UNIVERSITI PUTRA MALAYSIA

PREPARATION AND CHARACTERIZATION OF POLYLACTIC ACID/ POLY(ε-CAPROLACTONE)/CLAY/OIL PALM MESOCARP FIBER HYBRID COMPOSITE

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By

ENG CHERN CHIET

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

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in Fulfilment of requirement for the Degree of Master of Science

PREPARATION AND CHARACTERIZATION OF POLYLACTIC ACID/ POLY(ε-CAPROLACTONE)/CLAY/OIL PALM MESOCARP FIBER HYBRID COMPOSITE

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The research focused on incorporation of Oil Palm Mesocarp Fiber (OPMF) as filler in polylactic acid/poly(ε-caprolactone) (PLA/PCL) blends. The addition of clay as compatibilizer to improve mechanical and thermal properties of the composites. OPMF was bleached and then treated with silane coupling agent due to incompatibility of hydrophilic OPMF with hydrophobic matrix.

PLA/PCL blends were prepared by melt blending technique with the best ratio is 85/15. Fourier transform infrared (FTIR) revealed that there is physical interaction between PLA and PCL in blends. Thermogravimetric Analysis (TGA) showed that degradation of blends is two steps. Dynamic Mechanical Analysis (DMA) showed that blends exhibit two glass transition temperature (T_g) which indicated PLA/PCL is immiscible.

The PLA/PCL/clay composites were prepared by melt intercalation technique. X-ray diffraction (XRD) showed shifting of diffraction peak while transmission electron microscopy (TEM) indicated formation of agglomerate when clay was added. The addition of clay Nanomer® PGV makes blends become more flexible while addition of clay Montmorillonite K10 makes blends become stiffer. TGA thermograms proved that the presence of clay improve thermal stability of blends. Loss modulus shows that the addition of clay shifts two T_g in composites become closer to each other. Scanning

electron microscopy (SEM) micrograph revealed that clay Nanomer® PGV composites is more homogeneous and appear as single phase morphology.
The effect of 1% clay Nanomer® PGV on PLA/PCL/OPMF biocomposites was investigated. FTIR spectra showed that there is physical interaction between PLA, PCL, clay and OPMF in composites as peak shifting is observed. The addition of clay improves mechanical properties of biocomposites. TGA thermograms revealed that the addition of clay improves the thermal stability of the biocomposites. Loss modulus shows that the addition of clay shifts two T_g in composites become closer to each other and low tan δ indicate better fiber/matrix adhesion. SEM micrograph showed the addition of clay improves fiber/matrix adhesion as fiber breakage on the fracture surface.

FTIR spectra showed that bleaching of OPMF successfully removes hemicellulose from fibers while silanized unbleached and silanized bleached OPMF are less hydrophilic. Silanized bleached OPMF composites showed best mechanical properties in PLA/PCL/clay/OPMF hybrid composites. FTIR spectra indicated there is interaction between both silane treated OPMF with matrix. DMA showed that both silane treated OPMF shift two T_g in composites become closer while low tan δ peak show good fiber/matrix adhesion of bleached silane treated OPMF composites. SEM micrograph indicated that better adhesion between silanized bleached fiber with matrix as fiber breakage in the fracture surface. Water sorption test showed that silanized bleached OPMF is most water resistance with less water uptake.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

PENYEDIAAN DAN PENCIRIAN KOMPOSIT HIBRID POLILAKTIK ASID/ POLI(ε-KAPROLAKTON)/TANAH LIAT/SERAT BUAH KELAPA SAWIT

Oleh

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Penyelidikan ini memberi tumpuan kepada penambahan serat buah kelapa sawit (OPMF) sebagai pengisi dalam adunan polilaktik asid/poli(ε-kaprolakton) (PLA / PCL). Tanah liat ditambah sebagai agen keserasian untuk meningkatkan sifat-sifat mekanikal dan terma komposit. OPMF telah diubahsuai oleh peluntaran dan silana kerana ketidakserasian hidrofilik OPMF dengan hidrofobik matriks.

