



**CHEMICAL MODIFICATION OF PALM KERNEL
SHELL FILLED POLYLACTIC ACID
BIOCOMPOSITE FILMS**

by

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LIST OF SYMBOL

$^{\circ}\text{C}$	Degree Celcius
ΔH°_f	Heat of fushion for 100% crystalline PLA
ΔH_f	Heat of fushion for PLA
%	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
T_{dmax}	Dgradation temperature at maximum
T_m	Melting Temperature
T_c	Crystallization temperature
W/g	Heat flow
%/mm	Derivative weight loss
g/mol	Molecular weight

LIST OF ABBREVIATIONS

PS	Polystyrene
SEM	Scanning Electron Microscope
FTIR	Fourier Transform Infrared
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetric
DTG	Derivative Thermogravimetric
D-LFT	Direct long fibre thermoplastic molding
PP	Polypropylene
MAPP	Maleated polypropylene
PE	Polyethylene
PU	Polyurethane
LDPE	Low Density Polyethylene
rLLDPE	Recycle linear low density polyethylene
PET	Polyethylene terephthalate
PVC	Polyvinyl chloride
PLA	Polylactic Acid
PLA-GMA	Poly lactide-graft-glycidyl methacrylate
PHA	Polyhydroxyalkanoate
PEG	Poly(ethylene glycol)
PEA	Polyesteramides
PVOH	Polyvinyl alcohol
PCL	Polycaprolactone
PBS	Poly(butylene succinate)

PBAT	Poly(butylene adipate terephthalate)
PBSA	Poly(butylene succinate adipate)
PHB	Polyhydroxybutyrate
PHBV	Polyhydroxybutyrate cohydroxyvalerate
PVS	Poly (vinyl sulfate)
ABS	Acrylonitrile butadiene styrene
PKS	Palm Kernel Shell
OPKS	Oil palm kernel shell
OPS	Oil palm shell
php	Part per hundred polymer
ppm	Part per milion
MCC	Microcrystalline cellulose
TPS	Thermoplastic starch
HDPE	High density polyethylene
ASTM	American Society for Testing Materials
FDA	Food and drug administration
DDS	Drug delivery systems
DP	Degree of polymerization
ROP	Ring-opening polymerization
GRAS	Generally regarded as safe
NF	Nypa Fruticans
3-MPS	3-Mercaptopropyl trimetoxysilane
LA	Lactic Acid

LIST OF NOMENCLATURES

C	Carbon
C=O	Carboxyl
C-O	Carbonyl
C=C	Ethylene (IUPAC name: Ethene)
C-O-C	Ether
OH	Hydroxyl
Si-O-C	Silyl ethers
Si-C	Silane linkage (carbon silicon bond)
H ² O	Water
CO ₂	Carbon dioxide

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Pengubahsuaian Kimia Filem-Filem Biokomposit Polilaktik Asid Terisi Kernel Kelapa Sawit

ABSTRAK

Di dalam penyelidikan ini, kesan kernel kelapa sawit (KKS) yang tidak dirawat kimia dan yang dirawat kimia terhadap sifat-sifat mekanik, morfologi dan sifat-sifat terma telah dikaji. Biokomposit filem daripada serbuk kernel kelapa sawit dan polilaktik asid (PLA) telah di sediakan menggunakan teknik tuangan larutan di mana PLA dilarut di dalam cecair kloroform dan diaduk pada 600 rpm dan selepas itu di tuang dan diratakan di dalam piring petri. Keputusan kajian menunjukkan peningkatan kandungan KKS telah mengurangkan kekuatan tensil dan pemanjangan pada takat putus tetapi telah meningkatkan modulus elastisiti. Suhu peralihan kaca (T_g) dan kehabluran (X_c) adalah paling tinggi pada 10 bsp kandungan KKS. Suhu penguraian maksimum (T_{dmax}) telah meningkat dengan peningkatan kandungan KKS. Kestabilan terma mengalami peningkatan pada pengisi yang tinggi kerana kurangnya kehilangan berat kesuluruhan pada 600 °C. Kajian SEM menunjukkan interaksi antaramuka yang lemah diantara hidropilik KKS dan hidropik PLA. Pelbagai jenis modifikasi-modifikasi kimia seperti butil metakrilat (BMA), laktik asid (LA) dan 3-mecaptopiril-trimethoxy silana (3-MPS) yang telah dikaji dalam kajian in. Sifat-sifat biokomposit filem yg telah dirawat dengan BMA, LA dan 3-MPS telah menunjukkan penambahan kekuatan tensil dan modulus elastisiti tetapi pengurangan pada pemanjangan pada takat putus berbanding biokomposit yg tidak dirawat. Modifikasi kimia telah juga meningkatkan T_g dan X_c filem biokomposit. Analisis FTIR menunjukkan modifikasi kimia telah mengurangkan kumpulan hidroksi pada KKS. Interaksi antara muka dan lekatan adalah lebih baik di antara KKS dan PLA yang telah dibuktikan dengan kajian SEM.

