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<td>Polymer recycling, rotational moulding, plastic waste, circular economy</td>
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</table>
Original article

Sibele Piedade Cestari*, Peter Martin, Paul Hanna, Mark Kearns, Luis Claudio Mendes and Bronagh Millar

Use of virgin/recycled polyethylene blends in rotational moulding

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Abstract: Aiming to further plastics recycling via rotational moulding plastics processing, blends of virgin and recycled polyethylene sourced from post-consumer plastics were developed. Three different kinds of recycled high density polyethylene – from bottles, pipes and mixed household waste - were compounded with virgin medium density polyethylene in an extruder. The ideal amount of recyclate was chosen based upon the impact resistance of different contents (25, 50 and 75%) of recycled plastic with the 50/50 blend found to have the best performance. Compression moulded and rotationally moulded samples were analysed through falling dart impact test, flexural test, melt flow rate and differential scanning calorimetry analysis. The impact results of the compression moulded samples showed an increase in the impact resistance of the blends with higher melt flow index and lower degree of crystallinity. The rotationally moulded specimens displayed much lower impact resistance than the pure virgin plastic and a 20-30% reduction in the flexural moduli, which were ascribed to the crystalline structure of the part and issues in the blends' rotomoulding process. It was concluded that blending virgin and recycled polyethylene for rotational moulding can be an effective way to further plastics recycling inside the Circular Economy context.

Keywords: circular economy; plastic waste; polymer recycling; rotational moulding.

1 Introduction

The Circular Economy has a vital role in contemporary attempts to improve sustainability worldwide. All sectors of society pursue more sustainable actions and practices through industrial symbiosis, creative design, remanufacturing and cleaner production, aiming the transition to systems of more conscious consumption and production [1–3]. Inside this scenario, post-consumer plastics have received particular attention from researchers and funding agencies. Given the consequences for the environment (oceans pollution, presence of microplastics in the Arctic, microplastics found in the human placenta), tackling the plastics waste problem became a need [4–8].

As important as the environmental concern, the economic aspect of increasing the recycling rate of plastics should be considered [9–13]. In 1996, Vinci and La Mantia already highlighted that recycling thermoplastic polymer is a way to lower costs and consume less raw material [14]. But some precepts of the Circular Economy enclosed in the International Organization for Standardization (ISO) Standard on life cycle assessment (LCA) ISO 14040 [15] may prevent the reinsertion of post-consumer plastic in the production stream, leading to the wastage of still usable materials. Most studies on plastics recycling search to obtain a closed-loop lifecycle, believing that this is the most effective approach. But as stated by Geyer [16], "There is no engineering relationship or law of physics that requires primary production to decrease as recycling increases."

One of the biggest challenges to polymer recycling is the broad range of post-consumer plastics whose recycling has not yet been addressed. The existing sorting methods and recycling technology, that allows the continuous recycling of the most commonly used commodity polymers, cannot be applied indistinctively to all types of plastic [17]. In the United Kingdom, where the waste collection system leads to the existence of two separate household plastic wastes (bottles and mixed plastics/plastic films), there are already some developments being made in terms of transforming mixed household waste plastic into useful products [18].

Blending recycled plastics with virgin resins is one of the most straightforward approaches on recovering properties of a reprocessed polymer. La Mantia and coworkers (2021) studied blends of virgin and reprocessed polypropylene (PP), aiming to compare a predictive model of rheological
and mechanical properties with experimental results [19]. They concluded that the model was effective when applied to compatible blends. Alzerreca and coworkers (2015) blended virgin and recycled high density polyethylene (HDPE) to improve the quality of post-consumer recycled HDPE [20], and discovered the importance of contaminants, polypropylene and fillers in recycled materials. In terms of mixed plastics recycling, authors like Garofalo (2021) recently studied ways to improve the recycling of mixed polyolefin post-consumer plastics to be used in the piping sector [21]. Amongst the studied formulations and pre-treatments, they concluded that the batch previously washed with hot water, and compatibilised with 5% a PE/PP blend grafted with maleic anhydride, showed the best performance for the intended application.

Amongst several plastics processing techniques, the rotational moulding (or rotomoulding) is a relatively low-cost and competitive method to obtain hollow plastic products. It enables the production of complex shapes with even thickness and minimal need for external finishing, with no wastage of plastic in the process. Due to high processing temperatures and longer cycle times however, not all polymers can be thermoformed by this technique. Nowadays, the most frequently used polymer in rotomoulding is polyethylene (PE) in powdered grades, with flow properties especially tailored for this application and presenting better thermal stability [22–26].

