EFFECT OF SEPIOLITE CONTENT ON MECHANICAL, THERMAL AND FLAMMABILITY PROPERTIES OF ETHYLENE VINYL ACETATE NANOCOMPOSITE

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I lovingly dedicate this thesis to my wife Zhaleh and my Mother and Father who supported me each step of the way.

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ABSTRACT

Polymer/clay nanocomposites are a new class of composite materials consisting of a polymer matrix with dispersed clay nanoparticles. Ethylene vinyl acetate (EVA)/Sepiolite and EVA/modified Sepiolite (M-Sepiolite) nanocomposites were prepared by melt extrusion using a counter-rotating twin-screw extruder followed by injection molding in order to examine the mechanical, morphological, thermal and flammability properties of the nanocomposites. Sepiolite was modified with silane treatment (M-Sepiolite). Sepiolite content was various in EVA with 1, 3, 5 and 7 wt%. The mechanical properties of EVA/Sepiolite and EVA/M-sepiolite nanocomposites were studied through tensile. Scanning electron microscopy (SEM) was used to investigate the phase morphology of nanocomposites. The thermal properties were determined using Thermogravimetric analysis (TGA) and flame retardancy of nanocomposites was conducted by characterization for limiting oxygen index. The mechanical, thermal and flame retardancy properties of EVA / Sepiolite nanocomposite increased with the increase sepiolite content due to good dispersion and well interface interaction between Sepiolite and EVA. The mechanical, thermal and flame retardancy properties the EVA/M-Sepiolite nanocomposite was better than the mechanical, thermal and flame retardancy properties of EVA/Sepiolite nanocomposite, where the modification of Sepiolite caused extremely good interface interaction, as well as good dispersion and better adhesion between Sepiolite and polymer matrix.

ABSTRAK

Nanokomposit polimer/tanah liat adalah komposit kelas baru, terdiri daripada matriks polimer yang terisi dengan zarah tanah liat yang bersaiz nano. Nanokomposit etilena vinil asetat (EVA) and EVA/sepiolite yang telah diubahsuai (M-Sepiolite) disediakan melalui proses pengektrudan lebur dengan menggunakan ekstruder skru berkembar yang berputar bertentangan arah, diikuti dengan mesin pengacuanan untuk mengkaji sifat mekanik, morfologi, termal dan sifat untuk terbakar bagi komposit nano. Sepiolite telah diubahsuai dengan rawatan menggunakan silane (M-Sepiolite). Kandungan sepiolite di dalam EVA telah divariasikan kepada 1,3,4,5 dan 7 wt.%. Sifat mekanikal EVA/sepiolite dan komposit nano EVA/M-sepiolite telah dikaji menggunakan mesin tegangan dan mesin penguji kekerasan. Mesin pengimbas electron (SEM) telah digunakan untuk mengkaji morfologi komposit nano. Sifat termal telah ditentukan dengan menggunakan analisis termogravimetri, dan ujian rintangan untuk terbakar telah dijalankan dengan pencirian untuk indeks oksigen yang terhad. Sifat mekanikal, termal dan rintangan untuk terbakar komposit nano/Sepiolite telah meningkat dengan penambahan kandungan sepiolite disebabkan oleh penyebaran dan interaksi yang baik antara sepiolite dan EVA. Sifat mekanikal, termal dan kerintangan untuk terbakar bagi komposit nano EVA/M-Sepiolite adalah lebih baik daripada sifat-sifat yang dipunyai oleh komposit nano EVA/Sepiolite, di mana sepiolite yang telah diubahsuai menghasilkan interaksi yang amat baik, begitu juga dengan penyebaran antara sepiolite dan matriks polimer.

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CHAPTER 1

INTRODUCTION

1.1 Background

Polymer−clay nanocomposites are a new class of composite materials consisting of a polymer matrix with dispersed clay nanoparticles. The interest in such systems (organic−inorganic hybrid material) is due to the fact that, the ultrafine or nano dispersion of filler, as well as the local interactions between the matrix and filler, lead to a higher level of properties than for equivalent micro- and macro composites. In recent years, more attention has been given to incorporate nanomaterials into the polymer matrices to obtain high performance nanocomposites. Polymer/clay nanocomposites have drawn considerable interest because of their enhanced properties, including flame resistance, mechanical properties, gas barrier properties, thermal stability, and biodegradability when compared to pristine polymers (Wilson *et al.*, 2012) .Provided by only small amounts of nanometer-size clays homogeneously dispersed in a polymeric matrix.

