KENAF AND RICE HUSK FILLED POLYPROPYLENE COMPOSITES CONTAINING AMMONIUM POLYPHOSPHATE

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Specially dedicated to my beloved parents, family and friends…

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ABSTRACT

The main objective of this study was to investigate and compare the effects of kenaf and rice husk fibers content on flammability, mechanical, thermal and morphology properties of polypropylene (PP) composites. The effects of ammonium polyphosphate (APP) content on mechanical and flammability properties of untreated and treated fibers filled PP composites were also investigated. The composites were prepared using a co-rotating twin screw extruder for compounding process and followed by compression molding for test specimen preparation. Rice husk and kenaf fibers were treated by alkaline treatment to modify the fiber surface. Cone calorimeter and limiting oxygen index (LOI) test were performed to determine the flammability properties of the samples. Flexural and impact test were done to determine the mechanical properties of PP composites. In addition, thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) were done to investigate thermal stability of the composites. Addition of APP into PP composites was found to increase the LOI values that indicate the improvement of flame retardancy properties. Whereas, cone calorimeter results showed that incorporation of APP into fibers filled PP composites decreased the heat release rate (HRR) which indicated the improvement of flame retardancy system. Incorporation of APP into PP composites increased the flexural modulus of composites but decreased the flexural strength of composites. On the other hand, the flexural strength of PP composites was observed to increase with the addition of treated fibers. A decrease in impact strength was observed with the addition of APP to PP composites. Incorporation of treated fibers into PP composites also decreased the impact strength. From TGA results, it can be observed that the addition of APP into composites enhanced the thermal stability of composites at high temperature and increased the formation of char residue. The addition of APP in the fibers filled PP composites increased the crystallization temperature of composites. Morphological studies showed that both fibers that underwent alkaline treatment improved the fibers dispersion in the composites and also resulting in better mechanical properties. In this study, it was showed that addition of APP as flame retardant improved the flame retardancy of PP composites. Besides the improvement in flame retardancy properties, the alkaline treatment on rice husk and kenaf fiber has proven to show the better mechanical properties of PP composites.

ABSTRAK

Objektif utama bagi kajian ini ialah untuk mengkaji dan membandingkan kesan-kesan kandungan kenaf dan sekam padi ke atas sifat-sifat kebolehupayaan nyalaan, mekanikal, terma dan morfologi komposit polipropilena (PP). Kesan kandungan ammonium polifosfat (APP) ke atas sifat-sifat mekanikal dan kebolehupayaan nyalaan PP komposit berpengisi serat semulajadi (kenaf dan sekam padi) sebelum dirawat dan yang telah dirawat juga disiasat. Komposit-komposit telah disediakan dengan menggunakan alat penyemprit skru berkembar untuk proses penyebatian dan diikuti dengan alat acuan tekanan untuk tujuan penyediaan sampel ujian. Sekam padi dan kenaf telah digunakan sebagai serat semulajadi dan telah dirawat secara rawatan alkali bagi mengubahsuai permukaan serat. Ujian kon kalorimeter dan indeks pengehad oksigen (LOI) telah dilakukan untuk menentukan sifat-sifat kebolehupayaan nyalaan bagi sampel. Ujian lenturan dan hentaman telah dilakukan untuk menentukan sifat-sifat mekanikal. Tambahan pula, analisis termogravimetri (TGA) dan kalorimeter pengimbasan pembezaan (DSC) dijalankan untuk menyiasat kestabilan terma pada komposit. Penambahan APP ke dalam komposit PP berpengisi APP menunjukkan peningkatan nilai LOI yang mana menunjukkan penambahbaikan dalam sifat-sifat kebolehupayaan nyalaan. Sebaliknya, hasil ujian kon kalorimeter menunjukkan penambahan APP ke dalam komposit PP berpengisi serat semulajadi menyebabkan penurunan dalam kadar pembebasan haba (HRR) yang mana menunjukkan peningkatan dalam sistem rencatan nyalaan. Penambahan APP ke dalam komposit PP meningkatkan modulus lenturan bagi komposit tetapi sebaliknya menurunkan kekuatan lenturan komposit. Manakala, penambahan serat semulajadi yang telah dirawat dapat dilihat meningkatkan kekuatan lenturan bagi komposit PP. Penurunan dalam kekuatan hentaman telah dilihat berlaku apabila APP ditambah ke dalam komposit PP berpengisi serat semulajadi. Penambahan serat semulajadi yang telah dirawat juga menyebabkan penurunan dalam kekuatan hentaman. Daripada keputusan TGA menunjukkan penambahan APP ke dalam komposit meningkatkan kestabilan terma komposit pada suhu tinggi dan meningkatkan pembentukan sisa abu. Penambahan APP ke dalam komposit PP berpengisi serat semulajadi juga meningkatkan suhu penghabluran bagi komposit. Kajian morfologi menunjukkan bahawa kedua-dua serat semulajadi yang telah melalui rawatan alkali meningkatkan penyerakan serat-serat di dalam komposit dan ini mengakibatkan peningkatan dalam sifat-sifat mekanikal. Di dalam kajian ini, ia menunjukkan bahawa penambahan APP sebagai perencat kebakaran telah meningkatkan sifat-sifat kebolehupayaan nyalaan bagi PP komposit. Selain peningkatan dalam sifat-sifat kebolehupayaan nyalaan, rawatan alkali ke atas sekam padi dan kenaf juga telah terbukti menunjukkan sifat-sifat mekanikal bagi PP komposit yang lebih baik.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVATIONS

