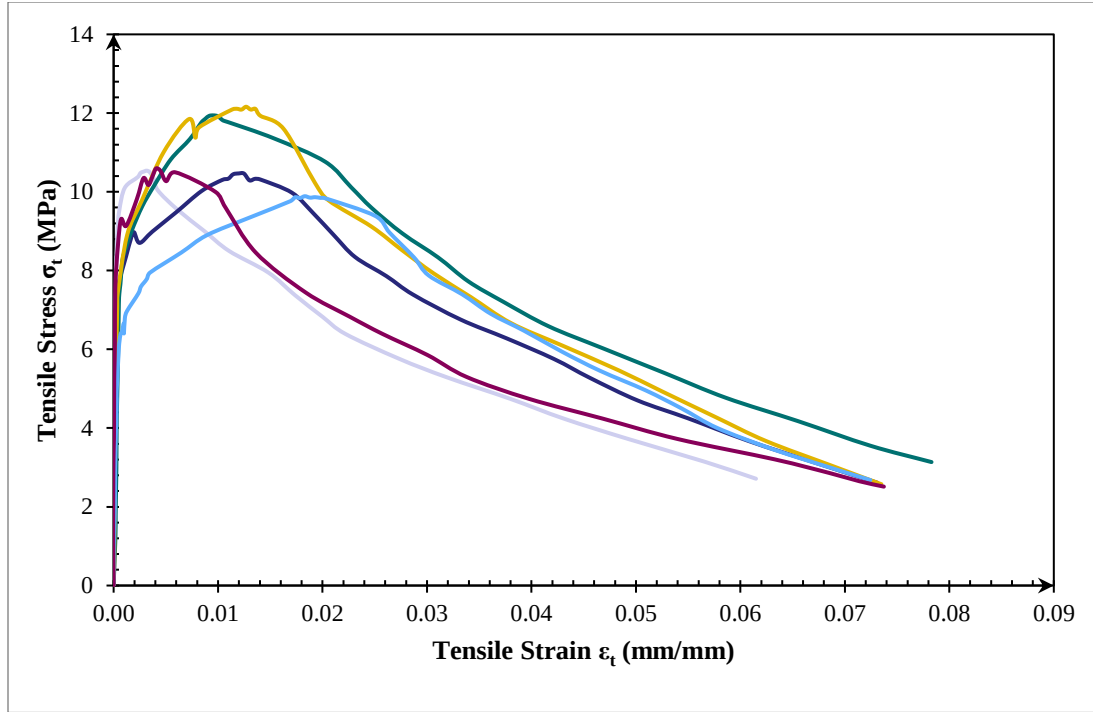


Figure 9.49: Tensile Stress-Strain Curve for DE3(repetition of six specimens)

Table 9.7: Tensile properties of DE3 batch

Specimen	f_{cr} (MPa)	ϵ_{cr}	f_{tu} (MPa)	ϵ_{tu}
T1	8.61	0.00041	10.59	0.0041
T2	6.66	0.00098	9.88	0.0183
T3	6.13	0.00046	8.83	0.0216
T4	7.42	0.00039	12.16	0.0127
T5	7.41	0.00055	11.94	0.0096
T6	7.6	0.00042	10.47	0.0120
T7	8.1	0.00022	10.54	0.0032
T8	8.11	0.00056	8.35	0.0035
T9	4.46	-0.00002	9.97	0.0055
T10	6.49	0.00035	10.64	0.0139
Average	7.63	0.0005	10.93	0.0091
SD	0.67	0.0003	0.91	0.0056

9.2.5. Comparison with Available Tensile Tests

Along with the above specimens in every batch, different specimen types were also cast and tested. For FD1 batch the results of the proposed setup were compared with direct tensile specimens of type SIA and GP (Figure 9.1), splitting cylinders (SP), and flexural specimens (AFNOR, 4PBT) from Hussain, (2021). Both flexural specimens were subjected to four-point bending, while their tensile strength was derived through inverse analysis proposed by Lopez (2016). Figure 9.50 shows the tensile strength obtained using different methods. It can be seen that for this batch the proposed

test demonstrated strengths similar to those of splitting, significantly higher than the remaining tests. Taking into consideration the strain distribution and the shape of the stress-strain curves previously presented, the high tensile resistance was attributed to the friction between the plaster layer and the loading rollers.

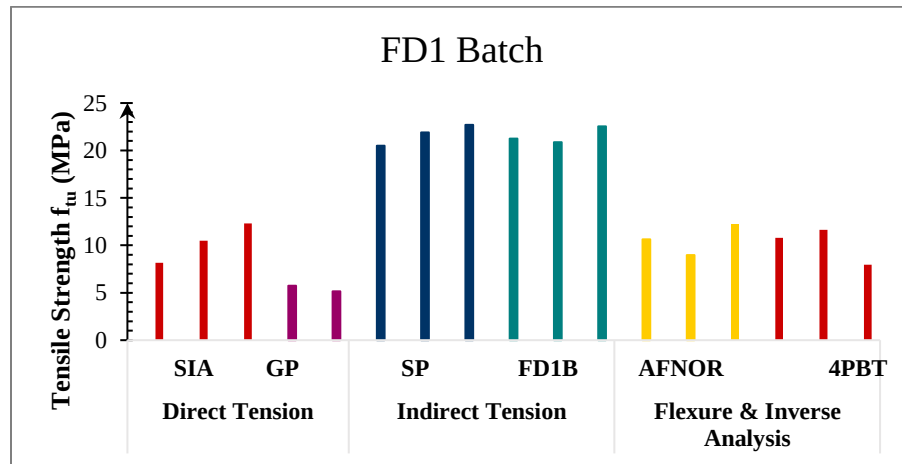


Figure 9.50: Comparison between tensile strengths obtained from different testing methods for FD1 batch

For FD2 batch, the comparison was performed with SIA, FHWA* and SP specimens. The FHWA* specimens are modified FHWA specimens (Figure 9.1) with half width in order to fit in the grips of the available UTM. Figure 9.51 illustrates the comparison between the different methods. The absence of plaster in the grooving area for full and half scale proposed specimens eliminates the friction, and the obtained results are comparable to those of direct tension. Furthermore, their response was further compared in Figure 9.52 that shows the tensile stress-strain response of the proposed tests and SIA and FHWA* specimens.

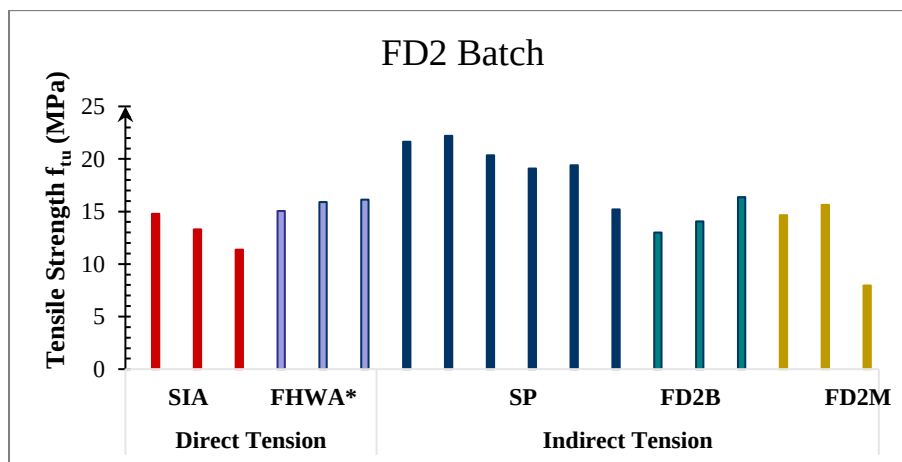


Figure 9.51: Comparison between tensile strengths obtained from different testing methods for FD2 batch.

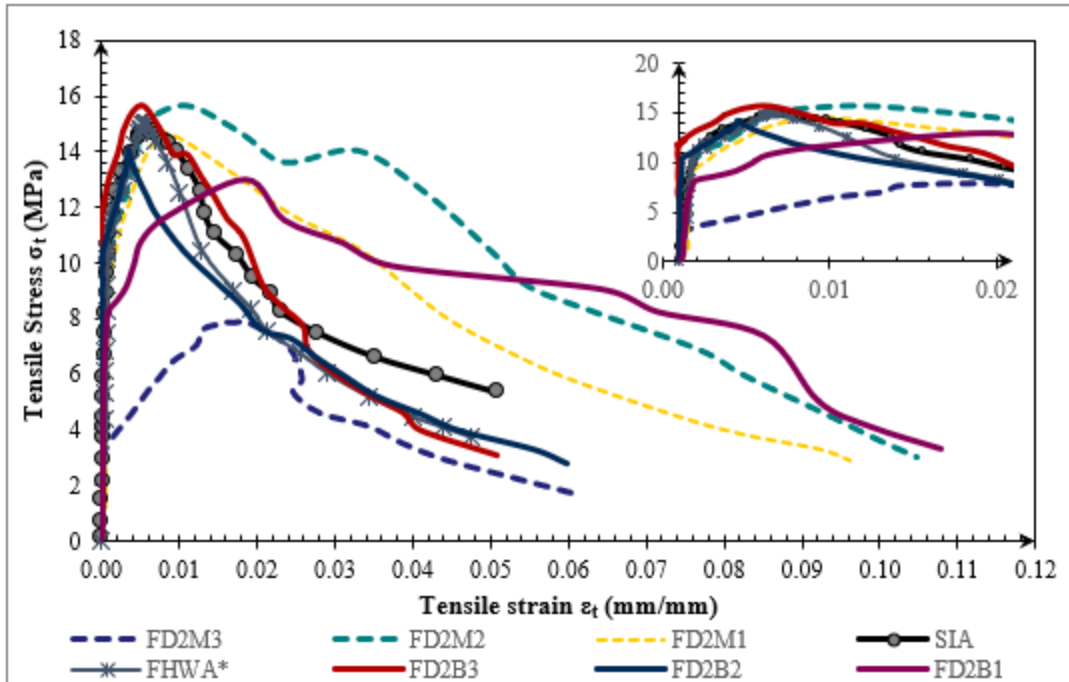


Figure 9.52: Comparison of tensile law of proposed specimens with direct tension specimens for FD2 batch. The insert zooms into the ascending branch and the onset of post peak response for some samples.

For DE1 batch, the results of DE1M-P and DE1M-R specimens were compared with direct tension and splitting tests and are shown in Figure 9.53. The use of pin supports increased the redundancy of the system resulting in an increase of strength of 60% compared to direct tension. For the rollers with greater diameter, the increase was 26%. Finally, the tensile law of the specimens is compared to the tensile law obtained by FHWA*. It is further shown that the pin supported specimens overestimate the tensile law especially after cracking.

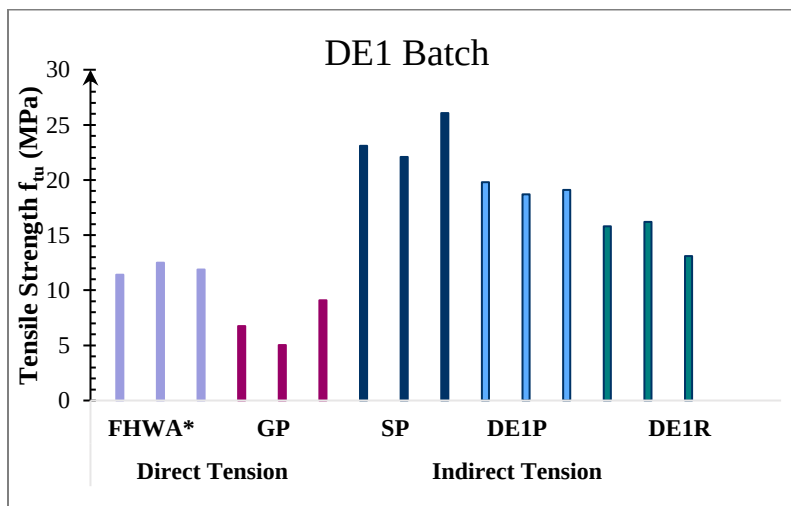


Figure 9.53: Comparison between tensile strengths obtained from different testing methods for DE1 batch.

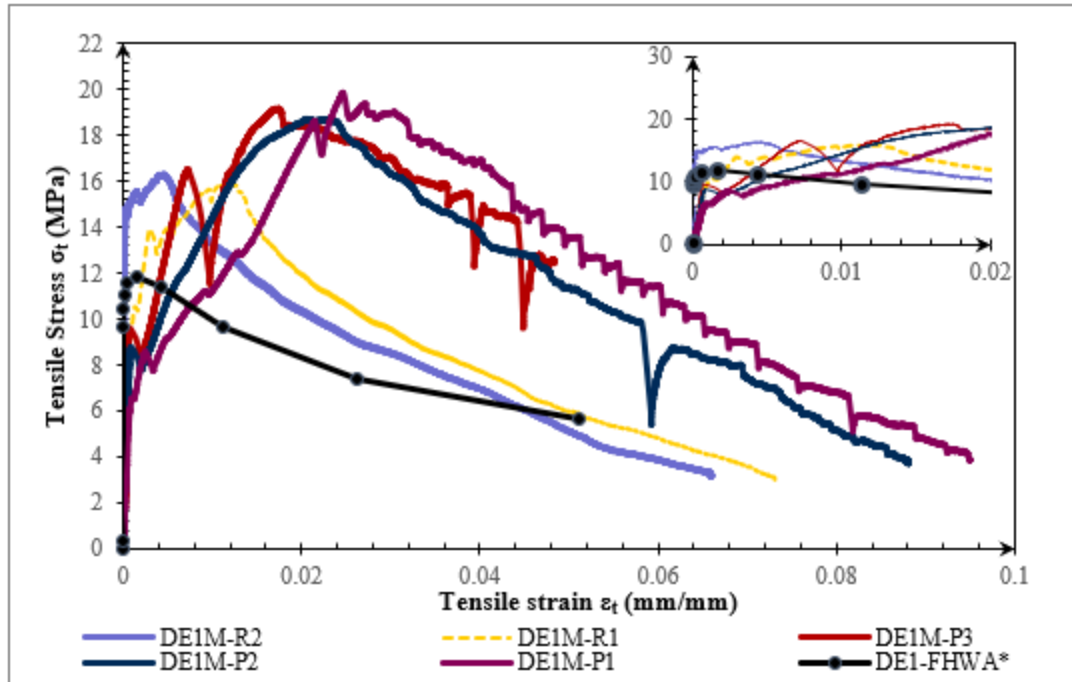


Figure 9.54: Comparison of tensile law of DE1M-P and DE1M-R specimens with direct tension for DE1 batch. The insert zooms into the ascending branch and the onset of post peak response for some sample

In case of batch DE2 (Figure 9.55), it is clear that the pin supports yield overestimated results. For rollers, the tensile strength is closer to the direct tension, but the response was problematic due to rotation of the spherical hinge and out of plane bending failure, especially for DE2S. Finally, the minimization of friction and the introduction of the new jig that prevents rotation yielded consistent results, with satisfying correlation with Direct Tension (Figure 9.56); this last figure highlights the gradual progressive improvement of the test setup developed and proposed in this thesis for tension characterization of THFRC.

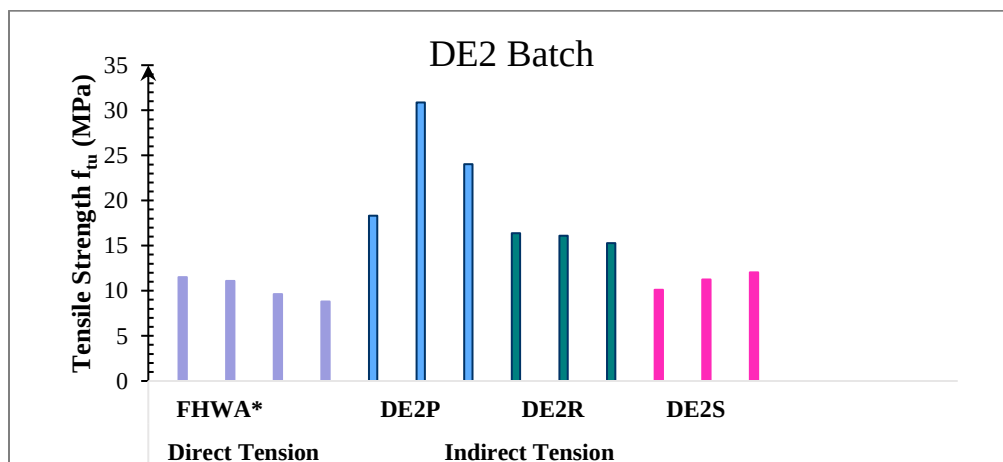


Figure 9.55: Comparison between tensile strengths obtained from different testing methods for DE2 batch

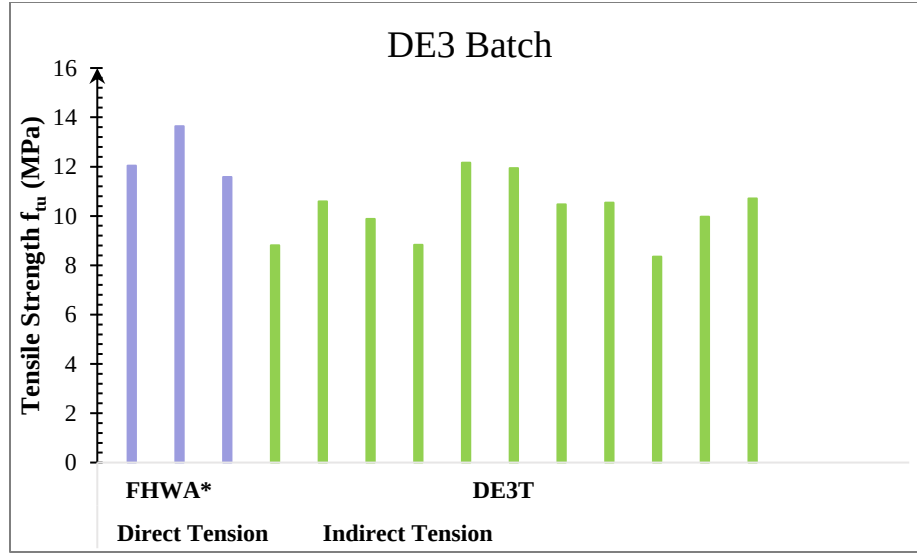


Figure 9.56: Comparison between tensile strengths obtained from different testing methods for DE3 batch

9.3. Conclusions

The results of experimental and analytical investigation on a new indirect tension test for THFRC presented and interpreted in this chapter lead to the following conclusions:

- The novel testing protocol developed and proposed in this thesis only needs conventional compression testing equipment and standard training of technical staff conducting the test – this is an important improvement over other quality control and characterization test setups, and it has the added advantage that may be implemented by most commercial testing facilities which would otherwise be exempted from the UHPFRC testing and quality control market.
- The geometry of the specimen which is based on strut and tie theory generates a well-defined central chord with constant cross section which is in pure tension, while the specimen is subjected to vertical compressive load in the same manner as in a splitting experiment. This is validated consistently through nonlinear FEA modelling and experiments.
- With the proposed test, the whole tensile law, namely pre-peak and post-peak branches, can be easily obtained without complicated inverse analysis as in case of flexure or uncertainties of the actual stress state as in case of conventional splitting test.
- Full and half scale specimens were tested, and no noticeable size effect was observed.

- The two types of unfavorable failure patterns observed were cracking in the vicinity of the central chord and out of plane bending. Both could be explained to the fiber distribution and orientation of the specimen, with the latter to be also linked to problematic support conditions which were subsequently corrected in the proposed test setup; the fiber distribution issue causes eccentricities as well in the direct tension tests and it is a sign of partial fiber segregation that occurs in overly flowable mixes.
- Roller supports were found to be more accurate over pin supports which lead to overestimation of strength due to indeterminacy. Additionally, the use of spherical hinge was linked to unfavorable out of plane rotations and thus leading to unreliable results. The latter was solved by using the base of conventional splitting testing frame.
- Friction has strong influence on the results and was minimized by use of brass plates in grooving areas and lubricated loading shafts. The latter in combination with the splitting frame yielded the most reliable experimental results which were also validated through DIC.
- With results repeatedly comparable to those of direct tension, the proposed modified splitting test can be used for both characterization and quality control of the tensile properties of UHPFRC. In that way complicated analysis procedures that require highly specialized personnel can be eliminated while facilitating standard testing equipment available in most commercial testing laboratories.

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Chapter 10: Multifunctional Nanoengineered Hybrid THFRC

10.1. Introduction

The prevalent applications of cementitious materials in the infrastructure combined with the performance deterioration due to aging, aggressive environmental exposure, loading conditions or various types of hazardous events have generated the necessity of developing new techniques of Structural Health Monitoring (SHM). The field of SHM involves a variety of methods which enable assessment and preservation in order to maintain sustainable structures. Existing techniques commonly involve strain, stress or crack sensors which are either embedded or attached to a structure. These non-intrinsic types of self-sensing structures use electric resistance strain gauges, optical fibers, piezoelectric materials, or Shape Memory Alloys (SMAs). However, drawbacks of non-intrinsic methods often include uncertainty on the degree of adhesion and possible local disturbance of the strain state in the interface between sensor and structure, challenges in scaling up or lack of continuity. A solution to these, is Intrinsic Self-Sensing Concrete (ISSC) which functions as both structural and sensing material thanks to incorporation of conductive fillers. As already described in Chapter 2, the self-sensing property is based on a unique capability of semiconductors known as piezoresistivity and describes the change of relative dimensions and resistance due to applied stress. This effect is the most commonly measured sensing signal in materials. More specifically the Fractional Change in Resistance or Resistivity (FCR) is used to assess the effectiveness of sensing.

In recent decades the research community has focused on the development of ISSC using conventional cementitious paste or mortar. With the advance of High-Performance Fiber Reinforced concretes, research interest gravitates towards UHPFRC materials that possess the inherit self-sensing capability due to the embedded distributed metal fibers. Although there are numerous studies on self-sensing capabilities of plain conventional concretes, there are limited studies on self-sensing of UHPFRCs. One of the first investigations was performed on reactive powder concrete (RPC) reinforced with a short-cut super-fine stainless-steel wire by Dong et al., (2016). The self-sensing performance of the ISSC was assessed by correlating the stress/strain response with FCR under external loads that included cyclic compression, monotonic compression until failure and monotonic flexure until failure. Although the sensing system was effective, the

20 μm diameter wire significantly compromised the flexural strength by as much as 50% compared to steel fibers that are conventionally used in UHPFRC.

