On the recyclability of polyamide for sustainable composite structures in civil engineering

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ABSTRACT

Previous studies have highlighted the potential application of Polyamide 6 (PA6) as excellent recyclable polymeric material, which is well suited to form carbon-fiber-reinforced, graphene-nanoplatelets-reinforced or metal-porous-polymer composite structures. The conventional screw extrusion process is one of the established melt processing techniques, having potential of enhancing mechanical, thermal and morphological properties of polymeric materials. PA6 is an important engineering material which exhibits excellent mechanical properties, chemical resistance, wear resistance, dimensional stability, and low coefficient of friction. In the present study attempts have been made to explore the behavior and characteristics of the recycled PA6 polymer through twin screw extrusion process towards increase in recyclability (as primary recycling process). The mechanical properties (tensile strength, Young’s modulus, peak load), metallurgical properties (porosity, wear and material loss) supported with optical microscopy were investigated to ensure the recyclability of PA6 as a properties enhancing extrusion process. It has been observed in the present study that, melt processing by screw extrusion at best settings of input process parameters enhances the material properties for applications in sustainable civil engineering.

Keywords: Polyamide, recycling, mechanical properties, wear, screw extrusion, composite structures, civil engineering

1. Introduction

Polyamide 6 (PA6) is being increasingly considered as a convenient matrix material in light-weight carbon-fiber-reinforced, graphene-nanoplatelets-reinforced structures or reinforcing fibers of cement mortars [1-4]. The recycling of such a material is attracting increasing attention, on considering that several advances have been made in the technology for recycling of plastic solid waste over the last 20 years [5]. Polymer recycling is majorly sub-divided in either chemical recycling or mechanical recycling. De-polymerization by chemical recycling is a way to replay original monomers [6]. However, complexity of systems and high cost of operation are some of difficulties of this method. The burning of polymers to recover energy is used too. In this situation, environment benefits are questionable [7]. So, chemical recycling is not considered as a reliable technique for feedstock preparations because of hazards and uneconomical processing, so mechanical recycling process is often preferred as feedstock preparations of fibrous polymer like PA6. Mechanical recycling is an interesting alternative which can combine technical viability, acceptable costs and environmental benefits. The contamination with impurities and others polymer as well as the degradation of material are the main difficulties for this method of recycling of polymers [8]. Mechanical recycling process is usually responsible for the reduction in mechanical properties such as tensile or impact strength, but this process is capable for preparing material that yield for the melt compounding and processing (e.g. fused deposition modeling, injection molding and compression molding). The PA6 is a semi-crystalline engineering thermoplastic with good mechanical properties, but suffers from high moisture uptake, poor resistance to thermal oxidation and UV radiation, and low impact strength under low-temperature or dry condition.
