INVESTIGATIONS ON RHEOLOGICAL PROPERTIES OF POLYMER-CERAMIC FIRE RETARDANT COMPOSITES

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Abstract

In the present work an attempt is made to develop fire retardant polymer material using the polymer nano composites. Fire retardant minimizes the risk of fire starting and if once started of spreading. Fire retardants exhibit an excellent property of reducing the spreading of fire, in turn reduces the fire hazards and are proven to save lives and protect property and are therefore an essential part of fire protection. Polymers and plastics are highly prone to fire and catch fire easily and continue to burn if fire retardants are not used. The safe application of plastics in our modern society would not be feasible without the use of flame retardants. The “side effects” of the fire retardants have to be taken into account in a balanced manner. This paper attempts to evaluate the rheological and DSC properties of fire retardant polymers.

Key words: Composites, Rheology, Dynamic Mechanical Analysis (DMA), Differential Scanning Calorimetry (DSC).

1.0 Introduction

The development of science and technology provides the availability of sophisticated products but concurrently increases the use of combustible materials [1]. Polymeric materials are commonly used in everyday life increasing fire hazards and so flame retardants are very often incorporated into them to limit their flammability. Fire-retardant polymers are polymers that are resistant to degradation at high temperatures. There is need for fire-resistant polymers in the construction of small, enclosed spaces such as skyscrapers, boats, and airplane cabins. In these tight spaces, ability to escape in the event of a fire is compromised, increasing fire risk. In fact, some studies report that about 20% of victims of airplane crashes are killed not by the crash itself but by ensuing fires.
Fire-safe polymers also find application as adhesives in aerospace materials, insulation for electronics, and in military materials such as canvas tenting.

As an example, in 2004, there were 508 fire-related deaths in the UK, compared with 593 in 2003 [2]. The highest number recorded was 1096 deaths in 1979. Through the 1980s and 1990s there was a general downward trend in fire-related deaths. This trend can be linked to the toughening of the legislation in terms of fire hazards combined with the growing use of flame retardants, global demand for flame retardant is forecast to increase by 4.8% per year to 2.2 million metric tons in 2009 [3] further there is a possibility that there will be increase in the demand for fire retardant materials in the future. The flammability behavior of polymers is defined on the basis of several processes and/or parameters, such as burning rates (solid degradation rate and heat release rate), spread rates (flame, pyrolysis, burn-out, smolder), ignition characteristics (delay time, ignition temperature, critical heat flux for ignition), product distribution (in particular, toxic species emissions)[4], smoke production, etc. The objective is then to inhibit or even suppress the combustion process acting chemically and/or physically in the solid, liquid or gas phase. One can interfere with combustion during a particular stage of this process, e.g: During heating, decomposition, ignition or flame spread. Some fire-safe polymers naturally exhibit an intrinsic resistance to decomposition, while others are synthesized by incorporating fire-resistant additives and fillers. Current research in developing fire-safe polymers is focused on modifying various properties of the polymers such as ease of ignition, rate of heat release, and the evolution of smoke and toxic gases.

Three approaches can be considered to reduce the flammability of polymers:

1. To use inherently flame retardent polymers (e.g. poly (tetrafluoroethylene), polyoxazoles, poly-(ether-ether ketone) or polyimides); [5]

2. To chemically modify existing polymers (e.g. copolymerisation of flame-retardant monomer into PET chains).[6]

3. Organic/inorganic hybrid polymers such as epoxy resin prepared from silsesquioxanes [7].

4. To incorporate flame retardants into polymers via usual procedures. [8]

2.0 Objective of present work:

- To prepare ceramic dispersed polymer composites with fire-retardant properties and sufficient mechanical properties to withstand the heat effect for reasonable time.
- The issue is to be addressed by addition of both micro and nano level size addition of the ceramics.

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Polymer - ceramic blends from the listed materials would be prepared with reinforcing ceramic particles mostly in the nano-metric scale size so that the required properties are achieved with small addition of the reinforcements.

To study the Synergistic effect of halloysite nanotubes on the flammability properties of acrylonitrile–butadiene–styrene composites.

3.0 Methodology:

In the present work it is proposed to use the following materials: for polymer matrix.

1. High density polyethylene (HDPE)
2. Epoxy

For the ceramic fire retardant it is proposed to use:

1. Clay
2. Carbonnano tube

Poly (acrylonitrile-co-butadiene-co-styrene) was obtained from Aldrich, containing acrylonitrile (25% pellets) and having a melt flow index of 6 g (10 min) −1 (230 °C/3.8 kg). Halloysite nanotubes HNTs (ultrafine grade) were obtained from Imerys Tableware Asia Limited, New Zealand (wt%): SiO2, (49.5%); Al2O3,(35.5%); Fe2O3, (0.29%); TiO2(0.09%), Ammonium polyphosphate (APP),Melamine polyphosphate (MPP) were obtained from Universal Chemtech. Co., Korea. Pentaerythritol (98%) was purchased from Aldrich. The extruded material was cut into required shape and was subjected to various tests as follows.

