UNIVERSITI PUTRA MALAYSIA

PREPARATION, CHARACTERIZATION, AND THERMAL AND PHOTO DEGRADATION OF POLYPROPYLENE/OIL PALM LEAVES FIBER COMPOSITE

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Master of Science

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Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

PREPARATION, CHARACTERIZATION, AND THERMAL AND PHOTO DEGRADATION OF POLYPROPYLENE/OIL PALM LEAVES FIBER COMPOSITE

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Chairman: **Nor Azowa Binti Ibrahim, PhD**
Faculty: **Science**

The fast increase in world population dramatically increases the need of packaging materials. Synthetic polymers are expensive, nonabrasive and nonrenewable materials. The increase usage of these materials has originated an issue of solid waste disposal. The accumulation of plastic waste in landfills is becoming a hazard for the environment, and ultimately for human. Therefore reduction of the uses of synthetic polymers especially for the above mentioned applications should be given priority.

Polypropylene (PP) is normally tough, flexible and suitable to be used in different applications such as packaging, labeling and textiles. While oil palm leaf fiber (OPLF) found to be good filler for thermoplastic polymer composites. In this study the use of OPLF as a reinforcement material for PP composites, and their mechanical and thermal properties, were investigated. Effects of Epolene E-43 as a compatibilizing agent for the composites on their mechanical, thermal properties and morphology were assessed. Moreover, the effect of iron (III) chlorides on the thermal and photo-oxidative degradation of the composites was also examined via thermogravimetric analysis (TGA) and UV-irradiation study of the doped PP/OPLF composites. OPLF was extracted from dried-oil palm leaf (OPL) and the composite sheet was formed by melt blending technique using internal mixer machines and compression molding. Composites of 10, 20, 30, 40, 50 and 60% by weight of OPLF were prepared.

The addition of Epolene-43 as compatibilizer improves the overall properties of PP/OPLF composites. The composite with 20w% fiber loading possess the best mechanical and chemical properties. Increase in fiber loading, in the compatibilized and uncompatibilized composites, reduces the tensile strength and elongation at break. However, it increases in tensile modulus, flexural strength and modulus of the composites. Water absorption increases with the increase of fiber loading by 10, 13, 15% for 40, 50 and 60w% fiber loading, but the addition of Epolene E-43 reduces water absorption. Interaction between the components of the composite is only physically as indicated by Fourier Transform infrared spectroscopy (FTIR) results except for 20w% fiber content. The addition of Epolene E-43 forms new ester bonds between the components of the composites. It was observed from differential

scanning calorimetric (DSC) data that the degree of crystallinity reduces by 44% and 36% for both 50w% of modified and unmodified fiber content composites, respectively, with the increase of OPLF content.

Thermo gravimetric analysis (TGA) results indicate that the PP/OPLF composites are more stable than the PP. Meanwhile dynamic mechanical analysis (DMA) shows that the presence of the OPLF enhances mobility but reduces the stiffness. Addition of iron (III) chloride accelerates thermal and photo degradation of PP/OPLF composites. Epolene E-43 is a good compatilizer as it reduces the composite void size and number as shown by field emission scanning electron microscopy (FE-SEM) micrographs indicating its addition improve interaction of OPLF and PP matrix.

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PENYEDIAAN, PENCIRIAN, DAN KEMEROSOTAN TERMA DAN FOTO SERAT DAUN POLIPROPILENA/KOMPOSIT KELAPA SAWIT

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Peningkatan pesat jumlah penduduk di dunia secara mendadak meningkatkan permintaan bahan pembungkusan. Polimer sintetik adalah mahal, menjejas alam sekitar dan tidak boleh diperbaharui. Peningkatan penggunaan bahan-bahan tersebut telah mewujudkan isu pelupusan sisa pepejal. Sisa plastik yang terkumpul di tapak pelupusan adalah bahaya kepada alam sekitar dan manusia. Oleh itu pengurangan penggunaan polimer sintetik terutamanya untuk aplikasi yang disebutkan di atas perlu diberi keutamaan.