Polyamides (PAs) are widely used in many engineering applications due to their very interesting mechanical properties and ease of processing, as they allow the injection of thick components with complex geometries [10]. As reported in literature PA6 is an important engineering material which exhibits excellent mechanical properties, chemical resistance, wear resistance, dimensional stability, low friction, etc [11-12]. So one of the most often used engineering plastic is PA6 because of its quite good mechanical (strength, hardness, toughness, damping) and tribological (sliding, wearing resistance) properties [13-14]. Hence, it has found various applications in industrial and domestic sectors, such as transport, aviation, sealing and coating, etc. [15]. For such applications, reinforced compounds and additives are used in order to increase the performance of the polymer materials. The additives can be classified according to the function as antistatic, fillers, and lubricants, fire resistant and anti-oxidation agents [16]. Reusing and recycling PA6 from waste can be achieved in different ways, including: (i) de-polymerization of PA6 chains into their monomers or oligomers [17-18] (ii) extraction and separation of polymeric components without significant degradation [19-20] and (iii) melt compounding of the waste stream with additives and modifiers [21-22]. PA6 fibers are tough, possessing high tensile strength, as well as elasticity and luster. These are wrinkle proof and highly resistant to abrasion and chemicals such as acids and alkalis. The fibers can absorb up to 2.4% of water, although this lowers tensile strength. The glass transition temperature of PA6 is 47°C [23]. The most common feedstock filament which is mechanically recycled is PA6 fibers known for its melt processing capabilities [24]. PA6 can be considered as human-made long chain polymeric material. It is widely used, economical materials characterized by excellent all-round properties, easy moulding and manufacturing. Traditionally PA6 is a very stable polymer and not readily degraded in the ambient environment. As a result, environmental pollution from synthetic plastics has been recognized as a large problem [25]. Government, researchers, and social organization worked on this issue to resolve it by improving reusability with objective of wire extrusion, additive manufacturing techniques or by making liquid fuel using thermal cracking process, [26-27]. Compared to the recycling process of metals, glass and alloys, PA6 polymers recycling is often more challenging because of low density. Various studies have been reported for significant improved properties of polymers with aid of micro or nano-composites. Silica nano powder (<30nm) at certain level incorporated to PA6 matrix confirmed better mechanical properties to the base polymer [28], Zinc oxide (ZnO) incorporated to the PA6/PBT (butylene terephthalate) blend matrix was resulted into finer morphology but caused deterioration in melt rheological properties [29]. Polyhedral oligomeric silsesquioxane (POSS) at 1-5wt% mixed with PA6/PP (polypropylene) blend ensures the valuable changes in the mechanical, morphological and thermal properties. Presence of POSS decreased the melt flow index, increased the yield and impact strength and improves the degree of crystallinity [30]. One of the studies reveals that the incorporation of calcium oxide (CaO) in PA6 provided the better thermal stability [31]. Thermoplastic polymers are mainly processed via melt processes like extrusion or molding (e.g. injection molding, screw extrusion) for distinguish applications like part fabrication or feedstock filament preparations. Some of the studies have been demonstrated the effect of single screw or double screw extrusion, resulted into the finer morphology, improved mechanical properties and thermally stable products [32-35]. Injection molding concept is widely reported as for development of multi-functioning component with provision of either no loss or improvement of material characteristics. Various destructive or non-destruction testing like; X ray diffraction (XRD), Tensile properties, hardness measurement, Differential scanning calorimetry (DSC), Scanning electron microscopy (SEM), Small Angle X-ray Scattering (SAXS) and Transmission Electron Microscopy (TEM) etc. have been justified the enhancement in material characteristics after melt processing [36-39]. Some of the recent studies highlighted the cross-linking of PA6 polymer chain [40], use of diamond nano-particles in PA6 matrix [41], effect of inducing water in PA6 and potential of PA6 for various industrial applications [42-43].

