

**PREPARATION AND CHARACTERIZATION OF
MODIFIED POLYPROPYLENE/ PALM KERNEL
SHELL (PP/PKS) BIOCOMPOSITES**

MURAD SALEM MOHAMED OMAR

UNIVERSITI MALAYSIA PERLIS

2012

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SHELL (PP/PKS) BIOCOMPOSITES**

by

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LIST OF SYMBOLS, ABBREVIATIONS OR NOMENCLATURES

APTES	3-aminopropyltriethoxysilane
ASTM	American Society for Testing and Materials
CO	Carbon Oxide
CO ₂	Carbon Dioxide
DMA	Dynamic Mechanical Analysis
DSC	Differential Scanning Calorimetry
EFB	Empty Fruit Bunch
FT-IR	Fourier Transform Infrared Spectroscopy
Ipp	Isotactic polypropylene
MA	Maleic anhydride
MAPP	Maleinated Polypropylene
MFI	Melt Flow Index
µm	Micrometer
mm	Millimeter
NaOH	Sodium Hydroxide
NMR	<i>Nuclear Magnetic Resonance</i>
°C	centigrade degree
OPKS	Oil Palm Kernel Shell
PKS	Palm Kernel Shell
PP	Polypropylene
PP-g-MAH	Polypropylene grafted Maleic Anhydride

rpm	revolution per minute
SEM	Scanning Electron Microscope
TGA	Thermo gravimetric analysis
TMA	Thermo-mechanical analysis
Vol%	volume per weight
Wt	Weight
ΔH_f	Enthalpy of fusion of the system
ΔH_f°	Enthalpy of fusion of perfectly (100%) crystalline polymer

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PENYEDIAAN DAN PENCIRIAN MENGENAI POLIPROPILINA/ISIRONG KELAPA SAWIT (PP/PKS) BIOKOMPOSIT

ABSTRAK

Kajian mengenai isirong kelapa sawit dan polipropilena biokomposit terdiri daripada empat bahagian. Bahagian pertama adalah untuk mengkaji kesan percampuran isirong kelapa sawit sebagai bahan penambah kepada polipropilena terhadap sifat-sifat mekanikal, penyerapan air, morfologi dan termal. Isirong kelapa sawit (penambahan 5-25 berat% bahan penambah) digabungkan dengan polipropilena dengan menggunakan pencampur dalam Brabender Plastograph pada suhu pemprosesan 180°C dan halaju pemutar 60 rpm. Keputusan awal menunjukkan bahawa kekuatan tegangan, kekuatan lenturan dan pemanjangan pada takat putus menurun dengan peningkatan penambahan pengisi tetapi modulus tegangan, modulus lenturan dan penyerapan air meningkat. Kajian morfologi menggunakan mikroskop elektron imbasan (SEM) menunjukkan interaksi antara muka yang lemah antara isirong kelapa sawit dan polipropilena apabila kandungan pengisi meningkat. Di samping itu, isirong kelapa sawit didapati menjadi ejen penukleusan yang lemah dalam kajian termal. Bahagian kedua kajian adalah untuk mengkaji kesan polipropilena dicantumkan maleic acetic (PP-g-MAH) sebagai pengganding terhadap sifat-sifat mekanikal, morfologi termal, dan penyerapan air pada polipropilena/isirong kelapa sawit. Bahagian ketiga kajian adalah untuk mengkaji kesan rawatan kimia terhadap sifat-sifat mekanikal, termal dan morfologi polipropilena/isirong kelapa sawit biokomposit dengan menggunakan natrium hidroksida (2 berat%). Bahagian akhir melibatkan rawatan pengubahsuaian pada polipropilena/isirong kelapa sawit biokomposit dengan menggunakan agen gandingan. Kesan pengubahsuaian kimia polipropilena/isirong kelapa sawit biokomposit meningkatkan kekuatan tensil, modulus tegangan, kekuatan lenturan, modulus lenturan dan penghabluran tetapi menurunkan penyerapan air. Kajian morfologi menunjukkan interaksi yang lebih baik antara pengisi dan matrik dengan penggunaan 2 berat% 3-aminopropiltriethoxysilane.