Polipropilena (PP) biasanya keras, fleksibel dan sesuai untuk digunakan dalam aplikasi yang berbeza, termasuk pembungkusan, pelabelan dan tekstil. Sementara serat daun kelapa sawit (OPLF) mempunyai ejen pengukuhan yang baik untuk komposit polimer termoplastik. Dalam kajian ini OPLF diguna sebagai bahan pengukuhan untuk komposit PP, dan sifat-sifat mekanikal dan termanya telah dikaji. Kesan Epolene E-43 sebagai agen penyerasian untuk komposit keatas sifat mekanik, terma dan morfologi telah dikaji. Selain itu, kesan daripada besi (III) klorida terhadap pemecahan oleh haba dan pengoksidaan foto komposit juga telah diperiksa menggunakan analisis Termogravimetri (TGA) dan kajian pemancaran sinar UV yang didopkan komposit PP / OPLF. OPLF telah diekstrak daripada daun kelapa sawit yang telah dikeringkan (OPL) dan kepingan komposit telah dibentuk oleh teknik pengadunan lebur menggunkan alat pencampur dalaman dan acuan pemampat. Komposit 10, 20, 30, 40, 50 dan 60 % daripada berat serat telah disediakan.

Penambahan Epolene E-43 menambahbaik keseluruhan sifat komposit PP / OPLF. Komposit dengan penambahan 20% serat mempunyai sifat mekanikal dan kimia yang terbaik. Peningkatan penambahan serat dalam komposit yang mempunyai bahan keserasian dan tidak menpunyai bala keserasian, telah mengurangkan kekuatan tegangan dan pemanjangan pada pemutusan. Walau bagaimanapun, ia meningkatkan modulus tegangan, kekuatan lenturan dan modulus komposit. Penyerapan air meningkat pada 10, 13 , 15% bagi penambahan serat pada 40, 50 dan 60% tetapi penambahan Epolene E-43 mengurangkan penyerapan air. Hanya interaksi fizikal yang berlaku antara komponen komposit seperti hasil yang diperolehi daripada spektroskopi inframerah transform Fourier (FTIR) kecuali pada 20% kandungan serat. Penambahan Epolene E-43 membentuk ikatan ester yang baru.

Ia telah dilihat melalui data kalorimetri pengimbasan pembezaan (DSC) di mana dengan peningkatan kandungan OPLF telah mengurangkan darjah penghabluran sebanyak 44% dan 36% untuk kedua-dua 50% daripada kandungan serat komposit yang telah diubahsuai dan tidak diubahsuai.

Hasil analisis termo gravimetrik (TGA) menunjukkan bahawa komposit adalah lebih stabil daripada PP. Sementara analisis mekanikal dinamik (DMA) menunjukkan bahawa kehadiran OPLF meningkatkan mobiliti tetapi mengurangkan kekukuhan. Penambahan besi (III) klorida mempercepatkan kemerosotan terma dan foto PP/ OPLF komposit. Epolene E-43 adalah bahan keserasian yang baik kerana ia mengurangkan saiz dan ruang kosong menunjukkan penambahan yang meningkatkan interaksi OPLF dan matriks PP. Seperti yang dapat dilihat dalam mikrograf pancaran mikroskop imbahan elektron (FE-SEM).

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I certify that a Thesis Examination Committee has met on 20 June 2014 to conduct the final examination of Khayat, Huwayda Abdulaziz A on her thesis entitled "Preparation, Characterization, and Thermal and Photo Degradation of Polypropylene/Oil Palm Leaves Fiber Composite" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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DECLARATION

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This is to confirm that:

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
DMA	Dynamic mechanical analysis
DMTA	Dynamic mechanical thermal analysis
DSC	Differential scanning calorimetry
DTG	Differential thermogravimetric analysis
E-43	Epolene E-43
FE-SEM	Field emission scanning electron microscope
FT-IR	Fourier transforms infrared
LMCT	Ligand-to-metal charge-transfer
MAH	Maleic Anhydride
MLCT	Ligand-to-metal charge-transfer
MPOB	Malaysian palm oil board
OPF	Oil palm fiber
OPL	Oil palm leaves
OPLF	Oil palm leveas fiber
PE	Polyethylene
PP	Polypropylene
SEM	Scanning electron microscopy
TGA	Thermogravimetric analysis
TPU	Taman Pertanian Universiti
USA	United states of America
UV	Ultraviolet
Mw	Molecular weight

rpm	Rotor speed (revolutions per minute)
Tg	Glass transition temperature
Tm	Melting temperature
Wt%	Weight percentage
Xc%	Percent crystallinity
ΔH_m	Heat of fusion