The literature review reveals that the use of thermoplastics with metallic and non-metallic fillers improves the rheological, mechanical, thermal and morphological properties. Only some of the studies have been reported which explain the use of melt processing (which confirms the property enhancement of the polymers). In the present study, application of twin screw extrusion process has been demonstrated to use this process as a recyclability tool without losing their mechanical, morphological and thermal properties.
2. Materials and Methods

2.1 Material Selection

For present study primary recycled PA6 in granules form has been selected as target material for investigation because of their all-round material properties and melt processing capabilities. The PA6 having poor biodegradability to the atmosphere that is the reason it is most likely to be recycled [44]. Fig. 1 shows the granules of PA6 imported from laboratory of Department of Civil Engineering, University of Salerno, Italy.

PA6 is semi crystalline polyamide which is most commonly known as nylon 6 or polycaprolactam. The PA6 fibers are tough, exhibits very high tensile properties, elongation and luster. Very good chemical resistivity and low wear rate enable it to be applicable in wide area. It is widely used for fabrication of gear, bearing, fittings and other automobile part via extrusion, molding or machining. Table 1 shows the mechanical, thermal and chemical characteristic of PA6 polymer.

**Table 1**

Properties of PA6 [45-49]

<table>
<thead>
<tr>
<th>Chemical formula</th>
<th>Tensile strength (kgf/mm²)</th>
<th>Flexural strength (kgf/mm²)</th>
<th>Chemical resistivity</th>
<th>Melting point (°C)</th>
<th>Glass transition temperature (°C)</th>
<th>Specific heat capacity (J/kg.K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(C₆H₁₁NO)n</td>
<td>4.22-16.88</td>
<td>274.27-773.59</td>
<td>Good to excellent</td>
<td>215</td>
<td>46</td>
<td>1600</td>
</tr>
</tbody>
</table>

2.2 Methods of extrusion melt processing

There is variety of screw extrusion processes available for producing feedstock filament for FDM. Single screw extrusion is a conventional process for producing feedstock filament but defects like, tiny pores, blow holes, non-
mixing are the major problems associated with this process. Twin screw extrusion has emerged as an advanced technique for producing feedstock filament free from defects. Twin-screw extruder are capable to ensure mixing, shearing, cooling, heating, compressing, transporting, shaping, pumping, etc. with very high level of flexibility. The main advantages of twin-screw extruders (intermeshing co-rotating) are their exceptional mixing capability that gives the remarkable characteristics to extruded products. In the twin-screw extrusion process, the raw materials may be solids (granules, powders & flours), slurries, liquids, and possibly gases [50-52]. Fig. 2 shows the available screw extrusion processes and their variant for producing feedstock filament.

3. Experimentation

3.1 DSC

In the present study twin screw extrusion was taken as melt processing technique to investigate the effect of process parameters on properties of feed stock filament. For the extrusion process temperature is one of the most significant factors for changes in material properties. So it was needed to evaluate the ranges of temperature on which the extrusion should be preceded and it must be decided via results based upon DSC. For determining of melting peak temperature of recycled PA6 granules thermal analysis was performed. For thermal analysis METTLER TOLEDO, Model DSC3, Swiss make with (SW 14.00) software was used in N2 gas environment. The typical DSC setup determines the behavior of applied samples by taking references from standard sample, both enclosed in a metallic crucible (Al or platinum). As shown in Fig. 3, DSC sensor uses two crucibles for heating and cooling, one for reference and another for sample.
For thermal analysis, 03 heating-cooling cycles were selected. In first cycle, initially heat the sample in comparison to reference from 30°C to 250°C at the rate of 10K/min, after that in the presence of N₂ gas (flow rate is 50.0 ml/min) cooling process was performed in which matter was cooled from 250°C to 30°C at the rate of 10K/min. After that in the second and third cycle, same process was repeated.

It has been observed (see Fig.3) that melting point temperature ($T_m$) of PA6 was 220.03°C in first heating cycle and 219.40°C in second and 218.91°C in third cycle respectively. This observation shows that for preparations of feed stock filament as a melt processing the working temperature must be greater than 220.03°C temperature. The primary recycled PA6 has passed through 03 consecutive heating cycles, having a matter of observation from thermal stability point of view. After 03 heating cycles it was observed that very little or ignorable changes of melting point and net energy intake (Integral energy) have occurred which shows the excellent stability of selected recycle PA6. Similar solidification rage in each cycles shows that there was very less degradation (See Fig. 4). So based upon these observations from the DSC results, the melt processing of recycled PA6 can be performed up to 250°C.

![Fig. 3 (a) DSC setup and (b) heating chamber for sample](image-url)
3.2 Design of experiment

Based upon the DSC result for primary recycled PA6, the temperature as one of the input process parameter for twin screw extrusion is employable to be taken in certain range. In the present study level of screw temperature was judicially taken under 250°C as 235, 240, and 245°C to check the effect of temperature variation over characteristics of PA6. Similarly applied load and torque with their level were judicially selected as shown in Table 2. For this study, commercial make: HAAKE Mini CTW, Germany, twin screw extruder has been used. The feed stock filament were prepared as per Taguchi L9 (3^3) orthogonal array. Table 2 shows list of input factors and their levels (based upon pilot experimentation) for the experimental study.