PREPARATION AND CHARACTERIZATION OF MODIFIED POLYPROPYLENE/PALM KERNEL SHELL (PP/PKS) BIOCOMPOSITES

ABSTRACT

The research on palm kernel shell (PKS) and polypropylene (PP) biocomposites consists of four parts. The first part is mainly to investigate the effect of incorporating PKS as filler on the mechanical, water absorption, morphology and thermal properties of PP. PKS (5-25 weight % filler loading) was compounded with polypropylene using Brabender Plastograph internal mixer at processing temperature 180°C and rotor speed 60 rpm. Preliminary results show that the tensile strength, flexural strength and elongation at break decreased with the increasing of filler loading but increased the tensile modulus, flexural modulus and water absorption. The morphology study using scanning electron microscopy (SEM) shows poor interfacial interaction between PKS and PP with the increasing of filler content. In addition, PKS was found to be a poor nucleating agent in thermal studies. The second part of the research is to study the effect of polypropylene grafted maleic anhydride (PP-g-MAH) as a compatibilizer on mechanical, thermal properties, morphology and water absorption of PP/PKS biocomposites were studied. The third part of the research is to study the effect of chemical treatment on mechanical, thermal properties and morphology of PP/PKS biocomposites were studied by using sodium hydroxide (NaOH) (2 wt. %). The final part involved subjecting the modification treatment on PP/PKS biocomposites by using coupling agent. The effect of chemical modification of PP/PKS biocomposites are improved tensile strength, tensile modulus, flexural strength, flexural modulus and crystallinity but decreased water uptake. The morphology study shows better interaction between filler and matrix with the usage of 2 vol. % of 3-aminopropyltriethoxysilane.

CHAPTER 1

INTRODUCTION

1.1 Research Background

In recent years, thermoplastic materials are being increasingly used for various applications (Romisuhani et al., 2010). Due to the high market demand, the cost of the thermoplastics has increased rapidly over the past decade. This situation made it necessary to use low cost fillers as a means of reducing the cost of the end product. However, the widely used inorganic fillers, such as glass fibre and mica are very expensive compared to natural fibres (Joseph and Thomas, 1996). Natural fillers-reinforced thermoplastic composites are more economical to produce than the original thermoplastics and, as a result, it may be possible to meet any future shortage of thermoplastics (Joseph and Thomas, 1996).

Moreover, the use of natural filler-reinforced in thermoplastic composites is highly beneficial, because the strength and toughness of the plastics can be improved. However, lack of good interfacial adhesion and poor resistance to moisture absorption made the use of natural fibre reinforced composites less attractive (Ishidi et al., 2011). This problem can be overcome by treating these fillers with suitable chemicals.

The most important chemical modification involves coupling methods. The coupling agent used contains functional groups, which can react with the filler and the polymer, the covalent and hydrogen bonds which improve the interfacial adhesion between fillers and polymer (Gassan and Bledzki, 1997).

It is therefore necessary to modify the fillers, the matrix, or both to enhance the adhesion between the fillers and matrix. It has been widely reported that the filler–matrix interfacial adhesion between natural fiber and polymer matrix can be improved with the addition of a maleic anhydride modified polypropylene (MAPP) as coupling agent to the matrix. There was also evidence that alkaline treatment to natural fiber to suggest that improvements in filler–matrix adhesion can be attained by means of alkali treatment of the fibres (Beckermann and Pickering, 2008).

Polymer–cellulosic filler composites are used primarily in building products such as decking, fencing, siding, and decorative trim. Other applications include infrastructure such as broad walks, marinas, and guardrails, transportation, like interior automotive panels, truck floors and head liners, and industrial and consumer applications such as pallets, playground equipment and benches. Natural filler-reinforced polymer composites are likely to be environmentally superior to glass filler reinforced polymer composites in most applications. Natural filler reinforcement composites following reasons: (1) natural filler production results in lower environmental impacts compared to glass filler. The production of natural filler-reinforced transport pallets uses 45% less energy, and results in lower emission of toxic gases (CO₂, methane, SO₂, and CO) than production of glass filler reinforcement transport pallets; (2) Natural filler reinforcement composites have higher filler content for equivalent performance, which reduces amount of more polluting base polymers (Demira et al., 2004). Biocomposite materials have been developed that offer certain mechanical and environmental advantages and also renewable-abundant resources. Biocomposite are defined as composites materials that build up by natural cellulose fillers as reinforcement fillers and starch or biopolymer as natural matrix. Biocomposites also called as green composites.

According to Marsyahyo and co-workers (Marsyahyo et al, 2008), the density of ramie fillers is much less than that of synthetic fillers such as E-glass fillers but ramie fillers has surface characteristic to be applied as superior reinforcement in composite material. The specific strength and specific modulus of natural fibres are comparable or even superior to E-glass fibres. Hence, there is an opportunity for using the natural fibres such as ramie) to replace the E-glass fibre for a composite reinforcement (Drzal et al., 2004). Drzal et al., (2004) suggested that in order to develop biocomposites with better mechanical properties, it is necessary to solve the problems by suitable treatments to enhance the compatibility between fillers and the matrix. Natural fillers are inexpensive, abundant and renewable, lightweight, degradable and abrasive to processing equipments (Marsyahyo et al, 2008).