Ethylene-vinyl acetate (EVA) copolymers with different acetate contents are extensively used in many fields, especially in the wire and cable industry. With different percentage of vinyl acetate in the copolymer it has different properties such as, thermoplastic use low percentage of vinyl acetate in copolymer, and high percentage of vinyl acetate in polymer is used on oil resistant and to be ready for hot melt adhesive; so, the percentage of VA is an significant matter in the copolymer. However, EVA copolymers are particularly flammable and emit a large amount of smoke while burning, which restrict their practical applications. The thermal degradation behavior of EVA has been studied by several researchers, and information such as structural changes, identification of volatile decomposition products, and degradation mechanism has been derived (Fu andQu, 2004; Wenwei *et al.*, 1994).

Nanoclay is widely used to improve the properties of EVA. EVA/clay nanocomposites can be easily prepared because the EVA contains polar group, vinyl acetate (VA), which can effectively interact with clay. They found that the interlayer distance of MMT increased with VA content increasing. But when VA content is beyond 15 wt%, there is no further increase of interlayer distance (Zhang *et al.*, 2003) .The clay can also be used to reinforce polymers because of the large interface area resulting in strong interaction between the polymer matrix and the nano filler (Chen *et al.*, 2011; Zhang *et al.*, 2003).

Considering the application, todays several method to reinforce EVA have been done for improvement physical and chemical properties improvement, for example EVA is blended with other polymers and reinforced with various filler or additive. Clay that is frequently used for improvement of EVA in last decades are organophilic clay (OMMT), ufunctionalized clay (Na-MMT) and sepiolite (Nyambo *et al.*, 2009; Wilson *et al.*, 2012).

Sepiolite nanofiller is one type of clay that have been use in polymer composites. The sepiolite has a fibroid hydrated magnesium silicate, with common composition $Mg_4Si_6O_{15}(OH)_2 \cdot 6H_2O$ (Huang, Chen, Yi, *et al.*, 2010) . The Sepiolite include a lot physic chemical properties such as High liquid absorption, mechanical strength in wet conditions, low weight, control of rheological properties in heat application systems, improving fire resistance (Galan, 1996).

Silane coupling has been predominantly employed since the early 1960 as the bonding method for the resin-filler interface. Although other coupling techniques, like micromechanical coupling have been investigated and applied, their success has been limited. An important advantage of silane coupling is that the hydrolysis (and reformation) of the chemical bond between silane coupling agent and filler material is a reversible process (Venhoven *et al.*, 1994).

Sepiolite is treated by various method and different material; sepiolite can be treating on surface or edge treatment and treat inside clay or change structure of clay with some matter. The discontinuity of the silica sheets gives rise to the presence of silanol groups (Si–OH) at the edges of the tunnels, which are the channels opened to the external surface of the sepiolite particles. The presence of silanol groups (Si–OH) can enhance the interfacial interaction between sepiolite and polar polymers. The distribution of nano clay is proven to be well dispersed in EVA within introduction of compatibilizing agent due to the interaction of hydrogen bonding between magnesium group in the organic surface of the sepiolite with the carbonyl group of EVA chain segment contributes to this process (Huang, Chen, Yi, *et al.*, 2010).

1.2 Problem Statement

Various methods have been employed on EVA in order to improve the properties of EVA especially mechanical and flammability properties. Nanoclays have widely being incorporated with EVA to explore the capability of nanofiller to enhance the properties.

According to Ahamad et al. (2011), the flame retardant, mechanical and thermal properties of ternary magnesium hydroxide/clay/EVA nanocomposites had significantly improved without losing its mechanical properties. The EVA/clay/$Mg(OH)_2$ nanocomposites showed better thermal, flame retardant and mechanical properties compared with nanocomposites of EVA filled only with the nano $Mg(OH)_2$ and this demonstrate the role of clay in the nanocomposites (Ahamad *et al.*, 2012).