Adunan PLA/PCL disediakan dengan kaedah pengadunan leburan dengan nisbah yang terbaik adalah 85/15. Spektra FTIR mendedahkan bahawa hanya ada interaksi fizikal antara PLA dan PCL dalam adunan. Termogram TGA menunjukkan bahawa penguraian berlaku dalam dua langkah. DMA mendedahkan bahawa terdapat dua T_g yang menunjukkan bahawa PLA / PCL adalah tidak bercampur dalam adunan.

Komposit PLA/PCL/tanah liat telah disediakan melalui teknik pengadunan leburan. Penambahan tanah liat menyebabkan pindahan puncak berlaku dalam XRD dan Mikrograf TEM menunjukkan penbentukan pengumpalan dalam komposit. Penambahan tanah liat Nanomer® PGV menyebabkan adunan menjadi lebih fleksibel manakala penambahan tanah liat montmorilonit K10 menyebabkan adunan menjadi lebih keras. Termogram TGA menunjukkan bahawa penambahan tanah liat meningkatkan kestabilan terma adunan. DMA menunjukkan penambahan tanah liat merapatkan dua T_g dalam adunan. Mikrograf SEM menunjukkan bahawa komposit tanah liat Nanomer ® PGV lebih homogen dan menpunyai morfologi satu fasa.

Kesan 1% tanah liat Nanomer® PGV pada PLA/PCL/OPMF biokomposit telah dikaji. Spektra FTIR mendedahkan bahawa terdapat interaksi antara PLA, PCL, tanah liat dan OPMF dalam komposit. Penambahan tanah liat meningkatkan sifat mekanik biokomposit. Termogram TGA menunjukkan bahawa penambahan tanah liat meningkatkan kestabilan terma biokomposit. DMA menunjukkan bahawa penambahan tanah liat merapatkan dua T_g dalam komposit dan tan δ yang rendah membuktikan perlekatan yang baik antara serat dengan matriks. Mikrograf SEM menunjukkan perlekatan yang baik antara serat dengan matriks.

Spektra FTIR menunjukkan bahawa pelunturan OPMF berjaya membuang hemiselulosa daripada serat manakala OPMF yang diubahsuai oleh silana menjadi kurang hidrofilik. Pelunturan silana OPMF komposit menunjukkan sifat mekanik yang terbaik dalam PLA/PCL/tanah liat/OPMF komposit hibrid. spektra FTIR menunjukkan bahawa terdapat interaksi antara OPMF dengan matriks. DMA menunjukkan bahawa penambahan perlunturan silana OPMF merapatkan dua T_g dalam komposit manakala tan δ yang rendah membuktikan bahawa perlekatan yang baik antara serat dengan matriks. Mikrograf SEM menunjukkan bahawa lekatan yang lebih baik wujud di antara perlunturan silana OPMF dengan matriks. Ujian kadar penyerapan air menunjukkan bahawa OPMF yang diubahsuai oleh perlunturan silana adalah paling rintangan tinggi terhadap air.

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I certify that an Thesis Examination Committee has met on 12 March 2014 to conduct the final examination of Eng Chern Chiet on his thesis entitled "Preparation and Characterization of Polylactic Acid/Poly(ε-Caprolactone)/Clay/Oil Palm Mesocarp Fiber Hybrid 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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This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of Supervisory Committee were as follows:

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SCHEMES

LIST OF ABBREVIATIONS

ASTM	American Standard for Testing and Materials
CEC	Cation exchange capacity
CPC	Cetyl pyridinium chloride
CSIRO	Commonwealth Scientific & Industrial Research Organization
DMA	Dynamic mechanical analysis
DP	Degree of polymerization
DTG	Differential thermogravimetry
EFB	Empty fruit bunches
ENR	Epoxidized natural rubber
FTIR	Fourier transform infrared
HDPE	High density polyethylene
IUPAC	International Union of Pure and Applied Chemistry
kN	Kilonewton
MMT	Montmorillonite
MPa	Mega Pascal
OPEFB	Oil palm empty fruit bunch
OPMF	Oil palm mesocarp fibers
PAN	Polyacrylonitrile
PBAT	Poly (butylene adipate-*co*-terephthalate)
PBS	Poly(butylenes succinate)
PBT	Poly(butylene terephthalate)
PCL	Poly(ε-caprolactone)