4.0 Experimentation:

The required polymer nano particle mixes were prepared in HAAKE mixer shown in Fig.1, to study the Synergistic effect of halloysite nanotubes on the flammability properties of acrylonitrile–butadiene–styrene composites. The following compositions of the required polymer Mixes with additives were made in the HAAKE mixer.
4.1 Haake Mixer

Fig. 1 HAAKE MIXER

HAAKE Rheomix Lab Mixer, torque rheometer platform are focused on batch testing of many highly viscous substances. Intelligent modular torque rheometer system are used for the simulation of industrial processes in the lab or pilot plants. The small-scale mixer can characterize materials like polymers, elastomers, additives and fillers to avoid problems in their production.

4.2 DMA (Dynamic Mechanical Analysis):

Fig2: DMA TEST RIG

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Rheological properties of the mixes were studied by DMA shown in Fig. 2. Dynamic mechanical analysis (abbreviated as DMA, also known as spectroscopy) is a technique used to study and characterize materials. It is most useful for studying the viscoelastic behavior of polymers. A sinusoidal stress is applied and the strain in the material is measured, allowing one to determine the complex modulus. The temperature or the frequency of the stress are often varied, leading to variations in the complex modulus, this approach can be used to locate the glass transition temperature of the material, as well as to identify transitions corresponding to other molecular motions.

Fig.3: General Schematic of a DMA instrument

4.3 Rheology:

Rheology plays an important role in influencing the quality of the polymer mixes. Thus rheological properties of the mixes were studied first. It is the branch of physics that deals with the deformation and flow of matter under stress. It is particularly concerned with the properties of matter that determine its behavior when a mechanical force is exerted on it.

The viscoelastic character of polymer melts reflects the entangled microstructure and plays an important role in property development and in flow stability.

The relationship between the structure and rheology of a polymer is of practical interest for two reasons:

- Firstly, rheological properties are very sensitive to certain aspect of structure and they are simpler to use than analytical methods, such as nuclear magnetic resonance.
• Secondly, it is the rheological property that governs the flow behavior of polymers when they are processed in the molten state.

4.4 DSC (Differential scanning Calorimetry):

Thermo analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature.

Various Applications:

• Fusion and Crystallization events
• Glass transition temperatures (Tg)
• Study oxidation
• Other chemical reactions.

5.0 Results and discussion:

Table 1: ABS with Halloysite Batches

<table>
<thead>
<tr>
<th>SI NO.</th>
<th>Wt % HNT</th>
<th>Wt % ABS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>1</td>
<td>99</td>
</tr>
<tr>
<td>2.</td>
<td>3</td>
<td>97</td>
</tr>
<tr>
<td>3.</td>
<td>4.75</td>
<td>95.25</td>
</tr>
</tbody>
</table>

5.2 Table 2: ABS with Cloisite 30B Batches

<table>
<thead>
<tr>
<th>SI NO.</th>
<th>Wt % Cloisite 30B</th>
<th>Wt % ABS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>1</td>
<td>99</td>
</tr>
<tr>
<td>2.</td>
<td>3</td>
<td>97</td>
</tr>
<tr>
<td>3.</td>
<td>4.75</td>
<td>95.25</td>
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</table>

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5.3 Table 3: Processing Conditions (HAAKE Melt Mix):

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Temperature (°C)</th>
<th>RPM</th>
<th>Time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>230</td>
<td>60</td>
<td>20</td>
</tr>
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</table>

RHEOLOGY:

Graph-1

Graph-2
DSC (Differential scanning Calorimetry)

Graph 1 shows the rheological properties of the ABS with Hallosite and Graph 2 shows the rheological properties of the ABS with Cloisite. Graph 3 shows the DSC properties of the ABS with Hallosite and Graph 4 shows the DSC properties of the ABS with Cloisite.

6.0 Conclusion:
The polymer – ceramic composites (ABS with Halloysite, ABS with Cloisite). Were Successfully prepared and the required specimens for the various tests were also prepared as per the ASTM standards. DMA (Dynamic Mechanical Analysis) is carried on various specimens. With the DMA test the material characterisation was carried out and the visco elastic properties of the composite material was evaluated. Using this technique the glass transition temperature was evaluated. Rheology tests on various configurations of the polymer specimens prepared are conducted as per standard. It was observed that the specimens with 3% Hallosite with Acrylonitrile-co-butadiene-co-styrene (ABS) showed excellent rheological properties over the other types of the specimens prepared. DSC (Differential scanning Calorimetry) for various combinations of specimens is carried.
7.0 Acknowledgements:

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References: