

**CHARACTERIZATION AND PROPERTIES OF PALM  
KERNEL SHELL FILLED LOW DENSITY  
POLYETHYLENE BIOCOMPOSITES**

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**UNIVERSITI MALAYSIA PERLIS**

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**Characterization and Properties of Palm Kernel Shell  
Filled Low Density Polyethylene Biocomposites**

**By**

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## LIST OF SYMBOLS, ABBREVIATIONS OR NOMENCLATURES

AA	Acrylic acid
ASTM	American Society for Testing and Materials
CAPE	Carboxylated polyethylene
COCA	Coconut Coupling Agent
DSC	Differential scanning calorimetric
DTA	Differential thermal analysis
EAA	Ethylene-acrylic acid
EBAGMA	Ethylene-butyl acrylate-glycidyl metacrylate
EDD	Ethylene diamine dilaurate
EFB	Empty fruit bunch
FTIR	Fourier transform infrared spectroscopy
HDPE	High density polyethylene
HDS	Hexadecyltrimethoxy-silane
HPLC	High performance liquid chromatography
LDPE	Low density polyethylene
LLDPE	Linear low density polyethylene
MAPE	Maleated polyethylene
MAPP	Maleated polypropylene
MPS	Methacryloxypropyltrimethoxy
MRPS	$\gamma$ -mercaptoproxytrimethoxy
Mt	Percentage of water absorption
OHF	Olive husk flour
PALF	Pineapple-leaf fiber
PEAA	Polyethylene co-acrylic acid
PLA	Poly (lactic acid)
PPEAA	Poly (propylene-ethylene acrylic acid)
PKS	Palm kernel shell
POFA	Palm oil fatty acid
PP	Polypropylene
PS	Polystyrene
PVC	Poly (vinyl chloride)

RHDPE	Recycled high density polyethylene
SEM	Scanning electron microscopy
$T_c$	Crystallization
TDM	Titanium-derived mixture
$T_g$	Glass transition temperature
TGA	Thermogravimetric analysis
$T_m$	Melting point
TS	Tensile strength
Wd	Original dry weight
Wn	Weight after immersed
WOWP	Waste office white paper
WP	Waste paper
$X_{biocom}$	Crystallinity of biocomposites

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**PENCIRIAN DAN SIFAT-SIFAT BIOKOMPOSIT TEMPURUNG KELAPA  
SAWIT (PKS) TERISI POLIETILENA BERKETUMPATAN RENDAH (LDPE).**

**ABSTRAK**

Biokomposit berdasarkan tempurung kelapa sawit (PKS) dan polietilena berketumpatan rendah (LDPE) telah dikaji. Biokomposit disediakan dengan menggunakan pencampur bilah-Z pada 180°C dengan kelajuan rotor 50rpm. Kesan pembebanan pengisi tempurung kelapa sawit (PKS) terisi di dalam LDPE keatas sifat-sifat mekanikal, penyerapan air, morfologi, sifat terma dan spektroskopi inframerah transformasi fourier (FTIR) telah dikaji. Keputusan menunjukkan bahawa peningkatan pembebanan PKS menyebabkan kekuatan tensil dan pemanjangan pada takat putus berkurang tetapi modulus Young dan penyerapan air didapati meningkat. Kajian morfologi menggunakan mikroskop elektron pengesanan (SEM) menunjukkan bahawa pembebanan pengisi yang tinggi menyebabkan interaksi antara muka adalah lemah antara PKS dan LDPE. Penghabluran biokomposit meningkat dengan semakin meningkatnya pembebanan PKS. Untuk meningkatkan sifat-sifat mekanikal biokomposit, polietilena ko-akrilik asid (PEAA) digunakan sebagai bahan pengserasi. Kesan modifikasi kimia ke atas biokomposit dengan polietilena ko-akrilik asid (PEAA), asid akrilik (AA) dan agen pengkupel kelapa (COCA) telah meningkatkan kekuatan tensil, modulus Young dan penghabluran tetapi mengurangkan pemanjangan takat putus dan penyerapan air. Kajian mikroskop elektron pengesanan (SEM) juga menunjukkan bahawa interaksi antara pengisi-matrik didapati meningkat dengan kehadiran PEAA, AA dan COCA. Keputusan FTIR menunjukkan nombor gelombang kumpulan hidroksil untuk kesemua biokomposit semakin berkurang yang mana menunjukkan bahawa interaksi diantara kumpulan hidroksil daripada PKS dengan bahan pengserasi dan agen pengkupel.

