Effect of Nanoclay on Flammability Behavior and Morphology of Nanocomposites from Wood Flour and Polystyrene Materials

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The flammability behavior of wood/plastic nanocomposites made from recycled polystyrene, wood flour, and nanoclay were investigated in this study. The wood flour was mixed, using the two weight ratios of 40 wt.% and 60 wt.% with recycled polystyrene, and nanoclay was added at 0 wt.% and 5 wt.%. A coupling agent was also added at up to 3 wt.% of the composite. The results showed that the oxygen index increased when higher contents of wood flour were added. Furthermore, it was found that the samples required more oxygen for ignition when the percentage of wood flour was increased. Similarly, it was found that the samples required a greater amount of oxygen for ignition with increasing nanoclay content. Therefore, the flammability of the sample decreased because the time to ignition took longer in the absence of sufficient oxygenation. X-ray analysis of the nanocomposites revealed that the morphological structure involved intercalation.

Keywords: Flammability; Oxygen index; Nanoclay; X-ray; Recycled polystyrene

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INTRODUCTION

Accidental fire has always been considered a disastrous phenomenon. One of the advantages to using nanoclays is a reduction in the rate of ignition, which depends on the ratio in which they are added to the wood and plastic combination. The weight ratio of nanoclays needs to be significantly smaller than the other components in order to improve the thermal properties of the composite. Nanocomposites demonstrate a noticeable enhancement of properties when nanoclay is added at 2 to 10 wt.%. Such composites are being developed for various modern applications that require certain mechanical, thermal, and electrical properties. The flame-retarding behavior of nanoclay can be evaluated *via* two different methods (Beyer 2002). Polymer-intercalation silicate nanocomposites provide a unique combination of reduced flammability and improved mechanical properties (Gilman 1999). When only 10 wt.% of the filler material is used in the composition, the dissolution temperature of this material can be 40 to 50 °C greater than that of the virgin polymer (Blumstin 1965). When the nanocomposite is heated, the polymer will dissolve and evaporate. This will lead to deterioration of the structure and the formation of a smaller intercalation spaces between the layered silicate or clay layers. The clay layer is believed to accelerate the formation of the char, which forms a layer of charcoal impregnated within the clay (Khosravian 2010). This special structure forms rapidly under pyrolysis and remains stable even long after pyrolysis is complete. These results imply that the charcoal from carbon clay soil can provide thermal protection for a substrate. In addition, natural fibers may be added to the thermoplastic polymers in order to produce wood polymer composites (WPCs). Thermal analysis of the WPCs is very important in the production process, since this parameter determines the maximum temperature that can be used during the process. Wood polymer composites are used only with synthetic polymers that can be processed below 200 °C, because the thermal stability of the wood is rather low. This limits the type of polymers capable of being processed and transformed into WPCs, thus confining the possible applications where WPCs could be used (Klyosov 2007). Improvements in the thermal properties of the WPCs may increase their acceptance for use in structural and outdoor applications.

Certain new nanocomposites retain their physical-mechanical properties and even sometimes enhance strength in addition to enhancing the polymer's resistance against fire (Sahraian 2003). In these systems, heat release, coaling content, and the carbon remainder decreases (Sahraian 2003). These materials, because of their high thermal capacity and low thermal conductivity, can absorb the heat of a flame and other heating processes in the polymer, as well as retard the ignition process and cool the polymer (Sahraian 2003; Khosravian 2010). Hindersinn (1990) demonstrated that the limiting oxygen index (LOI) is one of the primary methods for examining the flammability of different materials. However, this method is applicable only for materials that are capable of being burnt in the presence of oxygen. Beyer (2002) performed extensive research, which revealed that the rate of heat release is the only significant variable of a fire; therefore, it is referred to as the driving force. The principle of this measurement lies mainly in the consumption of oxygen, which indicates that there is a constant relationship between the amount of oxygen consumed and the content of heat released during the analysis of a polymer. Stark *et al.* (2010) showed from the LOI results that the oxygen index of the sample increased when increasing the content of the wood flour component. In other words, the sample would need more oxygen for ignition when the sample was burnt over a longer duration, which is consistent with the previous research. Hemmasi *et al.* (2013) studied the effect of nanoclay on the mechanical properties of a nanocomposite that was obtained from recycled polyethylene and bagasse flour. The XRD analysis showed that the resultant nanocomposite was of the intercalation type. The current research was conducted in order to investigate the effect of nanoclay on the flammability behavior of nanocomposites composed of wood flour and recycled polystyrene.