Chemical Modification of Palm Kernel Shell Filled Polylactic Acid Biocomposite Films

ABSTRACT

In this research the effect of untreated and treated palm kernel shell (PKS) on the mechanical properties, morphological analysis and thermal properties of polylactic acid (PLA)/PKS biocomposite films were studied. The biocomposite films from palm kernel shell powder and polylactic acid were prepared from solution casting method where the PLA was dissolved in the chloroform and was stirred at 600 rpm, then the solution were casted inside the petri dish. The result showed that the increased in PKS content had decreased the tensile strength, elongation at break but had increased the modulus of elasticity. The glass transition temperature (T_g) and crystallinity (X_c) were the highest at 10 php filler content. The decomposition temperature at maximum (T_{dmax}) was raised with increased in PKS content The thermal stability was improved at high filler content because of lower total weight loss at 600 °C. SEM analysis showed that poor interfacial interaction between hydrophillic PKS and hydrophobic PLA matrix. The different types of chemical modification using butyl methacrylate (BMA), lactic acid (LA) and 3-mercaptopropyl-trimethoxy silane (3-MPS) were investigated in this study. The treated biocomposite films with BMA, LA and 3-MPS exhibited high tensile strength and modulus of elasticity, however, the elongation at break lowered compared to untreated biocomposite films. The chemical modification also enhanced the T_g and X_c of the treated biocomposite films. The FTIR analysis showed the chemical modification of PKS had reduced the hydroxyl group of PKS. Better interfacial adhesion and interaction between PKS and PLA matrix were proven by SEM study. Amongst all the chemical modification that been used, the chemical modification using BMA shows the highest properties incomparison with the used of LA and 3-MPS as chemical modification.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Sustainability of bio-based eco-friendly products are the products with the potential and ecological acceptability that are gain from renewable resources which, have reuse abilities and act as biodegradable. (Mohanty et. al., 2002). Accordingly, another procedure on the development of environmental and economical manufacturing has being created, in addition to reuse and recycle of materials (Bismarck et. al., 2005), the business sector for biopolymers and biocomposites is increasing yearly. As indicated by European Bioplastics, the creation of bioplastics is expected to increase around 700,000 tons in 2010, to 1.7 million tons by 2015 (Kristy-Barbara, 2014). Numerous analysts these days concentrating on green and eco-accommodating materials with the terms, for example, renewable, recyclable, degradable and sustainable. This term is made because of ecological awareness. (John & Anandjiwala, 2009; Satyanarayana et al., 2009). Along these lines, natural bio-fibre composites are the option to glass-fibre with reinforcement of plastics. Thermoplastic biocomposites, made out of Polypropylene (PP), polyethylene (PE), polystyrene (PS) or biopolymers polylactic acid (PLA) with reinforcement of natural fibres have particularly comes into consideration for the pass couple of years.

