



**EFFECT OF KAPOK HUSK CONTENT AND
CHEMICAL TREATMENT ON PROPERTIES OF
LINEAR LOW DENSITY POLYETHYLENE ECO-
COMPOSITES**

by

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A thesis submitted in fulfilment of the requirements for the degree of
Master of Science in Materials Engineering

**School of Materials Engineering
UNIVERSITI MALAYSIA PERLIS**

2016

ACKNOWLEDGEMENT

Praise to Allah s.w.t for giving me a chance and strength in completing my master programmed. Firstly, I would like to acknowledge to the School of Materials Engineering department for allowed me to learn and gain knowledge during my postgraduate studies. Sincere thanks to all of them who gave their hand during my researched project.

I would like to express my special heartfelt gratitude and appreciation to my kind and supportive supervisor, Dr. Lim Bee Ying for her helps and support. Also, thank you to my late lecturer, Assoc. Prof. Dr. Ir Salmah Husseinsyah for her guidance and generous support. All of her scientific advices as well as her understanding in every phase of this research would be appreciate. Thanks also to her patience and encouragement to lead me on the right way. Without their help, I could not successfully finish my research.

Thank you to my beloved parents, Arshad bin Mohd Yasin and Rusidah Abd Rasid for their continuously supports and love during the progress of my studies. Not to forget my only one brother, Muhammad Farid bin Arshad, thank you so much. This thesis could not have happened without the love and consistent encouragement from all of them.

Appreciation also goes to all of my friends for supporting me during my research and useful helps and information to get through along this project completion.

I would like to expand my appreciation to all Universiti Malaysia Perlis's staff and technicians especially from School of Materials Engineering for their help from time to time during this project. Once again I would like to thanks to all for the supports and guidance offered to me along this project.

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LIST OF SYMBOL

| | |
|----------------------|--|
| $^{\circ}\text{C}$ | Degree Celcius |
| ΔH°_f | Heat of fushion for 100% crystalline LLDPE |
| ΔH_f | Heat of fushion for LLDPE |
| % | Percentage |
| μm | Micrometer |
| cm^{-1} | Reciprocal Wavelength (wavenumber) |
| cm^3 | Centimetre cubic |
| X_c | Degree of crystallinity |
| wt% | Weight percentage |
| lb | pound |
| MPa | Mega pascal |
| rpm | Revolutions per minute |
| M_t | Percentage of water absorption |
| W_o | Original dry weight |
| W_f | After immersed weight |
| T_{dmax} | Decomposition temperature at maximum rate |
| T_m | Melting Temperature |

LIST OF ABBREVIATIONS

| | |
|-------|---|
| LLDPE | Linear Low Density Polyethylene |
| KH | Kapok Husk |
| SEM | Scanning Electron Microscope |
| FTIR | Fourier Transform Infrared |
| TGA | Thermogravimetric Analysis |
| DSC | Differential Scanning Calorimetric |
| DTG | Derivative Thermogravimetric |
| PP | Polypropylene |
| PE | Polyethylene |
| LDPE | Low Density Polyethylene |
| PLA | Poly Lactic Acid |
| PHA | Polyhydroxyalkanoate |
| PVS | Poly (vinyl sulfatate) |
| ABS | Acrylonitrile butadiene styrene |
| PKS | Palm Kernel Shell |
| php | Part per hundred polymer |
| TPCS | Thermoplastic cassava starch composites |
| HDPE | High density polyethylene |
| RPF | Rambutan Peel Flour |
| ASTM | American Society for Testing Materials |
| rPP | Recycle Polypropylene |
| SA | Stearic Acid |
| UP | Unsaturated Polyester |

| | |
|-------|-----------------------------------|
| NF | Nypa Fruticans |
| MMA | Methyl Metacrylate Acid |
| rLDPE | Recycle Low Density Polyetyhylene |
| PEAA | Polyethylene Acrylic Acid |
| RH | Rice Husk |
| UCS | Ultimate compressive strength |
| WF | Wood Flour |
| VTMOS | Vinyltrimethoxysilane |
| DCP | Dicumyl Peroxide |
| CA | Citric Acid |
| CAPS | Citric Acid Modified Pea Starch |
| CARS | Citric acid Modified Rice Starch |
| MAA | Methacrylic Acid |
| PMMA | Poly (Methyl Methacrylate) |
| CPH | Cocoa Pod Husk |
| OPEFB | Oil Palm Empty Fruit Bunch |
| RC | Regenerated Cellulose |
| SDS | Sodium Dodecyl Sulfate |
| CSP | Coconut Shell Powder |
| LA | Lactic Acid |
| AHA | Alpha Hydroxy Acid |
| ETSS | Elasteriospermum tapos seed shell |