EXPERIMENTAL

Materials

Recycled polystyrene

Since municipal waste materials are not properly separated from each other, the seller has almost no information about the raw materials of post-consumer utensils, and the possibility of impurity in the raw materials of these utensils. Therefore, it was decided in this study to make recycled polystyrene experimentally. Polystyrene was obtained from the Tabriz Petrochemical Co., East Azerbaijan, Tabriz, Iran, with the specifications listed in Table 1. The polystyrene was passed twice through a twin-screw extruder at 180 °C and 100 rpm.

Table 1. Specifications of the Polymer Material

Composite	Manufacturer	Density (g/cm^3)	Tensile strength (kgf/cm^2)	Tensile modulus (kgf/cm^2)
Polystyrene (PS)	Tabriz Petrochemical Co.	1.07	265	280

Wood flour

Wood flour was used as a powder reinforcement for the polymer matrix. Spruce wood flour (Aria Cellulose Ind., Tehran, Iran), with the dimensions of 100 µm, was used for the composite material.

Coupling agent

Maleic anhydride polypropylene (MAPP) (Kimiya Javid Co., Isfahan, Iran), with a melt fluid index (MFI) of 1000 g/min and a concentration of 1.1% was introduced to improve the adherence between the hydrophobic polystyrene composite and the hydrophilic wood fibers.

Nanoclay

Cloisite 30B nanoclay, produced by Southern-Clay Products Inc., Gonzales, TX, USA, was used in the experiment. The clay soil of the Cloisite 30B contained natural montmorillonites, which were modified by a 4^{th} type ammonium salt.

Methods

Mixing

It was essential to use a dry lignocellulose material when mixing with thermosoft polymer because of the hydrophilic nature of the wood fibers (Kiaei *et al*. 2014). This limited the uniform distribution of the filler material in the polymeric matrix, in addition to enhancing the connection between the filler material and the polymer matrix. The presence of moisture in the lignocellulose material and the generation of steam upon processing and molding may cause delamination of the final product. Therefore, the wood flour was dried in an oven at 80 °C for 24 h. The wood flour and the nanoclay were used at two levels, while the coupling agent (MAPP) was prepared at 3 wt.% content. The mixing process was carried out using a HAAKE internal mixer equipped with a cam rotor type (SYS 9000, USA), at 190 °C and 60 rpm until a constant torque was reached (within 13 min). After the materials were mixed, the products produced an amorphous composite material that was ground after cooling, followed by a manual pressing for 5 min at 200 °C and 10 MPa. To balance the humidity, the samples were put for 14 days in conditioning room (relative humidity 60% to 65%, 20 °C temperature, Rate air flow 2 m/s).

Testing of flammability behavior

Limiting oxygen index (LOI) measurements were carried out by using of FTA flammability unit, (Stanton Redcroft S/N710, UK). The vertical burning test was applied for evaluation of LOI in accordance with ASTM D2863-13 (2013). In this regard, samples of 15 cm x 5 cm x 1.2 to 3.1 cm (length x width x height) were prepared. The samples were used to test the minimum oxygen content necessary for ignition with a basic flame in 3 min.

X-ray diffraction

X-ray diffraction (XRD; 3003 PTS, Seifert, Germany) was utilized to define the connection index and relative intercalation of the nanoclay particles. The examination was performed under CuKα irradiation, (λ=1.54 °A, 50 kV, 50 mA), steps of 0.02°, and a speed of 0.3°/sec in the angles of 2θ = 1 to 10°. The samples were formed into laminar shapes of 10 × 1 × 1 mm^3, and the power supply was set to 30 mA and 40 KV.