Developing blends of virgin and recycled plastic intended to be used in rotomoulding can be a pathway to increase recycling rates [27], however developing materials for the rotational moulding process can be challenging, as it demands specific flowability and thermal resistance. Therefore, in this work the ideal amount of recylcate, and the effect of three different types of recycled high density polyethylene (rHDPE), in blends with medium-density polyethylene (MDPE) were studied and their thermal and mechanical properties assessed.

2 Materials and methods

2.1 Materials

The virgin polymer used in the blends was a rotational moulding grade of medium-density polyethylene (MDPE), Revolve N-250, supplied by Matrix Polymers in powder. The selected recyclates were recycled high-density polyethylene (rHDPE) systematically recovered from bottles (01), pipes (02) and mixed household waste (03). Materials 01 and 02 were kindly donated by Van Werven Recycling company and provided in flakes. Material 03 was kindly donated by Impact Laboratories Ltd and provided in the form of pellets.

2.2 Methods

To determine the ideal amount of recylcate, the impact resistance and the melt flow rate (MFR) of MDPE blends having different contents of one of the rHDPEs (rHDPE01): 25, 50, 75 and 100% were assessed. After the results, blends containing 50% of each HDPE were prepared and falling dart impact test, flexural test, MFR, differential scanning calorimetry (DSC) and FEG-SEM analysis conducted.

2.3 Compounding and specimen preparation

Before compounding, the three recycled HDPEs were cryogenically ground in a Wedco single-stage grinding mill, to improve the blending with the MDPE powder. All the blends were compounded in a Collins ZK 25 co-rotating twin-screw extruder. The processing parameters were: temperature profile of 180°C in the feeding zone, 200°C in the five heating zones and the die, screw speed of 300 rpm, feeder rate of 5 kg per hour. Impact test specimens were prepared by compression moulding pellets in a Collins P200P platen press, under the five-stages processing conditions of Table 1, producing laminates of around 2.5 mm thickness. The differential scanning calorimetry and melt flow analysis were performed over pellets.
2.4 Rotational moulding
Test cubes of pure MDPE and the three 50/50 blends, measuring 330 x 330 x 300 mm, were produced on a Ferry Rotospeed 1600 carousel type rotational moulding machine equipped with a stainless steel mould of 440 x 330 x 330 mm, using ground pellets. The oven temperature was set to 300°C and a 'Rotopaq' temperature-monitoring system used to record the mould internal air temperature. In all cases, the test mouldings reached a peak internal air temperature of approximately 200°C resulting in a cycle time of approximately 30 minutes. Impact and flexural test specimens were cut from the test cubes, having a thickness of 3.5 mm (MDPE) and between 5-7 mm (blends).

2.5 Falling dart impact test
For the impact test, square specimens of 60 mm were prepared. The test was performed according to the British standard EN ISO 6603-2 [28] in a falling dart impact tester, weight of 18.63 kg, using eight specimens of each material. The compression moulded samples had around 2.5 mm thickness, and the rotomoulded samples had approximately 3.5 (MDPE) and 5-7 mm (blends) of thickness.

2.6 Flexural test
The flexural test was conducted according to the British standard EN ISO 178 standard [29], A method, 2.56 mm/min speed, 3.5% of deformation, deformation rate of 1%/min. Five rotational-moulded samples of each material (MDPE and 50/50 blends) of 120 x 15 mm, with around 3.5 (MDPE) and 5-7 mm (blends) thickness were tested.

2.7 Melt flow rate
The melt flow rate of the materials was carried out on a Kayeness Galaxy Model 7053 Indexer at 190°C using a load of 2.16kg in accordance with ASTM D1238 [30].

2.8 Differential scanning calorimetry
The DSC analysis was performed in a Perkin Elmer equipment DSC6 model, using pellets. The samples were analysed according to the following cycles: in the first cycle, the sample was heated from 30 to 200 °C at a heating rate of 10 °C·min⁻¹, keeping the material at 200 °C for 3 min; the second cycle was carried out at a cooling rate of 10 °C·min⁻¹ until 30°C; the third cycle repeated the same temperature range and heating rate of the first cycle. The crystallisation temperature (T_c) was obtained considering the cooling curve. The crystalline melting temperature (T_m) and the degree of crystallinity (X_c) of MDPE, rHDPEs and blends were obtained considering the second heating curves. The X_c was determined based on the ratio between the melting enthalpy (ΔH_m) of the polymers/blends and the 100% crystalline HDPE (293 J·g⁻¹), adjusted according to the content of each polymer in the blend [31].