Only recently, carbon nanomaterials have been used as a substitute of reactive powders in UHPFRC mixes to enhance the mechanical properties opening up great potential to allow UHPFRC to effectively sense stress, strain, and cracks (Wille & Loh, 2010). It is found that the addition of CNTs in UHPFRC substantially improved the electrical conductivity with pre-cracking self-sensing using the FCR method to be more effective under flexure than compression. This might be explained by the dense microstructure and the lack of pore water of this class of materials. The first is related to hydration products that fill the gaps between CNTs hindering the connection between the conductive materials (fibers and CNTs) while the pore water increases the conductivity (Lee et al., 2018; You et al., 2017). Similar response was observed by other researchers, who verified that the synergistic effect of hybrid reinforcement (steel fibers & CNTs) is important to a smooth self-sensing performance with minimized noise, while post-peak response in tension can be predicted through FCR in case of UHPSFRC with CNTs (Yoo et al., 2018)

As already mentioned, a limited number of studies have been performed on the self-sensing capabilities of carbon nanoengineered UHPFRC. In addition to that limitation, the current literature is focused on non-proprietary UHPFRCs containing carbon nanomaterials, while using conventional methods with embedded or attached electrodes to measure the electromechanical properties. In this study a proprietary nanoengineered UHPFRC mix is selected while a wireless approach for assessing the self-sensing ability is investigated compared to the conventional bulk resistivity methods. The piezoresistivity was explored under three types of loading: i) monotonic tension, ii) monotonic flexure, and iii) monotonic out of plane bending until failure. The first two load cases were studied with the 4-probe method via embedded electrodes while for the last, piezoresistivity was investigated using the Connectionless Electrical Pulse Response Analysis (CEPRA) test method. The correlation of stress and strain state with the change in resistance for direct tension and out of plane bending was performed via Digital Image Correlation.

10.2. Materials & Methods

10.2.1. Materials

The UHPFRC used in this study is a proprietary mix (ce200SF-GTM, ceEntek, Ltd.) Although the exact proportions of the mix design remain confidential, the dry premix contains only High Sulphate Resistant (HSR) cement and finely graded spherical sand. The mix proportions are shown in Table 10.1. The other liquid component consists of a CNF suspension (cePA3-86SD) that includes Carbon Nanofibers (CNF) and a superplasticizer diluted in potable water. The amount of CNFs is approximately 1.0 kg/m³ of UHPFRC which is an equivalent of 3.3 trillion fibers per m³ (ceEntek, 2022). Table 10.2 summarizes the properties of CNFs. The metallic microfibers are short straight copper coated steel fibers WSF0213 (Ganzhou Daye Metallic Fibres Co., Ltd), with properties listed in Table 10.3.

Table 10.1: Mix proportions for nanoengineered UHPFRC by mass

Mix Component	Quantity
ce200SF-G premix	1
cePA3-86SD CNF suspension	0.0078
Water	0.078
Steel Fibers WSF0213	0.07

Table 10.2: Properties of CNF

Diameter (nm)	Average Length (μm)	Surface Area (m ² /g)
50-200	10	120-130

Table 10.3: Specifications of Steel fibers

Tensile Strength (MPa)	Young's Modulus (GPa)	Specific Gravity (gr/cm ³)	Diameter (mm)	Length (mm)	Aspect Ratio
2600	200	7.8	0.2	13	65

10.2.2. Mixing, Casting & Specimen Preparation

For this investigation two batches were manufactured: a preliminary batch for assessment of mechanical properties such as tension, flexure and compression and a main batch for assessment of the self-sensing capability. For the first batch B1 a SP15Qt Gear Driven Planetary Mixer was used while for the second batch B2 (15 L of UHPFRC) it was replaced with a SP30Qt Gear Driven Planetary Mixer. First the premix was introduced to the mixer and mixed for 3 minutes. Prior to use, the CNF/admixture paste was stirred with an electric mixing drill and then diluted with water to form the CNF suspension. Then the formed suspension was slowly introduced to the mixer and

blended for 5 minutes at low speed. At the beginning the mix is dry and as the time progresses (Figure 10.1(a)), the small spherical agglomerations form larger pieces with viscous texture (Figure 10.1(b)). The fresh UHPFRC behaves like a non-Newtonian fluid hindering the mixing at medium or high speeds. More specifically it becomes stiffer when the force from the mixing blades is acting on it (Figure 10.1(e)) while it is very flowable when resting (Figure 10.1(f)). Although in most UHPFRCs the fibers are added at a stage where the fresh mix has high flowability, for this mix - due to its behaviour, the steel fibers are incorporated once a uniform mass of thixotropic UHPFRC is formed (Figure 10.1(c)) and then mixing continues for 3 minutes (Figure 10.1(d)). Before casting, the flowability of the mix is assessed per ASTM C1856, (2017) and the temperature of the fresh UHPFRC is measured. The static flow spread is 213 mm, and the temperature of the mix 27 °C. The increased temperature might be attributed to the difficulty of the mixer to mix the highly thixotropic UHPFRC.

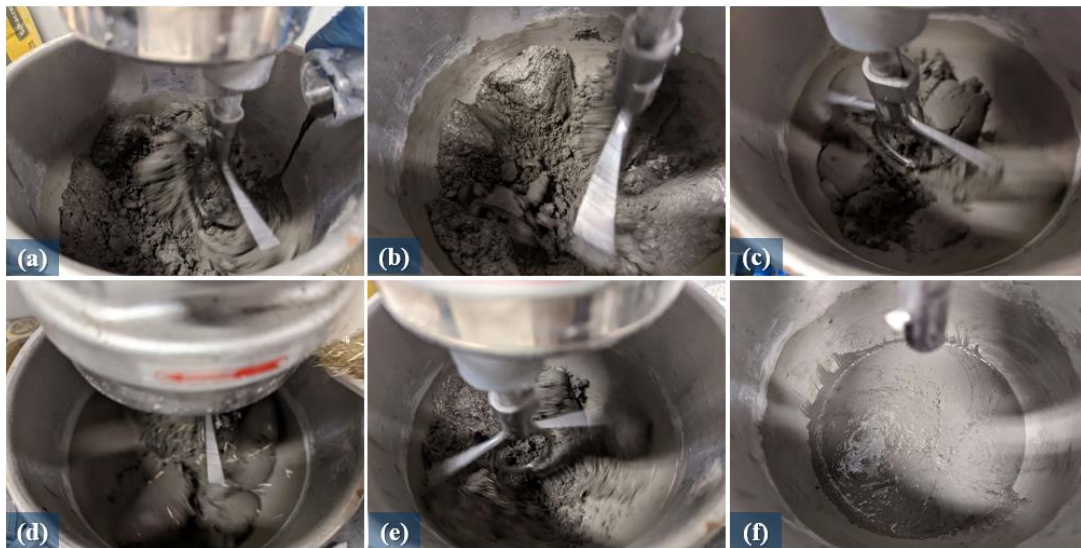


Figure 10.1: Mixing stages of UHPFRC with CNF (a): incorporation of CNF suspension, (b) change of consistency (c) thixotropic paste, (d) incorporation of steel fibers, (e) non-Newtonian fluid behaviour due to forces from the blade, and (f) flowable mix at rest

For the determination of mechanical properties, the specimens prepared included: two cylinders having dimensions of 75 mm x 150 mm to be tested in uniaxial compression (CC-1), two 75 (mm) x 75 (mm) x 280 (mm) flexural prisms (FP-1) and two 25 (mm) x 50 (mm) x 430 (mm) direct tension prisms (DT-1).

For the assessment of self-sensing capacity three different types of specimens were fabricated in triplets: (i) 75 (mm) x 75 (mm) x 280 (mm) flexural prisms (FP-2), (ii) 25 (mm) x 50 (mm) x 430

(mm) direct tension prisms (DT-2), (iii) panels with C shaped cross section for out of plane bending (OPB). Cylinders with diameter of 75 mm and height of 150 mm were also cast for determination of compressive strength only (CC-2). The dimensions of the OPB specimens are illustrated in Figure 10.2. The first three types of specimens are cast with flow from one end parallel to the larger dimension of the specimen as a common practice used to achieve fiber alignment while the cylinders are cast in one layer with a scoop.

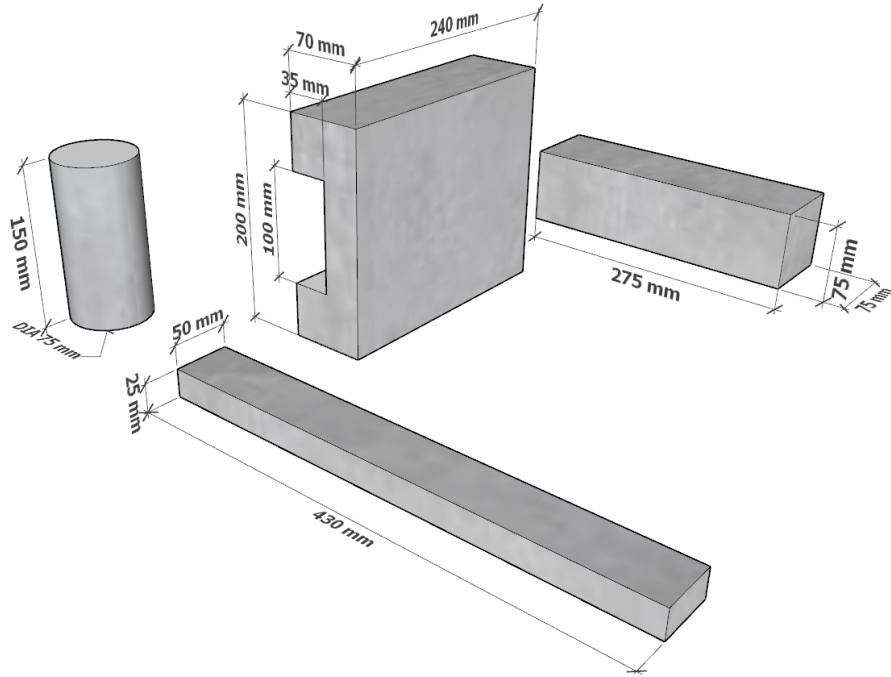


Figure 10.2: Specimens of B2 for electromechanical testing of nanoengineered UHPFRC

For FP-2 and DT-2 specimens, stainless steel wire mesh is used as electrodes and embedded in the concrete free surface. After casting, the specimens were covered with plastic sheets to prevent evaporation and shrinkage followed by demolding at 24 hours and wet curing for 28 days and then left to dry in ambient temperature and relative humidity of 60% till the age of testing. For self-sensing properties the testing was performed in mature age of a year, while for mechanical properties the testing was conducted at 60 days.

10.3. Mechanical properties

10.3.1. Compressive Strength

The UHPFRC cylinders were tested in accordance with CSA S6, (2018) and CSA-A23.2-9C, (2014) with a force control at rate of 1 MPa/sec on a Controls Compression Machine. Prior to

testing, the cylinders were ground until the surface of the cross section was leveled and smooth. Then the diameter on the top and bottom end surfaces were measured via a digital caliper and the average diameters were calculated. Table 10.4 includes the compressive strength for both batches. In all cases the failure was followed by loud sound while concrete pieces shattered in a brittle manner. As expected, the strength continues to develop even in ambient temperature and is increased by almost 10% after 10 months. The failure mode observed in the cylinders was a combination of Type 1 and Type 2 compressive failure as defined in CSA A23.2-9C. Namely there were well-formed cones on both ends or on one end and vertical cracks through caps on the other end (Figure 10.3).

Table 10.4: Compressive Strength of two batches of nanoengineered UHPFRC.

Batch	Sample	Compressive Strength (MPa)
1	CC-1A	157.5
	CC-1B	140.18
	Average CC-1 (SD)	148.18 (12.24)
2	CC-2A	155.71
	CC-2B	163.97
	CC-2C	175.57
	Average CC-2 (SD)	165.08 (9.97)



Figure 10.3: Fracture Pattern of nanoengineered UHPFRC in compression

10.3.2. Tensile Strength

For the determination of tensile strength, DT1 specimens were subject to direct tension by implementing a servo-controlled, closed loop Universal Testing Machine (UTM) at a loading rate of 0.15 mm/min. Prior to testing, and in order to avoid premature failure in the vicinity of the gripped area, CFRP sheets were applied to strengthen the ends. The cracking and ultimate tensile strength for the tested specimens are listed in Table 10.5. Both specimens formed cracks within the gauge length but close to the strengthen areas (Figure 10.4).

Table 10.5: Tensile Properties of nanoengineered UHPFRC for Batch 1

Sample	Apparent Cracking Load P_{cr} (kN)	Peak Load P_{max} (kN)	Cracking Strength f_{cr} (MPa)	Ultimate Strength f_{tu} (MPa)
1	6.6	10.1	5.28	8.08
2	7.0	11.75	5.6	9.4
Average (SD)	6.8 (0.28)	10.93 (1.16)	5.44 (0.22)	8.74 (0.93)



Figure 10.4: Failure of nanoengineered UHPFRC in tension

10.3.3. Modulus of Rupture Tests and Inverse Analysis

For the determination of flexural performance and back-calculation of tensile stress-strain law, a third point bending test was performed on FP-1 specimens in accordance with ASTM C1609, (2019). Two loading stages were adopted: a loading rate of 0.15 mm/min till the specimen reached to post peak load equal to 70% of maximum, and then 0.2 mm/min for the rest of the experiment until the post-peak residual strength had dropped to 20% of the peak load. More details regarding the test and experimental setup may be found in Chapter 6. Figure 10.5 illustrates the resistance curves of the tested specimens. Table 10.6 summarizes the flexural properties that can be found according with the ASTM 1609 (2019). The addition of CNFs have increased the stiffness of the material with the deflections at first peak and ultimate load to be lower as compared to the flexural performance of other proprietary UHPFRC mixes of similar strength found in the literature (Pantazopoulou et al., 2019).

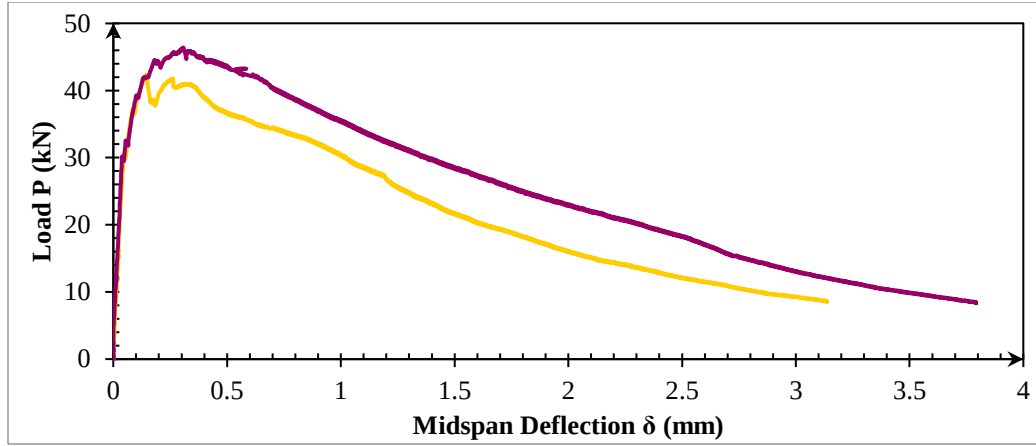


Figure 10.5: Resistance Curves of tested nanoengineered UHPFRC flexural prisms for Batch 1(repetition of two specimens)

Table 10.6: Summary of Experimental Flexural Performance Properties of Batch 1 nanoengineered UHPFRC

Sample	1	2	Average (SD)
First Peak Load P_1 (kN)	30.02	30.68	30.35 (0.46)
Peak Load P_p (kN)	46.39	42.38	44.38 (2.84)
First-peak deflection δ_1 (mm)	0.04	0.046	0.043 (0.005)
Peak Load deflection δ_p (mm)	0.3	0.16	0.23 (0.10)
Residual Load at L/600 P_{600}^D (kN)	44.93	39.87	42.4 (3.58)
Residual Load at L/150 P_{150}^D (kN)	28.28	21.52	24.9 (4.78)
First Peak Strength f_1 (MPa)	16.01	16.36	16.18 (0.25)
Peak Strength f_p (MPa)	24.74	22.60	23.67 (1.51)
Residual Strength at L/600 f_{600}^D (MPa)	23.96	21.26	22.61 (1.9)
Residual Strength at L/150 f_{150}^D (MPa)	15.08	11.47	13.28 (2.55)
Toughness T_{150}^D (J)	60.46	48.75	54.61 (8.27)
Equivalent Flexural Strength Ratio $R_{D,150}^T$ (%)	13.4	10.6	12 (2.0)

Apart from the flexural properties, an inverse analysis for determination of constitutive tensile law of nanoengineered UHPFRC from flexural tests was also performed. In the closed-form simplified inverse analysis proposed by Lopez (2017) and adopted by CSA S6 Annex A8.1 four cardinal points of the resistance curve are input to equations of Table 10.7 that are used to form a bilinear tensile stress-strain and tensile stress-crack width diagram. The points are selected based on an initial slope S_o (Figure 10.6). More details about the method can be found in Chapters 8 and 9.

Table 10.7: Derived Expressions of the Inverse Analysis Method (Annex 8.1 of CSA-S6 2018)

Property	Normalized Parameters
$E_{co} = \frac{7.2}{b} S_o$	
$f_{cm} = K_1 \frac{P_1 L}{bh^2}$	$K_1 = \frac{(P_1/P_2)^{0.19}}{1.63}$

$\varepsilon_{cr} = \frac{f_{crm}}{E_{co}}$	
$\varepsilon_{tu} = K_2 \varepsilon_{cr}$	$K_2 = \left(7.65 \frac{\delta_3}{\delta_1} - 10.53 \right)$
$f_{tu} = K_3 f_{crm}$	$K_3 = K_2^{-0.18} \left(2.46 \frac{P_3}{P_1} - 1.76 \right)$
$\varepsilon_{to} = K_4 \varepsilon_{cr}$	$K_4 = K_3^{-0.37} K_2^{0.88} \left(3.00 \frac{\delta_4^*}{\delta_3} - 1.80 \right)$
$w_o = \left(\varepsilon_{to} - \varepsilon_{tu} + \frac{10 f_{tu}}{3 E_{co}} \right) \frac{3h}{2}$	
$\delta_4^* = K_5 \delta_4$	$K_5 = 1 + \frac{0.6}{L} d_o$

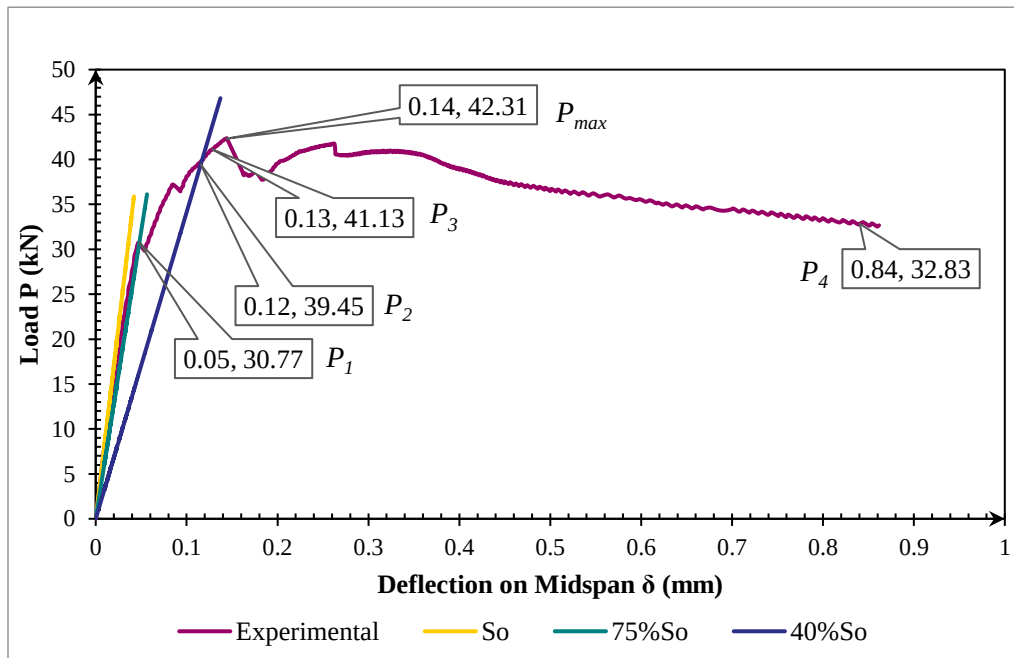


Figure 10.6: Evaluation of slopes and characteristic points for inverse analysis as per CSA Annex A8.1

The linearized constitutive law as obtained from the inverse analysis is illustrated in Figure 10.7. The material is strain hardening with a hardening ratio of 1.06. More specifically it has an average cracking strength 9.23 MPa and an average ultimate tensile strength of 9.8 MPa. The latter is slightly higher than the results observed in direct (uniaxial) tension, while the cracking strength from the inverse analysis is almost 70% higher than the one obtained from uniaxial tension. Regarding the ultimate strain, the difference between the specimens may be attributed to the lower ductility observed in FP1-B but also the randomness of the fiber orientation that is characteristic of this thixotropic mix.