Scanning electron microscope

Scanning electron microscopy (SEM) was used to determine the morphology of the composite and polymer mixtures. For this purpose, a Leo Oxford, model 440i (UK), scanning electron microscope was utilized.

RESULTS AND DISCUSSION

The oxygen index of virgin polystyrene is approximately 18 to 18.5. In comparison with virgin polystyrene, the oxygen index of the sample with recycled polystyrene was reduced by approximately 2%. Increasing the amount of wood flour and nanoclay led to higher oxygen indexes. Generally speaking, if a material has a higher oxygen index, it means that it actually requires more oxygen to ignite. Therefore, the greater the oxygen index, the more difficult it is to optimize the flaming conditions because of the thermal resistance of the material. Samples containing 40 wt.% wood flour had a smaller oxygen index in comparison with those composed of 60 wt.% wood flour (Fig. 1). The antioxidant content and the oxygen index are less in the recycled polymers; therefore a smaller amount of oxygen is required for burning, increasing the flammability of the composite (Khosravian 2010).

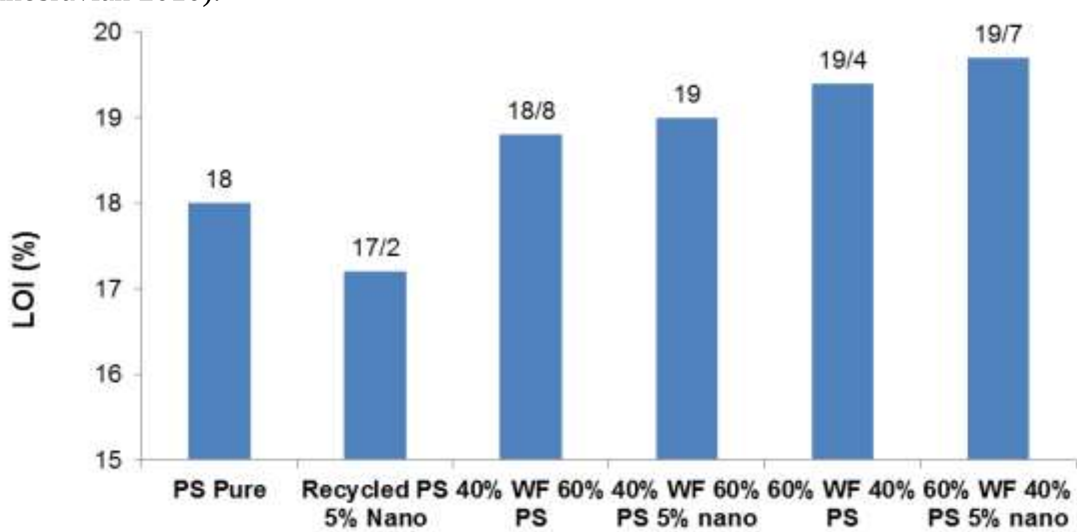

Fig. 1. Effect of wood flour and nanoclay on the oxygen index of nanocomposites

Flammability Behavior

Effect of wood flour on flammability behavior

Composite materials usually contain pores, which are created when steam or volatile materials mix together or during other manufacturing processes. These pores are interconnected with each other and develop a network of void spaces. This network allows oxygen to pass through these pores and enhance ignition. Materials that contain a higher content of cellulose fibers generally have a greater composite density and resistance to oxidation. Therefore, if the porosity of the composite is reduced, then the oxidation is restricted (Khosravian 2010). Increasing the oxygen index results in more oxygen required for burning the sample, making the ignition process more difficult. The oxygen index was increased in this study by increasing of the ratio of the wood flour to pure polymer, which was in accordance with the existing literature (Chiu and Wang 1998; Li *et al.* 2006).