### Table 2

Parameter selected for experimentation

<table>
<thead>
<tr>
<th>Levels</th>
<th>Temperature (in °C)</th>
<th>Load (in kgf)</th>
<th>Torque (in N-m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>235</td>
<td>5</td>
<td>0.10</td>
</tr>
<tr>
<td>2</td>
<td>240</td>
<td>10</td>
<td>0.125</td>
</tr>
<tr>
<td>3</td>
<td>245</td>
<td>15</td>
<td>0.15</td>
</tr>
</tbody>
</table>

Based upon Table 2, Table 3 shows the control log of experimentation based upon Taguchi L9 (3^3) orthogonal array which was 9 runs of experimentation.
Table 3
Control Log of experimentation

<table>
<thead>
<tr>
<th>Experiment No.</th>
<th>Temperature (in °C)</th>
<th>Load (in kgf)</th>
<th>Torque (in N-m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>235</td>
<td>5</td>
<td>0.10</td>
</tr>
<tr>
<td>2</td>
<td>235</td>
<td>10</td>
<td>0.125</td>
</tr>
<tr>
<td>3</td>
<td>235</td>
<td>15</td>
<td>0.15</td>
</tr>
<tr>
<td>4</td>
<td>240</td>
<td>5</td>
<td>0.125</td>
</tr>
<tr>
<td>5</td>
<td>240</td>
<td>10</td>
<td>0.15</td>
</tr>
<tr>
<td>6</td>
<td>240</td>
<td>15</td>
<td>0.10</td>
</tr>
<tr>
<td>7</td>
<td>245</td>
<td>5</td>
<td>0.15</td>
</tr>
<tr>
<td>8</td>
<td>245</td>
<td>10</td>
<td>0.10</td>
</tr>
<tr>
<td>9</td>
<td>245</td>
<td>15</td>
<td>0.125</td>
</tr>
</tbody>
</table>

Following Table no. 3 feed stock filament through extrusion process was prepared at different parametric conditions. For the investigation of tensile properties tensile test were performed on UTT (Universal tensile tester, Make: Shanta Engineering, India). Table 4 shows that different output parameters of PA6 feed stock filament for tensile properties.

Fig. 5 shows the extruded feedstock filament at different experimental conditions as per Taguchi L9 orthogonal array.

Table 4
Output parameters of PA6 feed stock filament for tensile properties

<table>
<thead>
<tr>
<th>Exp No.</th>
<th>Load at Peak (N)</th>
<th>Tensile Strength at Peak (MPa)</th>
<th>Youngs Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>193.55</td>
<td>6.35</td>
<td>263.17</td>
</tr>
<tr>
<td>2</td>
<td>181.39</td>
<td>4.37</td>
<td>221.93</td>
</tr>
<tr>
<td>3</td>
<td>61.29</td>
<td>5.15</td>
<td>201.29</td>
</tr>
<tr>
<td>4</td>
<td>164.84</td>
<td>5.54</td>
<td>264.84</td>
</tr>
<tr>
<td>5</td>
<td>53.13</td>
<td>5.93</td>
<td>213.13</td>
</tr>
<tr>
<td>6</td>
<td>205.16</td>
<td>9.02</td>
<td>285.16</td>
</tr>
<tr>
<td>7</td>
<td>66.85</td>
<td>6.6</td>
<td>206.85</td>
</tr>
<tr>
<td>8</td>
<td>183.67</td>
<td>8.93</td>
<td>283.87</td>
</tr>
<tr>
<td>9</td>
<td>163.85</td>
<td>6.16</td>
<td>263.35</td>
</tr>
</tbody>
</table>
Based upon Table 4, combined optimization of tensile properties (Peak Load, Strength at Peak, Youngs Modulus) was performed by using Minitab17 software. After optimization it was observed that experiment run no. 06 was considered as the best conditions for preparation of feed stock filament and experiment run no. 02 was the worst based upon mechanical strength point of view.