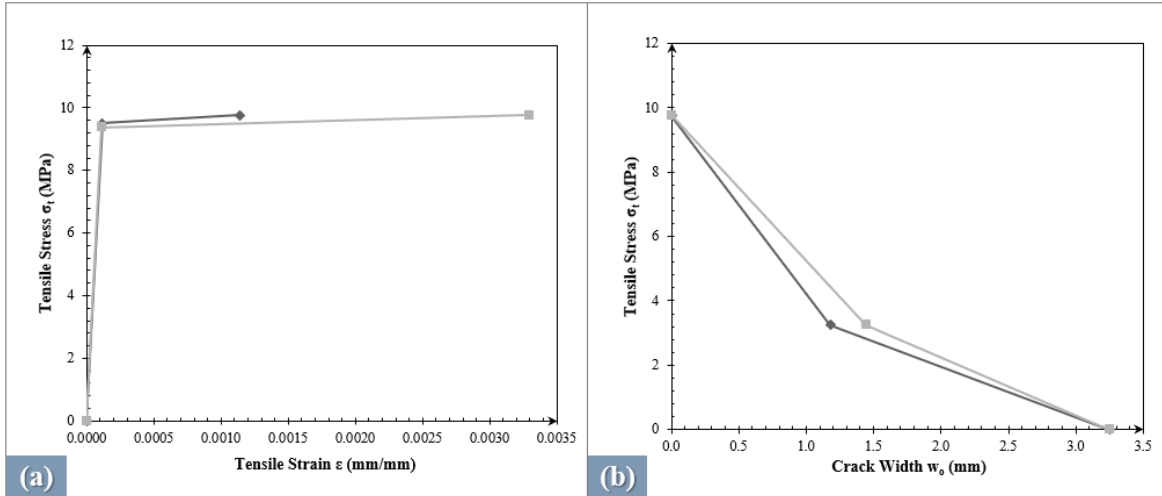


Figure 10.7: Linearized (a) Tensile Stress-Strain law and (b) Tensile Stress-Crack Width law from inverse analysis on Batch 1 flexural prisms of nanoengineered UHPFRC (repetition of two specimens)

10.4. Electrical Properties

Prior to the assessment of self-sensing performance, the effect of the two and four probe method on polarization was investigated, while resistivity in both directions was recorded via CEPRA. This preliminary work was performed in order to determine the appropriate time drift of polarization and study the sensitivity of CEPRA readings with regards to fiber distribution and boundary conditions.

10.4.1. Polarization of Electrical Resistance

As already discussed in Chapter 2, there are two established measurement techniques for the electrical resistance: namely the two and four-probe method. Generally, the four-probe method is preferred over the two-probe, as the contact resistance between the electrodes and the nanoengineered concrete is eliminated. For simple measurements that do not require complicated configuration, for both techniques, a direct current (DC) testing procedure is employed. Despite its simplicity, DC is associated with an effect known as electrical polarization. The latter refers to the separation of the center of positive charge and the center of negative charge in a dielectric material due to the induced electric field. Polarization causes extra charge, reducing the potential and thus the resistance over time. In order to assess properly the self-sensing performance of the produced nanoengineered UHPFRC, it is important to counteract polarization by applying electric current prior to testing till a plateau is reached. This time drift has to be determined prior to testing.

- *Experimental Setup and Procedure*

As mentioned earlier, the FP2 and DT2 specimens contained embedded electrodes in the form of stainless-steel wire mesh. The electrode configuration is shown in Figure 10.8. Since these specimens were designed for further mechanical testing, the placement was done in order to avoid premature failure and to not disturb with inclusions the constant moment region for DT2 and FP2 specimens, respectively. The four-probe method requires two outer input current electrodes and two inner output current electrodes.

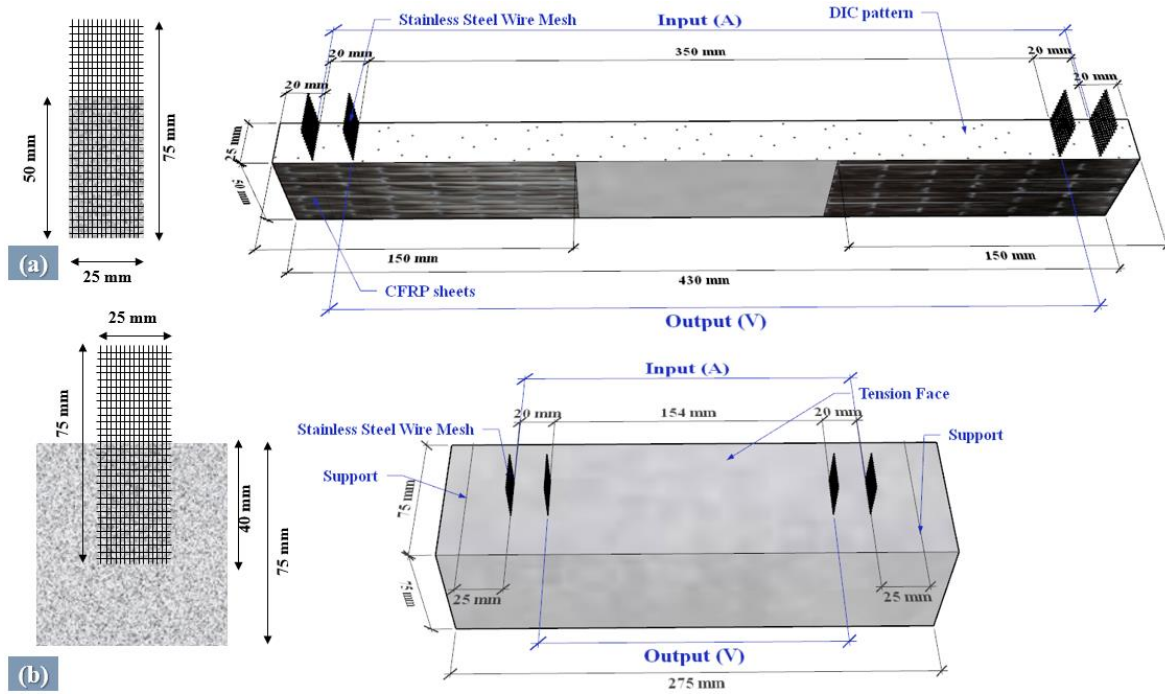


Figure 10.8: Electrode arrangement and geometry in (a) DT-2 specimens and (b) FP-2 specimens

As shown in Figure 10.9, a commercial digital multimeter (Keithley 2110-120 5.5) was employed to measure the polarization while the data were collected automatically by an instrument control software (Keithley KickStart Software). For both methods, the magnitude of the input current was maintained at 1 mA for 30 minutes to monitor the electrical polarization. The tests were conducted in an environment with a constant temperature (24 °C) and relative humidity (60%).

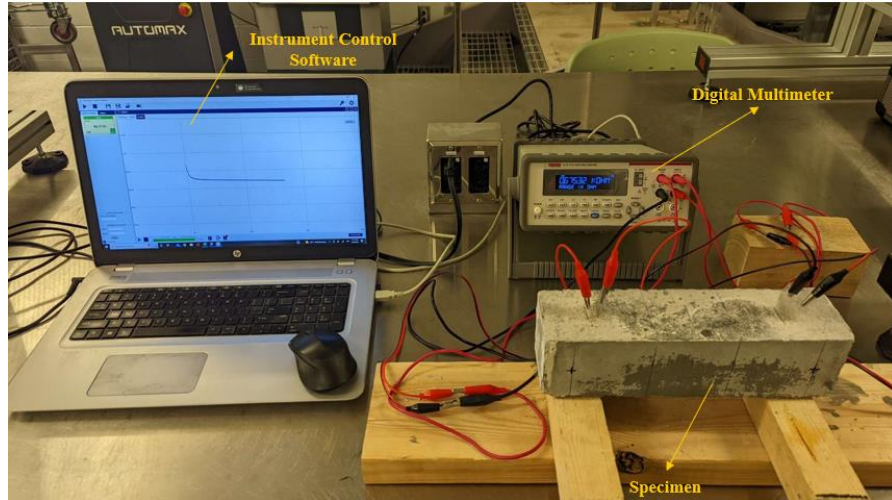


Figure 10.9: Instrumentation and experimental setup for measurement of polarization via four-probe method

Figure 10.10(a),(b) illustrates the change in electrical resistance of the nanoengineered THFRC due to electrical polarization before applying external load, for FP2 and DT2 specimens, respectively. In case of FP2 specimens, it can be observed that the electrical resistance dropped instantly at the beginning and then decreased with a slower pace until reaching the polarization time for stable measurement. However, for DT2 specimens, the reduction was steeper followed by an abrupt increase and then a plateau in shorter time compared to flexural prisms. The latter may be explained by better fiber alignment in case of direct tension specimens which was linked to wall effects. It was known that more conductive pathways reduce the time drift of polarization (Chung, 2021). The difference in range of resistance between the two different types of specimens was owing to different electrode configuration, and specimen size.

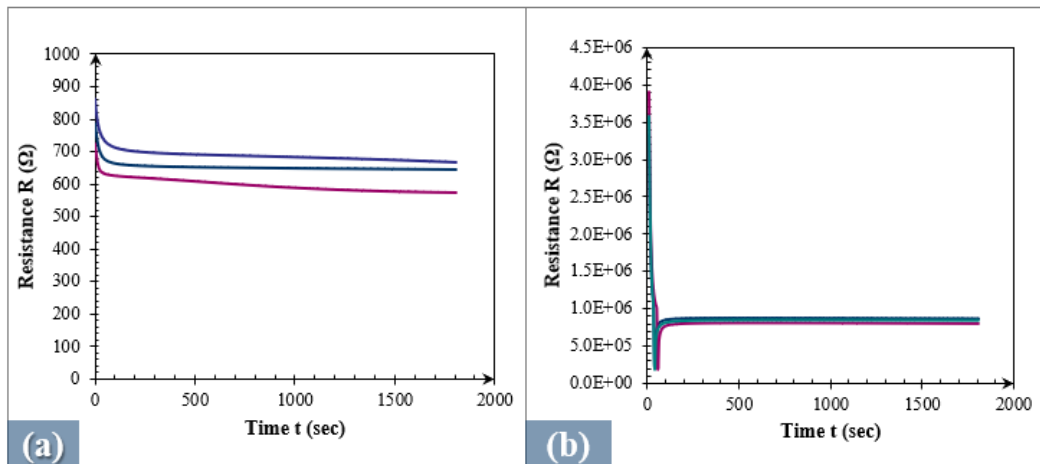


Figure 10.10: Change of electrical resistance due to polarization for (a) FP2 specimens and (b) DT2 specimens (repetition of three specimens)

10.4.2. Resistivity Measurements via CEPRA Technology

Resistivity measurements were also performed using Giatec iCOR®, a connectionless corrosion rate measurement device for reinforced concrete structures which can measure concrete resistivity, too. The measurement technique is based on a patented technology developed by Giatec Scientific Inc. and is known as Connectionless Electrical Pulse Response Analysis (CEPRA) (Ghods et al., 2016)). More specifically, in order to determine the low- frequency behavior of reinforced concrete systems, a narrow DC/AC pulse or a DC/AC step voltage is applied for a short period of time, while concurrently, the voltage of the system is recorded (© Giatec Scientific Inc., 2019). Since the concrete specimens did not contain reinforcement bars, the measurement of concrete resistivity was made according to the conventional Wenner Array method. The method is of the four-probe type, with equally distanced electrodes. The configuration of ICOR allows measurement of resistivity in x and y axis, simultaneously (Figure 10.11). The intrinsic electrical resistivity ρ can be calculated in terms of distance between electrodes, a , and the electrical resistance R , using Equation (10.1). The derivation of resistivity according to Wenner Array Method can be found in Appendix C.

$$\rho = 2\pi a R \quad (10.1)$$

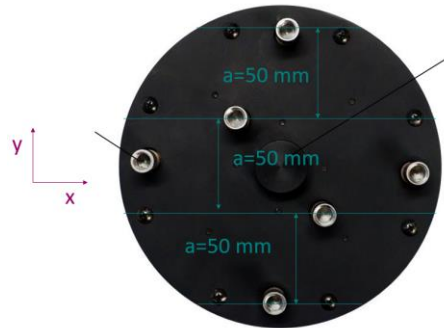


Figure 10.11: Giatec iCOR-Bottom view and electrode configuration

- *Experimental Setup and Procedure*

The experimental setup is illustrated in Figure 10.12. Initially, the sponge holders/electrodes should be filled with a conductive gel and then the completely moist contact sponges should be inserted in these tips. The tablet with iCOR application, which is connected to the device via Bluetooth technology, receives, analyzes, and stores the measured values, while it can also post-process test data and produce graphs and contour plots for concrete resistivity in real-time. Before

the measurement the user is asked to input parameters such as duration and direction of the measurement. For this study, simultaneous measurement in X and Y direction and a total duration of 12 seconds were chosen. An example of a measurement is shown in Figure 10.13. For each measurement, a fitted curve of the voltage measurement over time is performed and further employed for determination of concrete resistivity.

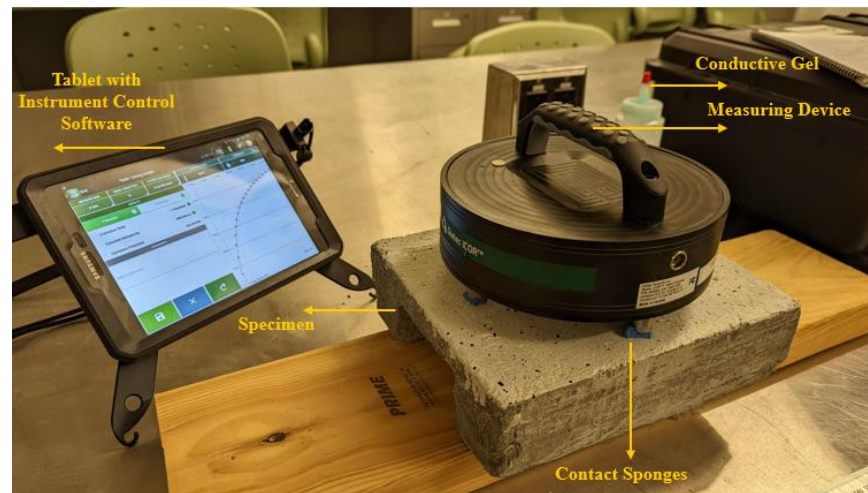


Figure 10.12: Experimental setup for concrete resistivity test via CEPRA Technology

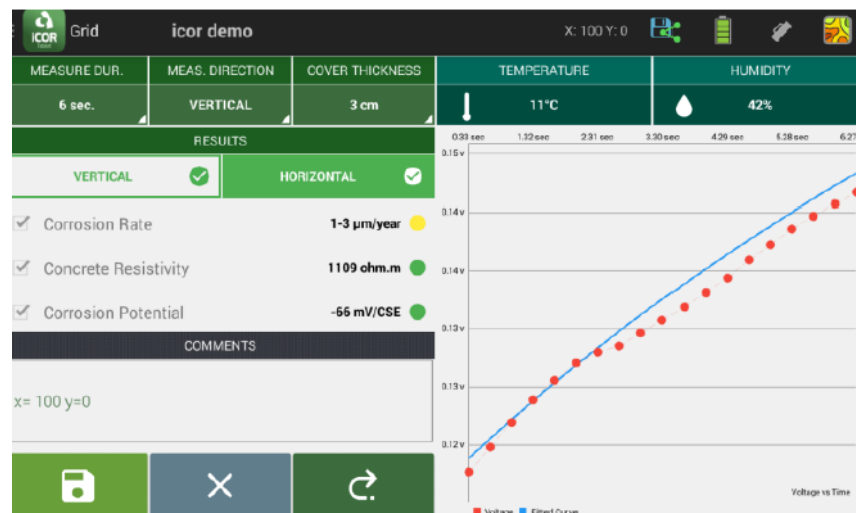


Figure 10.13: Example of measurement via CEPRA technology with iCOR®

- *Initial Resistivity, Effect of Fiber Distribution and Humidity*

For every OPB specimen, multiple measurements on the center and along the length of the specimens were taken without external mechanical load as shown in Figure 10.14. The center was chosen as the reference point for the tests with applied external load, while the measurements along

the length and in the direction of casting were performed to examine the effect of fiber distribution and boundary conditions on resistivity. The distance between each point in the case of measurement along the length was selected to be 10% of the length in the x direction (Figure 10.14(b)). Due to geometry limitations in the vertical direction, the measurements were performed in the midspan of the vertical length (L_y). Finally, since resistivity is highly dependable on humidity, 5 different humidity levels were studied, with intervals of 5% between 50-70% humidity for center resistivity for one specimen of the three.

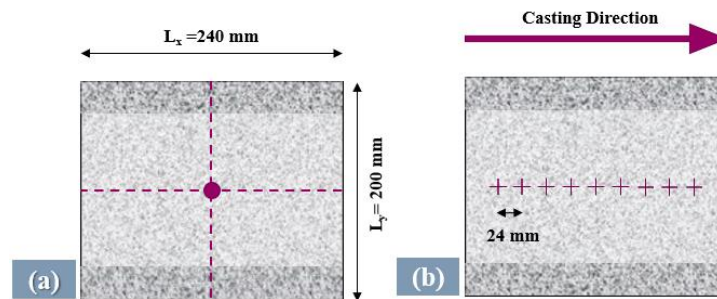


Figure 10.14: Wireless resistivity measurements without external load (a): in the center and (b) along the length

For the center resistivity nine measurements were taken at the center of each specimen and the average results are summarized in Table 10.8. The temperature and humidity were kept constant at 24 °C and 60%, respectively. The resistivity in Y direction is higher than in X, and this can be explained by the casting method which was flow from one end parallel to the X direction. In that way, there is higher fiber alignment in the direction of casting and thus lower resistivity. It is interesting to note that due to a formwork error, specimen OPB1 had a thickness of 30 mm instead of 35 mm and this is reflected to the ratio between directional resistivities. The smaller thickness imposed a wall effect leading to a more favorable fiber distribution in the X direction.

Table 10.8: Resistivity measurements in the center of OPB specimens

Sample	Resistivity in X ρ_x ($\Omega.m$)	Resistivity in Y ρ_y ($\Omega.m$)	ρ_y / ρ_x
OPB1	5858	7179	1.23
OPB2	6371	6457	1.01
OPB3	4841	5186	1.07

The fiber distribution effect is further investigated in case of resistivity measurements along the dimension parallel to casting. The temperature was 23 °C, while the humidity was 55%. Figure 10.15 illustrates the resistivity measurements in both directions along the horizontal dimension. For all specimens, moving farther from the casting point is associated with a decrease in resistivity

in the horizontal direction. At locations close to the boundary, both directional resistivities were increased with the value in the horizontal direction surpassing the value in the vertical. The latter can be explained by the wall effect which makes fibers to rotate about 90 degrees relatively to the flow. This agrees with fiber distribution visualization models examined in Chapter 4.

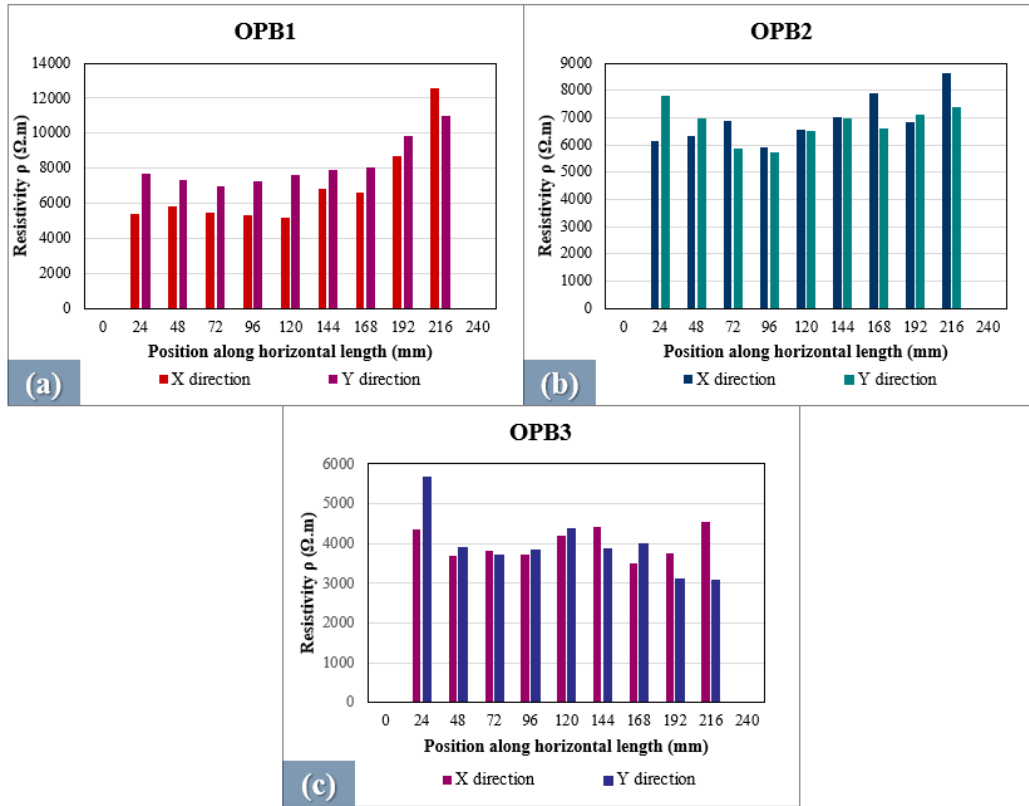


Figure 10.15: Effect of fiber distribution on resistivity for OPB specimens

Finally, the effect of humidity on resistivity is illustrated in Figure 10.16. As expected, the increase in humidity causes resistivity reduction with a more prominent effect after 65%.

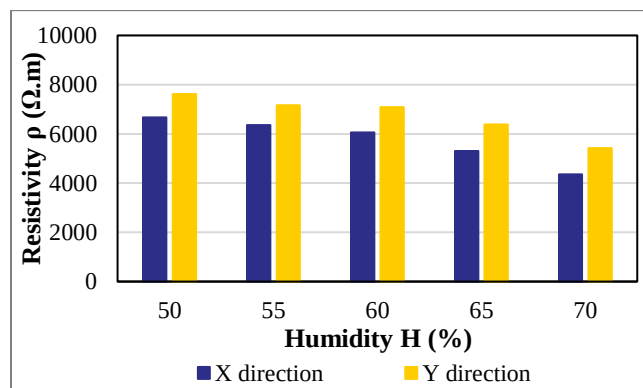


Figure 10.16: Effect of humidity on resistivity of OPB specimens

10.5. Self-Sensing Performance

For the assessment of self-sensing performance in tension three different mechanical tests were performed with real-time measurement of electrical resistance. Those tests include monotonic direct tension, monotonic pure bending and monotonic out of plane bending until failure. For direct tension and pure bending tests, the self-sensing performance is assessed through fractional change in resistance, while for out of plane bending, fractional change of resistivity is used. The measured resistance can be used as FCR according to Equation (10.2).

$$FCR = \frac{\Delta\rho}{\rho_o} \approx \frac{\Delta R}{R_o} \quad (10.2)$$

Here, $\Delta\rho$, ρ_o , ΔR and R_o are the change in resistivity, initial resistivity, change in resistance and initial resistance measured by the iCOR® or multimeter, respectively.

