

A study of the thermal and mechanical properties of recycled crosslinked HDPE

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Abstract— Crosslinked high-density polyethylene (XHDPE) is used to manufacture large and rigid products, such as fuel tanks, using a rotomolding process. Currently, defective and post-consumer parts are not recycled and sent to landfills. Hence, reuse of this material, through compression molding is the objective of this study. Thermal characterization indicated that the recycled XHDPE, in pulverized powder form, could melt although the heat of fusion was less than that of virgin material. However, while the virgin XHDPE could be compression molded at the melting temperature, the recycled XHDPE required higher temperature. The recycled XHDPE started to degrade at lower temperature than virgin XHDPE. Based on this data, optimal compression molding process conditions were chosen to prepare dense tensile test coupons. Both materials exhibited similar tensile properties demonstrating the recyclability of XHDPE.

Rotomolding; compression molding; recycled XHDPE.

I. INTRODUCTION

Rotomolding is a common manufacturing process used to produce large and strong parts with a uniform thickness. One of the main plastics used is the High-Density polyethylene (HDPE). For some applications, the HDPE must be crosslinked to make the parts stronger and increase the barrier resistance

HDPE is a thermoplastic with a density of 0.940-0.965g/cm³[1] and melting between 128°C and 138°C [2]. These properties are slightly changed after crosslinking. The crosslinking process results in bonding of the chain to itself and to other chains [3].

The crosslinked polyethylene is prepared using either a crosslinking agent (peroxide or silane) or electron beam radiation. During rotomolding, the crosslinking caused by the peroxide initiator increases the gel content (>80% as claimed by rotomolder using this materials), which reduces the flow of the material when heated above the melting temperature.

Due to this characteristic, XHDPE cannot be recycled and the industrial and post-consumer waste is currently sent to the landfill. Published studies on recycling this material include energy recovery [4], use of the ground material as a filler [5], degradation to low molecular weight polyethylene using solid

state shearing [6], molten state shearing [7], ultrasonic process [8], and supercritical process [9]. The degraded material was subsequently blended with other thermoplastic polymers to manufacture parts.

This research is focused on reuse of this material, without blends, through compression molding. The thermal behavior of the recycled XDPE is characterized and compared with the virgin XDPE to understand the issues in reusing this material. Based on this, the compression molding conditions are chosen to mold tensile specimens. The tensile properties of recycled XHDPE are compared with virgin XHDPE to evaluate former's recyclability. Preliminary results are presented and discussed here.

II. EXPERIMENTAL

A. Materials and Methods

Exxon Mobil's Paxton 7000 Series XHDPE was used in this study. Pulverized powder of the virgin and recycled material was supplied by a rotomolder in Manitoba. The melting characteristics of these two materials were studied using TA Instruments' Q2000 DSC (Differential Scanning Calorimeter). Samples were ramped at a rate of 10°C/min from -50°C to 250°C under nitrogen atmosphere. The degradation characteristics was studied using TA Instruments' Q500 TGA (Thermogravimetric Analyzer). Samples were ramped at a rate of 10°C/min from 25°C to 550°C using nitrogen and air as the purge gas. Based on these test results, compression molding temperature was chosen. Molding pressure was chosen based on preliminary trials. ASTM standard tensile test coupons were molded using these conditions and tested as per ASTM 638 using MTS Insight test machine.

III. RESULTS AND DISCUSSION

A. Differential Scanning Calorimeter Results

The results from DSC experiments, shown in Fig. 1, indicate that both materials melt around 130°C. A completely crosslinked material would not melt. The results suggest that XHDPE either did not completely crosslink during the rotomolding process or was de-crosslinked by the pulverization process.

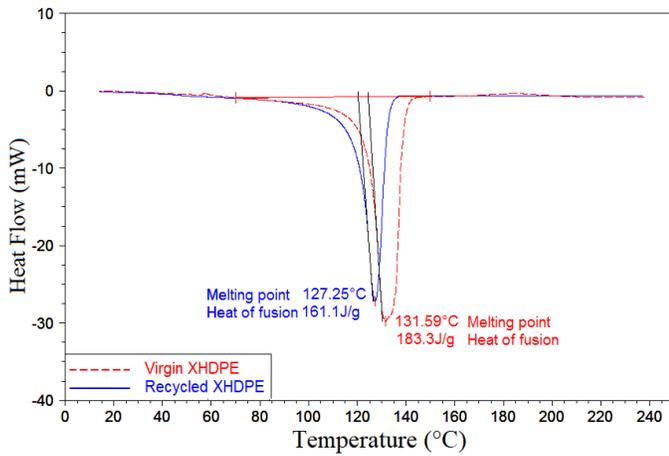


Figure 1. DSC test results for virgin and recycled XHDPE

The lower heat of fusion for recycled XHDPE than the virgin XHDPE suggests that there is residual crosslinking in the pulverized powders of recycled XHDPE. This result confirms that the recycled XHDPE, in pulverized powder form, can melt and hence is recyclable

B. Thermogravimetric Analysis

The results from TGA experiments shown in the Fig. 2 (purge gas – nitrogen) indicate that both the virgin and the recycled XHDPE degrade similarly.

The decomposition initiation and completion temperatures as well as the weight loss are similar for both materials. These temperatures are much higher than the molding temperatures.