Effect of nanoclay on flammability behavior

An acceptable way to improve ignition-retarding properties of the polymers is to use nanoparticles with layered silicates (nanoclay), such as organically modified montmorillonite. Nanoclay has been shown to significantly enhance the ignition retarding properties of the polymer/clay nanocomposites (Gilman 1999; Zhu *et al.* 2001; Morgan *et al.* 2002). It is generally believed that because of the barrier effect of the nanoclay, which impedes penetration of O_2 into the sample, destruction of the nanocomposite occurs at a higher temperature (Gilman *et al.* 2006). Meanwhile, the presence of nanoclay may prevent the release and movement of volatile and flammable gases, as well as movement from inside the composite towards the surface (Burnside and Giannelis 1995). Moreover, accumulation of the nanoparticles in the composite leads to stress concentration, such that the porosity increases because of reduced density in the stress region. As a result, the ignition is favored and the thermal stability decreases (Klyosov 2007). In other fire retardants, the reduction of flammability is associated with the loss of mechanical properties, whereas the mechanical properties are improved in the composites containing nanoclay (Morgan *et al.* 2002; Ray *et al.* 2003; Sahraian 2003; Zabih Zadeh 2005; Kord 2010).

Morphology

Morphological studies by XRD examination

X-ray diffraction patterns of the nanocomposites made from wood flour and recycled polystyrene were obtained for the 40 wt.% and 60 wt.% wood flour/polystyrene materials and the 0 wt.% and 5 wt.% nanoclay at the angles of 1 to 10°.

As shown in Fig. 2, the XRD peak of the modified nanoclay was formed in 2θ = 4.75° and a layer distance of 18.5°. Increasing of the content of nanoclay from 3 to 5 wt.%, with 40 wt.% recycled polystyrene and 60 wt.% wood flour, moves the XRD peak towards smaller angles, while the distance between the silicate layers increased by d = 48.21Å and 2θ = 1.83°, and d = 51.9Å and 2θ = 1.7°, respectively.

Based on Fig. 3, the XRD peak of the modified nanoclay was formed in 2θ = 4.75°, with a layer distance of 18.5Å. Increasing of the nanoclay content from 3 to 5 wt.%, with, moves the XRD peak towards smaller angles, while the layer distance between the silicate layers increased by d = 48.19Å and 2θ = 1.87°, and d = 46.2Å and 2θ = 1.91°.

This phenomenon led to more regular and effective organization of the recycled polystyrene within the clay silicate layers and also improved the interface with the wood flour, which can itself enhance the thermal properties of the nanocomposite. Thus, since the peaks associated with crystalline region of the clay did not disappear and the 2θ angle decreased slightly, the authors concluded that the nanocomposite exhibited a morphological structure of the intercalation type. The intercalation structure contained a polymer that could penetrate the clay layers and increase the distance between them. However, the layers retained their parallel spatial orientation within the matrix. During delamination, the clay layers are completely separated and individually distributed throughout the polymer. This structure allows for the maximum reinforcement of the polymer substrate (Medani 1999). As observed from the XRD patterns, the morphological structure of the nanocomposites exhibited characteristics of the intercalation type. In other words, the distance between the silicate layers of the nanoclay increased because of the penetration of the polymer chains. However, the clay layers were not completely disintegrated. Some peaks were also evident in the intercalation structures that were not completely disintegrated; these exhibited a shift to smaller 2θ angles or moved forward to greater 2θ angles (Kord 2011a). Therefore, one of the reasons for the occurrence of the intercalation structure in the nanocomposites may be attributed the increasing MFI content, which can lead to the reduction of the viscosity and molecular weight of the composite (Elloumi *et al.* 2010). The results of this section are in agreement with those reported by Kord (2011b), Danesh *et al.* (2012), Kord (2012), and Samariha *et al*. (2015).