10.5.1. Direct Tension

DT2 specimens were tested in direct tension in the same manner with DT1 specimens for determination of tensile strength. The exact same procedure was followed in terms of preparation of specimens and testing.

- *Experimental Setup*

Figure 10.17 shows the test set-up for measurement of electrical resistance of the nanoengineered UHPFRC during testing in direct tension. The universal testing machine (UTM) was employed with a loading rate of 0.15 mm/min, while the tensile elongation was obtained by virtual extensometers available in DIC analysis. For DIC analysis, the front face of the specimen was speckled while a digital camera was taking pictures every 6 seconds, (Figure 10.17(a)). The electrical resistance of the specimen under tension was measured using a digital multimeter via the four-probe method. The data from the multimeter were collected through an instrument control software (Figure 10.17(b)) which collected the data at the same frequency as the camera. Prior to testing, the electrical resistance was measured for 1 hr to ensure the elimination of polarization.

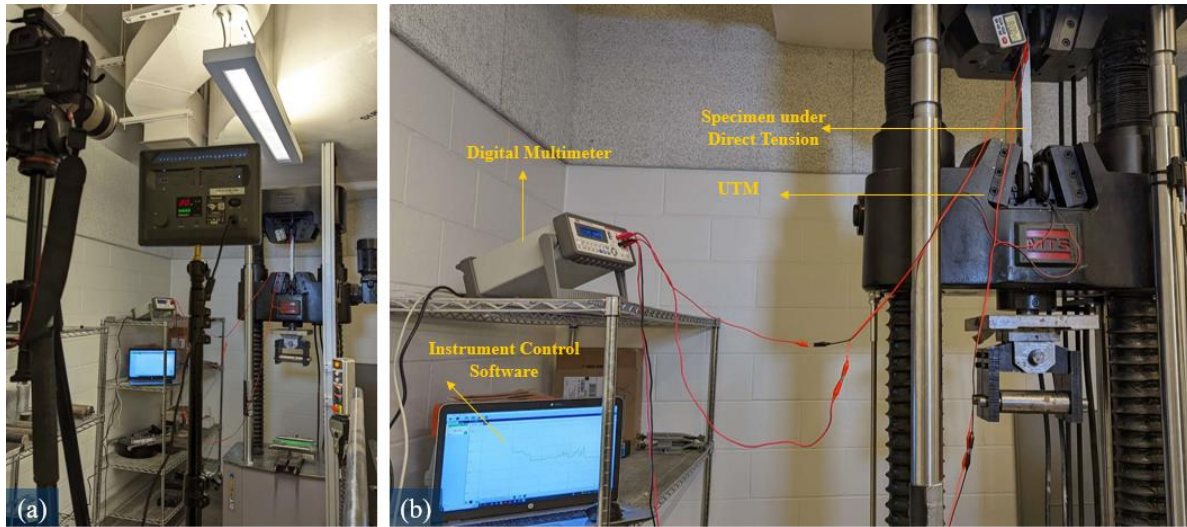


Figure 10.17: (a) General view of the set-up for electromechanical testing (b) Instrumentation used for self-sensing performance under direct tension

- *Comparative Tensile Stress- and FCR-axial Displacement Behaviour*

Figure 10.18 illustrates the tensile stress- and FCR- axial displacement of each specimen. To determine the strain, the tensile elongation obtained by virtual extensometers of DIC was divided by the gauge length of 100 mm. However, DIC fails to capture properly the strains in the elastic region and that is why the ascending branch is not prominent. When the stress increases while the specimen is in the elastic region, the resistance is decreasing. This can be explained by possible out of plane bending on the back face of the specimen which was not monitored. Another observation is related to the sharp changes in FCR at this stage, which are not related to sharp changes in stress. The latter might be linked to the fact that conductivity increases in the thickness due to Poisson's induced contraction, provided that the element has not yet cracked longitudinally. From DIC, the first cracking is observed to happen around 5 MPa. Note that for specimens DT2-A and DT2-B (Figure 18(a),(b)), the point of cracking in the stress curve coincides with the inflection point of FCR curve and the beginning of increase of the resistance. The increase of the resistance at the onset of cracking is due to the creation of discontinuities in the matrix and the disturbance of the conductive pathways created in the nanoengineered UHPFRC between the micro and nanofibers. Due to multiple cracking and the bridging of the cracks after the first cracking strength and till 60% post-peak, the FCR slope does not change considerably. Finally, specimen DT2-C (Figure 10.18(c)) other than the detection of localization, it did not demonstrate meaningful sensing characteristics, compared to the other specimens.

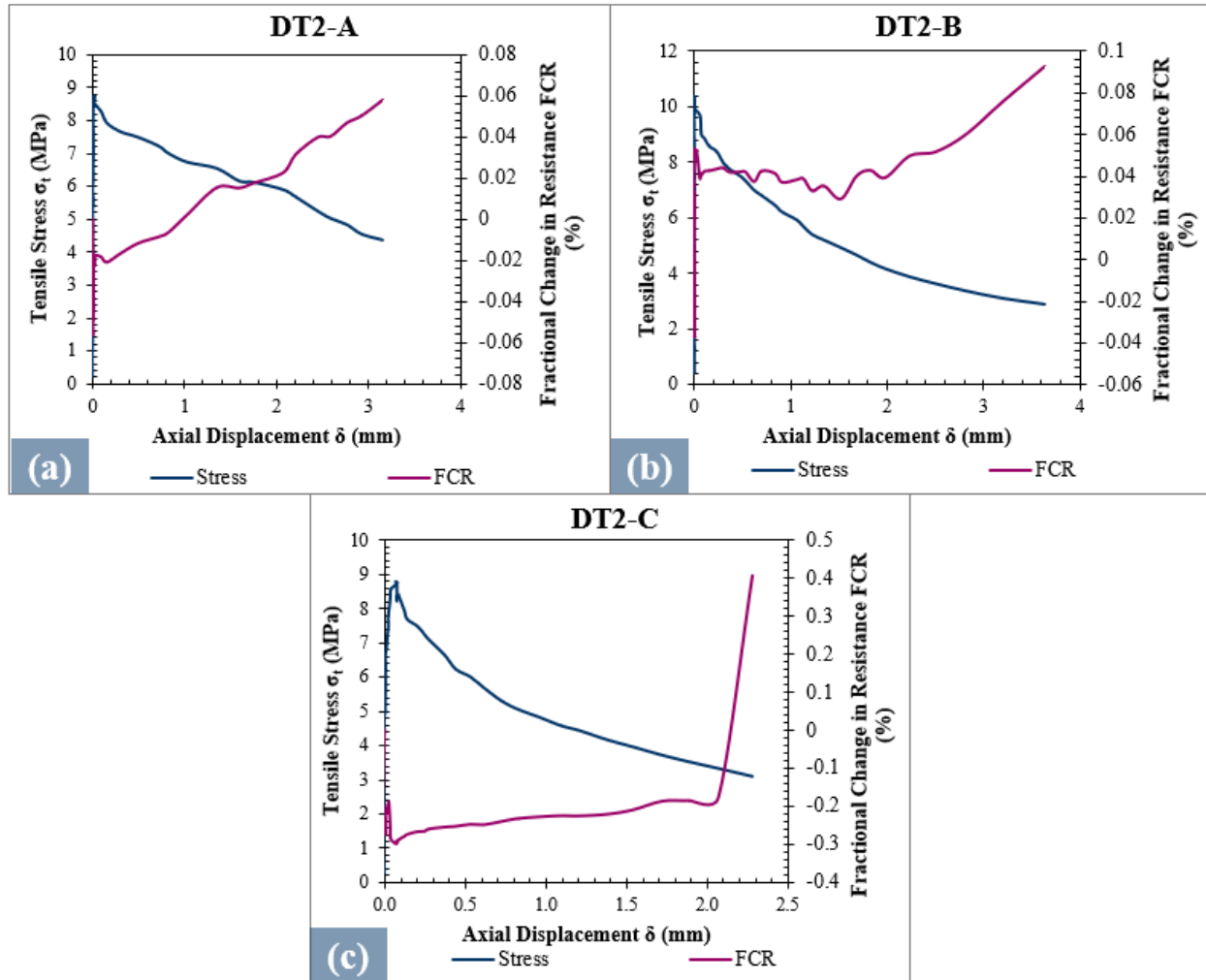


Figure 10.18: Tensile stress and FCR-axial displacement curves of nanoengineered UHPFRC for the three different DT2 specimens.

10.5.2. Pure Flexure

For the evaluation of self-sensing performance of the tension in pure bending, three FP2 specimens were tested according to the four-point bending test of ASTM C1609 following the same procedure described in Section 10.3.3.

- *Experimental Setup*

The experimental setup for the measurement of self-sensing capacities of the nanoengineered UHPFRC subjected to pure flexure is shown in Figure 10.19(a). Since the tension face is selected for monitoring, the specimen is rotated 180° from the casting side which had the embedded electrodes. The displacement of the midspan is measured via two LVDTs on both sides of the

specimen mounted on an aluminum frame. To avoid interference of the conductive loading system, or the instrumentation jig frame that holds the LVDTs, insulating tapes were placed at the connection lines (Figure 10.19(b)).

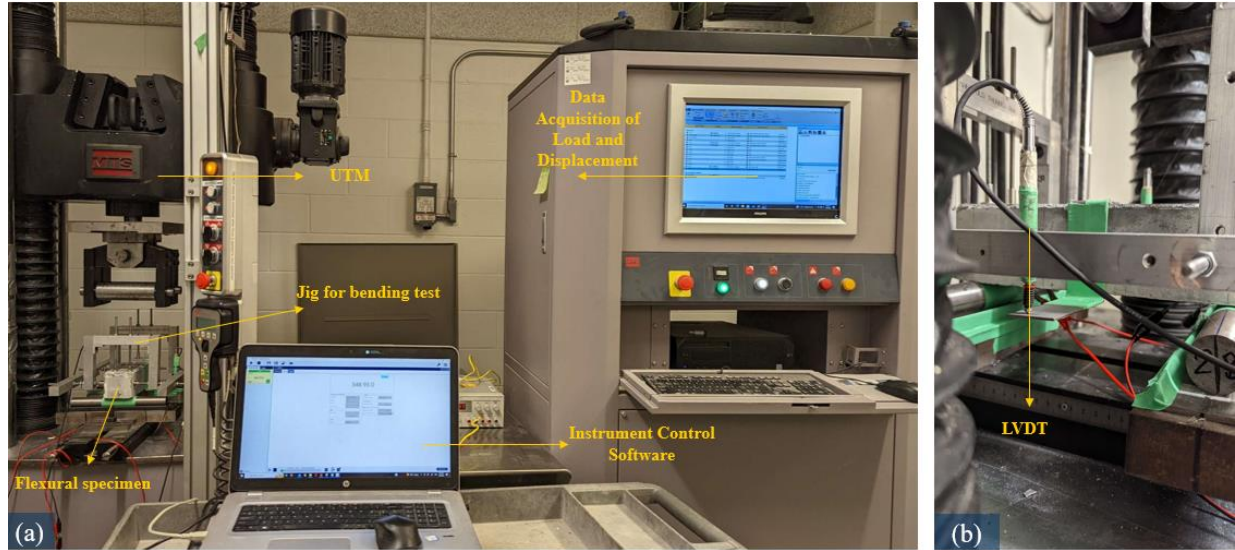


Figure 10.19: Experimental setup for four-point bending test (a) instrumentation ; and (b) flexural specimen with insulating tape at the points of contact with the loading system.

- *Comparative Flexural Load-Deflection and FCR-Deflection Behaviour*

The comparative flexural load-deflection and FCR-deflection behaviour of the FP2 prisms is presented in Figure 10.20. A similar trend with direct tension tests in terms of FCR is observed. In the elastic range up until cracking, the FCR decreases. The reduction could be explained by the depth of the electrodes which are above the neutral axis and therefore in the compression zone. Also, at this stage, the strains in the tensile face are not significant yet and thus the influence of compression dominates. In the end of linear elastic region, which can be associated with the onset of cracking, the FCR changes slope as the resistivity starts to increase drastically. The increase in FCR is more prominent at the descending branch for specimens FP2-A and FP2-C (Figure 10.20(a),(c)), while the jagged pattern in the flexural load curves which is associated with the pullout of fibers is also evident in the FCR curve. However, in case of FP2-B (Figure 10.20(b)) at post-peak the FCR is decreasing which does not agree with the expected response. The latter could be explained by the location of the crack, as it occurred between one input and output electrode close to the support (Figure 10.21).

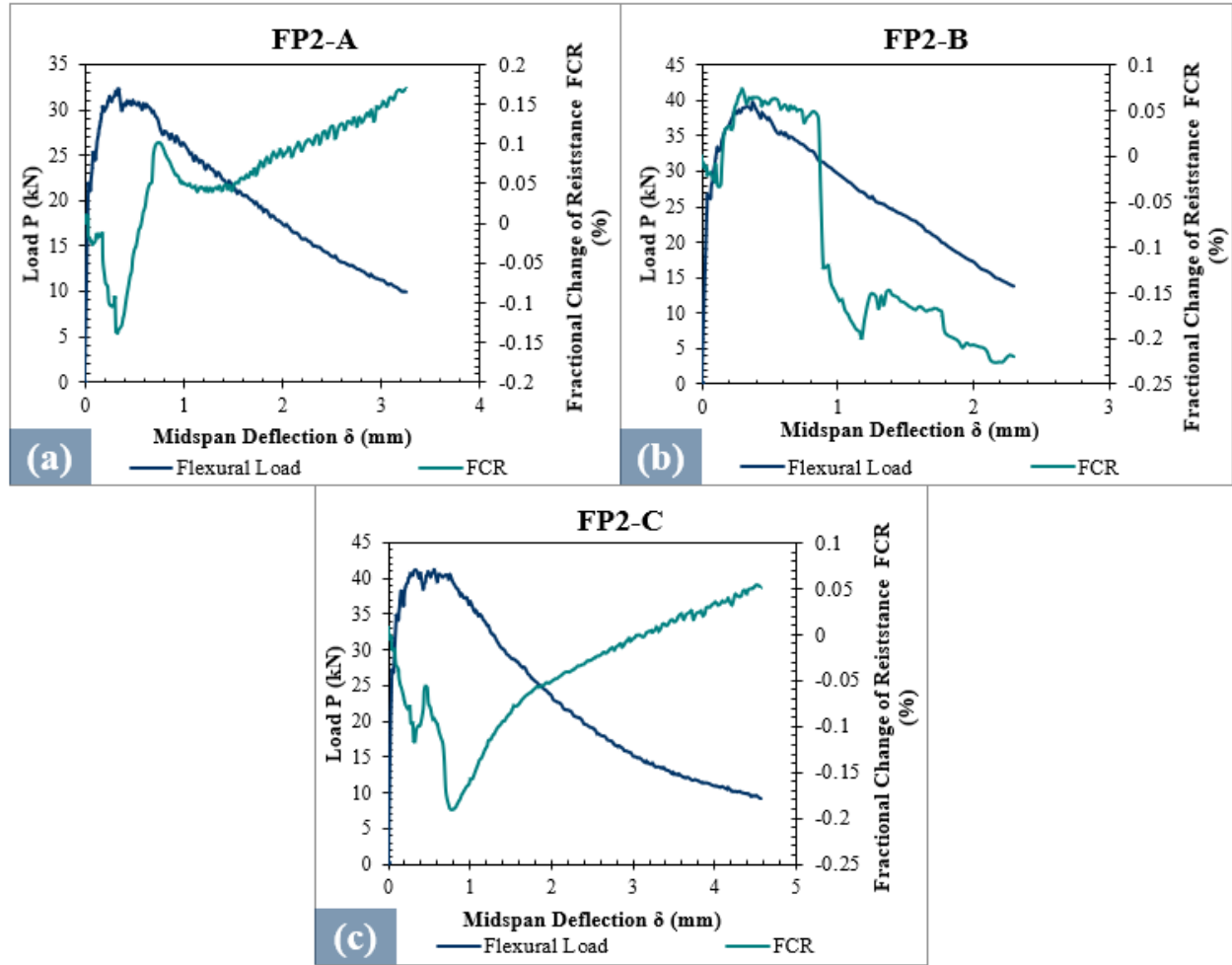


Figure 10.20: Comparative flexural load-deflection and FCR-deflection behaviour of FP2 specimens



Figure 10.21: Shear failure between an input and output electrode for specimen FP2-B

10.5.3. Out of Plane Bending

In order to assess the self-sensing performance of the wireless resistivity measuring device, out of plane bending test of a panel with a C cross section is proposed. The design of the cross-section

and type of test was performed based on limitations imposed by the electrode configuration of the measuring device and safety. The latter is crucial for real-time manual measurement of electrical properties of the externally placed device, under the presence of vertical load.

- *Experimental Setup and Procedure*

The experimental setup for the wireless sensing under out of plane bending is illustrated in Figure 10.22. The measurement is performed intermittently every 1 minute till the peak load. The device was set to measure both directional resistivities simultaneously, while the duration of the measurement was 12 seconds. Since resistivity is sensitive to temperature and humidity, same conditions were ensured for all specimens, namely 23 °C temperature and 50% relative humidity. DIC analysis was also employed to obtain the surface strain and deformation in both directions.

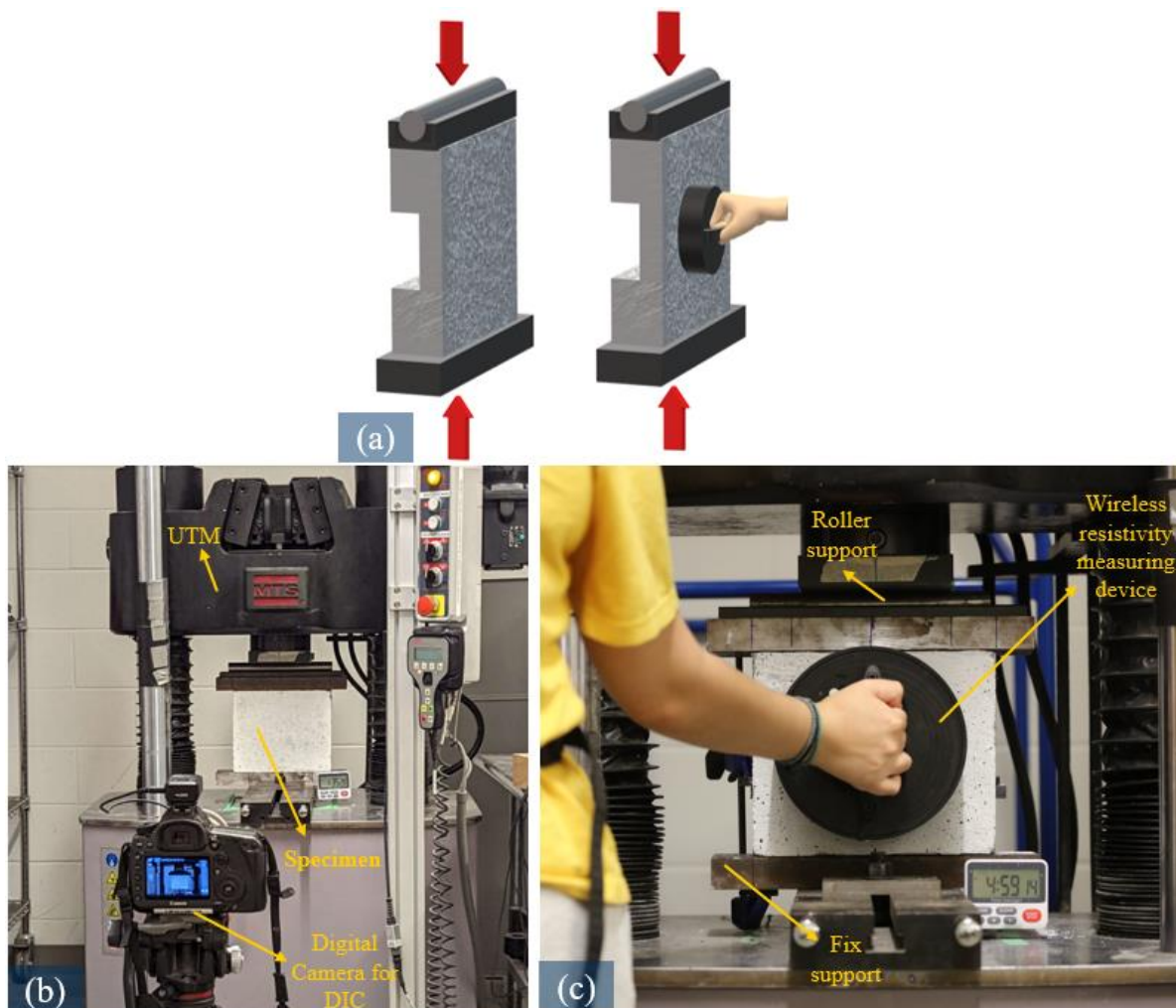


Figure 10.22: (a) Schematic illustration of the wireless electromechanical testing (b) instrumentation of the experimental setup (c) instant resistivity measurement via wireless device and support conditions.

In order to obtain the displacements in both directions and consequently the surface strain field, DIC analysis was performed. The displacements are found through Virtual Extensometers (VE) with their configuration as illustrated in Figure 10.23. All VE had the same gauge length equal to 150 mm, equal to the distance between outer electrodes in the wireless resistivity device. The average of VE1-3 and VE4-6 are used to determine the vertical and horizontal displacement, respectively.