**CHARACTERIZATION AND PROPERTIES OF PALM KERNEL SHELL  
(PKS) FILLED LOW DENSITY POLYETHYLENE (LDPE) BIOCOMPOSITES.**

**ABSTRACT**

Biocomposites based on palm kernel shell (PKS) and low density polyethylene (LDPE) was investigated. The biocomposites were prepared by using Z-Blade mixer at processing temperature 180 °C and rotor speed 50 rpm. The effect of filler loading of PKS as filler in LDPE on mechanical properties, water absorption, morphology, thermal properties and fourier tranformation infrared spectroscopy (FTIR) were studied. The results show that the increasing of filler loading have decreased the tensile strength and elongation at break but increased the Young's modulus and water absorption. The morphology study using scanning electron microscopy (SEM) shows poor interfacial interaction between PKS and LDPE with increasing of the filler loading. The crystallinity of the biocomposites increased with increasing of PKS loading. To improve the mechanical properties, compatibilizer, Polyethylene co-acrylic acid (PEAA) was used. The effect of chemical modification of biocomposites with polyethylene co-acrylic acid (PEAA), acrylic acid (AA) and coconut coupling agent (COCA) enhanced the tensile strength, Young's modulus and crystallinity of the biocomposites but reduced the elongation at break and water absorption. The morphology study (SEM) shows that the filler-matrix interaction was improved with incorporation of PEAA, AA and COCA. The FTIR results show that the wave number of hydroxyl group for all biocomposites shifted to lower wave number which indicates that interaction between the hydroxyl groups from PKS with compatibilizer and coupling agent.

# CHAPTER 1

## INTRODUCTION

### 1.1 Research Background

A biocomposite are composite materials comprising one or more phases derived from abiological origin. In terms of the reinforcement, this could include plant fibres such as cotton, flax, hemp, and the likes or fibres from recycled wood or waste paper, or even by-products from food crops. Regenerated cellulose fibres (viscose/rayon) are also included in this definition, since ultimately, they too come from a renewable resources (Paul et al., 2006). Biocomposites from plant and wood based fibers are used in a wide range of products, including aerospace, automotive, and building materials. Using biomass fibers to reinforce plastics has several advantages over synthetic ones. They offer environmental benefits because of their renewable nature and low energy consumption in production (Maya & Sabu, 2008).

© Biocomposites are obtained by the combination of biodegradable polymer as the matrix material and biodegradable fillers (e.g., lignocellulosic fillers). Since both components are biodegradable, the composite as the integral part is also expected to be biodegradable (Ave´rous et al., 2006). For short-term applications, biocomposites present strong advantages, and many number of papers have been published on this topic (Barreto et al., 2010; Fransisco et al., 2010; Ave´rous et al., 2006).

Currently, the main market for biocomposites are in the automotive and construction sectors which further development and improvement are still taking places. However, new opportunities and application will likely arise. Significant opportunities are likely to occur in the built environment at this sector which responsible for producing huge volumes of waste at a time. This environmental impact of industries is coming under close scrutiny.

The specific mechanical properties of natural fibres are comparable to those of traditional reinforcement. Thus, the intrinsic properties of natural fibres can satisfy the request of the global market especially for those industries concerned in weight reduction (i.e., automotive). That is why they can be a potential substitute for non-renewable synthetic fibres.

