Modification of Poly(ethylene terephthalate) Fibre by Polymer Blending with Poly(butylene terephthalate) Fibre: Part II—Sequential Melt-blending and Spinning

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The blend fibres prepared from poly(ethylene terephthalate) and 0-10% poly(butylene terephthalate) by sequential melt-blending and spinning show a single glass transition temperature, less than that of 100% PET. The melting point of the blend fibres is lower than that of 100% PET owing to decrease in crystalline order and partial transesterification. The thermal stability of the blend fibre decreases on addition of more than 3% PBT. Addition of 3% poly(butylene terephthalate) to poly(ethylene terephthalate) increases birefringence, initial modulus, sonic modulus and tenacity, which may be associated with higher inter-crystalline links.

Keywords: Melt-blending, Poly(butylene terephthalate), Poly(ethylene terephthalate), Polymer blending, Spinning

1 Introduction

Significant improvements in the properties of poly(ethylene terephthalate) [PET] fibre can be achieved by blending it with poly(butylene terephthalate) [PBT]. The relevant literature and the theoretical predictions of compatibility have been reported earlier. In this work, the blend fibres have been prepared from PET and 0-10% PBT by sequential melt-blending and spinning and their characteristics have been studied.

2 Materials and Methods

The polymers chosen for the present work were all commercial grades of PET and PBT in the form of chips. The characteristics of the chips have been reported earlier. The amount of PBT was restricted to maximum 10%, where the polymers are expected to be compatible. The blend compositions were 100/0, 97/3, 95/5 and 90/10, where the first number represents PET and the second one PBT weight per cent.

The melt-blending was done in a 3-neck flask at 295°C in nitrogen atmosphere with continuous stirring for 5 min. The blended chips were dried under vacuum at 70°C for 12 h. The fibres were prepared in a laboratory-scale melt-spinning unit under nitrogen atmosphere at 275, 274, 270 and 268°C respectively for 100/0, 97/3, 95/5 and 90/10 PET/PBT blends. The winding speed was kept at 50 m/min. The as-spun material was drawn in a laboratory-scale stretching unit to 3.5 x at 115°C.

Differential thermal analysis and thermogravimetric analysis of the fibres were carried out by using Stanton Redcroft's differential thermal analyser and thermogravimetric analyser at a heating rate of 10°C/min.

X-ray diffraction pattern of the powdered samples using CuKα radiation was used to measure the crystallinity of the fibres. The standard amorphous pattern was separately obtained for each fibre by getting diffractogram of 100% amorphous polymer blend in the film form. Birefringence was measured by using a Leitz microscope and a compensator.

The load-elongation curve was obtained under standard conditions on an Instron tensile tester (model 1112) at 50% per minute strain rate. Stress, strain and modulus were calculated. Sonic modulus was measured on a dynamic pulse propagation meter at 5 kHz.

3 Results and Discussion

Table 1 shows glass transition temperature (Tg), crystallization temperature (Tc), melting temperature (Tm), heat of crystallization (ΔHc) and heat of
fusion ($\Delta H_f$) values obtained from DTA thermograms. In all the cases, only a single value of $T_g$ is observed, which varies with the composition of blend. This implies that the minor component is in the amorphous state in the blends. A single glass transition temperature and lowering of its value in the blends in comparison with pure PET indicates that the two polymers are miscible in the amorphous state.

The crystallization temperature shows that there is a difference of 9.5°C between 100/0 and 90/10 PET/PBT blends. The lowering of the crystallization temperature is related to the lowering of the glass transition temperature of the blend fibres. The melting temperature decreases for the blends. The melting point depression may be the result of several phenomena such as compatibility in the amorphous state, smaller crystallite size and decreasing crystalline order. However, a large drop in melting temperature for 90/10 PET/PBT fibre may be owing to the partial transesterification of the blend as suggested by Li and Wong, and Herlinger et al., The degree of transesterification depends upon the time of mixing and it influences the melting temperature of the blend. For no transesterification, the melting points of each component, in each molar ratio, should be recognizable near each other.

The values of $\Delta H_f$ and $\Delta H_i$ increase with the addition of PBT component. $\Delta H_f$ is greater than $\Delta H_i$. It has been reported that the crystallization process is hindered in blends of PET and low percentage of PBT. Also, the spherulitic structure is disordered and the spherulites become larger with the addition of small amounts of second component. On heating to higher temperatures in DTA, the perfection of the imperfect crystallites of spherulites increases within very short period, which increases $\Delta H_f$ value. So, it might be possible that the addition of PBT formed more imperfect crystallites, which increase $\Delta H_f$ value.