LIST OF NOMENCLATURES

| | |
|-----------------|-------------------------------|
| C | Carbon |
| C=O | Carbonyl |
| C=C | Ethylene (IUPAC name: Ethene) |
| C-O-C | Ether |
| OH | Hydroxyl |
| CH ₂ | Methylene |
| CH ₃ | Methyl Group |

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Kesan Sekam Kekabu dan Rawatan Kimia Terhadap Sifat-sifat Eko-Komposit Linear Polietilina Berketumpatan Rendah

ABSTRAK

Penggunaan sekam kekabu (SK) sebagai isian di dalam linear polietilina berketumpatan rendah (LPEKR) telah dikaji. Kesan kandungan SK dan rawatan kimia terhadap sifat-sifat tensil, morfologi, terma, dan serapan air eko-komposit LPEKR/SK telah dikaji. Eko-komposit LPEKR/SK telah disediakan dengan menggunakan Brabender Plasticoder EC-Plus pada suhu 160°C dengan kelajuan rotor 50 rpm. Empat jenis rawatan kimia telah digunakan iaitu asid metakrilik (AMA), asid sitrik (AS), asid laktik (AL), dan sodium dodecyl sulfat (SDS). Keputusan menunjukkan penambahan SK ke dalam eko-komposit LPEKR telah mengurangkan kekuatan tensil, pemanjangan pada takat putus dan penghabluran. Walau bagaimanapun, modulus elastisiti dan serapan air dalam eko-komposit telah meningkat dengan peningkatan kandungan SK. Kehadiran SK telah menurunkan kestabilan terma LPEKR/SK eko-komposites berbanding LPEKR sendiri. Walau bagaimanapun, peningkatan SK meningkatkan kestabilan terma. Kajian morfologi terhadap eko-komposit telah menunjukkan interaksi yang lemah di antara pengisi SK dan matrik LPEKR. Kesan rawatan kimia terhadap SK telah meningkatkan sifat-sifat tensil dan terma dalam eko-komposit. Kekuatan tensil dan elastisiti modulus LPEKR/SK eko-komposit yang telah dirawat dengan AMA, AS, AL, dan SDS adalah lebih tinggi berbanding dengan eko-komposit yang tidak dirawat. Penghabluran dan kestabilan terma dalam eko-komposit LPEKR/SK yang telah dirawat juga adalah lebih baik berbanding eko-komposit yang tidak dirawat. Eko-komposit yang telah dirawat menunjukkan rintangan air yang lebih baik dari eko-komposit yang tidak dirawat. Interaksi yang lebih baik antara pengisi-matrik telah dibuktikan melalui kajian imbasan mikroskop elektron (IME). Analisa FTIR spektra menunjukkan perubahan kepada kumpulan berfungsi di dalam SK yang telah dirawat. Daripada keseluruhan jenis rawatan kimia, eko-komposit yang dirawat oleh AMA menunjukkan kekuatan tensil, modulus elastisiti, kestabilan terma, dan penghabluran yang paling tinggi berbanding dengan jenis rawatan yang lain.

Effect of Kapok Husk content and Chemical Treatment on Properties of Linear Low Density Polyethylene Eco-Composites