Mixing of natural fibres and polymer matrix resin can be characterized as biocomposites, it has been into the business sector due to awesome mechanical properties and it is vital for the ecological assurance of products and in addition enhancing the nature of product (John & Thomas, 2008; Siracusa et. al., 2008). The use of biopolymer network can hold the entire carbon substance and spare essential assets such as petroleum (Le Duigou et. al., 2010). Additionally, it diminishes in weight and cost, and gives less use of the oil assets (Mukherjee & Kao, 2011). Biocomposites were utilized as a part of the automotive business, building, furniture and packaging enterprises that due to the renewable items that draw consideration due to the expanded in ecological insurance, furthermore due to decrease in fossil assets such as oil and natural gases (Fowler Paul et. al., 2007). Thus, the use of biocomposites with petroleum-based polymer was just fractionally biodegradable. The biodegradation of this biocomposites was included natural filler while the byproduct is the polymer framework. This causes contamination to the earth. Reusing and recycle is one strategy to conquer this issue, yet just little quantities of petroleum-based polymer was really recyclable (Wu, 2009). There is an issue following a couple of many years of utilizing petroleum-based polymer, which is the deficiency of petroleum assets. The handling of petroleum-based polymer will give ecological impact, which is carbon dioxide (CO₂) emmersion. (Steinbuchel, 2005).

Bio-based or biodegradable polymers have an extensive variety of utilizations, for example, in the biomedical, packaging and agricultural fields. The most widely recognized of the biodegradable materials are mixes of thermoplastic starch (TPS) and aliphatic/aromatic polyesters, for example, poly(lactic corrosive) or polylactides (PLA), polycaprolactone (PCL), poly(butylene adipate terephthalate) (PBAT) and polyhydroxybutyrate (PHB) (Bastioli, 2005). PLA, which advantages both that originate

from renewable assets and being biodegradable in the degradable environment, has pulled in much consideration. It has turned into a financially suitable plastic item in industry and is utilized to deliver regular use articles and packaging materials, for example, plate, containers or biofilms for the medical industry (Van De Velde & Kiekens, 2002; Shibata et. al., 2003). PLA has great mechanical and processability properties furthermore great thermal stability, and constrained in nature that has been indicated by life cycle assesment (LCA). PLA is thermoplastic aliphatic polyester that is gotten from ring-opening polymerisation of lactide that were gotten from the maturation or fermentation of sugar feedstock (Mohanty et. al., 2005).

PLA is a brittle thermoplastic that has an exceptional enthusiasm as a polymer matrix in biocomposites material. PLA is an exceedingly versatile biopolymer and is highlighted that it is gotten from a renewable asset, for example, corn, sugar cane and sugar beet. (Yu et al., 2006). The creation of PLA devoured CO₂ by photosynthesis and it is considered eco-composite material (Rasal et al., 2010). In addition, the PLA can be compost over a timeframe by soil and also enzymatic degradation (Wu, 2009). Consequently, it can lessen the enviromental contamination. PLA can be utilized as a substitute for ordinary polymer as a result of great mechanical and thermal properties and has low effect on the nature, furthermore, it has great processability properties which, PLA can be prepared by blow molding, injection molding, and film casting (Ajmass et. al., 2008; Carroscio et al., 2010). In spite of the fact that the mechanical properties of PLA suited for some modern applications, it is considered excessively fragile for some commercial values applications yet think about having as a decent mechanical properties with others customary polymers. Carroscio et al., (2010) quote that PLA has tensile and yield modulus in contrast with traditional polymer, for example, polystyrene (PS) and polypropylene (PP). The principle detriments utilizing

PLA is that high cost, fragile in nature furthermore poor moisture barrier properties. (Madhavan et al., 2010). According to Madhavan et al. (2010) PLA has high dampness properties that causes hydrolytic compostable and it is real issues when processing and storing of the item base on PLA. Additionally, Rasal et al. (2010) examined that less active side chain which is present inside the PLA structure and ordered it as artificially inert polymer, which makes the surface adjustment more troublesome. It is conceivable to conquer the weakness of PLA by consolidating it with fillers, for example, natural fibres.