3 Results and discussion
3.1 Impact
Table 2 shows the impact of MDPE/rHDPE01 blends having 0%, 25%, 50%, 75% and 100% recycled material. The 25/75 and 50/50 blends' results displayed a peak impact energy similar to MDPE, suggesting that nearly the same impact property with up to 50% less virgin polymer can be achieved. For higher amounts of rHDPE, the blends became brittle and less resistant to impact, with a decrease of 42-74%. The trend curve in Figure 1 indicates that, within the studied blends, the 50/50 ratio presented the best relationship between impact resistance and recyclate content, representing the ideal amount of recyclate to prepare the remaining rHDPE02 and rHDPE03 mixes.
Concerning the 50/50 MDPE/rHDPE blends, the presence of recyclates has maintained/improved the impact resistance of pure MDPE (Figure 2A), being rHDPE03 the one with the highest percentage (18%) of increase. As the recyclates were sourced from different post-consumed artefacts, the higher impact resistance of rHDPE02 and 03 can be ascribed to a softer structure that may be a consequence of factors like the presence of other polymers/contaminants, broader polydispersion, degradation of polymeric chains, amongst others. These factors may have altered/hindered the crystalline packing of HDPE molecules, consequently reducing the degree of crystallinity and increasing the blend's impact resistance as a whole.

The impact resistance of the rotomoulded samples (Figure 2B) was much lower - around 85-87% -than that of pure MDPE. This decrease may be due to the lack of orientation and shear in the rotational moulding processing, if compared to the compression moulded samples. Plus, the crystallisation process of the blends inside the rotational-moulding mould happened in conditions (time and temperature) different than that of the platen press. Longer cooling times and higher temperatures might have led to the formation of a less cohesive structure with lower impact properties. As seen in literature, binary polymer blends having one component less crystalline than the other tends to segregate the amorphous diluent during the solidification process when given favourable time/temperature conditions [32–34]. Additional issues might have contributed to lower the impact properties of the rotomoulded blends. In comparison with the MDPE samples (Figure 3A), the blends samples were thicker (5-7 mm) than expected (4 mm), having a rough inner surface (Figures 3B to 3D). This uneven surface could indicate the heterogeneous structure formed in the rotomoulded part, where inner layers did not melt fully, creating a discontinuous structure that fractured more easily when submitted to the tensile efforts of the punctural impact test.

### 3.2 Flexural modulus
There was a decrease of 20-30% in the flexural modulus of all blends if compared to pure MDPE. Due to the high standard deviation of rHDPE01 results (Figure 5), no significant difference can be inferred from this test regarding the effect on the flexural modulus of each individual rHDPE. But there was a slightly higher flexural modulus in the rHDPE01 blend. In combination with the difference in the peak impact energy from the impact test, the rHDPE01 blend seems slightly stiffer than the other two blends. In general, the same issues that affected the impact test's samples – wall thickness and discontinuous structure of the part – might have influenced flexural results.

### 3.3 Melt flow rate
We notice a drastic decrease in the blend's flowability as we increase the rHDPE01 content (Table 3), which may be explained by the molecular structure of HDPE – higher molar mass and longer linear chains, with the tendency to entangle and reduce the flow capability of the molten blend. In binary polymer blends, the material with higher MFR tends to work as a diluent, increasing the flowability of higher molar mass polymer in the melt state [33].

In the 50/50 blends, the higher MFR results of rHDPE02 and rHDPE03 mixtures indicates that these recyclates might be mixed with other polymers of more flowability than HDPE. It may explain their higher impact results in the peak impact energy of the compression moulded samples for the rHDPE02 and rHDPE03 blends.