Depending of the molding conditions, the XHDPE may or may not come in contact with air. Hence, TGA test was repeated using air as a purge gas (Fig. 3).

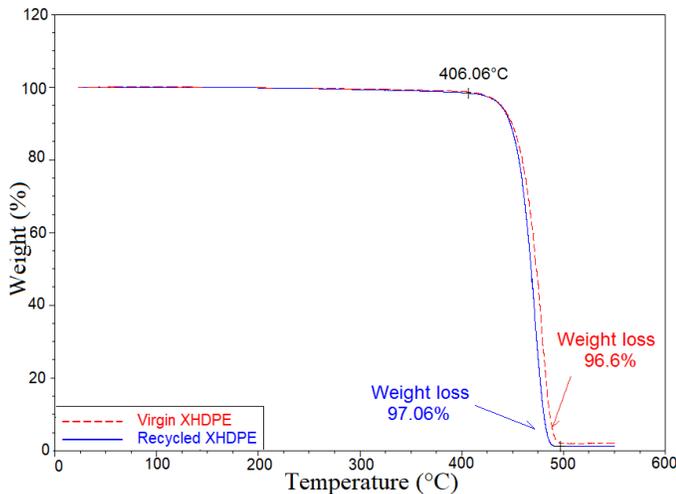


Figure 2. TGA test results for virgin and recycled XHDPE (Nitrogen)

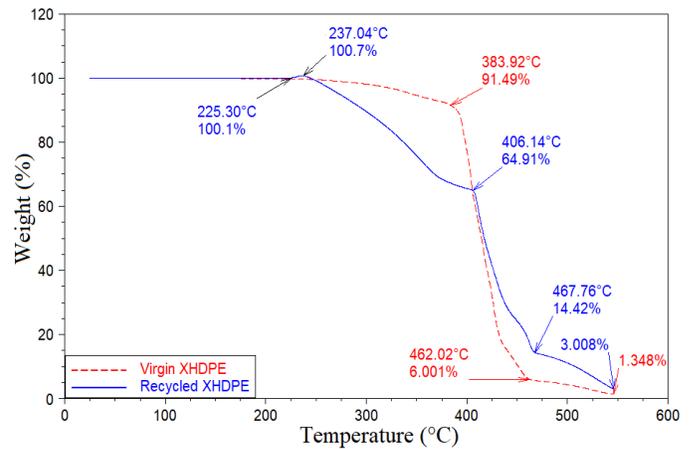


Figure 3. TGA test results for virgin and recycled XHDPE (Air)

The TGA results in Fig. 3 indicate that the recycled XHDPE is affected by the presence of air resulting in the formation of different products, suggested by the various slopes of the curve. Hence, the molding temperature was limited to 250°C.

C. Compression molding

Initial trials indicated that while virgin XHDPE could be melted and molded at the melting temperature, recycled XHDPE had to be molded at a considerably higher temperature and pressure. Tab. I presents the final compression molding conditions that resulted in a dense product without defects.

D. Mechanical properties

The tensile test results for both materials are presented in Fig. 4. The results show that the recycled XHDPE has a very similar stress versus strain curve when compared with the virgin material.

The tensile strengths of the virgin and recycled XHDPE are 20.25 MPa and 20.5 MPa respectively. The fracture strain of the virgin and recycled material are 344.5% and 273.6% respectively. While there is a slight decrease in the strain to failure of the recycled XHDPE, the tensile strength and modulus for both materials were similar.

TABLE I. COMPRESSION MOLDING SETTINGS

Material	Parameters		
	Temperature	Pressure	Time
Virgin XHDPE	150°C	0.286 MPa	30 min
Recycled XHDPE	230°C	2.86 MPa	30 min

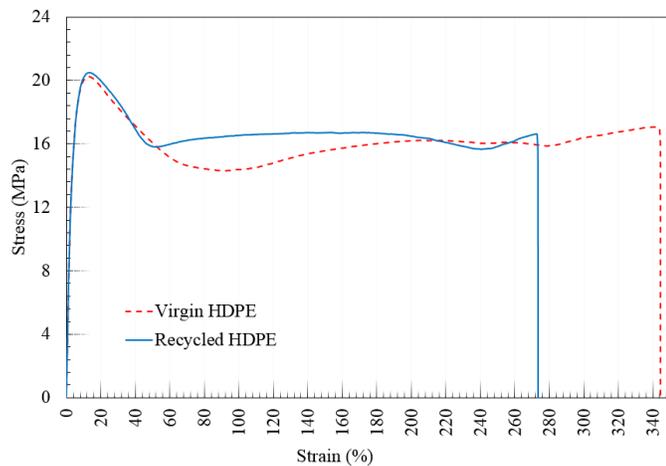


Figure 4. Tensile test results for virgin and recycled XHDPE

IV. SUMMARY

The thermal properties of Virgin and recycled XHDPE were investigated using DSC and TGA. Both materials exhibited melting around 130°C, although the heat of fusion for recycled material was less than that for the virgin material. The recycled material was successfully compression molded at a temperature and pressure higher than that used for the virgin material. Both materials exhibited similar tensile properties with the exception of strain to failure, which was slightly less for the recycled material. These results suggest that XHDPE can be reused through compression molding. Further studies are underway to study the mechanism of fusion of recycled XHDPE powder and the effect of pulverization on its recyclability.

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