3.3 Melt flow index

Melt flow index represents the material flow behaviour and quality of thermoplastic materials, [53]. Many researchers have investigated and established the relationship of melt flow index with numerous mechanical, chemical and thermal properties like, Yield stress, Viscosity, molecular weight distribution and shear [54-59]. The ASTM D1238 is applicable for most of the thermoplastic materials. The 2.16 Kg load was applied at 235°C through piston and material is collected per 10 min for determination of melt flow index The granule of feed stock filament is put into the pre-heated barrel of MFI tester. The weight as per the ASTM standard (D 1238-95) was put on the piston to expel the molten material from barrel and thereby made to exit out of die opening as extrudate and weighed to find MFI in terms of gm/10min. Melt flow analysis were performed to check the changes in the flow behavior of the material after melt processing. Fig. 6 shows that schematic of melt flow index tester.

![Fig. 6 Schematic of MFI Tester](image)

Testing has been conducted to determine the tensile behavior of the extruded feedstock filament as shown in Table 4. The tensile results was obtained best at experiment no. 6 and worst at experiment no. 2 following design of experiment from table 3. The best and worst feedstock filaments are then tested to check the changes in the flow rate of after melt processing (See Fig. 7).
3.4 Wear properties

The wear properties have been checked as material removal, coefficient of friction and wear rate of that specific samples. All the properties have been checked by 'pin on disk setup' with pin diameter 10mm at operating conditions mentioned in Table no. 5. Fig. 8 shows the experimental pin on disk setup and prepared pin of recycled granules, extruded filament of experiment no. 6 and 2.

Table 5

Operating condition for wear testing through pin on disk setup
<table>
<thead>
<tr>
<th>Static parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Load</td>
<td>1 kgf</td>
</tr>
<tr>
<td>Rotational Speed</td>
<td>250 RPM</td>
</tr>
<tr>
<td>Track diameter</td>
<td>76 mm</td>
</tr>
<tr>
<td>Cycle time</td>
<td>15 min</td>
</tr>
</tbody>
</table>

4. Result and discussions

In the present study the analysis has been made from thermal, mechanical and morphological point of view to investigate the effect of melt processing on PA6 for potential application. The discussions have been justified with results of DSC, tensile properties, melt flow as rheological properties, porosity as morphology and wear properties.

4.1 Influence of extrusion process parameters on tensile properties.

Following the control log of experimentation based Taguchi L9 orthogonal array. The Young’s modulus with their SN (Signal to noise) ratios has been analyzed. Table 6 shows the value of Young’s modulus and their SN ratios for different experimental conditions.

<p>| Table 6 Outputs of Young’s modulus for different experimental condition |
|-----------------------------|-----------------|-----------------|-----------------|-----------------|</p>
<table>
<thead>
<tr>
<th>Experiment No.</th>
<th>Temperature (°C)</th>
<th>Load (kg)</th>
<th>Torque (N-m)</th>
<th>Youngs Modulus (MPa)</th>
<th>SN Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>235</td>
<td>5</td>
<td>0.100</td>
<td>263.17</td>
<td>48.4047</td>
</tr>
<tr>
<td>2</td>
<td>235</td>
<td>10</td>
<td>0.125</td>
<td>221.93</td>
<td>46.9243</td>
</tr>
<tr>
<td>3</td>
<td>235</td>
<td>15</td>
<td>0.150</td>
<td>201.29</td>
<td>46.0764</td>
</tr>
<tr>
<td>4</td>
<td>240</td>
<td>5</td>
<td>0.125</td>
<td>264.84</td>
<td>48.4597</td>
</tr>
<tr>
<td>5</td>
<td>240</td>
<td>10</td>
<td>0.150</td>
<td>213.13</td>
<td>46.5729</td>
</tr>
<tr>
<td>6</td>
<td>240</td>
<td>15</td>
<td>0.100</td>
<td>285.16</td>
<td>49.1018</td>
</tr>
<tr>
<td>7</td>
<td>245</td>
<td>5</td>
<td>0.150</td>
<td>206.85</td>
<td>46.3131</td>
</tr>
<tr>
<td>8</td>
<td>245</td>
<td>10</td>
<td>0.100</td>
<td>283.87</td>
<td>49.0624</td>
</tr>
<tr>
<td>9</td>
<td>245</td>
<td>15</td>
<td>0.125</td>
<td>263.35</td>
<td>48.4107</td>
</tr>
</tbody>
</table>