ABSTRACT

The utilization of kapok husk (KH) as filler in linear low density polyethylene (LLDPE) was studied. The effect of KH content and chemical treatments on tensile properties, morphology, thermal properties and water absorption of LLDPE/KH eco-composites were investigated. The LLDPE/KH eco-composites were prepared by using Brabender Plasticorder EC-Plus at temperature 160 °C and rotor speed 50 rpm. The four types of chemical treatments used were methacrylic acid (MAA), citric acid (CA), lactic acid (LA) and sodium dodecyl sulfate (SDS). The results indicated that the addition of KH in LLDPE eco-composites had decreased the tensile strength, elongation at break and crystallinity of the eco-composites. However, the modulus of elasticity and water absorption of eco-composites increased with increasing of KH content. Presence of KH has lowered the thermal stability of LLDPE/KH eco-composites compare to neat LLDPE. However, the increment of KH content improved the thermal stability. The morphological studies of eco-composites exhibited poor interfacial interaction between KH filler and LLDPE matrix. The effect chemical treatment of KH had increased the tensile and thermal properties of eco-composites. The tensile strength and modulus of elasticity of treated LLDPE/KH eco-composites with MAA, CA, LA, and SDS were higher than untreated eco-composites. The crystallinity and thermal stability of treated LLDPE/KH eco-composites were also improved compared to untreated eco-composites. The treated eco-composites had better water resistance than untreated eco-composites. The better filler-matrix interaction has been proven by scanning electron microscope (SEM). Spectra FTIR analysis showed the changes of functional group of treated KH. From various chemical treatments, the treated eco-composites with MAA showed the highest tensile strength, modulus of elasticity, thermal stability, and degree of crystallinity compared to others treated eco-composites.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Polymer eco-composites have been concerned an expanding studied in the course of recent decades for both industrial applications and fundamental research (Thomas et al., 2014; Petchwattana et al., 2013). Recently, a remarkable interest towards eco-composites has been attracted due to increasing environmental consciousness and demands of governmental authorities. The terms “eco-composites” are subjects to eco-friendly natural filler filled polymer matrix (Gaceva et al., 2007). The complex combination between polymer matrices and eco-friendly natural filler is usually made up for enhancement of properties of the products. Moreover, polymer eco-composites are more economic and environmentally friendly compare to a raw polymer matrix. Expanding in environmental awareness all throughout the globe has constrained the researchers to create new green materials that enhance the environmental quality of products (Gupta & Gupta, 2007; Chun et al., 2015b). In addition, several commercial programs towards momentum of eco-composites have been done such as the expanding worldwide natural and social worry, consumption of petroleum assets, and new ecological controls (Bagoeva et al., 2007).

Polymer matrix is basically inert to microorganisms or the chemicals in an environment (Kahar et al., 2012; Prachayawarakorn et al., 2010). Due to this, to eliminate a high volume of polymer waste may take a long time to degrade which prompted to natural issues. A developing effort has merged the polymer researchers to expand ecological materials lately by the addition of natural filler (Luo et al., 2014)

instead of synthetic fillers (Hamid et al., 2015; Chun et al., 2014; Sébastien et al., 2015). Towards the unstable economic worldwide nowadays, customers definitely preferred a high quality product beneficial with a low cost. Finding addition materials for plastic could involve lower cost by using natural filler as reinforcement to the polymer eco-composites (Nitayaphat et al., 2009). They have been consolidated in polymer matrices with the major objective of giving improvement in properties and a decrease in cost of the final products (Leaversuch., 2002). These also prevent the waste of potentially useful materials and reduced the consumption of raw polymer matrix with addition of natural filler (Choi et al., 2006; Ashori et al., 2009; Ashori et al., 2010).

The plastic industries are a key enablers of innovation of many products and technologies in other sectors of the economy such as engineering, medicine, automobile, sports, domestic, packaging, etc (Nwanonenyi et al., 2013). This is because of their simplicity of handling and low energy consumption during fabrication and their inertness, which makes them appropriate to be utilized as a part of all fields (De Carvalho et al., 2002). Moreover, the unique characteristics of plastics also allowed them to make a strong contribution to a more environmentally sustainable and resources efficient in worldwide (Qaiss et al., 2014; Malha et al., 2013). The increasing demand for plastics industries is expected to be one of the key factors driving the expansion of plastics market (Petchwattana & Covavisaruch., 2014; Manikantan et al., 2012). Linear low density polyethylene (LLDPE) matrix is one of a polymer with numerous applications, for example, film applications (e.g. stretch cling film, silage film), plastic bags, liquid paper board coatings, wire and cable, injection moulding parts and pipes (Geng et al., 2006). Generally, linear low density polyethylene is versatile polymer among polyethylene. It is a vital commercial polymer which is generally utilized for various applications as a part of current innovation. LLDPE has greater mechanical

properties which have higher tensile strength, impact and puncture resistance (Hemati et al., 2011; Nestore et al., 2012; Kontou & Niaounakis., 2006). Meanwhile, Nwanonenyi et al., (2013) has reported that LLDPE has very flexible, elongates under stress, and has better environmental stress cracking resistance properties.

Some researchers have been reported that the addition of hemp as natural filler in LLDPE eco-composites (Nestore et al., 2013) improved the tensile strength and flexural strength of the eco-composites. This is expected since natural fillers are one of the most prospective materials which have been used to improve the properties of the polymer eco-composites (Pérez et al., 2012; Wang et al., 2008; Fauzani et al., 2014; Moayad & Khalaf., 2015) instead of biodegradable (Kaith & Kaur, 2011), low toxicity, low manufacture costs, low disposal costs and renewability (Puppi et al., 2010). Natural filler proposed a number of advantages over synthetic fillers in various technical applications (Nacher et al., 2007; Yarysheva et al., 2007). It is mainly due to their renewable origin, lightweight, moderately high specific strength, no health hazards, and more economic (Pervaiz et al., 2003). Probably, the most attractive properties the utilization of natural filler filled polymer matrix is the ecological effect, arising out from the fact that these natural fillers can be effortlessly degrade toward the end of their life cycle (Kahar et al., 2012; Prachayawarakorn et al., 2010).