CHAPTER 1

INTRODUCTION

1.1 Background of Research

The widespread utilization of non-natural plastics, especially as packaging materials in various food products, has caused many complications to health, society, environment and economy. Therefore, the replacement of this material is the top priority concern. Recently, waste management faced a problem of rapidly reduce of landfill field and dramatic increase in the cost of plastic waste disposal. Moreover, the production and waste incineration of petrochemical-derived synthetic polymers, such as polyethylene and polypropylene cause greenhouse gas emission. The requirement of using safe and degradable materials is a prominent research subject and serious legislation aspects. Polymer researchers found out that green chemistry could be used to produce biodegradable polymers due to their biodegradability, renewability and environment-friendly products.

The industrial composite packaging manufacturers should consider of using reinforcement plant fibers as it may reduce the usage of synthetic polymers. Several studies on the properties of natural fiber reported that the benefit of these materials lies in their low weight, free formability and low density, which leads to have high specific strength (Jawaid and Abdul Khalil, 2011). The usage of natural fillers as reinforcement in composite could produce cheap and environmental friendly products, which reduce the environmental pollution because of their naturally degradable constituents (Yang *et al*., 2007).

Tropical countries, such as Malaysia disposed large quantities of oil palm leaves (OPL) from the pruning process of palm oil trees. Lignocellulosic fiber of OPL can considered as prospective reinforced fiber, since it has numerous advantages, such as biodegradability, renewability and environmental friendly (Arbelaiz *et al*., 2005a). However, there is a lack of information about the use of OPL as reinforcement fillers in composites. The fast progress in the research of natural based fiber composite has continued vigorously to find out methods of using lignocellulosic material as reinforcing fillers in synthetic polymers, such as polyethylene (PE) and polypropylene (PP). They have higher mechanical properties than lignocellulosic material because of its hydrophobic properties (Yang *et al*., 2007). The large differences between hydrophilic lignocellulosic fiber and hydrophobic polymer matrix lead to weak interfacial bonds between them, which reduce the mechanical properties of the resulted composites (Cantero *et al.*, 2003). Coupling or compatibilizing agents have also positive result in better adhesion between lignocellulosic fiber and PP matrix (Colom *et al.*, 2003). Several effective coupling agents for lignocellulosic/PP composite have been established, such as graft copolymers of PP and maleic anhydride (MAH) (Felix and Gatenholm, 2003; Li *et al*., 2001; Sathe *et al*., 2003). The compatibility between fibers and PP matrix has been improved by using these coupling agents and the diffusion of flax fiber in PP was also enhanced (Van De Velde and Kiekens, 2001; Arbelaiz *et al*., 2005a). Different compatibilizers, such as Epolene E-43 (maleic anhydride modified-poly-propylene) (García-López *et al*., 2007; Rozman *et al*., 2001), poly[methylene (polyphenyl isocyanate) (Pickering and Ji, 2004), gmethacryloxypropyltrimethoxysilane (A-174), vinyltri (2-methoxy ethoxy) silane (A-172) (Yazdani *et al*., 2006), g-aminopropyltriethoxysilane (Liu and Wang, 2007), poly(propylene-acrylic acid) and poly(propylene±ethylene acrylic acid) have been studied for lignocellulosic fiber/PP.

Among all the studied coupling chemicals, Epolene E-43 (E-43) is the most efficient one due to its three main advantages: (i) its large marketable availability, (ii) its alkoxysilane groups content at one end which is able to react with large amount of hydroxyl groups in the surface, and (iii) it has large number of functional groups that capable to modify of the matrix function. E-43 was reported to enhance the mechanical properties of the composite, since it has high maleic anhydride (MAH) groups, which tends to form chemical bonding with hydrophilic lignocellulosic fiber, whereas it could smooth the progress of wetting the hydrophobic polymer chain, which can enhance the compatibility between both components (Yang *et al*., 2007). There is a scarce of information regarding the impact of fiber loading and E-43 on mechanical and thermal properties of composites. Thus, this study was focusing to determine the optimum ratio of mixing PP with oil palm leaves (OPL) lignocellulose and to identify the influence of coupling agent on the mechanical and thermal properties of the composites. Maleic anhydride from E-43 coupling agent was used to evaluate the improvement in the interfacial bounding between PP matrix and OPL lignocellulosic fiber and their mechanical and thermal characteristics were assessed by performing several analysis.