Natural fibres are subdivided based on their origins, coming from plants, animals or minerals. All plant fibres are composed of cellulose while animal fibres consist of proteins (hair, silk, and wool). Plant fibres include bast (or stem or soft sclerenchyma) fibres, leaf or hard fibres, seed, fruit, wood, cereal straw, and other grass fibre. Over the last few years, a number of researchers have been involved in investigating the exploitation of natural fibres as load bearing constituents in composite materials. Natural organic fibres from renewable natural resources often the potential to act as a biodegradable reinforcing materials alternative for the use of glass or carbon fibre and inorganic fillers. These fibres often several advantage including high specific strength and modulus, low cost, low density, renewable nature, biodegradability, absence of associate health hazards, easy fibre surface modification, wide availability, and relative non – abrasiveness (Al-Kaabi et al., 2005; Bledzki & Gassan, 1999; Thwe & Liao, 2002; Kim et al., 2004).

Many researches have been done on virgin thermoplastic and natural fibre composites, which have successfully proven their applicability to various fields of technical application, especially for load-bearing application. Thermoplastics have been compounded with natural fibres such as polyethylene (PE) with flax fibre (Foulk et al., 2004), PE with henequen (Herrera-Franco et al., 2005), PE with wood flour (Bengtsson & Oksman, 2006), PE with curaua (Araujo et al., 2008), polypropylene (PP) with kenaf (Tajvidi, 2005), PP with jute fibre (Cabral et al., 2005; Li & Sain, 2005), poly (vinyl chloride) (PVC) with wood/flour (Guffey & Sabbagh, 2002), PVC with baggase fibre (Zheng et al., 2007), polystyrene (PS) with sisal fibre (Antich et al., 2006), and poly (lactic acid) with flax fibre (Oksmana et al., 2003), PLA with abaca fibre (Bledzki et al., 2009).

The interest in cellulose fibers as reinforcement agents in composite materials with polymer matrices has increased dramatically in the last decade, as detailed in recent reviews (Aparecido et al., 2009). Lignocellulosic materials, which predominantly consist of cellulose, lignin and hemicelluloses, in the production of the plastic composites has gained momentum in recent years which have stimulated much interest in the manufacture of composite during the past decade, i.e. used as filler material instead of conventional filler such as mica, clay and glass fiber.

© Palm Kernel Shell is one of the lignocellulosic biofibres derived from waste palm kernel oil. Palm Kernel Shell is an agricultural by-product from palm oil mills, and it is one of the main agricultural wastes in Malaysia. Palm Kernel Shell can be utilized to a great extend as construction material. At present, it uses is limited mostly to fuel for burning and as finishes traditional mud houses. There is a great interest to find some value-added applications for Palm Kernel Shell.

Low-density polyethylene (LDPE) is important thermoplastic due to their having a good combination of properties, such as fluidity, flexibility, transparency and a glossy surface. LDPE is used mainly as a food packing material, sheet and film.

However, the most important issue associated with these composites is the interfacial adhesions between the natural reinforcing fillers and matrix polymers. The compatibility problem may be due to the fact that the polyolefin is non-polar and hydrophobic, whereas the natural polymer, which is a lignocellulosic material, is polar due to the –OH groups in the cellulose. This results in poor adhesion and prevents the reinforcing filler from acting effectively in the composite. The good properties of these composites can be obtained by improving the compatibility between these two materials. In order to solve these problems, studies have been performed on surface modification or treatment of filler using a compatibilizing agent or coupling agent in order to reduce the hydrophilicity of the filler.

Cellulosic fillers can be modified by physical and chemical methods. Extensive studies are currently being performed on polyolefins (polypropylene and polyethylene) and various natural reinforcing fillers, in conjunction with various chemicals that could affect the interface. Among various ways of chemical modification for polyolefin/natural fibre composites were used such as effect of crosslinking on polyethylene (Cousin et al., 1989; Benedetto et al., 1989), effect of silane coupling agent (Raj et al., 1989a; Raj et al., 1990; Raj et al., 1989b), and the effect of different coupling agent using poly[methylene (polyphenyl isocyanate)] and silanes (A-172, A-174, A-1100) (Maldas et al., 1989). The functionalized polyolefins are most often tested because of their efficiency and commercial availability.