Natural fillers, depending upon the source, are classified into three types which are animal, plant and mineral. Many natural plant types of filler found in Malaysia are obtained from crop residues and by products of the agricultural industry such as empty fruit bunch (Rozman et al., 2003), coconut shell powder (Chun et al., 2013a) and cocoa pod husk (Chun et al., 2014). Kapok is one of the abundance natural filler in Malaysia which has heavily cultivated to obtain kapok cotton for pillow and mattress productions. In the meantime, a numerous of kapok husk has disposed as a by-products. Contribution

to a waste management and to overcome the environmental problem, kapok husk has been utilized as new natural fillers in polymer eco-composites field. Kapok husk has been reported contain high number of cellulose compositions which expected to improve the eco-composites properties (Kobayashi et al., 1977).

Along with these properties, lignocellulosic fillers also have a few drawbacks. Lignocellulosic of natural fillers are prone to absorb moisture in the environment, which affects the properties of eco-composites. The main disadvantage of natural fillers is their hydrophilic natures, which lower the compatibility with hydrophobic polymeric matrices during eco-composites fabrication (Boujmal et al., 2014; Salleh et al., 2014).

However, there is an effort to develop the nature properties of these natural fillers which is chemical treatment. A number of chemical treatments can be done to obtain good properties by improving the compatibility between the polymer matrices and natural filler such as silane treatment (Singha and Takur, 2009), alkaline treatment (Farahani et al., 2012) and esterification (Salmah et al., 2011). Filler treatment is important in natural filler based eco-composites as well as to improve the wettability, dispersion, and filler-matrix interaction. Chemical treatment is performed to overcome the incompatibility between hydrophilic lignocellulosic filler and hydrophobic polymer matrix. The strong interfacial bonding between polymer matrix and natural filler obtained by chemical treatment will improve the physical, mechanical, and thermal properties of the composites system (Essabir et al., 2013).

1.2 Problem Statement

Many previous researchers have reported on the development of linear low density polyethylene (LLDPE) with natural fillers eco-composites. However, the utilization of LLDPE with kapok husk filler eco-composites has not been reported. In

this project, kapok husk (KH) has been used as filler in LLDPE matrix. In order to cut cost usage of neat LLDPE polymer matrix, using of local KH filler can enhance the value of agriculture waste materials which are abundant in Malaysia. The KH could have promising future as new filler in polymer eco-composites not because of their low cost, low density, environmental friendliness and good mechanical properties but also in a context of valorising abundant and unexploited in Malaysian resources.

However, the main problem of LLDPE/KH eco-composites is their incompatibility between hydrophobic LLDPE and hydrophilic KH. Thus, the chemical treatments were used to improve the interfacial interaction between both materials and also reduced the hydrophilicity of natural fillers itself. In this study the different types of organic compound were used such as citric acid, lactic acid, methacrylic acid, and sodium dodecyl sulfate.

1.3 Objectives of Study

The objectives of this study are:

1. To study the effect of kapok husk (KH) filler content on tensile properties, morphological study, water absorption test, and thermal properties of LLDPE/KH eco-composites.
2. To investigate the effect of organic acids chemical treatments such as citric acid, lactic acid and methacrylic acid on properties of LLDPE/KH eco-composites.
3. To study the effect of sodium dodecyl sulfate on properties of LLDPE/KH eco-composites.

1.4 Scope of Study

The scope of this project covers several testing which had been done to investigate the properties of the eco-composites. Tensile properties, morphological study, water absorption test, thermal properties and Fourier Transform Infrared (FTIR) were conducted. Tensile properties of the eco-composites were determined by tensile test in order to measure the tensile strength, elongation at break, and modulus of elasticity of the eco-composites. The morphology of LLDPE/KH eco-composites was carried out by Scanning Electron Microscopy (SEM). Meanwhile, thermal properties performances of LLDPE/KH eco-composites were investigated using thermogravimetry analysis (TGA) and differential scanning calorimetric (DSC). The FTIR was used to determine the functional group of untreated and treated LLDPE/KH eco-composites. Moreover, the water absorption test investigates the ability of the eco-composites to absorb the water.