PE	Polyethylene
PHB	Poly(b-hydroxybutyrate)
PHBV	poly(3-hydroxybutyrate-co-3-hydroxyvalerate)
phc	parts per hundred contents
PLA	Polylactic acid
POME	Palm oil mill effluent
PP	Polypropylene
PPDO	poly(p-dioxanone)
PS	Polystyrene
PVC	Poly(vinyl chloride)
rpm	Revolution per minute
RWF	Recycled wood fiber
SEM	Scanning Electron Microscopy
STA	Simultaneous Thermal Analyser
Tan δ	Tangent delta
TEM	Transmission electron microscopy
T_g	Glass transition temperature
TGA	Thermogravimetric analysis
wt%	Weight percentage
XRD	X-ray diffraction
˚C	Degree Celcius

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Conventional petrochemical based polymer such as polypropylene (PP), polyethylene (PE) and polystyrene (PS) are widely used in many areas. However, these polymer cause major side effects to environment as these non degradable polymers tend to accumulate in disposal system. Although many techniques such as incineration, recycling and landfill disposal had been practised as ways for polymer disposal, there are still some drawbacks of these techniques. Incineration of polymer will release toxic gases which hazardous to human's health while recycling of polymer will not yield quality product due to heterogeneous nature of polymers. Due to limitation of space, landfill disposal also less preferred. Therefore, biodegradable polymer attracted the attention of researcher as biodegradable polymer seems to be the best solution to this problem.

A wide range of synthetic or natural polymers degrade by hydrolytic (polyglycolide, polylactides, polydioxanone, polycaprolactone, polyhydroxyalkanoates) or enzymatic (polysaccharides, protein, polyamino acids) route (Nair and Laurencin, 2007). Although these polymers have wide range of mechanical properties and degradation rate, inappropriate stiffness or degradation rate restrict their application, Therefore, blending with other polymers or adding plasticizer need to be done to tune the properties of these polymers according to application requirements (Vieira *et al*., 2011).
Polylactic acid (PLA) is a versatile polymer made from renewable agricultural raw materials, which are fermented to lactic acid. PLA has good mechanical properties, thermal plasticity and biocompatibility. However, PLA is a stiff and brittle material which restricts its applications. Therefore, it is necessary to use plasticizers to improve the elongation and impact properties of PLA (Meinander *et al*., 1997). Blending PLA with other flexible biodegradable polymers such as poly (ε-caprolactone) (PCL) can plasticized PLA to reduce the brittleness.

The properties of PLA/PCL blends can be further improved by adding reinforcement filler such as nanoclay. Clay is a nanomaterials with primary particles with at least one dimension in the nanometer scale (Yuan and Wu, 2007). Many studies have shown that the use of nanoclay increase tensile strength, tensile modulus, thermal failure resistance and impact resistance of composites (Peter and Woldesenbet, 2008). These properties are very crucial in the application for the production of panels, barrier and coating materials in automobile, packaging, civil and electrical engineering.
Natural fibers have been widely applied as reinforcement filler in polymer composites due to natural fibers have many notable benefits than synthetic fibers. Besides, natural fibers are environmentally friendly, totally biodegradable, renewable, cheap and

comparatively lower density (Taj *et al.*, 2007). In addition, the use of lignocellulosic fibers can reduce the cost and also enhance the biodegradability of polymers (Iannace *et al.*, 1999). The reduction in cost enables biodegradable polymers to be more competitive to other conventional polymers. However, poor wettability, incompatibility with some polymer matrices and high moisture absorption by the fibers limit the uses of natural fibers as reinforcement filler in polymer composites (Wambua *et al.*, 2003). Therefore, modification of natural fibers is needed to improve interfacial adhesion between natural fibers and polymer matrix.