Regular natural fibre as a substitution to artificial fibre in polymer grid offers a decent mechanical execution and eco-friendly properties. The utilization of natural fibre composites is expanding nowadays. The blend of intriguing mechanical and physical properties together with their environmental friendly character has excited enthusiasm for various modern applications (Mohanty et. al., 2005; Pickering, 2008; Graupner et. al., 2009). Fibres characterise as fillers for PLA biocomposites. It has good processibility properties, to give better thermal properties, hydrolysis resistance, and enhances physical properties of PLA. The examinations on PLA biocomposites by utilizing natural and altered cellulose fibres have demonstrated that their mechanical properties scale corolated with the added fibres. (Wan, 2001; Mathew et al., 2005). The use of the natural fibre fortified with PLA biocomposites can adjust the mechanical and thermal stabilities are vital to decrease the expense, change the biodegradability, and furthermore broaden the utilization of these biocomposites. The exploration on PLA biocomposites, which included the utilization of cocout fiber (Wu, 2009), bamboo fiber (Kang and Kim, 2011), kenaf fiber (Yussuf et al., 2010), banana fiber (Shih and Huang, 2011) and hemp fiber (Pickering et al., 2011). A large portion of this examination utilizing PLA with natural fibre demonstrates positive change in modulus of elasticity

(Yussuf et al., 2010; Shih and Huang, 2011). Utilizing the fibre as filler inside PLA network can see change on the thermal properties. Perinovic et al., (2010) demonstrated that PLA biocomposites with lignocelulosic filler (olive stone) enhanced the thermal properties in correlation with using pure PLA.

In the course of the most recent times, various analysts have been included in contemplating the usage of fibre as biocomposite materials (Azwa et. al., 2013). The utilization of the lignocelulosic piece has increases enthusiasm for enhancing biocomposite materials. This interest is because of recyclability, efficiency and incredible quality properties of the fibre itself (John and Thomas, 2008). The lignocelulosic filler likewise has fabulous properties in comparision with mineral filler, for example, mica and talc. It has high quality properties, low density, low cost, negligible hazardous, biodegradability, and great eco-friendly properties (Tserki et al., 2005; John and Anandjiwala, 2009; Fronaco and Gozalez, 2005; Park et al., 2008). Chiefly, linocelulosic filler originates from the plant part, for example, coconut shell (Bledzki et al., 2010) and rice husk (Yang et al., 2007a). The significant issue confronting by lignocellulose is that, it is actually absence of good interfacial attachment when incorporate with polymer matrix network. The subsequent impact will be the decrease in mechanical properties of the biocomposites. On the surface of lignocelulosic has a many polarized hydroxyl group that if wetting occur on the filler surface makes it hard to bond with the non-polar polymer matrix grid (Tserki et al., 2005; John & Anandjiwala, 2009). It likewise indicates poor scattering in the polymer matrix grid because of inalienable filler-filler interaction, which result from intermolecular hydrogen bond (Pickering et al., 2003).

On the other hand, chemical modifications were done to lignocelulosic fibre hoping to enhance the matrix–fibre grip or adhesion. Some chemical modification could

prompt in decreasing moisture absorption properties and diminish its hydrophilicity of the lignocellulosic fibre composites. The vast majority of the chemical modification of fibre include silanization (Lee, et al., 2008), acetylation (esterification) (Danyadi et al., 2010), and graft copolymerization (Kim et al., 2011). Their chemical characteristic permits them to respond with the fibre surface, which bonds between the fiber and polymer matrix. Study from Lee et al. (2008) utilizing silane treatment improves the thermal and mechanical properties of PLA/wood flour/talc biocomposites.

Oil palm (*Elaeis guineensis*) is one of the plantations that are developed throughout Malaysia, Indonesia and Africa. The quantity of the collected oil palm are progressively increase every year. These few parts of the plant were collected as by-product, for example, empty fruit bunch, palm oil mill gushing, palm fruit fibre and palm kernel shell. The palm kernel oil is reaped and will left buildup residue of PKS. Apporximately 15 to 18 tons of fresh fruit bunch are created every year and PKS contains around 64% of the natural product group waste (Adewumi, 2009). Curently, PKS is utilized as ignition materials because of high lignin content which generally contain high burning quality. Interestingly high lignin content left ignition and eventually deposited that will make contamination to the earth.