As observed from Fig. 9, the temperature at 240°C, Load at 15 kgf and Torque at 0.100 N-m contributed mostly for change in the Young’s modulus. The mentioned levels of input parameters have given best result under consideration.
Table 7 shows the analysis of variance for SN ratio based upon Table 6. As observed from Table 7, Torque is the most important parameter with contribution of 83.49% for Young’s modulus. The P value for torque was calculated as 0.030 which is lesser than 0.05. The P value for torque suggested that it is the significant parameter for extrusion purposes. Table 8 shows the ranking of input process parameters.

**Table 7**

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
<th>Adj SS</th>
<th>Adj MS</th>
<th>F</th>
<th>P</th>
<th>Percentage Contribution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>2</td>
<td>1.4706</td>
<td>1.4706</td>
<td>0.73530</td>
<td>4.85</td>
<td>0.171</td>
<td>12.43</td>
</tr>
<tr>
<td>Load</td>
<td>2</td>
<td>0.1789</td>
<td>0.1789</td>
<td>0.08947</td>
<td>0.59</td>
<td>0.629</td>
<td>1.51</td>
</tr>
<tr>
<td>Torque</td>
<td>2</td>
<td>9.8783</td>
<td>9.8783</td>
<td>4.93915</td>
<td>32.56</td>
<td>0.030</td>
<td>83.49</td>
</tr>
<tr>
<td>Residual</td>
<td>2</td>
<td>0.3034</td>
<td>0.3034</td>
<td>0.15170</td>
<td></td>
<td></td>
<td>2.57</td>
</tr>
<tr>
<td>Total</td>
<td>8</td>
<td>11.8312</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>100.0</td>
</tr>
</tbody>
</table>

Note: DF- Degree of freedom; Seq SS- Sum of square; Adj SS- Adjacent sum of square; F-Fishers value; P- Probability

**Table 8**

Response Table for Signal-to-Noise Ratios Larger is better

<table>
<thead>
<tr>
<th>Level</th>
<th>Temperature</th>
<th>Load</th>
<th>Torque</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>47.14</td>
<td>47.73</td>
<td>48.86</td>
</tr>
<tr>
<td>2</td>
<td>48.04</td>
<td>47.52</td>
<td>47.93</td>
</tr>
<tr>
<td>3</td>
<td>47.93</td>
<td>47.86</td>
<td>46.32</td>
</tr>
<tr>
<td>Delta</td>
<td>0.91</td>
<td>0.34</td>
<td>2.54</td>
</tr>
<tr>
<td>Rank</td>
<td>2</td>
<td>3</td>
<td>1</td>
</tr>
</tbody>
</table>

**4.1.1 Optimized value of Young’s modulus**

For optimization following formula based upon Taguchi design has been used:
\[ \eta_{opt} = m + (m_{A2} - m) + (m_{B3} - m) + (m_{C1} - m) \]

Where ‘m’ is the overall mean of S/N data, \( m_{A2} \) is the mean of S/N data for Composition of materials at level 2 and \( m_{B3} \) is the mean of S/N data for rotational speed at level 3, \( m_{C1} \) is the mean of S/N data for temperature at level 1.

\[ y_{opt}^2 = \frac{1}{10} \eta_{opt} \]

for properties, lesser is better

\[ y_{opt}^2 = \frac{10 \eta_{opt}}{10} \]

for properties, Larger is better

Calculation,

Overall mean of SN ratio (m) was taken from Minitab software.

\[ m = 47.70 \]

Now from response table of signal to noise ratio, \( m_{A1} = 48.04 \), \( m_{B3} = 47.86 \), \( m_{C3} = 48.86 \).

From here,

\[ \eta_{opt} = 47.70 + (48.04 - 47.70) + (47.86 - 47.70) + (48.86 - 47.70) \]

\[ \eta_{opt} = 49.36 \text{ db} \]

\[ y_{opt}^2 = \frac{10 \eta_{opt}}{10} \]

\[ y_{opt} = 293.77 \text{ MPa} \]

Based upon peak plot of parameters shown in Fig 6, the confirmatory experiment for Young’s modulus was conducted for 240\(^\circ\)C temperature with 0.100N-m torque and 15 kg load applied. The observed value was 291.32 MPa which is close to the predicted value.