Silane is bifunctional molecule which used to modify surface of natural fibers. One end of the silane which consists of alkoxysilane groups are capable to react with OH rich surface of fibers while another end have many functional groups which can be designed as a function of the polymer matrix to be used. This provides a good interaction between natural fibers and matrix or even formation covalent bonds between them (Castellano *et al.*, 2004).

1.2 Biodegradable Polymer

Recently, the call for biodegradable and biocompatible polymers is increase tremendously as the increasing of global awareness to minimize the environmental pollution due to the waste of non biodegradable plastic. Therefore, biodegradable polymers are ideal alternatives to replace non biodegradable polymers due to there are able degrade hydrolytically and enzymatically to form CO_2 and H_2O or CH_4 and H_2O. These degradation products can be naturally transform to non toxic products as biodegradable polymer are more prone been attack biologically in natural environment. Most of the biodegradable aliphatic polyester are produced by enzymatic synthesis and chemical synthesis of natural polymer based products by polycondensation and ring-opening polymerization (Kim and Kim, 2008).

Biodegradable polymers such as polylactic acid (PLA), poly(ε-caprolactone) (PCL), poly(p-dioxanone) (PPDO), poly(butylenes succinate) (PBS), poly (hydroxyalkanoate)s such as poly(b-hydroxybutyrate) (PHB), poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) and starch are abundantly available (Luckachan and Pillai, 2011). Although biodegradable polymers have wide range of mechanical properties and degradation rate, inappropriate stiffness or degradation rate limit their application (Vieira *et al.*, 2011). Besides, relatively expensive, small scale and some properties restrictions of these polymers compared to common polymers such as polystyrene (PS), polypropylene (PP), polyethylene (PE) and poly (vinyl chloride) (PVC) leads to biodegradable polymers are not preferred in the conventional of plastic industry.

1.2.1 Polylactic Acid

Polylactic acid (PLA) is one of the most favourable biodegradable polymers due to its mechanical properties, thermoplasticity, biocompatibility and biodegradability. PLA is produce from lactic acid monomer, a fermentation product of molasses, potato starch or corn starch. Lactic acid monomer is a basic chiral molecule which occurs in two enantiomers, L- and D-lactic acid. Polymerization of lactic acid monomer into PLA can be done by direct condensation or by formation of cyclic dimer intermediate (lactide). Direct condensation required usage of solvent to react under high vacuum and temperature while the polymerization by lactide is solvent free. Poly (L-lactic acid) is high crystalline polymer with low D- content (<2%), while fully amorphous PLA can be achieved by addition of comparatively high D- content (>20%). PLA is hard polymer with glass transition temperature in the range of 60 °C to 70 °C and melting temperature between 170 °C to 180 °C (Gupta *et al*., 2007). The structure of PLA is shown in Figure 1.1. PLA is a comparatively brittle and stiff polymer with low deformation at break. Therefore, modification of PLA is needed in order to compete with other flexible polymers such as polypropylene or polyethylene (Balakrishnan *et al*., 2010). There are many techniques to modify PLA such as copolymerization (Fukuzaki *et al*., 1990), blending with other polymers (Chen *et al*., 2009), addition of plasticizers (Silverajah *et al*., 2012), addition of nucleating agents (Phuphuak and Chirachanchai, 2013) and forming composites with fiber or nanoparticles (Ochi, 2008).

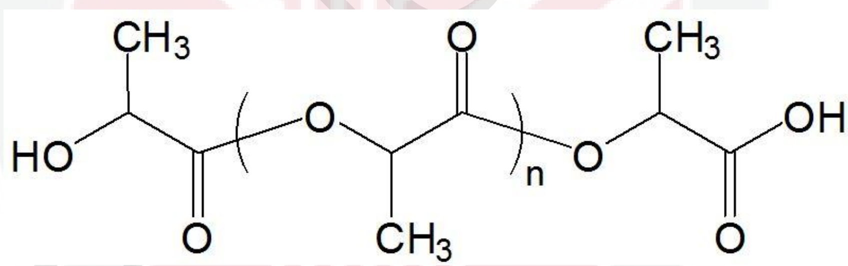

Figure 1.1: Chemical structure of polylactic acid (Petinakis *et al*., 2013)