Initial decomposition temperature (IDT), integral procedural decomposition temperature (IPDT) and maximum decomposition temperature (TD$_{max}$) for the blend fibres were calculated from TGA thermograms and are given in Table 2. 97/3 PET/PBT blend fibre shows higher thermal values than the pure PET fibre. The values decrease with further increase in PBT content. The decrease in thermal stability values can be attributed to the introduction of more flexible butyl groups which make the chain prone to degradation at a comparatively lower temperature. Thermal degradation of polyester has been investigated by many workers, which originates in the cleavage of polyester chain.

Crystallinity decreases and birefringence increases by blending (Table 3). Higher birefringence values indicate high orientation for the blended filaments. Since PBT is present in small quantities, it may not be able to crystallize independently. Instead, it will restrict the crystallization process, resulting in a higher orientation at the expense of crystalline areas.

Table 4 shows the mechanical properties of blends. The tenacity value slightly increases for 97/3 PET/PBT fibre. Elongation, sonic modulus and initial modulus are also high for this fibre. The high modulus value can be due to high orientation. The improvement in properties in terms of modulus

<table>
<thead>
<tr>
<th>Sample</th>
<th>IDT °C</th>
<th>IPDT °C</th>
<th>FDT °C</th>
<th>TD$_{max}$ °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>100/0</td>
<td>439</td>
<td>549</td>
<td>576</td>
<td>512</td>
</tr>
<tr>
<td>97/3</td>
<td>444</td>
<td>539</td>
<td>637</td>
<td>522</td>
</tr>
<tr>
<td>95/5</td>
<td>425</td>
<td>535</td>
<td>594</td>
<td>512</td>
</tr>
<tr>
<td>90/10</td>
<td>415</td>
<td>527</td>
<td>567</td>
<td>500</td>
</tr>
</tbody>
</table>

IDT—Initial decomposition temperature.
IPDT—Integral procedural decomposition temperature.
FDT—Final decomposition temperature.
TD$_{max}$—Maximum decomposition temperature.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Crystallinity %</th>
<th>Birefringence</th>
</tr>
</thead>
<tbody>
<tr>
<td>100/0</td>
<td>59</td>
<td>0.0824</td>
</tr>
<tr>
<td>97/3</td>
<td>57</td>
<td>0.0978</td>
</tr>
<tr>
<td>95/5</td>
<td>53</td>
<td>0.0897</td>
</tr>
<tr>
<td>90/10</td>
<td>52</td>
<td>0.0825</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample</th>
<th>Tenacity g/d</th>
<th>Elongation %</th>
<th>Initial modulus g/d</th>
<th>Sonic modulus g/d</th>
</tr>
</thead>
<tbody>
<tr>
<td>100/0</td>
<td>2.48</td>
<td>41</td>
<td>150</td>
<td>123</td>
</tr>
<tr>
<td>97/3</td>
<td>2.50</td>
<td>48</td>
<td>178</td>
<td>126</td>
</tr>
<tr>
<td>95/5</td>
<td>2.33</td>
<td>43</td>
<td>187</td>
<td>117</td>
</tr>
<tr>
<td>90/10</td>
<td>2.22</td>
<td>40</td>
<td>144</td>
<td>105</td>
</tr>
</tbody>
</table>

$T_g$—Glass transition temp.; $T_c$—Crystallization temp.; $T_m$—Melting temp.; $\Delta H_f$—Heat of crystallization; and $\Delta H_i$—Heat of fusion.
and strength may be because of the formation of inter-crystalline links\textsuperscript{17,18}. Li and Wong\textsuperscript{10} have shown that the morphology, domain structure and number of tie points have a significant impact on the orientation efficiency of PET/PBT blended film and its mechanical properties.

90/10 PET/PBT fibre shows a lower modulus than PET. The modulus of PBT fibre is 6-7 times lower than that of PET fibre owing to more contracted chain conformation in the crystalline region\textsuperscript{19}. This results in a lower modulus. The tenacity of this blend shows a lower value owing to phase segregation and independent crystallization\textsuperscript{12}.

4 Conclusions

4.1 The blend fibres show single glass transition temperature, less than that of 100% PET, indicating miscibility of the two polymers in the amorphous state.

4.2 90/10 PET/PBT blend has a comparatively low melting point owing to partial transesterification.

4.3 Addition of more than 3% PBT to PET decreases the thermal stability of the blend fibre.

4.4 97/3 PET/PBT blend shows better thermal stability and improved mechanical properties, such as tenacity, initial modulus and sonic modulus, which may be because of the formation of higher inter-crystalline links.

References