Synthetic polymers have high heat stability. However, in the presence of moisture or oxygen the absorption of thermal, light or mechanical energy can lead to decrease their physical and mechanical properties (Bernstein *et al*., 2005). Related to several studies, the degradation mechanisms of thermal and photo- chemical oxidation are similar, except for the initiation step of the oxidation reaction (Tidjani and Wilkie, 2001). Thus, stabilization is important to avoid thermal and photo-oxidative degradation for long-term use of PP products.

Thermal oxidation of polyolefin occurs mainly through the abstraction of a hydrogen atom of the methylene groups (Carroccio *et al*., 2007; Cerruti *et al*., 2003). The impurities such as catalyst residues and metal ions or sensitizers, i.e. chromophores, carbonyl or peroxides, which are formed during high temperature of polymer processing, favoured the light absorption to break carbon-hydrogen bond (Gijsman *et al*., 1999). Furthermore, the decomposition of hydroperoxides plays an important role in starting branching reactions during continued oxidation (Allen *et al*., 2004; Tidjani and Wilkie, 2001). Specifically, the activation energy of the bimolecular decomposition reaction is often quite low and even at low temperatures weakening properties can occur (Gugumus, 2002). Moreover, the oxidation chains usually have a short length in polyolefin oxidation system, which may cause the lowering of stabilizing effectiveness of conventional chain- breaking antioxidants (Lánská, 1996; Rychly *et al*., 1997).

Several transition metal salts are able to form organization complexes with methylene groups of polyolefin (Cerruti and Carfagna, 2010; Nielsen *et al*., 2002), leading to affect its thermal and photo-oxidative stability (Gupta and Jais, 1993; Lánská *et al*., 2005). Polypropylene has a feature of forming coordination complexes with an extensive variety of metal cations leading to consistent chemical structure, presence of contributing oxygen atom in each segment and high chain flexibility (Rabek *et al*., 1992; Schellenberg, 2009). The obligated behavior of ions to PP depends mostly on the active charge density on the chain, the conformation of polymer, size of the cation and valence, ratio of the polymer molar: the polarity of salt and solvent.

However, in the presence of oxidizing agents PP is sensitive to UV-irradiation. In recent years, several studies found that transition metal salts had accelerating the photo- oxidative degradation of this polymer (Kaczmarek, 1996; Kaczmarek *et al*., 2001; Kaczmarek *et al*., 2002; Morlat-Therias *et al*., 2005; Rabek *et al*., 1992; Roy *et al*., 2009). Iron salts ($FeCl_3$) has been reported as the most effective types of polyolefin stabilizing systems include inorganic inhibitors even at low concentrations (3w%) (Kaczmarek *et al*., 2001). The influence of iron salt on photo oxidation of PP exposed to UV irradiation for time periods of weeks has been investigated. Changes in spectroscopic properties were observed. The importance of this analysis lies in understanding the real polymer aging behaviour. The effect of iron salt on the thermal and thermo-oxidative degradation of PP/OPLF composites has also been investigated.

1.2 Problem Statements

Among limitation of using (PP) is nonrenewable origin raw material and non-biodegradable. This research aimed to prepare biocomposite by incorporating oil palm leave fiber (OPLF) into the PP matrix. Maleic anhydride PP (Epolene-43) was added to increase compatibility between nonpolar PP and polar OPLF while oxidant $FeCl_3$ was added to increase oxidative ability of the composite.

1.3 Scope of Study

The aim of this study is to produce new biodegradable composite from polypropylene and oil palm leaves fiber by using melt-blending technique. In this study natural fibers were extracted from OPL and incorporated in polypropylene with different ratio that included 10, 20, 30, 40, 50, 60 w%. Mechanical properties (tensile and flexural), thermal properties (thermogravimetric analysis and differential scanning calorimetric analysis), morphology of the blends (scanning electronic microscopy), physical testing (water absorption and photo-degradation analysis) and other characterization (dynamic mechanical analysis) of the composites were evaluated.

1.4 Objectives of Study

The objectives of the study were:

1. To prepare and characterize PP/OPLF composites and to determine the optimum ratio of PP and OPLF based on mechanical properties.

2. To study the effect of E-43 coupling agent on the mechanical and thermal

properties of the composites.

3. To investigate the thermal and photo degradation and stability of composites.

4. To identify the effect of iron salt on the stability and degradation of the composites.

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