1.2.2 Poly(ε-caprolactone)

Poly(ε-caprolactone) (PCL) can be produced by ring opening polymerization of ε-caprolactone using different types of catalysts such as anionic, cationic and co-ordination catalyst. It also can be synthesis through free radical ring opening polymerization of 2-methylene-1-3-dioxepane. PCL is hydrophobic, flexible semi-crystalline polymer with glass transition temperature of -60 °C and melting point range of 59 °C to 64 °C. PCL have exceptional blend compatibility which can be blended with other polymers to enhance stress crack resistance, dye-ability and adhesion (Woodruff and Hutmacher, 2010). The structure of PCL is shown in Figure 1.2. High ductility of PCL can be regards as a good plasticizer for PLA compared to low molecular weight plasticizers as PCL does not move to the surface of the blend and the physical properties cannot be depreciate (Yeh *et al*., 2009).

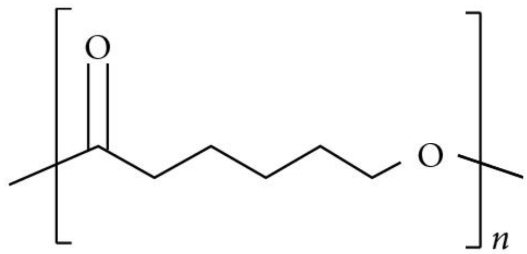

Figure 1.2: Chemical structure of poly(ε-caprolactone) (Odermatt *et al*., 2012)

1.3 Green Composites

Green composites are materials possess environmentally friendly attributes that are technically and economically practical while minimizing the production of pollution. Due to strong worldwide needs for creating a resource circulating society, development of green composites attract a lot of attention from many researchers (Abdul Khalil *et al*., 2012). Extensive research been conducted to develop a new type of totally biodegradable green composites by combing natural fibers with biodegradable polymers. These composites are ecofriendly, fully biodegradable and maintainable in which they are really green in all aspect. Besides, they can be readily disposed of or composted without causing side effects to environment. The major applications of these green composites are large scale produced consumer products with short life cycle or designed for short term usage (Netravali and Chabba, 2003).

1.4 Problem Statements

Recently, many studies been conducted by academic or industrial researcher on biodegradable polymer in order to replace conventional non-biodegradable polymer which cause major drawback to the environment. However, the cost of biodegradable polymer is comparatively higher than petrochemical based non-biodegradable polymer which limits its application. The incorporation of cheap natural fibers as reinforcement filler into biodegradable polymer is an alternative to reduce its cost.

Polylactic acid (PLA) is one of the most promising biodegradable polymers from renewable agriculture recourses that draw many attentions from researchers. However, PLA is hard and brittle which needed to plasticize in order to make it more competitive with other flexible polymers. Plasticized PLA with flexible poly (ε-caprolactone) (PCL) seem to be an alternative as PCL able to plasticize PLA from brittle to ductile which makes it more comparable to other flexible polymers. However, PLA/PCL blend is immiscible polymer blends. Therefore, incorporation of nanoclay can acts as physical compatibilizing agents to improve miscibility of PLA/PCL blends probably by intercalation of clay in blends.

Studies on natural fibers as reinforcement filler in polymer matrix become main focus for research all over the world due to its advantages such as ecofriendly, totally biodegradable, sufficiently available, renewable and not expensive. Although natural fibers seem to be promising reinforcement filler in polymer matrix, poor interfacial adhesion between polar hydrophilic fiber and non polar hydrophobic polymer matrix restrict its development. Chemical treatment of natural fiber is one of the best solutions as modification will reveal more active groups on the fiber surface which promote more efficient coupling with polymer matrix.

1.5 Objectives of The Study

The objectives of the study were:

1. To prepare PLA/PCL/clay/OPMF hybrid composite.
2. To investigate the effect of clay and fiber modification on the properties of PLA/PCL/clay/OPMF hybrid composite.
3. To characterize the morphology, thermal and mechanical, chemical and physical properties of hybrid composite.

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