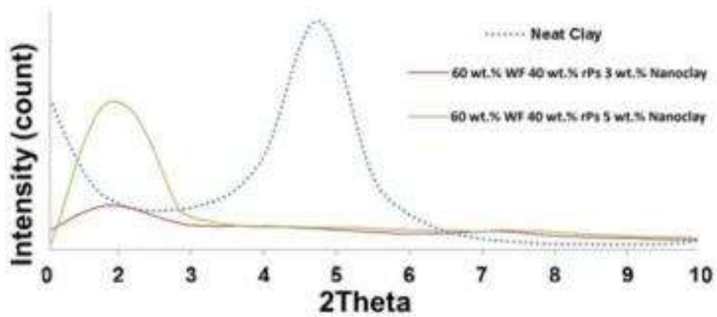

Fig. 2. XRD pattern of a composite made from 60 wt.% recycled polystyrene and 40 wt.% wood flour

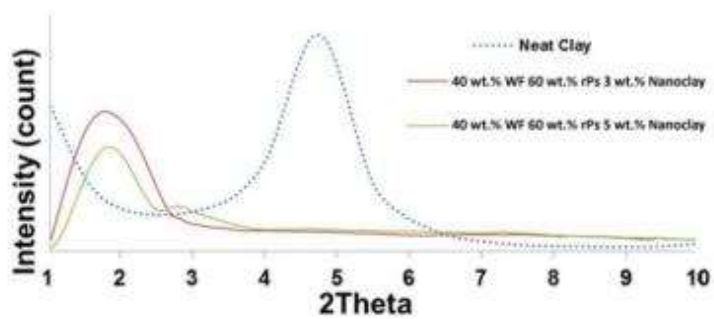

Fig. 3. XRD pattern of a composite made from 40 wt.% recycled polystyrene and 60 wt.% wood flour

Morphological studies by SEM

SEM micrographs showed the distribution and compatibility between the filler materials and the matrix. The void spaces implied a reduction in the oxygen index. Gilman (1999) found that the gaseous data could be used to infer the quantity of charcoal, which was reinforced with the clay soil. Therefore, it is suggested that the charcoal layer may be responsible for thermal stability. The data obtained from this study also supported the performance of the charcoal layer. With the clay soil acting as a barrier to release the oxygen, this extended the time for ignition of the nanocomposites. It was understood in this investigation that numerous reports have shown varying performance of delamination or intercalation of the structure of nanoclay.

Micrographs of the 40 wt.% wood flour and 0 and 5 wt.% nanoclay composites are shown in Figs. 4 and 5. It was noted that increasing the content of nanoclay decreased the porosity of the composite material.

The nanoclay acted by filling in pores to reduce the ignition potential and thermal stability (Khosravian 2010). Furthermore, the nanoclays prevented volatile and flammable gases from being released and moving from the pores to the surface of the composite, thus potentially adding to the oxygen index (Burnside and Giannelis 1995).

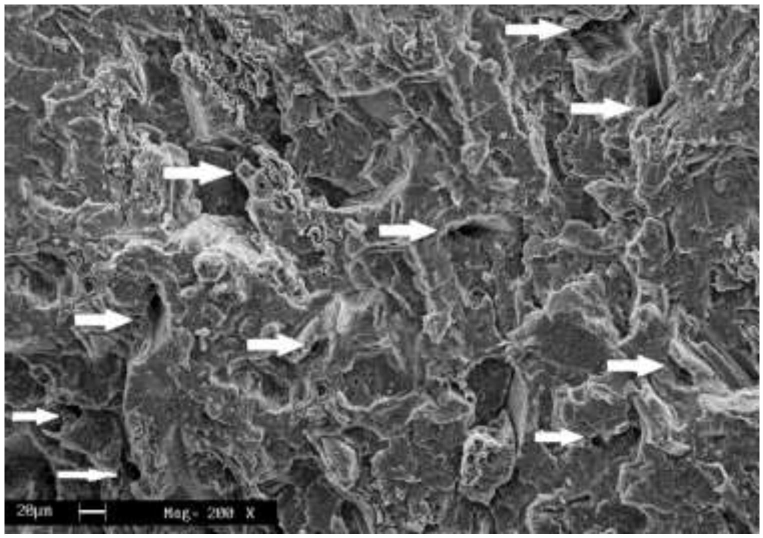

Fig. 4. SEM image of a sample containing 40 wt.% wood flour, 60 wt.% polymer, and 0 wt.% nanoclay (pores have been decreased according with increasing nanoclay content)