In the previous research the flame retardant and thermal properties of EVA-nanocomposites are improved by combination of nanofiller with aluminium trihydrate. The thermal stability of EVA / nanofiller nanocomposites is increased by very small amount of nanofiller within the polymer matrix. Nanocomposites show a delay of the degradation; the peak of heat release measured by a cone calorimeter is dramatically reduced. Char configuration of the nanocomposites has been improved and is responsible for the very good flame retardancy (Beyer, 2001b).

Yaru Shi et al. (2009) reported on EVA/nanoclay nanocomposites were made by polymer modified clay master batch approach for study of mechanical properties and flame retardancy of EVA/MMT nanoclay nanocomposites. The mechanical properties of nanocomposites is enhanced by increased two time of tensile strength and tripled Young’s modulus, in addition the toughness of EVA/nanoclay nanocomposites decreased insignificantly than EVA. The flame retardancy of nanocomposites increased with rising of content of nanoclay (Shi *et al.*, 2009).

Use of the compatibilizer and clay treatment are also considered for further improvement of polymer properties .According to Qianping Ran et al. (2005) , EVA-MMT nanocomposite was provided by melt intercalation where the MMT was treated with interfacial method by swelling agent hexadecy 1 trimethylammonium bromide (CTAB) and was diffused in the poly(ethylene vinyl acetate). The CTAB-MMT was able to strengthen the EVA. The optimal tensile and tear strengths of the EVA/ CTAB-MMT composites were obtained with 5wt% CTAB-MMT in the composites. Compared with the EVA/MMT composites, the interfacial interaction of the EVA/CTAB-MMT nanocomposites was markedly enhanced. The EVA/CTAB-MMT nanocomposites exhibited higher thermal stability (Ran *et al.*, 2006).

There is no work reported on the mechanical, thermal and flame retardancy of EVA/Sepiolite nanocomposites and effect of modified Sepiolite on the properties of EVA nanocomposites. The Sepiolite, which is a natural clay mineral, is attractive filler for polymer because of its acicular form, low cost, and abundance. Furthermore the surface of Sepiolite is easily modified(Volle *et al.*, 2012).

In this study, Sepiolite nanofiller incorporated with EVA and the silane treatment conducted on Sepiolite nanoclay. The mechanical properties, thermal and flammability properties of EVA/sepiolite nanocomposite evaluated.

1.3 Objectives of study

This research focuses on the effect of Sepiolite and M-Sepiolite on EVA nanocomposite for the improvement of flame-retardant, mechanical properties and thermal properties.

The main objective of this research can further divided as below:

1. To investigate the effect of sepiolite content on the mechanical, thermal and flammability properties of EVA / Sepiolite nanocomposite.
2. To determine the effect of silane treatment of sepiolite on the mechanical, thermal and flammability properties of EVA / sepiolite nanocomposites.

1.4 Scopes of study

In order to achieve the objectives of this research, the following activities have been carried out:

1. Literature review

   Literature research on background of copolymer EVA, clays and clay minerals and sepiolite, and also the effect of nanoclay sepiolite for improve flammability and properties EVA/clay the nanocomposite.

2. Sample preparation

   a. Modification sepiolite with silane treatment

   b. Mixing Sepiolite and M-Sepiolite with EVA with the amount of Sepiolite various from 1-7 wt% in EVA.

   c. Prepare the sample with melt extrusion

3. Sample characterization

   a. Morphological

      i. Field Emission Scanning Electron Microscope (FESEM) was used to investigate the surface and cross section structure and morphology quality of EVA and EVA Sepiolite and M-Sepiolite nanocomposites.

      ii. X-ray Diffraction (XRD) was employed for the investigation of bundle Sepiolite and M-Sepilite aggregation and delamination behavior in EVA matrix.

   b. Mechanical test

      i. Tensile test was used to identify tensile properties such as tensile strength, Young’s modulus and elongation at break.

   c. Thermal analysis

      i. Thermo gravimetric analysis (TGA) was carried out to determine thermal stability properties such as the initial degradation temperature at 5% mass loss T5%, Final degradation temperature Td and char residual.

d. Flammability test
    i. Limiting Oxygen Index Test (LOI) was done to obtain Flame retardancy properties like Limiting oxygen index (LOI) value.

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