STUDIES ON THE MORPHOLOGICAL, THERMAL AND RESISTIVITY PROPERTIES OF RECYCLED POLY (ETHYLENE TEREPHTHALATE) COMPOSITES WITH NANOCLAY FILLERS

S. Rajarajan¹, J. Raja ²

¹ Research Scholar, Sathyabama University, Jeppiaar Nagar, Chennai-600 119.
² Professor and Head/IT, SSN College of Engineering, Kalavakkam, Chennai-603 110.

Abstract

Nanocomposites based on polymer-clay mixtures are a growing area of interest due to their potential in flexible applications. In this study, new nanocomposite materials were produced from the components of recycled thermoplastic as the matrix and montmorillonite as the filler by using a co-rotating twin screw extruder. During this study, recycled Poly(Ethylene Terephthalate), rPET, was mixed with organically modified quaternary alkyl ammonium montmorillonite in the contents of 1, 2, and 5 weight %. Three types of clays were evaluated during the studies. For comparison, 2 weight % clay containing samples were prepared with three different clay types, Cloisite 15A, 25A, 30B. Even though, 2wt% Cloisite 25A shows better mechanical properties, 2 wt% Cloisite 30B is better in respect of electrical properties.

Key words: Nanocomposite, Thermoplastic Nanocomposites, rPET, Montmorillonite.

I. INTRODUCTION

Polymer layered silicate nanocomposites have become an important area studied more widely in academic, government and industrial laboratories. These type of materials were first reported as early as 1950, [1]. However, it was not widespread until the period of investigation on this type of structures by Toyota researchers, [2-5]. This early work of Toyota group was based on the formation of nanocomposites where montmorillonite was intercalated with ε-caprolactam in situ. Polymeric materials can be filled with several inorganic and/or natural compounds in order to get the wide array of property enhancements, e.g., increased stiffness and strength, improved solvent and UV resistance, greater dimensional stability, decreased electrical conductivity, enhanced gas barrier properties. The property improvements of clay based nanocomposites are due to the nanoscale nature of the formed system resulting in very high surface areas.

From an industrial approach, owing to high costs of development, synthesis and commercialization of new polymers, most researchers look for new materials by reinforcing or blending existing polymers, so the tailor made properties of the materials can be achieved, [6]. Poly(Ethylene Terephthalate) (PET) is a low-cost, and high performance thermoplastic that finds use areas in a variety of applications, such as in fabrics and soft drink bottles, reinforcement of tyres and rubbery goods, food and beverage packaging. PET has excellent surface characteristics, and high heat deflection temperature. PET regrinds from post consumer soft drink bottles have slightly reduced molecular weight and structure related properties as compared to pure polymer.

In this study, the aim is to produce nanocomposite materials from recycled PET regrinds as the matrix with the addition of organically Modified Montmorillonite (MMT) clays as the filler, and observe the effects of clay content and clay type on sample properties, like thermal, morphological and resistivity.

II. MATERIALS

A. Organoclays

Experiments were carried out with three different types of montmorillonites, namely Cloisite 30B, 15A, and 25A. These organoclay structures show differences in selection according to what degree of polarity they have. There is an increase in relative product hydrophobicity and a decrease in product polarity in the order of 30B, 25A, and 15A. Thus, the hydrophobicity resulted from different natures of organic modifiers affects the chemical compatibility between the polymer and the filler.

Following sections describe the product properties of these clay types. Cloisite 30B is a montmorillonite modified with a ternary ammonium salt, whereas Cloisite 15A and Cloisite 25A are montmorillonite modified with a quaternary ammonium salts.

TABLE 1 Properties of Organoclays

<table>
<thead>
<tr>
<th>Clay Structure</th>
<th>CATION EXCHANGE CAPACITY (CEC)</th>
<th>% MOISTURE</th>
<th>% WEIGHT LOSS ON IGNITION</th>
<th>DENSITY, g/cc</th>
</tr>
</thead>
<tbody>
<tr>
<td>MT2 ET0H</td>
<td>90 meq/100g</td>
<td>&lt; 2%</td>
<td>30%</td>
<td>1.98</td>
</tr>
<tr>
<td>2M2H T</td>
<td>125 meq/100g</td>
<td>&lt; 2%</td>
<td>43%</td>
<td>1.66</td>
</tr>
<tr>
<td>2MH TL8</td>
<td>95 meq/100g</td>
<td>&lt; 2%</td>
<td>34%</td>
<td>1.87</td>
</tr>
</tbody>
</table>
B. **PET Resin**

PET soft drink bottle regrind was obtained in the form of flakes with the following general physical properties.

**TABLE 2 Contaminants in Recycled PET Resin**

<table>
<thead>
<tr>
<th>CONTAMINANT</th>
<th>VALUE (PPM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVC</td>
<td>60</td>
</tr>
<tr>
<td>Polyethylene</td>
<td>5</td>
</tr>
<tr>
<td>Metal pieces</td>
<td>0</td>
</tr>
<tr>
<td>Adhesive</td>
<td>10</td>
</tr>
<tr>
<td>Paper pieces</td>
<td>3</td>
</tr>
</tbody>
</table>

**TABLE 3 Properties of Recycled PET Resin**

<table>
<thead>
<tr>
<th>MATERIAL PROPERTIES</th>
<th>VALUE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Intrinsic Viscosity, [η]</td>
<td>0.750 g/cm.s</td>
</tr>
<tr>
<td>Glass Transition Temperature, Tg</td>
<td>60 ºC</td>
</tr>
<tr>
<td>Melting Temperature, Tm</td>
<td>255 ºC – 260 ºC</td>
</tr>
</tbody>
</table>

C. **Injection Molding**

Keeping the temperature of the injection nozzle constant at 275ºC, previously extruded and chopped samples of R-PET/organoclay blends were injection molded. Adjustments in mold fill time (3-8 sec), molding cycle time (3-5 min), and holding pressure (6-8.5 bars) were made in order to obtain the best-molded part appearance for each composition. Optimum values were 3 sec for fill time, 3 min for cycle time and 8.0 bars for holding pressure.

The samples obtained at the end of these production steps were analyzed and tested according to ASTM Standards.
Fig.2 SEM of recycled-PET neat Resin

Fig.3, 4 and fig.5 shows the fracture surfaces of samples containing 1, 2, and 5 weight % of Cloisite 30B. In these photos, the main difference is the dispersion of the clay platelets. Due to the nature of high clay loading, 5-weight % sample exhibits a structure having large clay aggregates, which act as stress concentrators. This causes localized stresses on the surface of the clay leading to worse impact strength. In sample with 1 weight % clay, long crack propagation lines and clay aggregates, small white objects on the photo, can be observed. In comparison with the sample with 2-weight % clay loading, the surface looks smoother implying easier crack propagation. 2 weight % one has more tortuous structure preventing easy crack propagation.

Fig.4 SEM of recycled-PET+ 2 wt % Cloisite 30B

When compare the samples containing 2 weight % of 30B (Fig.4), 2 weight % of 15A (Fig.6) and 2 weight % of 25A(Fig.7) clay types, it is observed that smaller distances between crack propagation lines and more disperse and tortuous characteristic in the composition containing 30B clay type. But, this structure gives lower impact energy than the one containing 25A clay type. The reason could be that, in the samples of 30B adhesion strength between the matrix and the filler may not be as good as the samples containing 25A montmorillonite, thus cracks in samples with 30B may need lower energy to propagate in spite of better dispersion. 15A samples give the lowest impact energy.

Fig.5 SEM of recycled-PET+ 5 wt % Cloisite 30B

Fig.6 SEM of recycled-PET+ 2 wt % Cloisite 15A

Fig.7 SEM of recycled-PET+ 2 wt % Cloisite 25A

B. Thermal Studies

Differential scanning calorimeter analysis was performed in
order to evaluate the changes in Glass Transition temperature (Tg) with increasing clay content. Glass transition temperature is largely related to the molecular mobility of polymer chains.

According to the results, 2 weight % 30B yields better resistivity than other two 15A and 25A clay blends. This shows obviously the material structure plays a vital role in determining the resistivity of polymer nanocomposites. As a whole, intercalation or exfoliation of the nano-filler into the rPET matrix decides the resistivity.

V. CONCLUSION

Based on SEM, the main differences between the structures due to the dispersion of clay particles, and the compatibility of montmorillonite types with the recycled PET base resin. It is observed that the most tortuous path was there at 2 weight% clay loading implying the highest degree of dispersion and exfoliation. At lower clay content (1%), the amount of clay was not enough to improve the properties. At higher clay content (5%), the clay remained mostly as aggregates giving rise to poor properties. Among the clay types, generally the 25A type of montmorillonite produced the highest tortuous structure in SEM micrographs. The tortuous surface, small and the heterogeneous crack propagation pathways on micrographs are the evidence of exfoliation of silicate layers in the matrix implying enhancements both in physical and thermal properties.

DSC analysis showed that the incorporation of clay particles into the base polymer caused changes in Tg values. The maximum increase in Tg values was observed in the sample containing 2 weight % of 25A clay type. The Tg increased from 81.8oC (value of r-PET) to 83.2oC.

Resistivity results shows 2 weight%30B clay type is better for dielectric application. Since, SEM studies and DSC studies are favourable for 2 weight% 25A clay type, this clay and composition may be good mechanically, whereas resistivity tests prefer 2 weight% 30B clay as a good electrical insulator.

REFERENCES


S.RAJARAJAN graduated from Mohammed Sathak Engineering College, Kilakarai during the year 1991 in Electronics and Communication Engineering Discipline. He obtained his Master's degree during the year 2002 with the specialization in Applied Electronics from Sathyabama Institute of Science and Technology, Chennai. He is doing his research work in the field of NanoElectronics. Currently he is the Assistant Professor, in the department of Electronics and Communication Engineering of Sathyabama university, Chennai-600 119. He has published about 17 papers to his credit in National and International Conferences. He has guided about 12 M.E./M.Tech. projects so far.