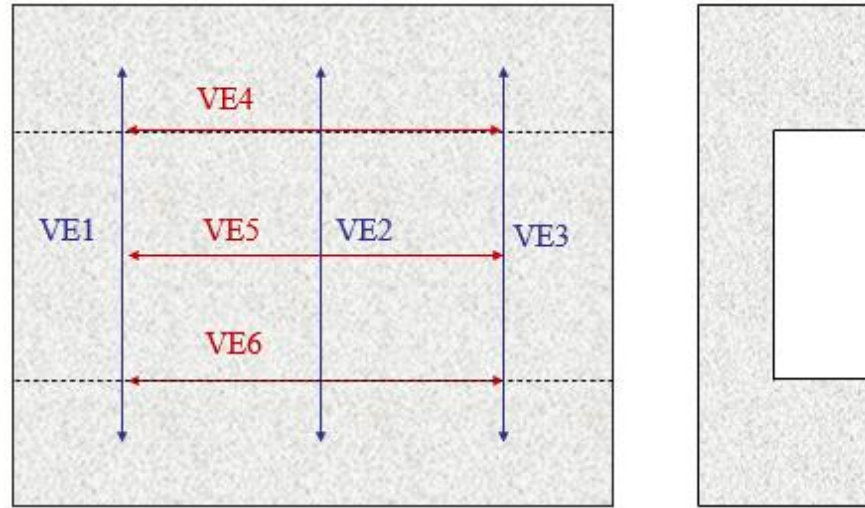


Figure 10.23: Configuration of VE for DIC Analysis

- *Comparative Load-Deflection and FCR-Deflection Behaviour and Correlation with DIC*

The comparative load-deflection and FCR-deflection curves for both directions are presented in Figure 10.24. In the vertical direction, the inflection points, and abrupt jumps of the FCR curve are related to changes of slope in the load curve. Upon reaching the ultimate load, FCR in the vertical direction drops dramatically and beyond that point the resistivity measurements are not meaningful anymore. In case of self-sensing in the horizontal directional, there are no consistent results either with the FCR curve of specimen OPB 2 to predict better the response, compared to the other two specimens.

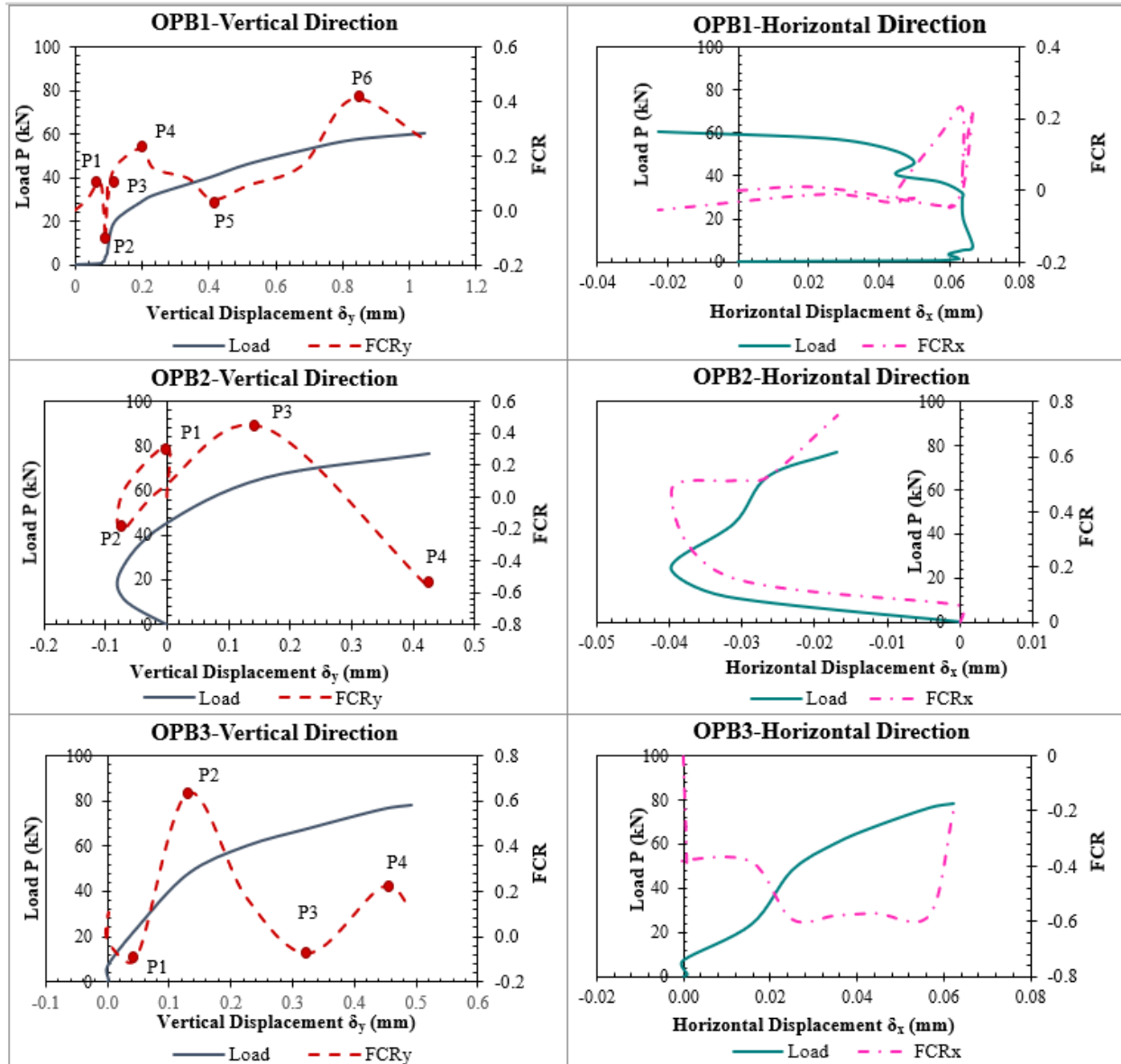


Figure 10.24: Comparative load-deflection and FCR-deflection behaviour in both directions for OPB specimens

The inflection points of FCR in the vertical direction can also be correlated with the surface strains obtained from DIC. Figures 10.25-10.27 represent the surface strains in the vertical direction at the inflection points of the corresponding FCR curve. For specimen OPB1 (Figure 10.25), P4 represents the onset of cracking, while the compressive strains near the supports at P5 could affect the resistivity, reducing the FCR. After that, at P6, multitracking occurs which is linked to the increase of the resistance. In case of OPB2 (Figure 10.26), the presence of compressive strains at the vicinity of the roller support could lower the resistivity for P2. However, the highest FCR at P3 predicts again the onset of cracking, with P4 to be an unreliable result as the failure occurs.

Finally, for OPB3 (Figure 10.27), the first cracking is associated with the highest positive FCR, while after the cracking, the rest of the specimen unloads and resistance is affected by compressive stresses at the vicinity of the roller (P3).

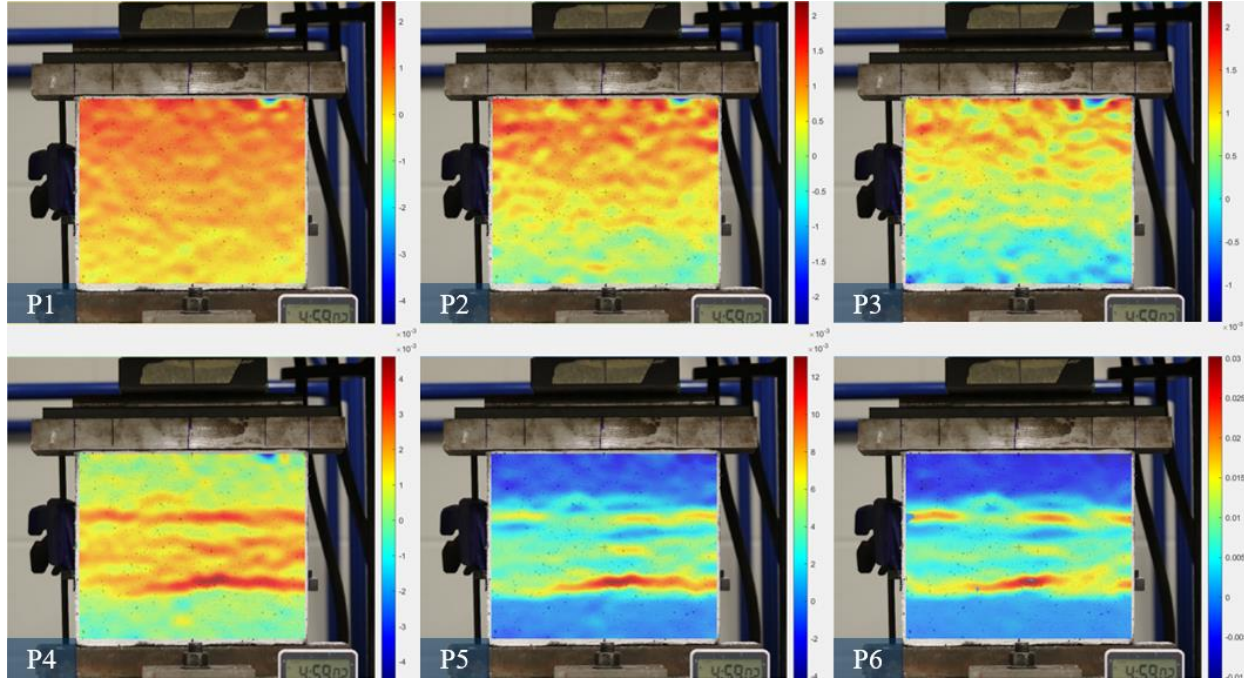


Figure 10.25: Surface ε_y strain fields for specimen OPB1 at the inflection points of FCR curve.

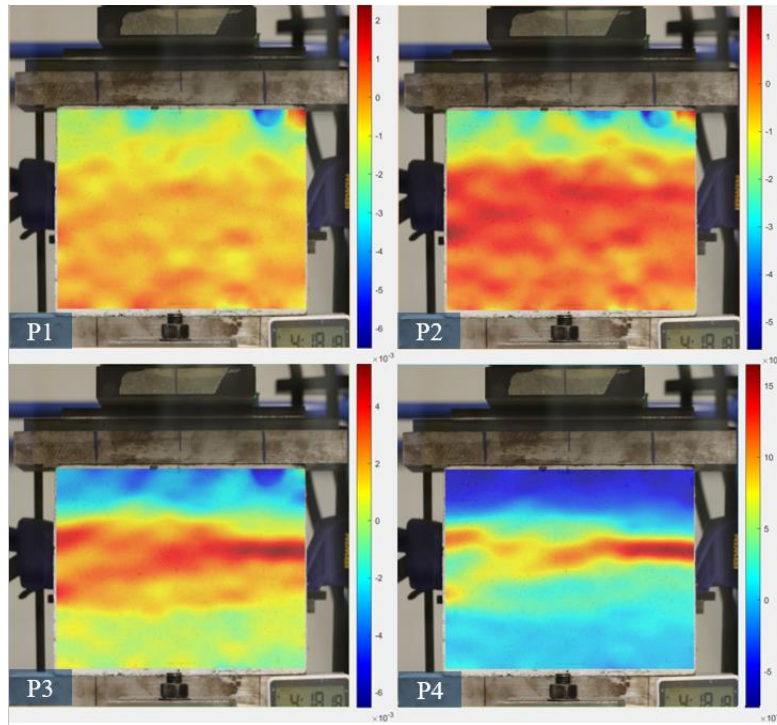


Figure 10.26: Surface ε_y strain fields for specimen OPB2 at the inflection points of FCR curve.

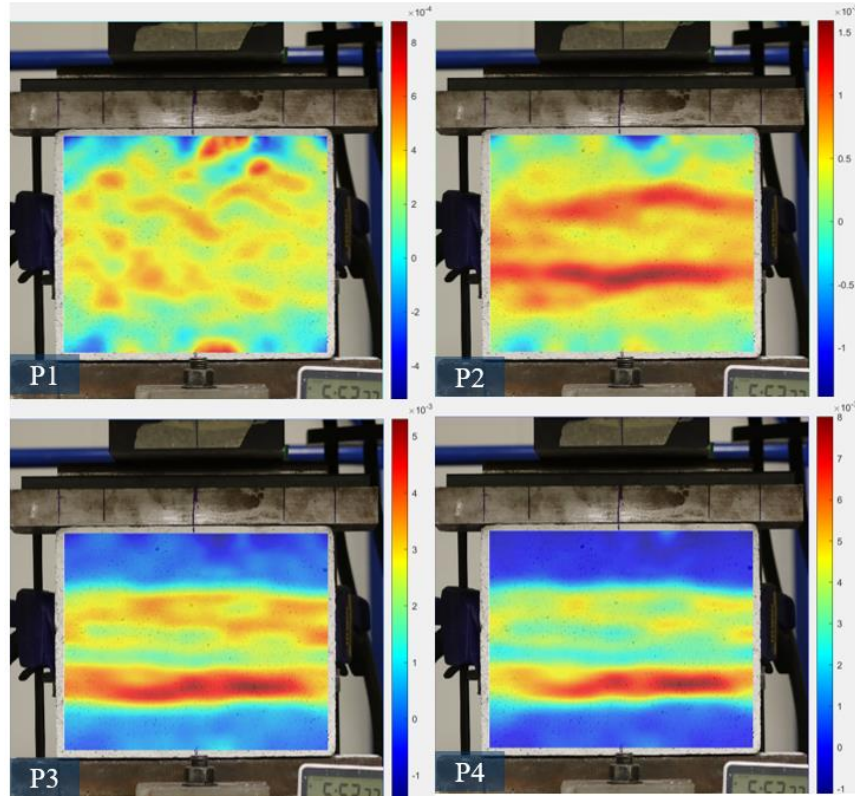


Figure 10.27: Surface ε_y strain fields for specimen OPB3 at the inflection points of FCR curve.

10.6. Conclusions

The proprietary nanoengineered UHPFRC of this study was subjected to mechanical and electromechanical testing in order to assess mechanical properties and mainly its self-sensing capabilities. The material exhibits a compressive strength of 148 MPa at 28 days, and an ultimate tensile strength of 8.7 MPa with a tension hardening ratio of 1.6. The presence of CNFs induced a highly thixotropic behaviour in the fresh state while also enhancing the first peak strength in flexural tests, compared to other UHPFRC of same compressive and tensile strength. Regarding the self-sensing performance of this investigation the following could be concluded:

- The micro-fiber distribution, which is highly dependent on casting method and specimen geometry has a significant effect on the electrical properties of the material. Namely, specimens with better fiber alignment had less polarization time drift. Moreover, the effect was also evident in case of directional resistivity measurements, as the resistivity in the direction of casting was systematically higher than the other direction.

- The wireless resistivity device can also be used as a nondestructive method for assessment of fiber distribution.
- The FCR curve could be used to predict the onset of cracking, as in all the specimens a sharp increase in the resistance was observed.
- The FCR curve is not sensitive in the elastic region.
- The proposed wireless sensing approach was able to capture the first cracking. However, the nature of the test and the limitations of the specimen geometry perplexed the interpretation of results. Further research is needed.

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Chapter 11: Conclusions and Future Work

11.1. Summary

The objective of this dissertation was the development and characterization of sustainable, high performance and multifunctional building materials. Firstly, a variety of industrial by-products and natural raw materials were characterized physically and chemically with the objective to use them in the development of a novel sustainable and eco-friendly structural concrete. Then, through exploitation of geopolymer chemistry and particle packing theory, a cementless geopolymer matrix for THFRC that eliminates CO₂ emissions by 60-90% was developed and fully characterized in accordance with North American Standards in terms of mechanical, physical and durability properties. The development of embedded reinforcement in the proposed material used as a matrix in THFRC was investigated using direct tension pullout tests; furthermore, the behavior of the material under complex stress states was studied experimentally and analytically using push-off tests. Besides geopolymer THFRC, emphasis was given more generally to other strain hardening fiber reinforced concretes in order to resolve some of the knowledge gaps in the field. These included the study of fiber orientation and rheological properties and their effects on the mechanical behaviour of tension hardening materials using stereological mathematical models and an innovative transparent model concrete. The latter facilitated the assessment of the effect of reinforcement, fiber volume, viscosity, boundary conditions and casting methods on mechanical properties of tension hardening concrete materials. Most importantly, this thesis contributed towards the solution of the conundrum of straightforward tensile characterization of the material. To this end, a novel simplified indirect tensile test was developed and validated experimentally and numerically. The latter was accomplished through advanced computational simulations and experimental validation and refinement of the test. NLFEA were also performed to assess current inverse analysis techniques for tensile characterization of UHPFRCs; a constitutive model was adapted in the context of explicit time integration in order to replicate the response of THFRC's after strain localization using an advanced computational platform. Finally, a multifunctional nanoengineered hybrid UHPFRC was studied in terms of electrical, mechanical, and self-sensing properties by employing a novel wireless measuring technique.

11.2. Contributions

11.2.1. Development and Characterization of THFRGC in terms of Mechanical, Durability Performance and Documentation of Structural Behaviour

The geopolymer THFRC developed in this study comprises in its entirety industrial by-products and natural weathered minerals and it represents an original contribution to the Concrete Materials field. The development of a such material will have a great impact on the environment since a considerable reduction of carbon footprint would be achieved whereas utilization of industrial wastes instead of harmful disposal would be attained. The variety of aluminosilicate raw materials tested as possible binders in this thesis is promising as it is not limited to the conventional raw materials and industrial wastes used in most of the studies available in the literature. The raw minerals are a product of Ontario and are therefore adapted to local sourcing of materials. No addition of cement was necessary whereas flowability and stability of the mix was attained for a satisfactory workability time avoiding the perils of flash setting.

The developed material was then characterized in terms of mechanical, physical and durability properties according to North American Standards designed for OPC concretes. This experimental campaign that is based on the tests performed to obtain a material identity card in North America, could be considered as a stepping point in the direction of developing and implementing geopolymer THFRCs in North American concrete industry.

Finally, the structural behaviour investigation regarding bond and development of reinforcing bars in geopolymer THFRC and push-off tests under multiaxial stress states performed for this dissertation further promote its application as a structural material. Bond strength was characterized and was found to be consistent in terms of parametric dependency and magnitude with other THFRCs, whereas the entire bond-slip relationship of the geopolymer THFRC was obtained using a direct tension pullout test providing an essential link for modelling and calculating the response of complete structural components comprising THFRGC. The tests under multiaxial stress states enabled the derivation of failure criteria for this material, both for simple design calculations of shear strength, as well as a component of a thorough plasticity model formulation with explicit time integration. The latter was shown to successfully perform in nonlinear modeling studies for components that experience crack formation and subsequent severe strain localization in tension.

11.2.2. Study of Fiber Orientation in THFRCs Using a Transparent Model Concrete Fluid

A technique using translucent fluid model concrete was used for visualization of fiber orientation in FRCs with density of fiber that is commensurate to the concrete developed in the present study. The study expanded the investigated parameters that affect the orientation of fibers, wall effects on orientation, as the effects of reinforcement, rheology, casting method and fiber content. A collection of data was created from several saw-cut specimens of various THFRCs tested previously, with the objective to check against the data the available models for the number of fibers crossing an arbitrary cross section. Great dispersion and discord was found between the published models and the experimental data. To mitigate this uncertainty, an expression for the fiber effectiveness coefficient was developed and calibrated from the data. This expression forms the cornerstone for explicit determination of the tensile strength of the material as well as the internal confinement that may be relied upon as a result of the action of fibers.

11.2.3. Development of a Novel Indirect Tension Test for THFRCs

A novel tensile test for THFRCs was developed based on strut and tie model that combines the convenience of a splitting tensile test with the accuracy of a direct tension test using conventional compression testing equipment. The test also allows the determination of the full response of the material. The test was validated using advanced nonlinear finite element modeling according to the explicit time integration procedure discussed earlier, so as the design the performance of the specimen form throughout the range of strains developed in the tests; it was also validated using a large collection of tests conducted in house with the objective to refine the design, but also against other standardized tension tests for THFRC in order to verify its convergence.

11.2.4. Experimental Assessment of Self-Sensing Capabilities of Multifunctional Nanoengineered THFRCs Using Wireless Technologies

To date, the assessment of self-sensing performance has been limited to conventional methods with embedded or attached electrodes to measure the electromechanical properties. In this study a proprietary nanoengineered THFRC mix was used to test a wireless approach for assessing the self-sensing ability. The wireless tests were compared to the conventional bulk resistivity

methods. This was a novel exploratory step towards the application of wireless approaches to self-sensing of THFRC. It was shown that the method reliably detects the onset of cracking of THFRC.

11.3. Conclusions

Some of the most important findings:

- Full characterization of raw materials in terms of physical and chemical properties is needed for the development of geopolymer concretes. Optimization should not only be performed in terms of particle packing density but also and most importantly chemical composition, for a stable microstructure. Mineral wastes from the quarry industry in Ontario was tested and shown to be ideally reactive for geopolymerization. The entire novel material developed in the study comprises entirely waste products thereby minimizing the environmental impact.
- The chemical admixtures developed for OPC concretes are not ideal for geopolymer concretes, as they are specifically designed targeting hydration mechanism which is different from geopolymerization.
- The developed geopolymer THFRC is lightweight, self-compacting but also shows a thixotropic nature ideal for additive manufacturing. In terms of mechanical properties, the material shows a compressive strength of 50 MPa and a tensile strength of 8 MPa, while being highly ductile. Due to the passive confinement provided by the fibers, the average axial deformation at 50% post-peak in compression is 0.028 while the lateral is 0.038. For tension the material is considered to be tension hardening according to CSA S6 Annex 8.1 as the hardening ratio is 1.13, and the ultimate tensile strain 0.002. However, the lower modulus of elasticity compared to OPC concretes of the same strength implies that further research is needed towards further optimization.
- The novel developed geopolymer THFRC demonstrated low chloride ion penetrability, satisfying freeze-thaw and deicing salt-scaling resistance according to North American Standards. Meanwhile it outperformed UHPFRCs in case of abrasion and sulphate resistance. However, higher drying shrinkage and water absorption was observed, and these are the directions for further optimization.