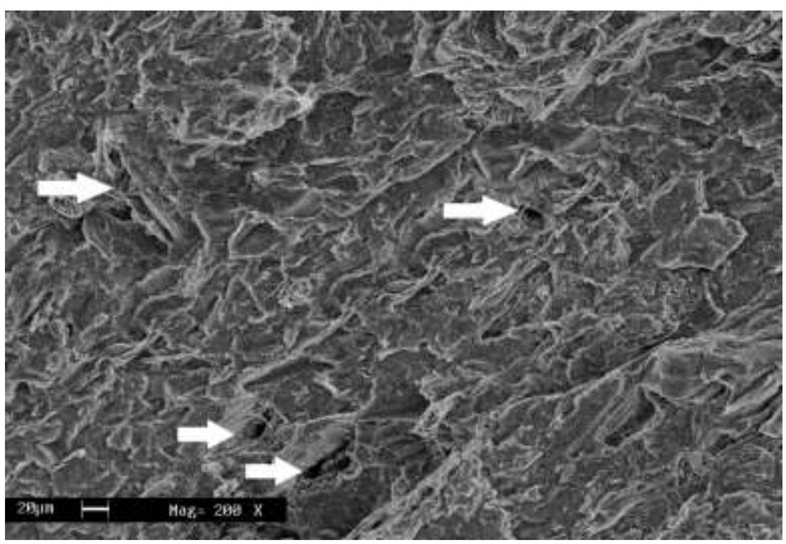

Fig. 5. SEM image of a sample containing 40 wt.% wood flour, 60 wt.% polymer, and 5 wt.% nanoclay (the pores have been decreased according with increasing nanoclay content).

Figures 6 and 7 illustrate the structure of the 60 wt.% wood flour composites with the addition of 0 wt.% or 5 wt.% nanoclay, respectively. It was found that as the nanoclay content was increased, this led to finer pores and voids, as well as a closer connection between the polymer and the lignocellulose material.

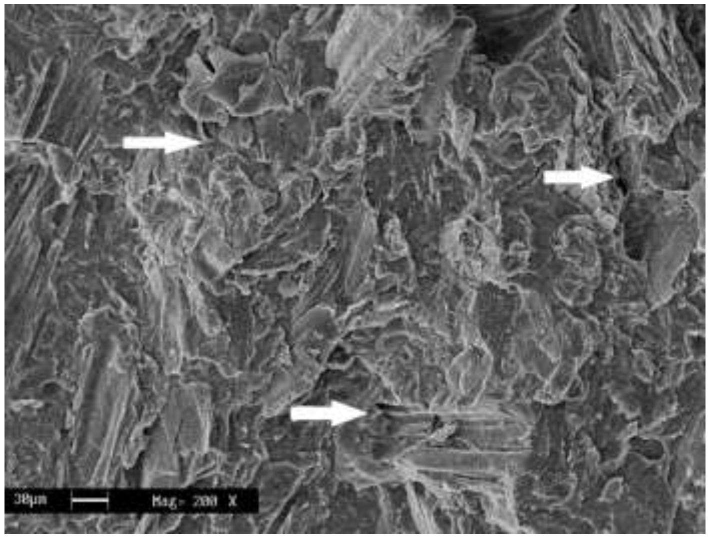

Fig. 6. SEM image of a sample containing 60 wt.% wood flour, 40 wt.% polymer, and 5 wt.% nanoclay (pores have been decreased according with increasing nanoclay content)