### 3.4 Differential scanning calorimetry
The DSC analysis of MDPE/ rHDPE01 blends (Table 4) showed the mutual interference between the materials in the degree of crystallinity ($X_c$) of the blend. The MDPE/ rHDPE01 blends displayed single $T_m$ temperatures peaks (127, 129 and 131°C) closer to the pure rHDPE01 (133°C), showing that the crystalline material melting in the blend was predominantly HDPE structures. The lowest
content of rHDPE01 seems to have hindered the crystallisation process of the MDPE molecules, and this MDPE/HDPE ratio has prevented the packaging of the long linear chains of HDPE [35]. But in the 50/50 blend, the crystallinity of the blend raised by 20%, denoting that the MDPE has worked as a diluent and facilitated the motion of rHDPE01 molecules in the melt towards the growing crystals. The $T_c$ temperatures showed that the blend's crystallisation process was dominated by HDPE molecules, which crystallised first at 119°C and solidified the polymer mass, creating a barrier to the movement of MDPE chains towards the crystallisation centres. The crystallisation dynamics of HDPE occurs much faster than that of less linear PE structures, and the less-crystalline MDPE's amorphous phase furthured the diffusion of the crystallising front of HDPE. This quick solidification process of rHDPE01 that left MDPE more amorphous can be related to the better impact results of the 50/50 blend. Regarding the other two 50/50 MDPE/rHDPE blends, the one with rHDPE02 showed the lowest $T_m/\Delta H_m$ and more considerable decrease in the $X_c$ (11%), denoting a HDPE with a less organised crystalline structure [33].

Differently from rHDPE01, the DSC curves of rHDPE02 and 03 (Figure 6) show a small melting peak around 160°C, typical of polypropylene (PP) ones, as seen in literature [36, 37]. This peak indicates the presence of some PP in the 02 and 03 blends and may explain their higher MFR and impact results.

4 Conclusions

In the blends having different MDPE/rHDPE01 contents, the $T_m$ temperatures obtained in the DSC analysis suggested that HDPE has dominated the crystallisation process of the blends, leaving the MDPE more amorphous and compensating for the stiffness HDPE in the impact resistance. Regarding the different types of rHDE, the results highlighted the presence of another polymer mixed to the rHDPE02 and 03 - probably PP, due to a peak observed around 160°C. The blends containing these less crystalline recyclates showed higher MFR and better impact performance. Using the impact resistance results of compression moulded samples as the criterion, the scanning for the ideal amount of recyclate in the rHDPE01 blends showed that the 50/50 ratio displayed results similar to those of MDPE. The other 50/50 blends had slightly higher impact resistance, probably containing some PP in the rHDPE02 and 03 recyclates. In this case, where the impact resistance is one of the most important mechanical properties of rotemoulded parts, the usually unwanted PP contamination of the recycled HDPE has had a positive effect in the blend's intended application. Although the rotemoulded samples did not achieve the same performance of the compression moulded ones in the impact test, measures like reducing the wall thickness of the part and the particle size of the ground powder could lead to more reliable results. Improving the formulation of virgin/recycled PE blends for the rotemoulding industry can be a pathway to increase the use of post-consumer plastics in the Circular Economy.

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References


17. La Mantia F.P., Mistretta M.C., Titone V. An additive model to predict the rheological and mechanical properties of polypropylene blends made by virgin and reprocessed components. *Recycling*, 2021, 6, 2.


Table 1: Processing conditions of the platen press.

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<th>Pressure (MPa)</th>
<th>Time (s)</th>
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<tr>
<td>1</td>
<td>190</td>
<td>0</td>
<td>60</td>
</tr>
<tr>
<td>2</td>
<td>200</td>
<td>5</td>
<td>30</td>
</tr>
<tr>
<td>3</td>
<td>220</td>
<td>1</td>
<td>30</td>
</tr>
<tr>
<td>4</td>
<td>220</td>
<td>1</td>
<td>30</td>
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<tr>
<td>5</td>
<td>20</td>
<td>2</td>
<td>120</td>
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Table 2: Impact resistance of the materials.