- A translucent fluid model concrete with rheological properties manipulated to simulate THFRCs can be used to illustrate efficiently the effect of casting method, fiber content, presence of reinforcement and framework size on fiber orientation in THFRCs.
- The available models for fiber orientation and effectiveness factors do not match well the experimental evidence, although this parameter is cornerstone in practical design guidelines for THFRC.
- Regarding bond-slip behaviour, the confinement effect of the fibers changed the failure mode from splitting to a hybrid splitting-pullout. Bond strengths attained are as high as 1.34 to $1.88\sqrt{f_c}$ (depending on cover thickness), in the same range obtained from similar tests performed in UHPFRC.
- The presence of the steel fibers enhances the shear strength of the geopolymer concrete by acting as a partial replacement of transverse reinforcement while they also provide passive internal confinement through the mass of the material with magnitude equal to 0.78 of the tensile strength in case of a 2-D fiber orientation.
- Constitutive models used in Non-Linear Finite Element Formulations were able to simulate accurately the response of four-point bending tests.
- Quasi-static explicit dynamics analysis can be used in models with complex material behaviour such as UHPFRC and provide accurate results even for the stage beyond strain localization in tension.
- From the numerical simulations it was observed that the constitutive law in tension of UHPFRC obtained from inverse analysis procedures described in CSA S6 Annex 8.1 is not an intrinsic property.
- The novel indirect tensile test proposed in this study shows promise as an alternative to current tensile testing regimes for both characterization and quality control. It only requires a conventional compression testing machine and standard training of the technical staff conducting the test. Moreover, the full response of the material can be easily determined, while there are no uncertainties of the actual stress state in the cross section of interest due to the geometry and loading conditions of the specimen.
- The electrical properties and thus self-sensing ability of nanoengineered UHPFRC are strongly influenced by fiber orientation. FCR which is an established quantity to measure

sensitivity was found to predict the onset of cracking, while it was observed that it was not sensitive enough in the elastic region of a direct tension test.

- The wireless resistivity device used in this study can be used as a non-destructive method for assessment of fiber orientation, but also for assessment of self-sensing performance. It was found that the method was able to predict accurately the onset of cracking. Further research is needed to address issues regarding specimen geometry.

11.5. Areas of Future Research

While sustainable high performance and multifunctional building materials appear to be excellent candidates for decarbonization of the concrete industry, continued research is needed for innovation and further application. In this regard, the longer ranging research plans include the following:

1. The geopolymer THFRC developed in this study, due to its thixotropic nature is a promising material for exploring the next frontier of additive manufacturing.
2. Geopolymer concrete is an ideal material type for development of exoplanetary applications because of the similarity of Martian and Lunar Regoliths to the reactive precursors needed for the technology. Further investigation of innovative materials such as extraterrestrial soil simulants as alternative precursors is therefore of great interest in space exploration. .
3. An extensive campaign of accelerated tests of exposure of elements to aggressive conditions (fire, corrosion, and carbonation) would be needed for a thorough characterization of the developed geopolymer THFRC.
4. Further research and patenting after optimization of a standardized formwork fabrication of the proposed modified splitting test is needed in order to promote its use in codes.
5. Assessment of self-sensing performance of multifunctional nanoengineered UHPFRC in extreme environmental conditions (fire, freeze thaw and corrosion) in order to better comprehend the factors that affect the sensitivity of the smart material.
6. Formulation of advanced computational multi-level and multiphysics simulations to study microstructure, rheology, piezoresistivity and constitutive behaviour of UHPFRC materials.

Appendix A: Geopolymer Concrete Trial Mixes

A.1. Characterization of Mineral Powders

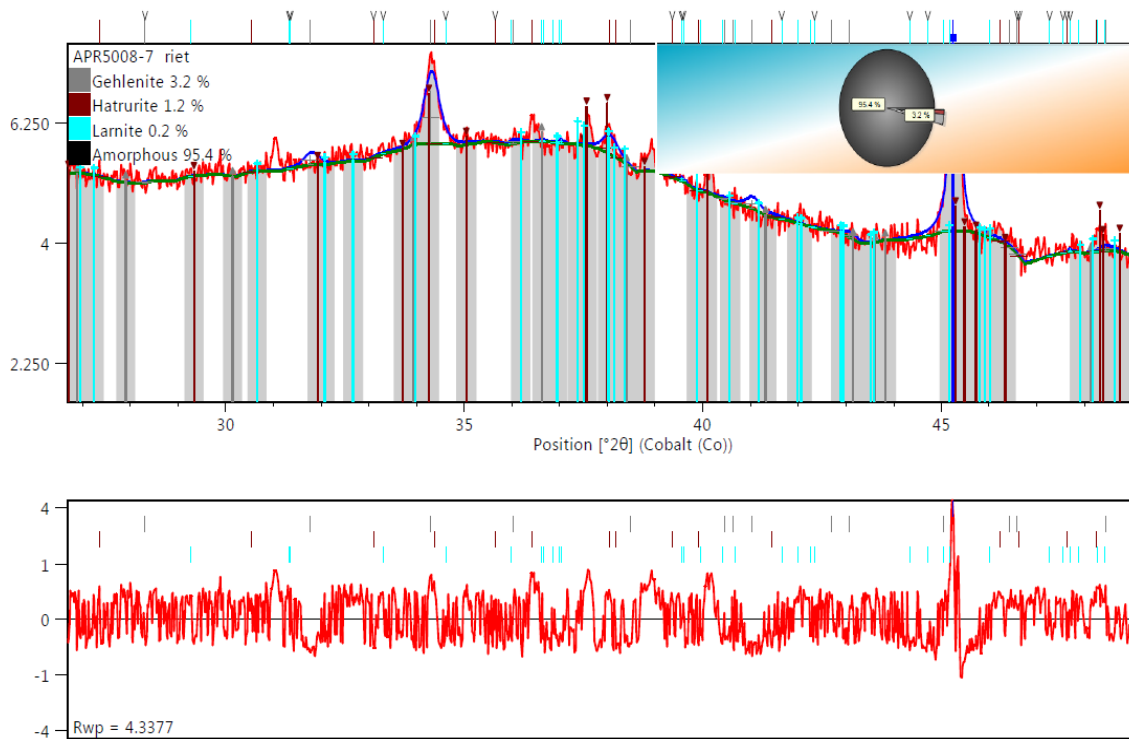


Figure A.1: XRD of GGBFS

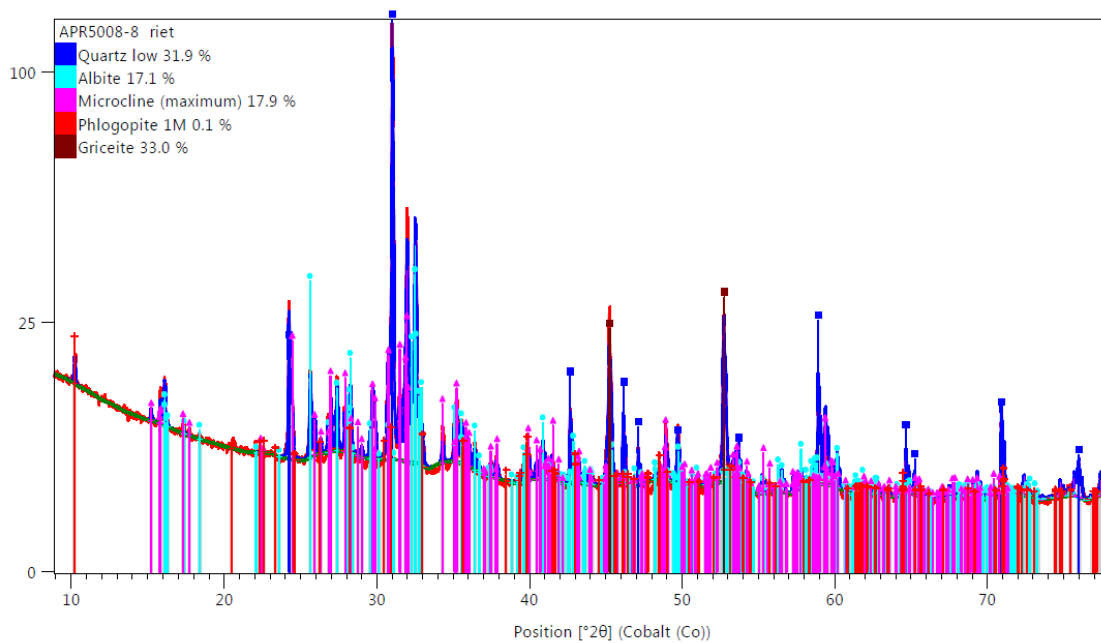


Figure A.2: XRD of FP

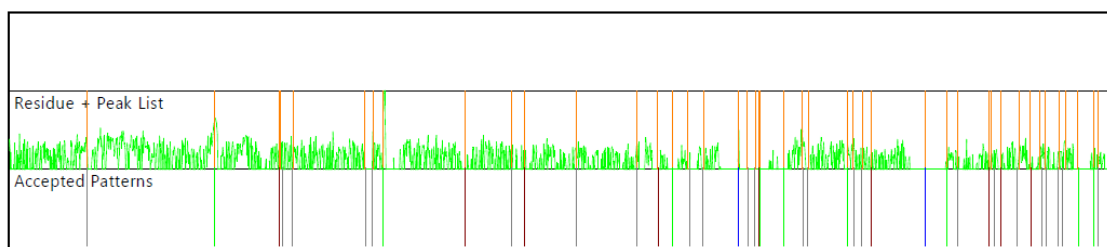
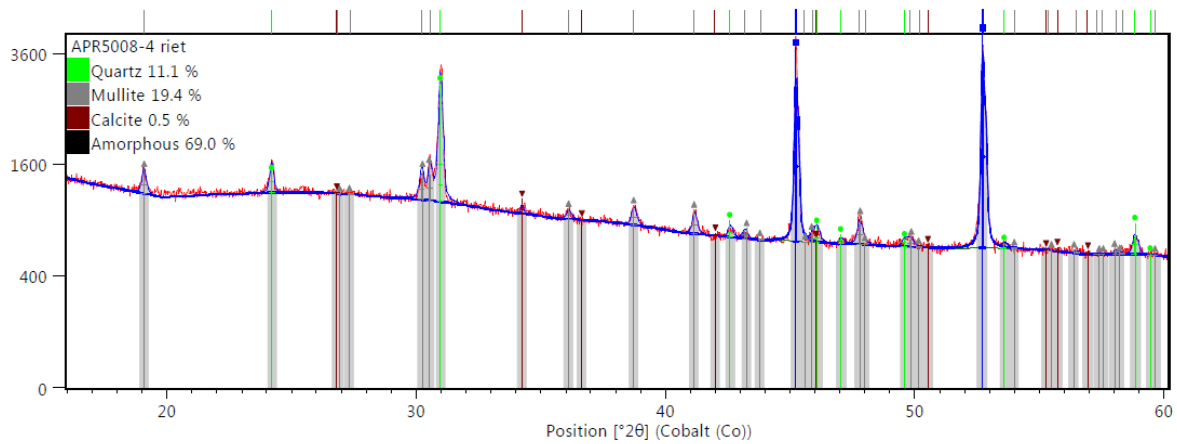


Figure A.3: XRD of FA-F

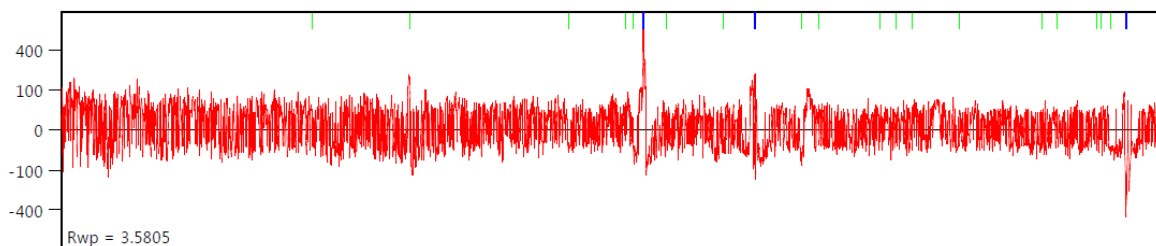
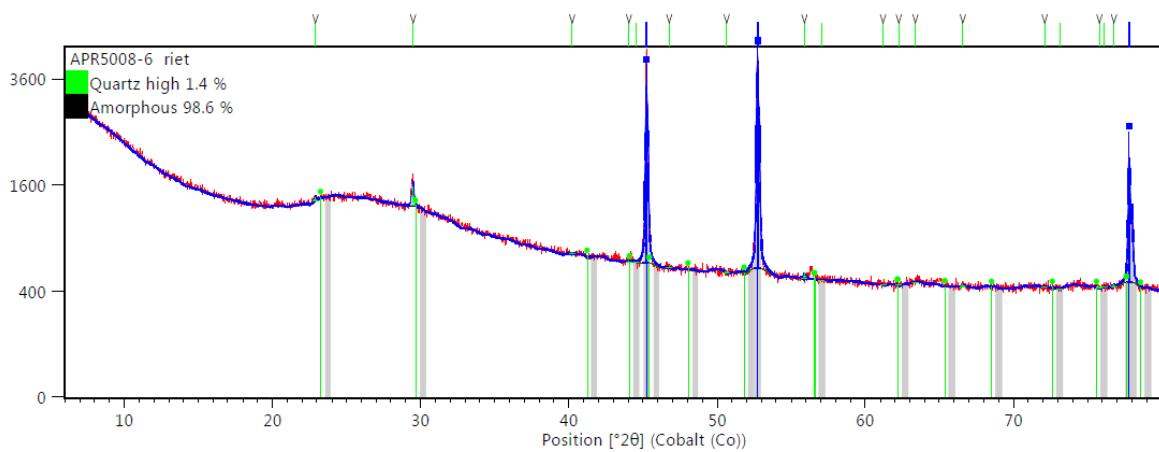


Figure A.4: XRD of MK-750

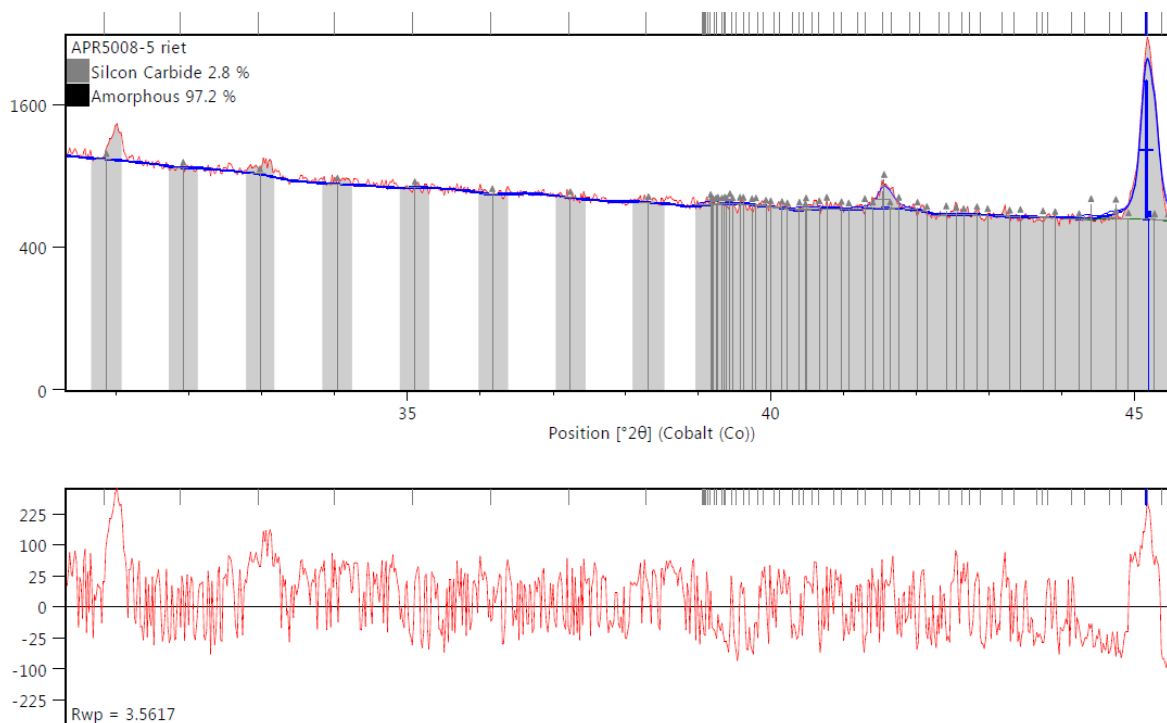


Figure A.5: XRD of DSF

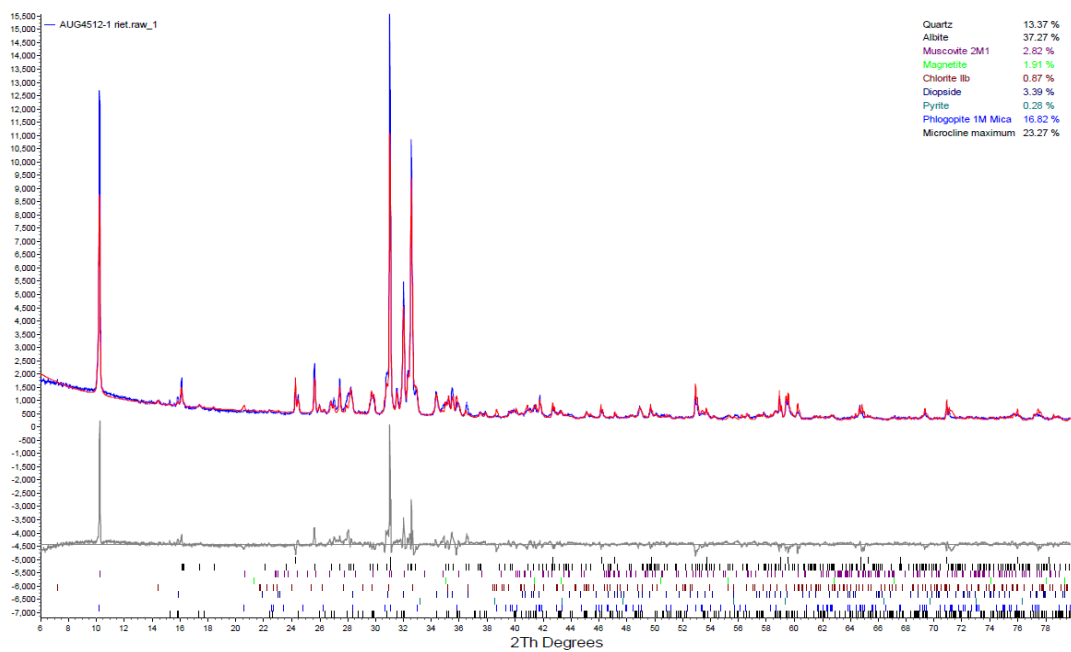


Figure A.6: XRD of GS

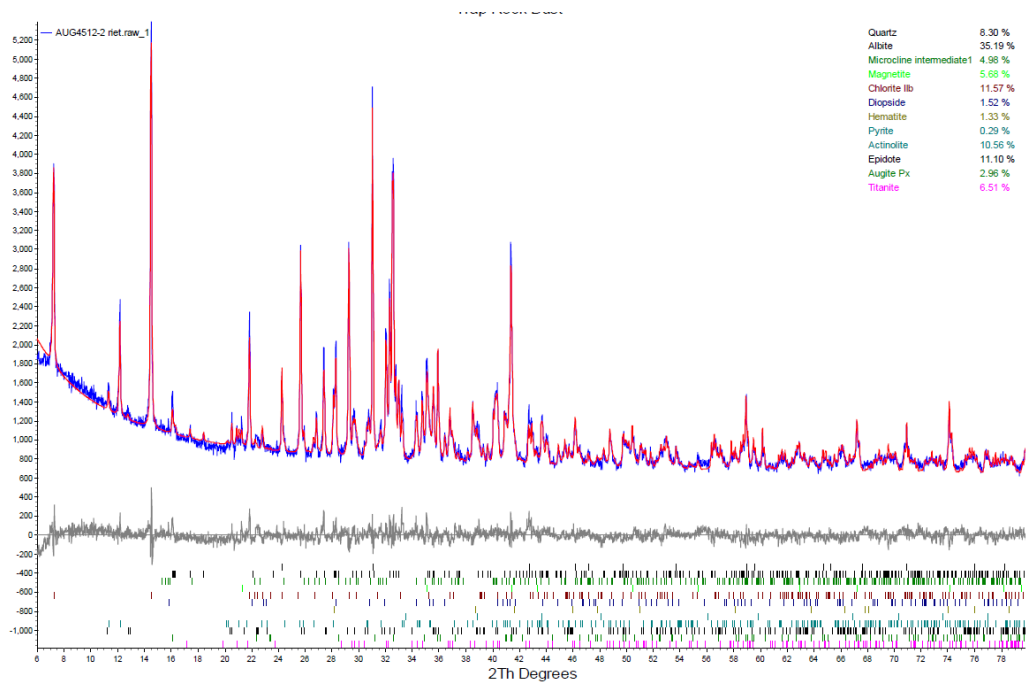


Figure A.7: XRD of TRD

A.2. Trial Mixes for Development of Geopolymer Concrete Matrix

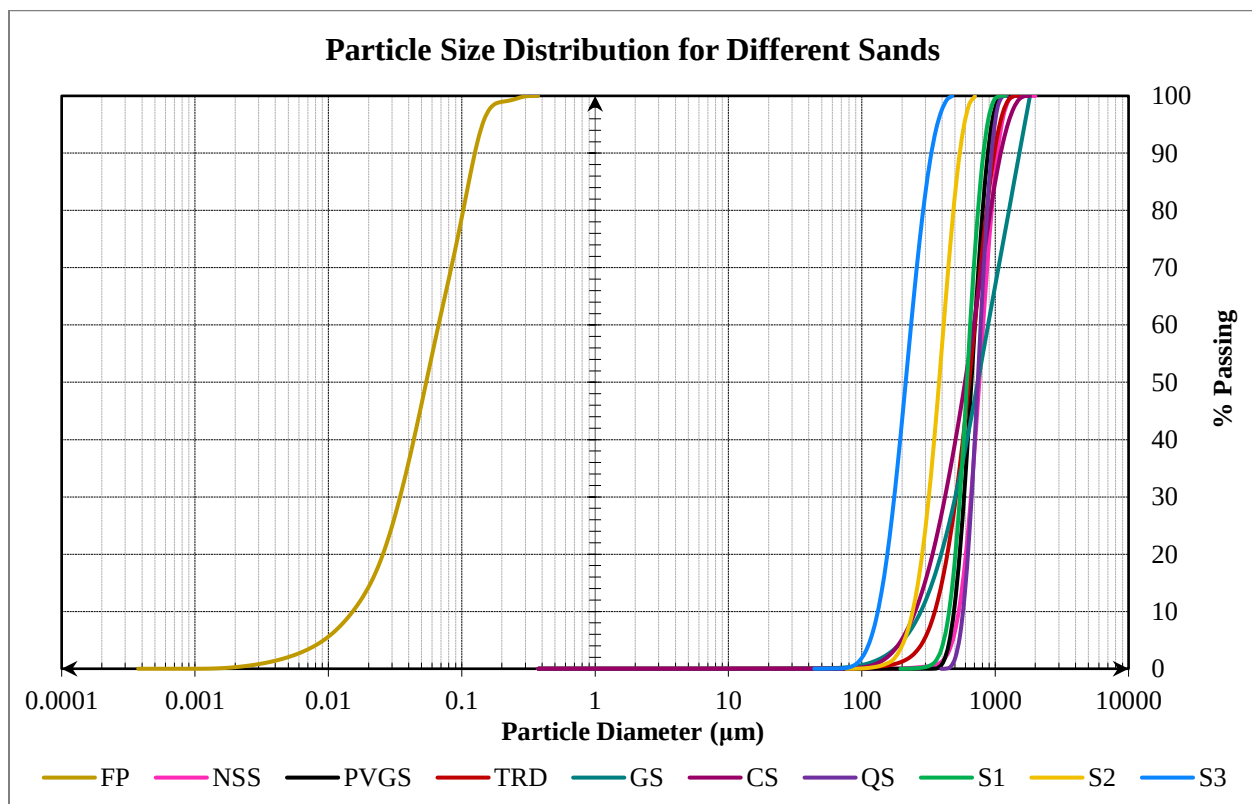


Figure A.8: Particle Size Analysis for Different Sands

Table A.1: Trial Mixes with Different Alkaline Solution (gr/L)