The improvement in the mechanical properties of the composites can be achieved by increasing the filler volume fraction up to the certain degree of filler loading depending on the packing arrangement of the filler. The filler matrix interaction may be improved by making chemical or physical modification to the filler. Chemical modification of natural filler such as acrylation, silylation and other treatments reduce and other treatment reduces their moisture sensitivity. Much remain to be done to change or modify and improve bulk and surface characteristics in order to improve composites properties. The strong interfacial bonding strength obtained by improving the compatibility between the hydrophilic filler and hydrophobic matrix polymer can improve the physical, mechanical and thermal properties of the composite system (Kim, 2008).

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## 1.2 Research Objectives

The prime objective of this study is to determine the properties and characterizations of palm kernel shells (PKS) filled low density polyethylene (LDPE) on a different composition. Several different compositions of LDPE/PKS biocomposites were prepared in order to study:

1. To study the effect of palm kernel shell loading on mechanical properties, water absorption, morphology, and thermal properties of LDPE/PKS biocomposites
2. To study the effect of compatibilizer (PEAA) on properties of LDPE/PKS biocomposites
3. To study the effect of chemical modification (acrylic acid) on properties of LDPE/PKS biocomposites
4. To study the effect of coconut coupling agent on properties of LDPE/PKS biocomposites

The testing of LDPE/PKS biocomposites has been done by tensile test, water absorption, morphology, thermal analysis using differential scanning calorimetry (DSC), and fourier transform infrared spectroscopy (FTIR) Analysis.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Polymer Composites

Composites materials have been utilized to solve technological problems for a long time and these materials start capturing the attention in industries with the introduction of polymeric-based composites. Since then, composites materials have become common engineering materials and are designed and manufacturing for various application including automotive components, sporting goods, aerospace parts, consumer goods, and in the marine and oil industries. As early as 1908, the first composite materials were applied for the fabrication of large quantities of sheets, tubes and pipes for electronic purposes (paper or cotton to reinforce sheets, made of phenol- or melamine-formaldehyde resins). In 1896 for example, aeroplane seats and fuel-tanks were made of natural fibers with a small content of polymeric binders (Bledzki & Gassan, 1999).

The growth in composite usage also came about because of increase awareness regarding product performance and increase competition in the global market for lightweight components. Today, it appears that composites are materials of choice for many engineering applications. Daniel et al., (2003) has defined composites as an engineering material that made from a mixture of two or more constituent material. There are two categories of constituent materials which are matrix and reinforcement. At least one portion of each type is required. The matrix material surrounds and

supports the reinforcement materials by maintaining their relative positions. The narrower definition of composites become more specific and can be restricted to those combinations of materials that contain high strength/stiffness fiber reinforcements supported by a high performance matrix material.

However, based on definition by (Matthew & Rawlings, 1994), this definition is not sufficient and three other criteria have to be satisfied before any material can be classified as composite. First both constituents have to be present in reasonable proportions, say greater than 5%. Secondly, it is only when constituents phases have different properties, and hence the composites properties are noticeable different from properties of constituents. Therefore, we have come to recognize these materials as composites.

Polymer composite materials are widely used in practice and are an object of numerous, either scientific or scientific-applied, studies. Polymer composites featuring both stiffness and strength have been of great commercial interest for a century now. Regarding structure and properties, polymer composite comprise such materials as glass and carbon plastics, polymer concretes and concrete polymers, varnish-dye materials, wood-polymer materials, reinforced elastomers, etc. Their significance is due to a number of useful properties that pure, unfilled polymer do not possess. The introduction of fillers into polymers provides for the change of material properties, thus gaining a definite profit (Yatehko et al., 2001). Although inorganic fillers such as calcium carbonate, mica, fiberglass, talc and clay have been widely used, composites containing organic fillers derived from renewable resources have aroused broad interest because of growing environmental concerns and the increasing costs of inorganic fillers. In particular, cellulose-based fillers, such as natural fibres and cellulose fibres,