<table>
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<tr>
<th>Material</th>
<th>Peak energy (J)</th>
<th>Peak impact energy (J.mm⁻¹)</th>
<th>Total impact energy (J.mm⁻¹)</th>
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<tr>
<td>MDPE/rHDPE01 100/0</td>
<td>19.4 ± 1.2</td>
<td>7.9 ± 0.2</td>
<td>15.6 ± 0.4</td>
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<td>MDPE/rHDPE01 75/25</td>
<td>17.2 ± 1.2</td>
<td>7.5 ± 0.5</td>
<td>14.6 ± 0.6</td>
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<td>MDPE/rHDPE01 50/50</td>
<td>21.3 ± 2.1</td>
<td>8.0 ± 0.8</td>
<td>16.4 ± 0.8</td>
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<td>MDPE/rHDPE01 25/75</td>
<td>14.0 ± 3.7</td>
<td>4.6 ± 1.2</td>
<td>10.3 ± 2.0</td>
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<tr>
<td>MDPE/rHDPE01 0/100</td>
<td>8.3 ± 0.8</td>
<td>2.9 ± 0.4</td>
<td>7.5 ± 1.6</td>
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<td>MDPE/rHDPE02 50/50</td>
<td>19.5 ± 1.4</td>
<td>9.1 ± 0.4</td>
<td>16.7 ± 0.5</td>
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<tr>
<td>MDPE/rHDPE03 50/50</td>
<td>19.9 ± 1.0</td>
<td>9.3 ± 0.3</td>
<td>15.6 ± 0.3</td>
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Table 3: Melt-flow rate of the materials.

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<tr>
<th>Material</th>
<th>MFR (g.10 min⁻¹)</th>
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<tr>
<td>MDPE/rHDPE01 100/0</td>
<td>6.37</td>
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<td>1.95</td>
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<td>MDPE/rHDPE01 50/50</td>
<td>0.67</td>
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<td>MDPE/rHDPE01 25/75</td>
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<td>MDPE/rHDPE03 0/100</td>
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Table 4: Differential scanning calorimetry data of the materials.

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<th>$T_c$ (°C)</th>
<th>$\Delta H_m$ (J.g⁻¹)</th>
<th>$X_c$ (%)</th>
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<tr>
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<td>122</td>
<td>105</td>
<td>133</td>
<td>45</td>
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<td>43</td>
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<td>MDPE/rHDPE01 50/50</td>
<td>129</td>
<td>112</td>
<td>159</td>
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<td>133</td>
<td>114</td>
<td>170</td>
<td>58</td>
</tr>
<tr>
<td>MDPE/rHDPE02 50/50</td>
<td>128</td>
<td>112</td>
<td>119</td>
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<td>MDPE/rHDPE02 0/100</td>
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<td>113</td>
<td>114</td>
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<td>MDPE/rHDPE03 50/50</td>
<td>128</td>
<td>112</td>
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<tr>
<td>MDPE/rHDPE03 0/100</td>
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<td>113</td>
<td>123</td>
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Figure 1: Trend curve of peak impact energy of MDPE/rHDPE01 blends.

Figure 2: Peak impact energy of (A) MDPE and 50/50 MDPE/rHDPE blends, and (B) rotomoulded MDPE and 50/50 MDPE/rHDPE blends.

Figure 3: Perforated impact samples of (A) MDPE, and 50/50 (B) MDPE/rHDPE01, (C) MDPE/rHDPE02 and (D) MDPE/rHDPE03 blends.

Figure 4: The inner surface of rotomoulded parts of 50/50 MDPE/rHDPE blends.

Figure 5: Flexural moduli of rotomoulded MDPE and 50/50 MDPE/rHDPE blends.

Figure 6: 2nd heating DSC curves of 50/50 MDPE/rHDPEs blends.
Figure 1. Trend curve of peak impact energy of MDPE/rHDPE01 blends

25x17mm (600 x 600 DPI)
Figure 2. Peak impact energy of (A) MDPE and 50/50 MDPE/rHDPE blends, and (B) rotomoulded MDPE and 50/50 MDPE/rHDPE blends

100x55mm (300 x 300 DPI)
Figure 3. Perforated impact samples of (A) MDPE and 50/50 (B) MDPE/rHDPE01, (C) MDPE/rHDPE02 and (D) MDPE/rHDPE03 blends

68x69mm (72 x 72 DPI)
73x69mm (72 x 72 DPI)
69x69mm (72 x 72 DPI)
63x69mm (72 x 72 DPI)
Figure 4. The inner surface of rotomoulded parts of 50/50 MDPE/rHDPE blends

133x99mm (72 x 72 DPI)
Figure 5. Flexural moduli of rotomoulded MDPE and 50/50 MDPE/rHDPE blends

93x56mm (330 x 330 DPI)
Figure 6. 2nd heating DSC curves of 50/50 MDPE/rHDPEs blends

170x112mm (158 x 158 DPI)