Materials	SSSH1.5	PSPH1.5	PSPH1.7
FA-F	474	474	474
DSF	345	345	345
GGBFS	620	620	620
PS	-	320	-
KOH	-	59	-
SS	320	-	320
NaOH	45	-	45
Extra Water in AS	30	30	30
QS	557	557	557
CS	1050	1050	1050
S1	453	453	453
Extra Water	81	30	81
HRWRA P1	20	20	20
Initial Setting Time (min)	8	11	8
f_c at 7 days (MPa)	45.12	28.89	26.78
f_c at 14 days (MPa)	53.87	36.71	42.23
f_c at 28 days (MPa)	57.53	45.36	51.18



Figure A.9: Efflorescence observed on early mixes with SSSH1.5

Table A.2: Trial Mixes with Different Inert Sands (gr/L)

Materials	IS-1	IS-2	IS-3	IS-4	IS-5	IS-6	IS-7
FA-F	865	865	865	865	865	865	865
DSF	180	180	180	180	180	180	180
GGBFS	144	144	144	144	144	144	144
PS	219	219	219	219	219	219	219
KOH	52	52	52	52	52	52	52
Extra Water in AS	96	96	96	96	96	96	96
QS	538	-	-	-	-	-	-
CS	-	538	-	-	-	-	-
PVGS	-	-	538	-	-	-	-
NSS	-	-	-	538	-	-	-
S1	-	-	-	-	538	-	-
S2	-	-	-	-	-	538	-
S3	-	-	-	-	-	-	538
Extra Water	13	13	13	13	13	13	13
HRWRA-N	13	13	13	13	13	13	13
WRA	18	18	18	18	18	18	18
f_c at 56 days (MPa)	67.71	55.39	40.5	53.8	48.63	48.76	55.04

Table A.3: Comparative Trial Mixes for Inert and Partially Reactive Sands (gr/L)

Materials	IS-8	PRS
FA-F	907	865
DSF	144	180
GGBFS	95	95
MK-750	75	75
PS1.7	355	355
Extra Water in AS	43	43
S3	801	-
GS	-	178
TRD	-	574
FP	-	25
Extra Water	13	13
HRWRA-P1	17	17
Steel Fibers	156	156
f_c at 28 days (MPa)	64.30	79.80

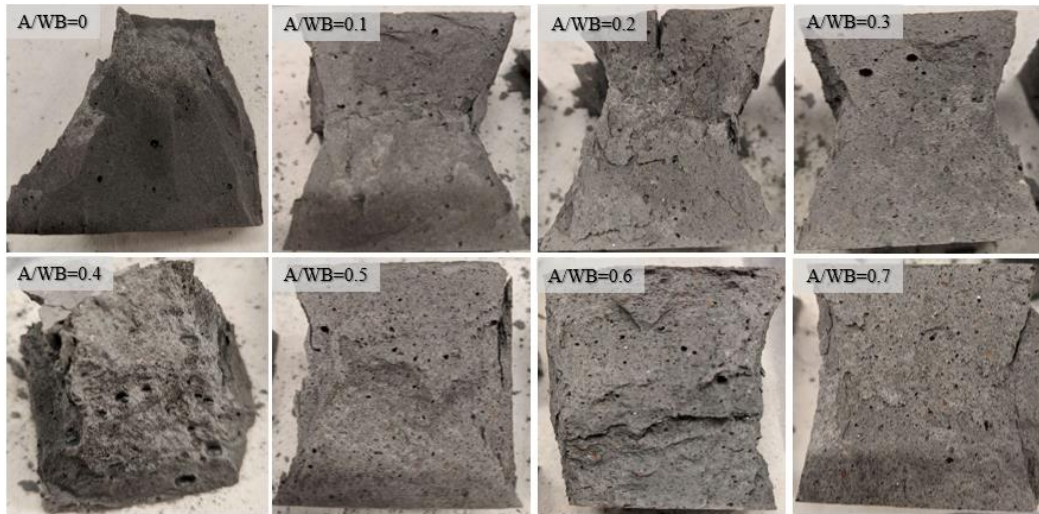


Figure A.10: Fractured cubes for different Aggregate contents with respect to the wet binder (dry binder and alkaline solution)

Table A.4: Selected Trial Mixes using Particle Packing Theory (gr/L)

Materials	PP1	PP2	PP3
FA-F	350	880	350
USF	160	146	85
GGBFS	150	90	100
MK-750	180	92	130
PS1.7	360	378	250
Extra Water in AS	70	72	50
GS	360	178	425
TRD	800	574	50
FP	400	26	-
Extra Water	10	10	10
HRWRA-P1	5	5	5
Setting time (min)	16	29	18
Spread (mm)	120	136	104
$f_{c,ave}$ at 14 days (MPa)	60.04	57.33	64.63

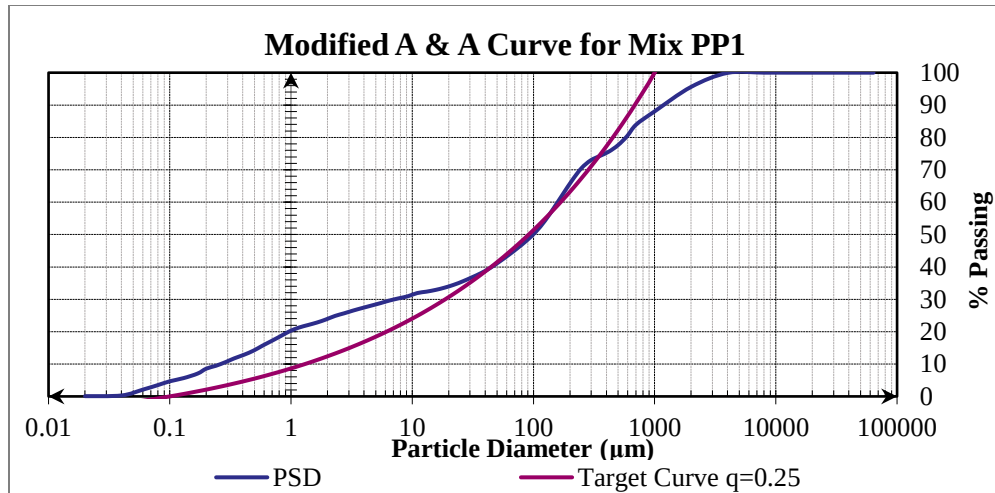


Figure A.11: Particle Packing for Mix PP1

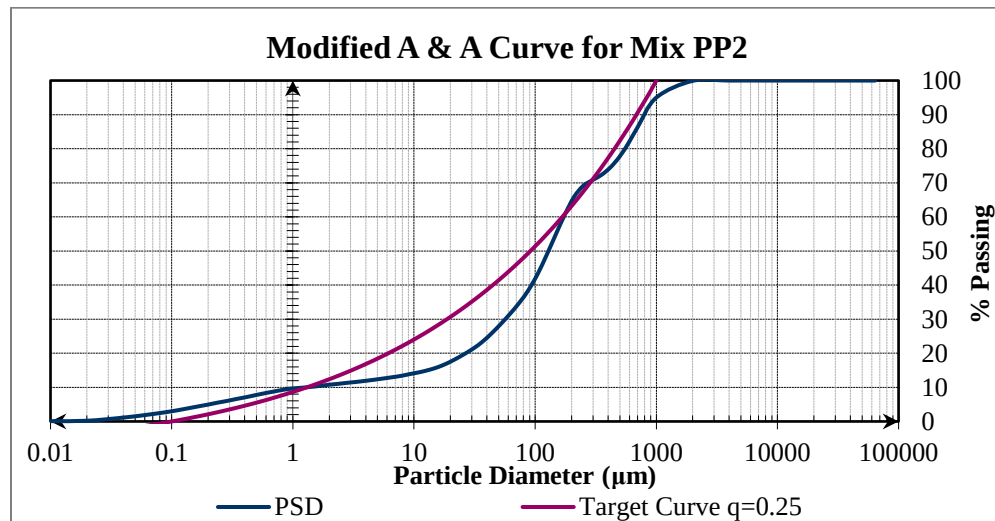


Figure A.12: Particle Packing for Mix PP2

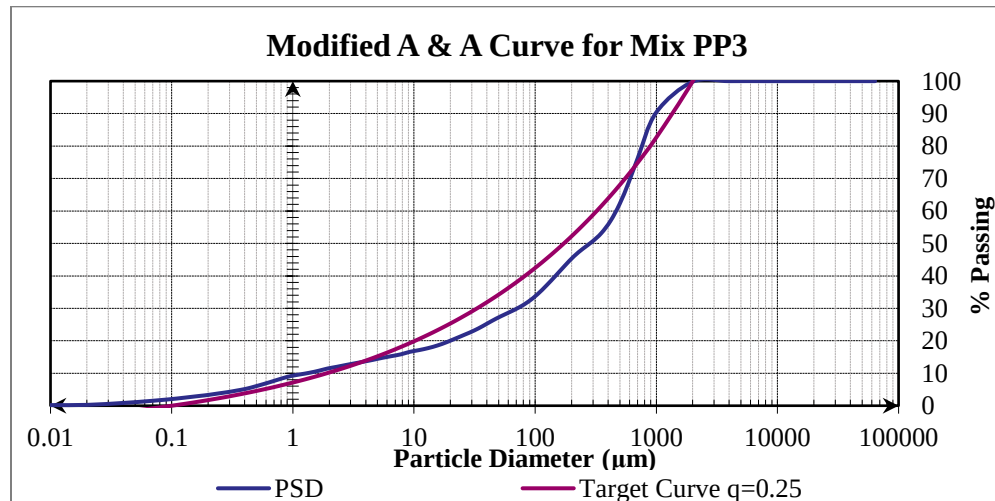


Figure A.13: Particle Packing for Mix PP3

Table A.5: Selected Trial Mixes for partial substitution of FA-F with MG and PM (gr/L)

Materials	0.5FA-F/0.5MG	0.85FA-F/0.15PM
FA-F	440	480
MG	440	-
PM	-	410
USF	146	146
GGBFS	90	90
MK-750	92	92
PS1.7	378	378
Extra Water in AS	72	72
GS	550	550
TRD	168	168
FP	26	26
Extra Water	10	10
HRWRA-P1	5	5
Setting Time (min)	45	40
$f_{c,ave}$ at 14 days (MPa)	54.74	50.24

A.3. Stoichiometry of the Developed THFRGC Compared to HPFRGCs from the Literature

Table A.6: Selected THFRGCs mix designs found in literature

Materials	[1 ¹¹]	[2 ¹²]	[3 ¹³]
FA-F	40	388	172
GGBFS	180	310	688
SF	230	78	45
MK-750	80	-	-
AS	400	93	359
Sand	980	1052	905
Water	80	194	87
MG	260	-	-
PM	45	-	-
Fresh Density (kg/m ³)	2295	2115	2256

¹¹ Developed THFRGC in this Thesis (the chemical composition of MG, MK-750, and PM are given in Chapter 3)

¹² Al-Majidi, M. H., Lampropoulos, A., & Cundy, A. B. (2017). Tensile properties of a novel fibre reinforced geopolymer composite with enhanced strain hardening characteristics. *Composite Structures*, 168, 402–427. <https://doi.org/10.1016/j.compstruct.2017.01.085>

¹³ Liu, Y., Zhang, Z., Shi, C., Zhu, D., Li, N., & Deng, Y. (2020). Development of ultra-high performance geopolymer concrete (UHPGC): Influence of steel fiber on mechanical properties. *Cement and Concrete Composites*, 112, 103670. <https://doi.org/https://doi.org/10.1016/j.cemconcomp.2020.103670>

Table A.7: Chemical composition of mineral powders and alkaline solutions used in the selected literature (% wt.)

Study	[1]	[2]	[3]	[1]	[2]	[3]	[1]	[2]	[3]	[1]	[2]	[3]
Material	FA-F			GGBFS			SF			AS		
SiO ₂	56.5	59	54.22	35.2	35	33.81	94.5	95	95.38	23.52	24.6	24.31
Al ₂ O ₃	23.5	23	31.18	8.37	12	14.78	0.41	-	-	-	-	-
CaO	9.1	2.4	1.24	38.9	40	38.81	0.49	-	1.84	-	-	-
Fe ₂ O ₃	3.55	8.8	2.63	0.68	-	0.36	0.16	-	0.61	-	-	-
MgO	1.08	1.4	0.47	10.7	10	7.09	0.3	-	0.26	-	-	-
Na ₂ O	2.13	0.7	0.49	0.31	-	0.26	0.15	-	0.26	-	-	17.93
K ₂ O	0.84	2.8	1.34	0.59	-	0.44	0.59	-	0.44	21.48	17.2	-
H ₂ O	-	-	-	-	-	-	-	-	-	55.00	58.2	57.75

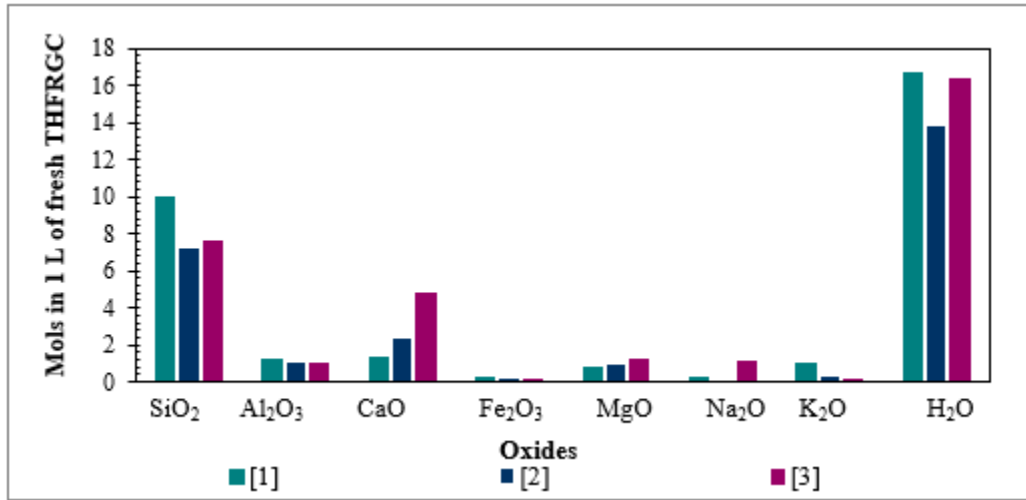


Figure A.14: Mols of main oxides in 1 L of fresh THFRGC

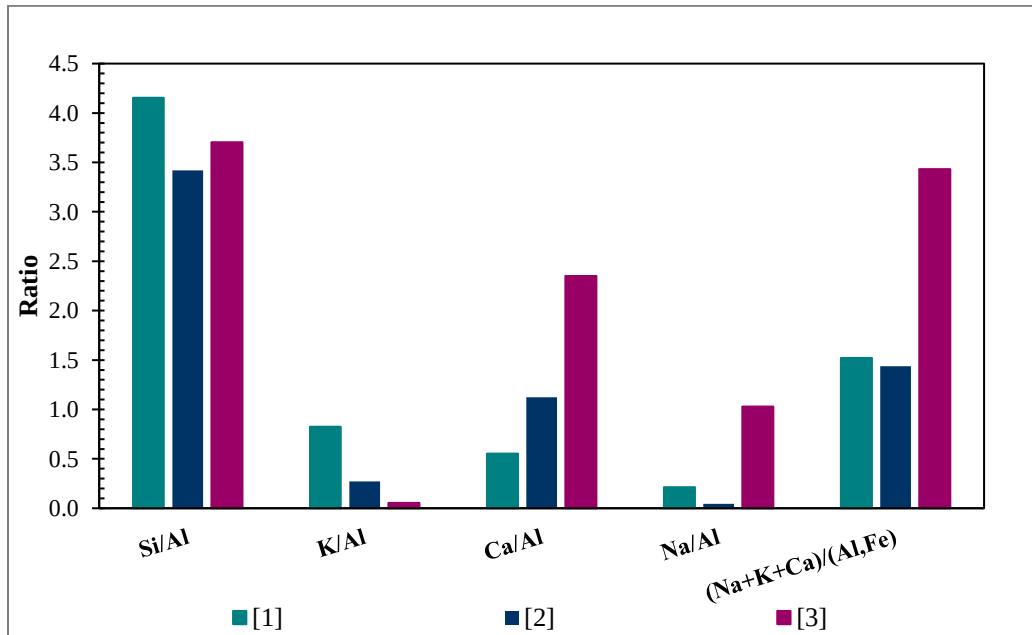


Figure A.15: Atomic Ratios in poly(sialate) structure of THFRGCs

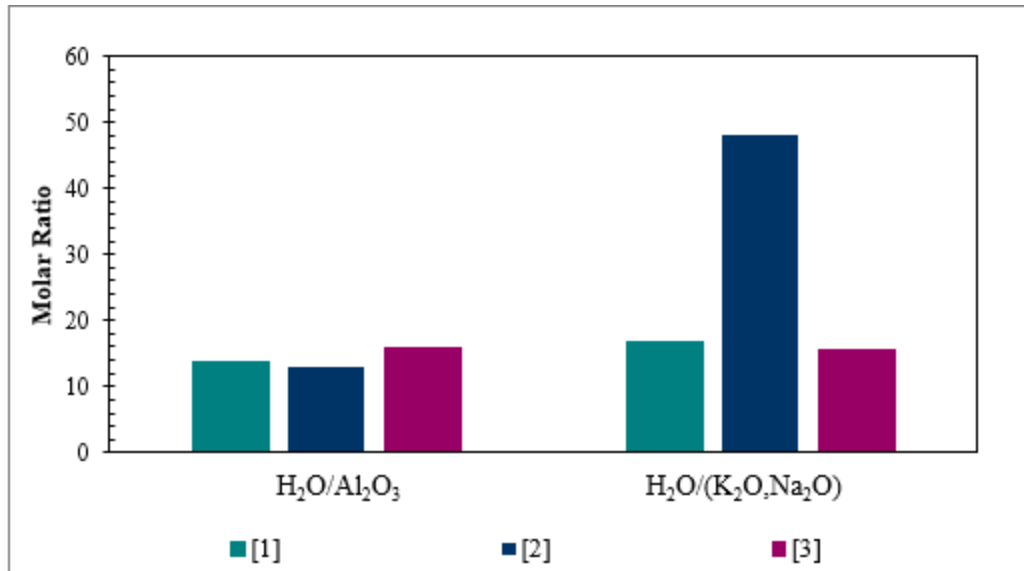


Figure A.16: Molar water to alkali ratio in poly(sialate) structure of THFRGs

A.4. Informative on Chemical Admixtures

- *High-Range Water Reducing Admixtures*

High-Range Water Reducing Admixtures, also known as superplasticizers (SPs) which are used to lower the water demand and increase workability, are categorized based on their mechanism and chemical base. Melamine and naphthalene-based SPs are considered as second generation, as their fluidization mechanism is based on electrostatic inter-particle repulsion. Modified Polycarboxylates are classified as the third and latest generation, as their mechanism is a combination of electrostatic repulsion and steric repulsion resulting from lateral ether chains on the SP molecule (Rixom & Maivaganam, 1999; Nematollahi & Sanjayan, 2014).

- *Air Detraining Admixtures*

Air Detraining Admixtures are used when excess release of gas from aggregates or entrainment of excess air due to poor consolidation/ high viscosity occurs. Common chemical base is water-insoluble alcohols (octyl alcohol, 1-octanol, caprylic alcohol), silicones and tributyl phosphate with the latter to be the most common (Kosmatka *et al.*, 2002). Their target is to destabilize air in fresh concrete, allowing it to escape during mixing.