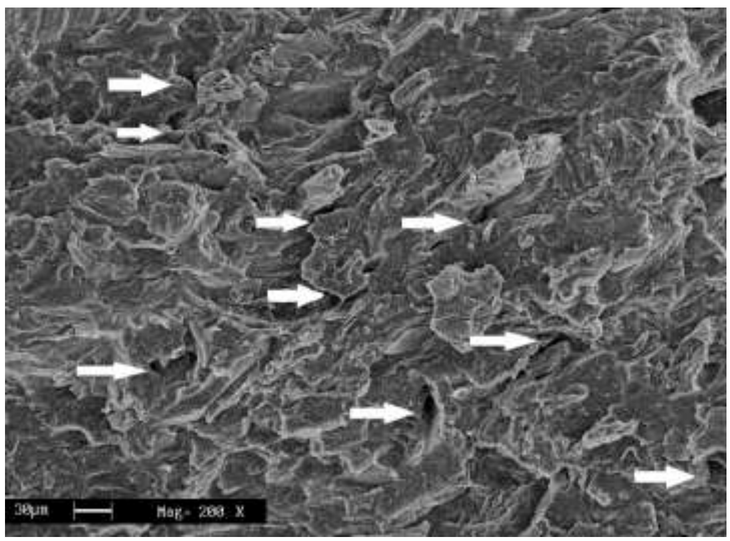

Fig. 7. SEM image of a sample containing 60 wt.% wood flour, 40 wt.% polymer, and 0 wt.% nanoclay (pores have been decreased according with increasing nanoclay content)

Larger voids on the fracture surface were found in the sample with 0 wt.% nanoclay in comparison with the sample containing 5 wt.% nanoclay. The composite with 5 wt.% nanoclay incorporated a smaller number of voids, indicating a weaker connection between the wood flour and the polymer matrix (Guo *et al.* 2007). It has been reported that the nanoclay must be matrix-spread in order to retard ignition. Generally speaking, the ignition retarding mechanism of the silicate-charcoal nanocomposite requires that charcoal disintegrates the substrate material and reduces mass loss of the disintegration products. When the nanoclay was introduced at 5 wt.%, a significant amount of delamination up-regulated the mechanism of retarding the ignition. Meanwhile, the coupling agent used in the nanocomposites was effective for the delamination process. Delhom (2009) showed that the amount of absorbed oxygen, which determines the time to ignition, is generally dependent on the oxygen pressure, the temperature, and the contact area.

A material with an increased surface area (*e.g*. porous, spongy, and powdery) may absorb greater amounts of oxygen; therefore, is more susceptible to ignition. It was observed that the nanoclay acts as a barrier against penetration from oxygen into the nanocomposite structure. This was because the pores and holes are filled in and resist penetration by oxygen.

The fibers were surrounded by the matrix material. The adhesion between the two phases causes the fibers to be fixed in their place. Recycling of the polymer and increasing the MFI content may provide enhanced covering of the particles by the polymer and increase its surface area. Fink *et al*. (2000), Huda *et al*. (2006), and Naeimian (2008) investigated the morphology of composites made from lignocellulose/ polymer fibers using SEM imaging. The micrographs revealed the direction, distribution, and uniform distribution of the reinforcements, indicating that the coupling agents facilitated stress transitioning between the matrix material and the fibers. This also improved the distribution and uniform distribution of the reinforcements in the matrix of the recycled polystyrene. A correlation relationship was identified between the thermal properties of the composites and their microstructural characteristics. Properties of each plastic-reinforced nanoparticle were dependent on the type of reinforcement, arrangement of the particles, and connection of the particles with the polymer phase. The micrographs obtained from SEM of the fractured surface contributed to the discovery of a relatively satisfactory distribution and compatibility between the filler and the matrix. The empty spaces implied a reduction of the oxygen index.

CONCLUSIONS

1. The LOI of pure polystyrene was 4.6 % more than recycled polystyrene.
2. The LOI was increased by increasing the wood flour content from 40 wt.% to 60 wt.%. The LOI was increased 1 and 1.5 percent by increasing nanoclay content from 0 to 5 wt.% in samples with 40 and 60 wt.% wood flour content.
3. Increasing of the nanoclay and wood flour increased the oxygen index.
4. Morphological examination of the nanocomposites by XRD revealed that the formed structure is of the intercalation type.
5. Pores in the composite structure decreased with increasing nanoclay and wood flour components, which justified the subsequent increase of the oxygen index.

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