APP	-	Ammonium Polyphosphate
ASTM	-	American Standard Test Method
DSC	-	Differential Scanning Calorimetry
H_2O	-	Water
LOI	-	Limiting Oxygen Index
MA	-	Maleic Anhydride
MAPP	-	Maleic Anhydride grafted Polypropylene
MAH-g-POE / POE-g-MAH	-	Maleic Anhydride grafted Poly(ethylene-co-octene)
NH_3	-	Ammonia
PHC	-	Per Hundred Composites
PHRR	-	Peak Heat Release Rate
POE	-	Poly(ethylene-co-octene)
PP	-	Polypropylene
RHF	-	Rice Husk Flour
TGA	-	Thermogravimetry Analysis
THE	-	Total Heat Evolved
TTI	-	Time to Ignition
WF	-	Wood Flour
WRHA	-	White Rice Husk Ash

LIST OF SYMBOLS

T_c	-	Crystallization Temperature
T_g	-	Glass Transition Temperature
T_m	-	Melting Temperature
%	-	Percentage
°C	-	Degree Celcius
wt%	-	Weight over Weight Percentage

CHAPTER 1

INTRODUCTION

1.1 Research Background

Polymeric materials reinforced with natural fibers provide advantages of high stiffness and strength to weight ratio as compared to conventional construction materials (Saheb and Jog, 1999). Natural fiber reinforced composites have gained popularity nowadays because of their processing advantage and good technical properties. Practically, the types of thermoplastics that can be used with natural fiber are limited due to low thermal stability of natural fiber. Polypropylene (PP) is one of the commodity thermoplastics which have good properties such as good rigidity and tensile strength and also inactive toward acids, alkalis and solvent compared with other thermoplastic materials. PP is used for wide range of application such as household goods, packaging, and automobile due to its good processability, relatively high mechanical properties, and low cost compared with other thermoplastics.

Synthetic polymers which are widely used today are petroleum based and it is expected that petroleum production will decrease resulting in an increase in the market value of the polymer based products. However, the addition of natural fiber into polymer matrix can reduce the cost production of polymer material and also increased the market value of natural fiber. The advantages using natural fiber as reinforcements in composites are low density and non abrasiveness nature which resulting in low production cost and better specific properties. Hemicelluloses

content in natural fibers which contribute to biodegradability properties for the natural fibers (Akil *et al*., 2011).