- *Workability Retention Admixtures*

Workability-retaining admixtures allow concrete to maintain its fresh characteristics without adversely affecting the setting time or hardened state properties. The mechanism is similar to that of a time-release dispersant that continuously provides a special polymer to the surfaces of the reactive materials' grains to prevent them from clustering thus resulting in workability retention (Nmai & Daczko, 2012).

- *Rheology Controlling/ Viscosity Modifying Admixtures*

Viscosity Modifying Admixtures (VMA) or Rheology Controlling Admixtures (RCA) enhance the cohesion of the concrete in cases where high paste content mixtures are used, namely in SCCs. They are commonly used to prevent bleeding and segregation due to the ability of VMA molecules to absorb water and swell and thus preventing immobilizing the reactive material grains. Their chemical base is usually starches or polysaccharides, with most common to be modified cellulose, Welan Gum or Diutan Gum. The latter mainly affect the rheology of the fluid state with strong impact on the rheology of concrete, while starches influence the particle interactions (Schmidt *et al.*, 2013).

- *Hydration Controlling and Retarding Admixtures*

Hydration Controlling or Retarding Admixtures are used to prolong the setting time of concrete. Usually, they are a combination of polycarboxylate ethers or sulphonates with molasses, sucrose, or gluconate to provide the retardation. Pure retarders are mainly based on gluconate and or sucrose nevertheless they also demonstrate plasticizing properties. Other retarders with less plasticizing action are phosphates and acids like citric or tartaric. Their mechanism is quite complex and consists mainly of two processes: (i) the adsorption of the retarder's molecules on the surface of reactive materials' grain and thus the retardation of silicate hydrate formation in case of OPC and (ii) the chelation of calcium ions in solution which prevents precipitation of calcium hydroxide. It should be noted that such admixtures do not reduce the exotherm of mass concrete, but only delay its onset. To manipulate the latter, less reactive materials should be used or cooling the constituents could be effective (Dransfield, 2003).

Appendix B: A Preliminary Characterization of Partially Optimized THFRGC

B.1. Mix Design & Fresh Properties

Table B.1: Mix Design (gr/L)

Materials	P-F	P-NF
FA-F	880	880
DSF	145	145
GGBFS	49	49
MK-750	91	91
PS1.7	355	355
Extra Water in AS	71.1	71.1
S2	200	200
S3	67.5	67.5
GS	100	100
TRD	300	300
Extra Water	15	15
HRWRA-P1	5	5
Steel Fibers	156	-
Dynamic Spread (mm)	200	183
Final Setting Time (min)	32	36

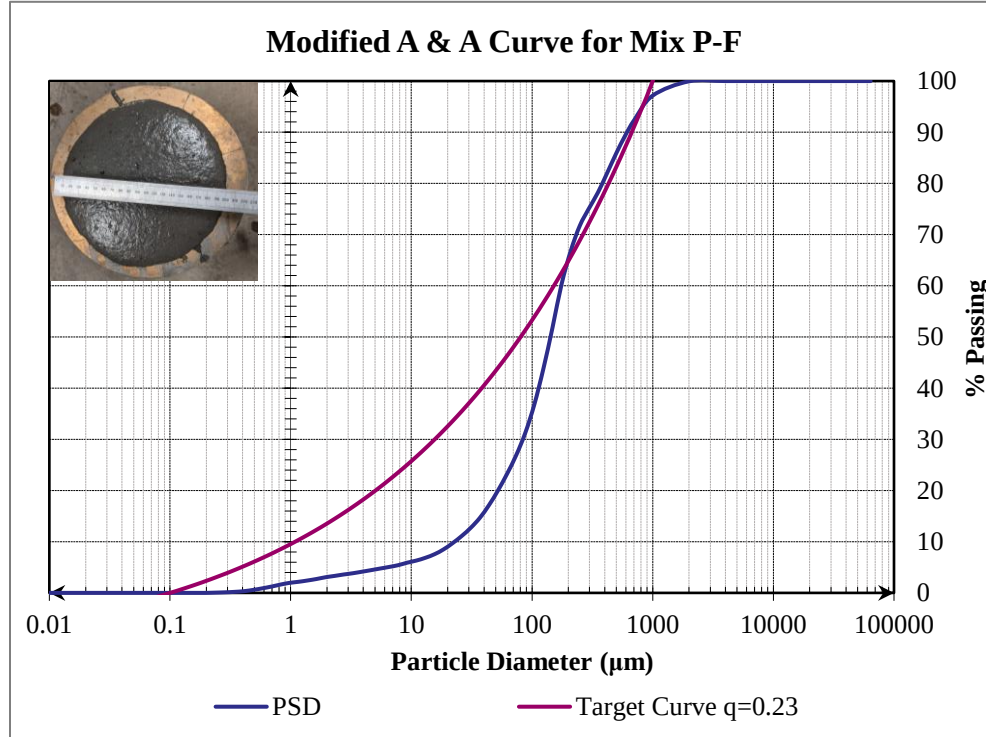


Figure B.1: Particle Packing of the only chemically optimized preliminary THFRGC mix

B.2. Mechanical Properties

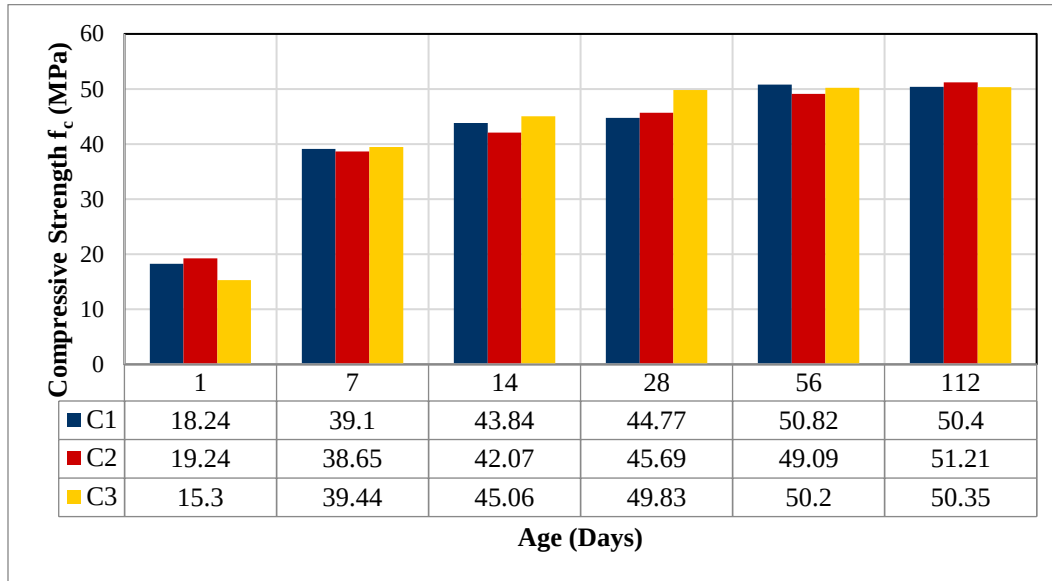


Figure B.2: Compressive strength of P-NF 75 mm x150 mm cylinders over age



Figure B.3: Fractured specimens from P-F batch



Figure B.4: Fractured specimen from P-NF batch

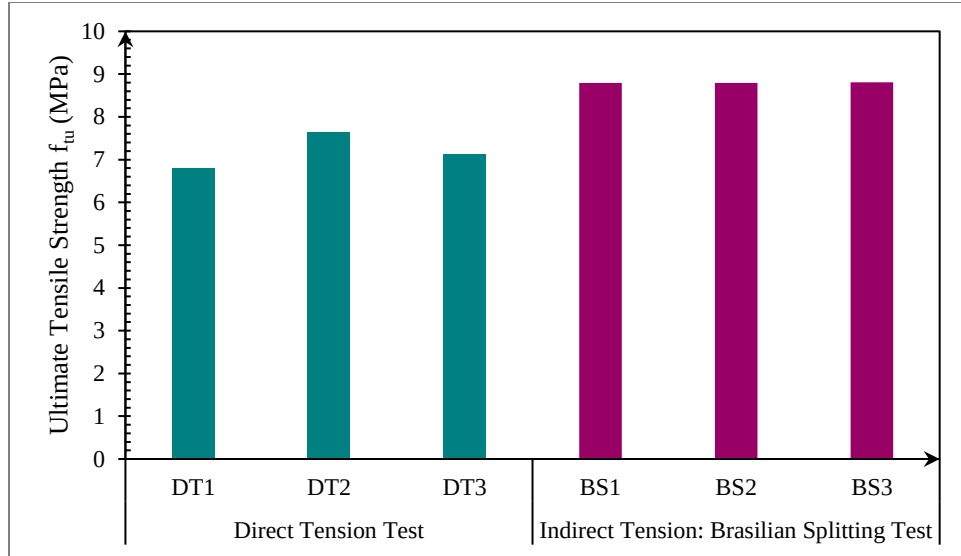


Figure B.5: Tensile Strength of P-F batch obtained from direct and indirect tension tests

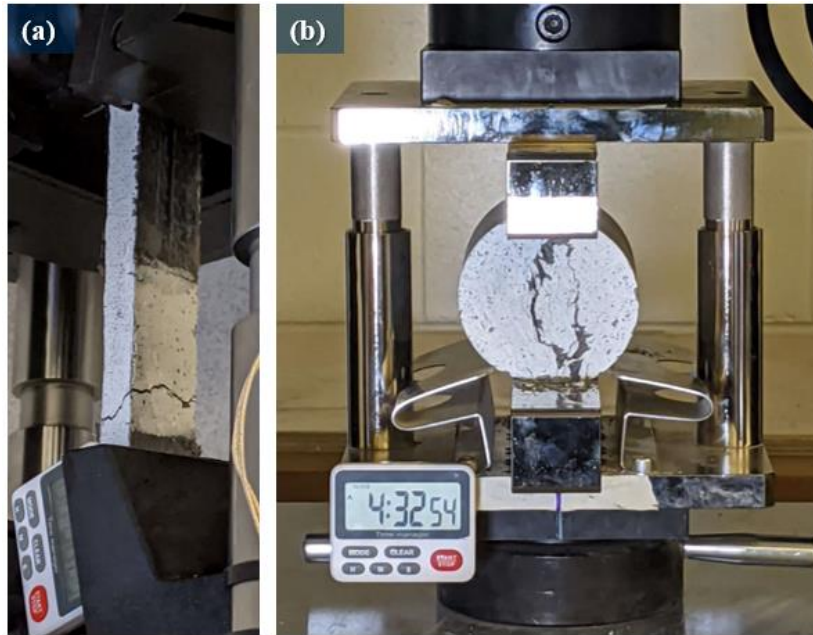


Figure B.6 Fractured specimen and experimental setup for (a) Direct Tension Test and (b) Brazilian Splitting Test

Table B.2: Flexural Performance of P-F batch

Specimen	P_{cr} (kN)	P_{max} (kN)	$\delta @ P_{cr}$ (kN)	$\delta @ P_{max}$ (kN)	σ_{cr} (MPa)	$\sigma_{f,max}$ (MPa)
SP1	10.64	21.23	0.062	0.27	5.67	11.32
SP2	13.2	16.06	0.082	0.23	7.04	8.56
SP3	15.7	22.52	0.162	0.47	8.37	12.01
Average	13.18	19.94	0.102	0.32	7.03	10.63
SD	2.53	3.42	0.053	0.129	1.35	1.82

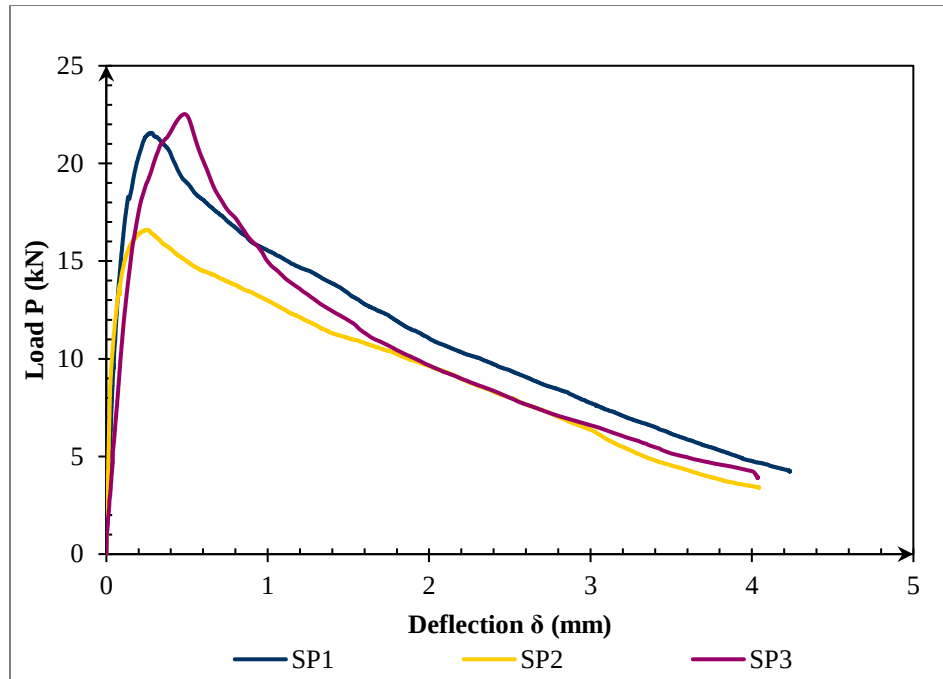


Figure B.7: Resistance curve of 75 mm x 75 mm x 280 mm prisms from P-F batch according to ASTM C 1609.

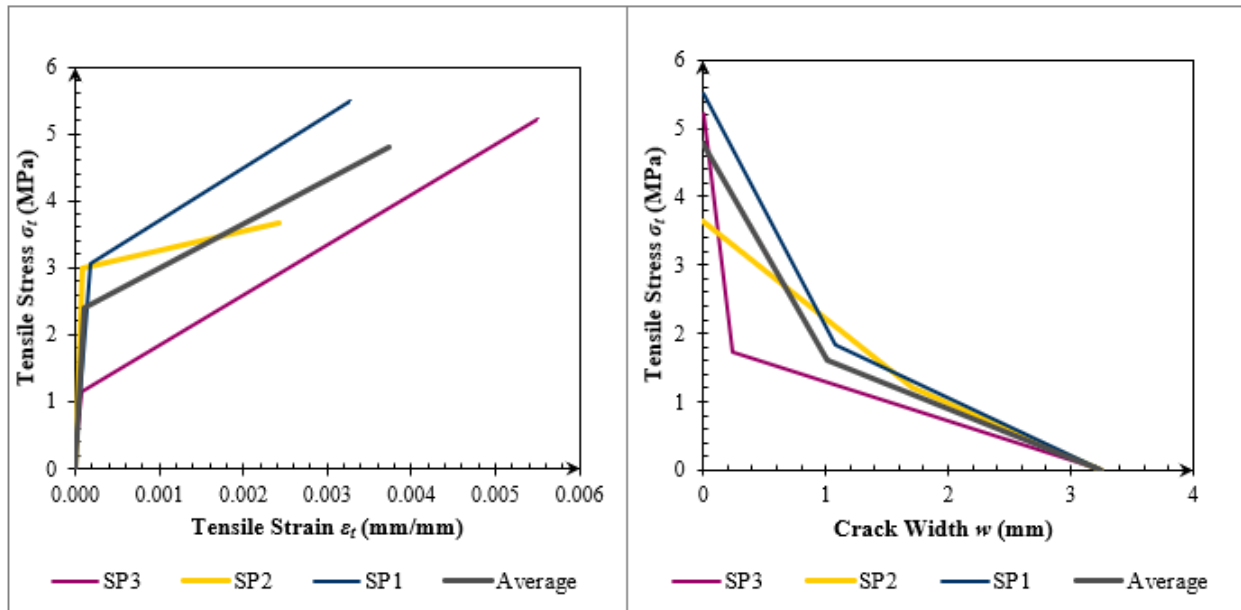


Figure B.8: Linearized Tensile stress-strain and crack width law obtained according to the inverse analysis prescribed in CSA S6, Annex 8.1

B.3. Physical Properties

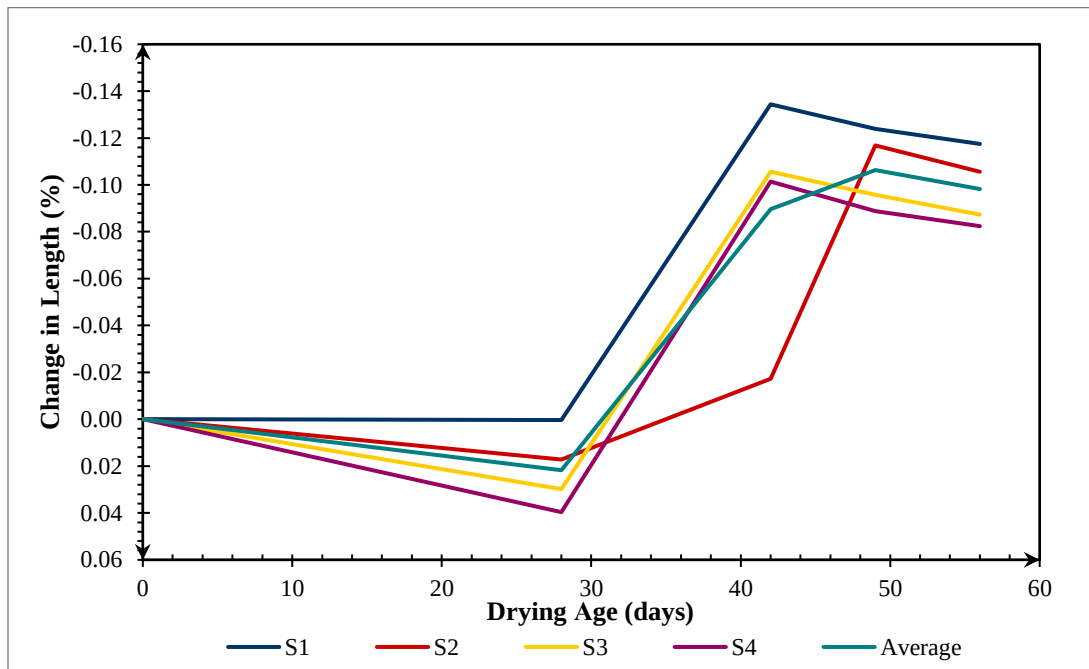


Figure B.9: Drying shrinkage of P-F batch according to ASTM C157

Appendix C: Informative on the Derivation of Resistivity According to the Wenner Array Method

An arbitrarily sharp probe contacts and injects current (through an applied voltage) into a semi-infinite volume (infinite in all directions except towards the probe) of a conductive material as shown in Figure C.1. The current travels outwards from the point of contact through concentric hemispherical shells of equipotential, each of which have current density (J) of:

$$J = \frac{I}{2\pi r^2} \quad (\text{C.1})$$

Where r is the radial distance from the probe ($2\pi r^2$ being the surface area of the hemisphere).

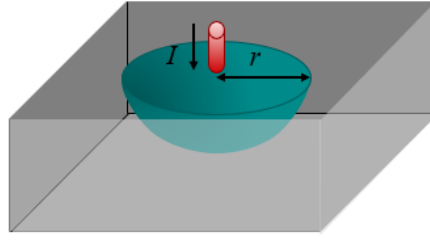


Figure C.1: A probe injecting current I into a semi-infinite volume of conductive material. The green hemisphere is a shell of the injected current with radius r .

By applying Ohm's Law ($E = \rho J$) with the electric field across each shell equal to the voltage drop over the shell thickness, or $-dV/dr$ (this term is negative as voltage decreases with r), and with the thickness of the shell tending towards zero, Equation (C.2) is obtained:

$$\Delta V = V - V' = \frac{\rho I}{2\pi} \left(\frac{1}{r} - \frac{1}{r'} \right) \quad (\text{C.2})$$

By applying the boundary condition that V approaches zero as r approaches infinity, the equation simplifies to:

$$V = \frac{\rho I}{2\pi r} \quad (\text{C.3})$$

Now there are four probes (labelled 1 to 4) in contact with the semi-infinite conducting material, which are in a line with equal spacing (α). They are set up so that current is injected through probe 1 and collected by probe 4 as shown in Figure C.2.

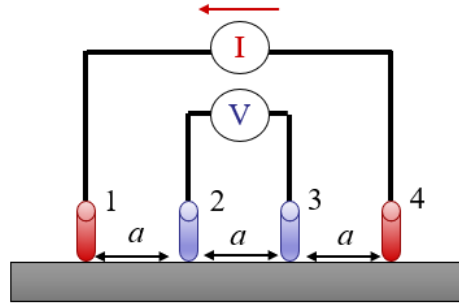


Figure C.2: A schematic diagram of a four-point probe. The four probes have equal spacing (a) and are shown in contact with a surface. A current (I) is injected through probe 1 and collected through probe 4, whilst the voltage is measured between probes 2 and 3

If equivalent boundary conditions are assumed for each probe, the voltage at any point is equal to the sum of the voltage due to each probe separately, i.e.:

$$V = \frac{\rho I}{2\pi} \left(\frac{1}{r_1} - \frac{1}{r_4} \right) \quad (\text{C.4})$$

When r_1 and r_4 are the radial distances from probe 1 and probe 4, respectively. Measurements of the voltage are then made between probes 2 and 3. Using Equation (C.4), the voltage at probes 2 and 3 is:

$$\begin{aligned} V_2 &= \frac{\rho I}{2\pi} \left(\frac{1}{a} - \frac{1}{2a} \right) \\ V_3 &= \frac{\rho I}{2\pi} \left(\frac{1}{2a} - \frac{1}{a} \right) \end{aligned} \quad (\text{C.5})$$

Hence, the change in voltage (ΔV) between probes 2 and 3 is:

$$\Delta V = V_2 - V_3 = \frac{\rho I}{2\pi} \left(\frac{2}{a} - \frac{1}{a} \right) = \frac{\rho I}{2\pi a} \quad (\text{C.6})$$

Therefore, the resistivity between the probes is:

$$\rho = 2\pi a \left(\frac{\Delta V}{I} \right) = 2\pi a R \quad (\text{C.7})$$