Other filler that attract materials engineer in recent years is lignocellulosic fillers. These lignocellulosic materials consist of lignin, hemicellulose and cellulose have become an alternative over conventional filler like glass filler, calcium carbonate and others. This is due to its environmentally friendly because lignocellulosic materials are derived from plants. Palm kernel shell (PKS) is one of the lignocellulosic materials which are derived from palm oil plants. PKS are being considered as agricultural waste because only its kernels were extracted for palm oil that used for daily cooking.

Polypropylene play important role in composite whereby it protect the fillers from environment threat. The usage of polypropylene in plastic manufacturing industrial increased- every year compared to other low cost polyolefins because of its high temperature resistance, easy to process and high crystallinity (Peacock and Calhoun, 2006).

1.2 Problem Statement

Each year, the agricultural field in Malaysia produces more than 70 million tons of agricultural products (Wei, 2008). These are agricultural products, which are the most abundant biomass resources are oil palm fiber, palm oil empty fruit bunch (EFB) and coconut shell.

The tropical climate in Malaysia, where there is adequate rainfall and sunlight throughout the year contributes to the high rate of biomass production (Wei, 2008). Unfortunately, the number resulting from the production of by-products creates problems of waste management, where the removal of by-products in large quantities it is difficult and expensive (Wei, 2008). Taking into account the environmental effects of this bio-waste, the number of investigations and studies has considered the conversion of biomass in these high-value products, which serve as raw materials to support other industries (Wei, 2008). In order to reduce this problem, PKS waste can be used to combine with polymer to form biocomposites.

However, a homogenous dispersion of PKS with polymer is often difficult to achieve due to the strong tendency of the fillers to agglomerates. Since the compatibilizer is expensive the filler was treated by using chemical treatment. In order to overcome this problem, the chemical treatment by using NaOH , coupling agent and PP-g-MAH copolymer was employed as compatibilizer which can formed boundary layer between the filler and matrix thus, improved the adhesion of the filler with matrix.

1.3 Objectives of Study

The primary objective of this work is to characterize and determine the properties of Polypropylene (PP)/palm kernel shell (PKS) biocomposites on a different composition of PKS loading with an aim of developing a new natural filler material as useful filler in thermoplastic based composites. PP/PKS biocomposites are prepared to study:

1. The effect of palm kernel shell (PKS) as filler on mechanical, thermal properties and water absorption of PP/PKS biocomposites.
2. The effect of polypropylene grafted maleic anhydride (PP-g-MAH) on mechanical, thermal properties and water absorption of PP/PKS biocomposites.
3. The effect of sodium hydroxide (NaOH) on mechanical, thermal properties and water absorption of PP/PKS biocomposites.
4. The effect of silane coupling agent (3-aminopropyltriethoxysilane) on mechanical, thermal properties and water absorption of PP/PKS biocomposites.

1.4 Scope of Study

The research focuses on the influence of different filler loading on the mechanical, thermal and physical properties of the biocomposites. PKS fillers were used as reinforcement in the PP matrix. The filler content was varied from 0, 5, 10, 15, 20 and 25 wt%. To improve the fibre-matrix adhesion, chemical treatment by NaOH with 2 wt%, silane agent 2 vol% and PP-g-MAH 2 wt% as a compatibilizer were performed on the fillers. The processing of the natural filler biocomposite involves Brabender Plastograph® EC Plus and Compression molding process. The prepared composites were tested for its mechanical properties (tensile and flexural). The thermal properties

which include TGA and DSC were studied. The water absorption was studied to PP/PKS biocomposites. The morphology was closely observed by Scanning Electron Microscope (SEM) of chemical treated fillers and tensile fractured surfaces of the biocomposite specimens.

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CHAPTER 2

LITERATURE REVIEW

1.1 Introduction

These days various synthetic polymers are being prepared combined with various reinforcing fillers in order to improve the mechanical properties and obtain the characteristics demanded in actual application (Yang et al., 2004). Polymer composites based on natural fillers are currently receiving great attention as innovative materials for industrial applications in several sectors, such as automotive, building, appliance, packaging and biomaterials. The main advantage of employing natural fibers is that these are biodegradable and renewable, and exhibit low cost, low density and high toughness (Yu et al., 2006). Fillers which merely increase the bulk volume and hence, reduce price, are known as extender fillers while those which improve mechanical properties, particularly tensile strength are termed as reinforcing fillers (Isaac et al., 2012).

Particulate filled polymer composites have been used in increasing quantities in various applications, e.g., in the automotive industry, for appliances or as garden furniture. The properties of these composites are basically determined by the behavior of the matrix, the properties of the filler, the composition, the particle spatial distribution and the interfacial interaction (Michael et al., 2010).