Similar studies have been performed for peak tensile strength and peak load to ensure the accuracy of the experimental values. The predicted and experimental values for all the tensile properties have been calculated (See Table 9)

**Table 9**

Predicted mechanical properties and experimentally determined values

<table>
<thead>
<tr>
<th>Composition/Proportion</th>
<th>Attribute</th>
<th>Output parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extruded PA6</td>
<td>( \eta_{opt} \text{ (dB)} )</td>
<td>Young’s Modulus (MPa)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>49.36</td>
</tr>
<tr>
<td></td>
<td>Predicted value (( y_{opt} ))</td>
<td>293.77</td>
</tr>
<tr>
<td></td>
<td>Actual value at optimized setup</td>
<td>291.32</td>
</tr>
</tbody>
</table>

4.2 Influences of melt processing on thermal properties.

As shown in Table no. 4 the best tensile value was observed for experiment no. 6 and poorest tensile properties were obtained in experiment no. 2. The thermal changes have been evaluated in comparison with un-extruded grains of PA6. Fig. 10 shows the relative DSC comparison of (1) Un-extruded PA6, (2) Extruded at experiment no. 6 and (3) extruded at experiment no. 2. The results show that the extrusion as melt processing has great effects on the melting point behavior. Experimentation conducted in 3 continuous cycle of heating and cooling explains that extrusion as melt processing lead to increase in the melting temperature and also relative increase in the solidification temperature. Changes occurs in the DSC curve shown in Fig. 10 suggested that the PA6 granules (un-extruded) was least stable from melting and solidification point of view, whereas extruded PA6 were more stable.
4.3 Wear analysis

Wear analysis has been performed on the pin on disk setup to investigate the effects of melt processing on the wear behavior of PA6 as material removal rate, wear in microns, frictional force required and coefficient of friction. Fig. 11 shows the wear track for different samples. Thick wear track for un-extruded PA6 appeared as highest wear indicator, whereas wear track for experiment No. 6 shows thin wear track, hence ensures better wear properties.
4.3.1 Material removal rate (MRR)

The MRR is an indication of the wear characteristics of the materials. More is the material removal means more will be the wear of the material. The MRR has been evaluated by calculating differences in the initial weight and weight after wearing through pin on disk.

\[
\text{MRR} = \text{initial weight of the pin before wear} - \text{final weight of the pin after wear}
\]

The MRR results shown in Fig. 12 suggest that extruded PA6 samples under the desired conditions can reduce the material removal rate as compared to the un-extruded one.

4.3.2 Wear Rate (µm)

Wear rate was appeared similar to the result of the MRR and other properties. The wear rate of extruded sample at exp. No. 6 was measured lowest and at exp. No. 2 highest. The impact of miss-selection of process parameter can lead to increase in the wear rate compared to the un-extruded one as shown in Fig. 13.
4.3.3 Frictional Force

It is not necessary in every cases that higher is the wear rate means lower is the requirements of the frictional force. It is depends upon the nature of the material and molecular weight of the material sometimes. In the present case the result was came as expected and shows that MRR is directly proportional to the frictional force (See Fig. 14).

![Frictional force (N)](image)

Fig. 14 Comparison of different samples considering Frictional force

4.3.4 Coefficient of friction ($\mu$)

Similarly to the wear rate, the coefficient of friction is not measurable of the wear rate or MRR of the material and it depends upon the some other properties like hardness and toughness. But in the present case coefficient of friction followed the MRR and wear rate (See Fig. 15). The coefficient of friction can be evaluated by considering expression given below by putting values of frictional force and applied load.

\[ F = \mu N \]

Where, \( N = m.g \)