There are many types of natural fibers that have been used as reinforcement in the composites such as jute, bamboo, kenaf and others. Kenaf (Hibiscus Cannabinus, L.family Malvaceae) is one of lignocellulose plants that can be developed over a great extent of weather conditions (Nishino *et al*., 2003). Kenaf which is one of commercial plants in Malaysia was chosen as natural fiber for plastic reinforcement due to its ability to increase the stiffness of the composites significantly (Sameni *et al*., 2003). A study by Rowell *et al*. (1999) reported that the tensile and flexural modulus of 50 wt% kenaf fibers filled PP composites is approximately similar or higher than 40 wt% glass fibers filled PP composites.

Rice husk is one of agro-wastes from rice milling process. This agricultural by-product is an excellent thermal insulating due to the significant amount of silica content (Marti Ferrer *et al*., 2004). Like kenaf, rice husk which also contain cellulose, hemicelluloses and lignin increased the modulus of the composites. The hemicelluloses content in rice husk can help in biodegradation process. As the objective of this study is to produce biodegradable polymer composite, rice husk is one of the suitable fillers to be incorporated into polymers. According to Marti-Ferrer *et al*. (2004), the lignin and hemicellulose contents in rice husk fiber are lower than wood fiber which makes the rice husk able to be processed at a higher temperature of 250 °C. A similar study by Sain *et al*. (2004) also reported that incorporation of rice husk into PP composites increased the modulus of the composites.

As stated above, the addition of natural fibers into PP can improve the mechanical properties of the composite. However, addition of natural fibers into PP can also affect the flame retardancy of the composites. Due to natural fiber being more prone to flame, naturally addition of natural fiber into composites will increase the flammability of the composites. To overcome this, flame retardant is added into PP to increase flame retardancy of the composites. Several studies have been done on flammability of natural fiber filled PP composites (Zhang *et al*., 2012 and Abu

Bakar *et al*., 2010). The studies showed that the addition of flame retardants enhanced the flame retardancy of the PP composites by disrupting the burning process of the composites. With the improvements in flame retardancy, the applications of PP composites will further widen into areas such as in automotive and housing sectors.

Natural fibers which are hydrophilic have poor interfacial properties with polymer matrix, which is hydrophobic in nature (Li *et al*., 2007). Therefore, fiber surface modification is needed to improve the interaction between fiber and matrix. The natural fibers surface contains many reactive hydroxyl groups. Hydroxyl groups in natural fibers can also be found in the lignin and in the free and bound water present in most commercially available materials. Fiber surface modification by using chemicals can activate the hydroxyl groups, thus introduce the new moieties that can help the natural fibers to interlock with the matrix (Li *et al*., 2007).

There are various chemical treatments which are used to modify the fiber surface such as mercerization, isocyanate treatment, acrylation, acetylation, silane treatment and others. In this study, mercerization or alkaline treatment are used to improve the fiber-matrix adhesion and strength. In alkaline treatment, sodium hydroxide (NaOH) was used to activate the hydroxyl groups in natural fibers, thus influencing the molecular orientation of the cellulose crystalline due to the extraction of lignin and hemicellulosic compounds (Kalia *et al*., 2009).

During processing, the agglomeration occurred is probably due to poor compatibility between natural fiber and PP and high moisture absorption which reduce the effectiveness of natural fiber (Saheb and Jog, 1999). The presence of very fine fibers caused a high interfacial surface between polar filler and nonpolar matrix. This leads to the worsening bonding between both of them and thus decreased the tensile strength of the composite (Rosa *et al*., 2009). As a way to overcome this limitation, a compatibiliser such as maleic anhydride grafted polypropylene (MAPP) is mixed together with natural fibers filled PP composites. A study by Rosa *et al*. (2009) proved that addition of compatibilizer increased the mechanical properties of the composites. Incorporation of MAPP in natural fibers filled PP composites

accommodates polar interactions such as acid-base interactions and become a bridge between the hydroxyl groups and the lignocelluloses natural fiber.

There are some requirements which are needed for the flame retardants to be effective in PP. The flame retardant must be thermally stable up to the PP processing temperature which is below 260 °C in the fibre-forming process (Zhang and Horrocks, 2003). Other than that, there must be no leaching and immigration properties present thus it could be well-matched with PP. The additive also should be kept hold its flame retardant properties whenever it exhibits in the fiber. The flame retardant addition into PP also can decrease the toxicity of gas and smoke that develop from combustion process to only at permissible level. Furthermore, the flame retardant level should be fairly at low level, typically around 10 wt% to at least achieve a minimum effect on the composites properties (Zhang and Horrocks, 2003).