Generally, the properties of filled polymers change with the dispersion state, geometrical shape, and surface quality of the filler particles as well as the particle size.

The majority of cellulosic feedstocks are lignocellulosic materials of different polarity to thermoplastics. Lignocellulosic materials such as agricultural residues have

been widely used as reinforcing fillers in natural filler-thermoplastic polymer bio-composites. These bio-composites, which are made using lignocellulosic materials as reinforcing fillers, are inexpensive, biodegradable, thus causing minimal environmental pollution, and non-toxic to the human body (Yang et al., 2005).

2.2 Lignocellulosic materials as fillers

Fillers are used to improve the working properties of thermoplastics, such as the strength, rigidity, durability, and hardness (Othman et al., 2006).

The addition of natural filler in polymeric composites aims to produce unique characteristics of great versatility, light weight, biodegradability and recyclability with good specific properties. In comparison with other fillers such as traditional glass fiber and inorganic mineral fillers, wood is inexpensive, low density and non-abrasive to processing machinery (Qiu et al., 2006).

Studies are ongoing to find ways to use lignocellulosic fibers in place of synthetic fibers as reinforcing fillers. These natural fillers are especially being sought since the production of composites using natural substances as reinforcing fillers is not only inexpensive but also able to minimize the environmental pollution caused by the characteristic biodegradability (Premalal et al., 2002), enabling these composites to play an important role in resolving future environmental problems. The need for materials that are non-toxic to the human body and have appropriate characteristics for specific purposes is ever increasing due to the lack of resources and increasing levels of environmental pollution. Thus, research is proceeding to develop composites using various recycled wastes (Son et al., 2001, and Son et al., 2004), especially in developing composites using most environmentally friendly agro-wastes (lignocellulosic materials)

as reinforcing fillers and thermoplastic polymers as matrixes. The convenience of these composites lies in the fact that the ingredients are obtained easily from natural wastes and hence the composites can be made relatively easily. They can be used to resolve environmental problems and to produce products with various physical properties and effective functions. Lignocellulosic materials as reinforcing fillers in plastics, in place of the previously used inorganic substances and synthetic fibers, offer a major benefit in terms of environmental protection.

Biodegradable lignocellulosic filler possess several advantages compared to inorganic fillers, such as lower density, greater deformability, smaller abrasiveness, high stiffness, reduce dermal and respiratory irritations, good thermal properties, enhance energy recovery and relatively lower cost. It is shown that incorporation of fillers as reinforcing materials significantly changes various properties of thermoplastics (Coutinho et al., 1999).

2.3 Palm Kernel Shell

Malaysia is the second largest oil palm exporting countries in the world. The demand for vegetable oil in the international market is on the rise. Every year, palm oil industries produce large volume of OPKS as waste material after the production of palm oil (Ramli et al., 2003) stated that nearly 5 million ha of oil palm trees is expected by the year 2020 in Malaysia alone. This will increase the production of both palm oil and its wastes.

Palm kernel shell (PKS) is one of the lignocellulosic material families obtained from crude palm oil producing factories in Asia and Africa.

PKS are hard stony endocarps that surround the kernel and the shells come in different shapes and sizes, they are light and naturally sized. Being hard and of organic origin, they will not contaminate or leach to produce toxic substances. Normally, the shells are flaky and of irregular shape that depend on the breaking pattern of the nut. PKS are available in large quantities in palm oil producing countries in Asia and Africa. Malaysia alone produces nearly 4 million tons of PKS annually and this is likely to increase as more production is expected in the near future (Alengaram et al., 2008). The shell of the nut of Palm kernel tree do not have economic value, so when incorporated into a polymer matrix, it results into a material of appreciable cost benefit.

2.4 Polypropylene

Polypropylene was the first synthetic stereo regular polymer to achieve industrial importance and it is presently the fastest growing fibre for technical end-uses where high tensile strength coupled with low-cost are essential features; it has shown consistent growth of about 5% per annum for the last 10 years. In 1999, worldwide consumption of polyolefin fibres exceeded 5.5 million tonnes and they accounted for 18% of the world's synthetic fibre production. Polypropylene fibres have been widely used in apparel, upholstery, floor coverings, hygiene medical, geotextiles, car industry, automotive textiles, various home textiles, wall-coverings (Zhang et al., 2003).

Polypropylene is semi-crystalline polymer which consists of both crystalline and amorphous phase. It was discovered in 1950s and used in wide variety of application such as indoor/outdoor carpet fibers, thermally insulating fabric, disposable diaper, children's toys, dish washer-safe food container and others. It became favourable plastic among the manufacturers because of its durable, toughness, strength, easy processing and high melting point compare to other inexpensive polymers. However,