Then,

\[ \mu = \frac{F}{m.g} \] \hspace{1cm} (considering \( g = 9.8 \))

taking weight of 1Kg in the present case, the coefficient of friction,

For granules, \( \mu = 1/9.8 \)

\( \mu = 0.102 \)

Similarly all the samples were evaluated their coefficient of friction. Fig. 15 shows that increase in the frictional force in present case lead to the decrees in the coefficient of friction. The extruded sample at experiment no. 6 exhibited the lesser coefficient of friction as compared to the others.
4.4 Shore D hardness

In the present study the shore D hardness test performed by a portable Shore D durometer. The material which was evaluated worst wear properties having better hardness property. It has been found that the material which id having the better tensile property also have the best hardness and lesser wear property. Fig. 16 shows the relative comparison of the samples under consideration of shore d hardness.

4.5 Influences on morphological characteristic

The microstructural examinations have been performed to ensure the reason of changes in behavior of the extruded samples supported by mechanical and thermal analysis. Extruded sample with experiment no. 6 exhibited better mechanical, wear and thermal properties. The micrograph for experiment no. 6 shows that there is a uniform surface appearance and having lesser surface irregularities as compared to the both of the samples. The un-extruded surface having more pores as compared to the both extruded samples that is the reason why extruded sample was more stable and exhibited better mechanical, wear and thermal properties (See Fig. 17). The cross examinations have been performed with support of percentage porosity at the samples surface and grain size at that surface.
Fig. 17 Microstructure of the different sample surfaces at 100X magnification

Fig. 18 shows the nature of the surfaces of the samples which was tested mechanically and thermally. At experiment no. 6 which was exhibited better mechanical and wear properties having lowest porosity on their surface. The porosity results were also supported by exploring grain size number which is irrespective of the grain size. The samples with lowest porosity exhibited the greatest grain size number. It is an important consideration that extrusion as melt processing refined the molecules arrangement and provided more strength and stability.

![Morphological characteristics](chart.png)

Fig. 18 Relative comparison of different samples considering percentage porosity and grain size number

5. Conclusions

Following conclusions have been made in relation of effect of melt processing on the thermal, mechanical, wear and morphological characteristics.

- The extrusion as the melt processing has been considered as the one of the techniques for primary recycling of the polyamide material with potential application in engineering applications.
- In context of the changes in mechanical behavior, the extrusion process given a product with better mechanical and metallurgical properties. The best setting for parameter i.e. at experiment no. 6 has given
the best result and improves the tensile and hardness properties. Further from thermal analysis point of view, it was observed that primary extrusion leaves the material with better thermal stability.

- From morphological point of view, it was also ascertained that primary recycling through twin screw extrusion (as melt processing technique) has also refined the surface properties.

At global level the recycling of polymeric materials is becoming an attractive field (by reinforcing various fillers in waste polymers to enhance their physical/mechanical properties). So far sand, fiber, ash, rice husk and wood husk have been used for such reinforcements. Further, many areas have to be explored in the field of plastic recycling by reinforcement of metallic/ceramic fillers like SiC, Al and Fe in powder form and other agricultural waste (like banana fibers, almonds shells etc.) availabe depending upon the geographical locations to enhance the mechanical and tribological properties by preparing composite structure. An alternate route through a fused deposition modelling may be used for preparation of functional/non-functional prototypes by using recycled PA6 filaments. Reinforcement in PA6 can be done by primary and secondary recycling techniques. With this process filament wire can be prepared and fed into the open source three dimensional printers and various direct applications (like rapid tooling) can be satisfied in coming future as new composite structures.

We address studies on the use of recycled PA6 for the additive manufacturing of innovative composite materials and structures with arbitrary geometry at different scales [1-4], [60-70] to future work. Special interest bears the high environmental and mechanical potential of recycled nylon fibers for the reinforcement of sustainable cement mortars [4], as well as the design and rapid prototyping of multiscale geometries for reinforcing elements of sustainable composite materials with hierarchic structure [71-74].

Acknowledgement

The authors are highly thankful to DST (Government of India) File No. TSG/NTS/2014/104 for providing financial assistance to carry out the research work.

References