As mentioned previously, several researches have been done on flammability of natural fiber filled PP composites. One of them is the study on wood fiber filled PP composites by Zhang *et al.* (2012). In the study, wood fiber has been used as natural fiber with incorporation of ammonium polyphosphate (APP) as flame retardant into PP composites. APP as an effective flame retardant was proven when the flame retardancy of the composites was increased with addition of APP. Besides that, Abu Bakar *et al*. (2010) also investigated and compared the flammability of wood fibers filled PP composites containing various flame retardant.

1.2 Problem Statements

Previous studies on mechanical properties of PP composites with kenaf and rice husk as natural fibers have been done. The results showed that using kenaf and rice husk as reinforcement in PP composites contributed to good mechanical properties and biodegradability properties (Nishino *et al*., 2009 and Rosa *et al*., 2009). Because of drawback such as high moisture absorption, researches also have been done on treatment of natural fibers to increase the mechanical properties of

composites (Li *et al*., 2007). Recently, there is a demand for composites which not only possess good mechanical properties but also have good flame retardancy properties. However, the incorporation of natural fibers into composites decreased the flame retardancy due to natural behaviour of natural fibers which is more prone to flame (Sain *et al*., 2004). Therefore, flame retardant was added to improve the flame retardancy of the composites. Generally, there are not much research being done on flammability of natural fibers filled PP composites and especially, there is still no report on flammability properties of treated rice husk and kenaf fibers filled PP composites. The chemical treatment and compatibilizer were used in order to achieve satisfying mechanical properties. In a previous study, magnesium hydroxide was used as flame retardant to increase the flame retardancy in rice husk filled PP composites (Sain *et al*., 2004). In this study, the flammability, mechanical and thermal properties of rice husk and kenaf fibers filled PP composites containing APP as flame retardant were investigated.

1.3 Objectives of Study

The overall objective of this study is to develop a new composite material based on kenaf/rice husk and PP, which has good mechanical properties and enhanced flammability resistance.

This can be further divided into:

1) To compare the effect of natural fiber types (kenaf and rice husk) on flammability, mechanical and thermal properties of PP composites.
2) To determine the effect of APP loading on mechanical and flammability of natural fibers filled PP composites.
3) To investigate the effect of chemical treatment of natural fibers on mechanical and flammability of APP filled PP composites.
4) To compare the effectiveness of maleic anhydride grafted poly(ethylene-co-octene) (POEgMAH) and MAPP as compatibilizers for natural fibers and PP.

1.4 Significance of Study

In this study, a new thermoplastic composite filled with natural fibers with good flammability resistance and mechanical properties is expected to be developed. Incorporation of natural fibers in polymeric material will produce materials which are suitable for wood replacement. In addition, the natural fibers that were used are among domestic plants which can be easily found in Malaysia. The application of these fibers can help to provide a cost reduction of the product due to the relatively cheaper price of natural fibers. The product will also have another property value added which is flame retardancy with the addition of APP as flame retardant.

1.5 Scope of Study

a) In this study, rice husk and kenaf fiber were used as natural fibers. The samples preparation involved the following stages:

   i. Natural fibers were grinded and sieved to obtain <500 µm of size fiber. 500 µm of size fiber range was chosen to reduce the tendency of agglomeration.
   ii. Some of the natural fibers were undergo chemical treatment with NaOH.
   iii. Melt extrusion blending
   iv. Compression moulding

b) Flexural test and impact test were carried out to determine the mechanical properties of the composites.

c) Limiting oxygen index (LOI) test and cone calorimeter test were done to investigate the flammability properties of the composites.

d) As for thermal characterization, thermogravimetry analysis (TGA) and differential scanning calorimetry (DSC) were done to analysis thermal stability and properties of the composites.

e) SEM (Scanning Electron Microscopy) was carried out to analyze microstructure and morphology of the composites.

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