UNIVERSITI PUTRA MALAYSIA

EFFECT OF FIBER LOADING AND SILANE TREATMENT ON PROPERTIES OF POLY (BUTYLENE ADIPATE-CO-TEREPHTHALATE) /OIL PALM EMPTY FRUIT BUNCH FIBER BIOCOMPOSITES

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Chairman: Dr. Nor Azowa Ibrahim, PhD

Faculty: Science

In this study, green composites of oil palm empty fruit bunch (OPEFB) fiber (100-200 µm) with poly(butylene adipate-co-terephthalate) (PBAT) using vinyl tris (2-methoxyethoxy)silane as crosslinking agent were prepared by melt blending technique. The effect of fiber loading and addition of vinyl tris (2-methoxyethoxy)silane on the mechanical and thermal properties of PBAT/OPEFB biocomposites were investigated. The composites were prepared by using 10, 20, 30, 40 and 50 % by weight of fiber. Different fiber loadings were tested to determine the optimum amount of OPEFB required producing good composite. Consequently, the composite with 40% fiber loading, blending temperature and time of 120 ºC and 12 minutes, respectively and rotation speed of 30 rpm were found as the best formulation and condition for preparation of the composite.

Mechanical properties of the composite were assessed by tensile, flexural and impact strength (notched and unnotched). In addition, water absorption and degradation study were also carried out. The composites showed the increase in tensile strength and modulus, flexural strength and modulus, unnotched and notched impact strength with the increment of fiber loading.

The presence of silane led to increase in tensile properties, flexural strength, unnotched and notched impact strength but reduction in flexural modulus. The composite with 4% silane loading showed the best mechanical and physical properties.

FTIR spectra of the composites indicated that there were interaction between the components of the composites and new bonds formed after addition of additive. Thermal stability of the composites decreases with increasing of fiber content. The presence of silane on the composites did not show any significant improvement in thermal stability as shown by Thermogravimetric Analysis. The SEM micrographs of the fractured surfaces gave clear indication of the effect of silane in reduction of void size and close interaction of PBAT and OPEFB fiber was clearly demonstrated for composite with additive which resulted in improvement of mechanical properties of composite. Dynamic mechanical analysis results showed that storage modulus and loss modulus of composites increased with increasing fiber loading. Furthermore, T_g of PBAT positively shifted after the addition of fiber, indicated there is interaction

between PBAT and OPEFB. The crosslinked composite presented higher T_g than non crosslinked composite. With the increased of fiber loading, the water absorption was also increased, and the addition of additive led to less water absorption. The biodegradability of composites was evaluated by the soil burial test for three months. The results showed that the composites were degraded faster than pure matrix due to cellulosic materials is easily attacked by microorganisms. It was also observed that addition of silane as well as the fiber loading tends to promote the degradation rate of PBAT. However the rate of biodegradation of PBAT was slower than expected.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untak ijazah Master Sains

Pengaruh Kandungan Serat dan Rawatan Silana Terhadap Sifat Biokomposite Poli(Butilena Adipate-Ko- Tereftalat/Sirat Tandan Kosong Buah Kelapa Sawit

Oleh

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Dalam kajian ini, bahan komposit hijau serat tandan kosong sawit (OPEFB) (100-200 µm) dengan poli(butilena adipate-ko-tereftalat) (PBAT) menggunakan vinil tris (2 metoksietoksi)silana sebagai ejen rangkai silang telah disediakan dengan teknik pengadunan lebur. Kesan kandungan serat dan tambahan vinil tris(2 metoksietoksi)silana pada ciri-ciri mekanikal dan terma biokomposit PBAT/OPEFB telah dikaji. Bahan komposit tersebut telah disediakan dengan menggunakan 10, 20, 30, 40 dan 50 % serat. Muatan serat berbeza telah diuji bagi menentukan kandungan optimum serat. Diperlukan bagi mendapatkan komposit yang baik dengan 40% muatan serat, dengan suhu dan masa pengadunan masing-masing pada 120 ºC dan 12 minit dan kelajuan putaran 30 rpm telah didapati sebagai formulasi dan keadaan terbaik untuk penyediaan komposit.

Sifat mekanikal seperti regangan, tegangan, lenturan dan kekuatan hentaman (tidak gerigis dan bergerisis) telah dijalankan. Penyerapan air dan kajian degradasi juga dijalankan. Bahan komposit menunjukkan peningkatan dalam kekuatan tegangan dan modulus, lenturan kekuatan dan modulus dan kekuatan hentaman tidak gerigis dan bergerisis dengan tambahan pemuatan serat.

Kehadiran silana pada muatan 1, 2, 3, 4 dan 5 % meningkatkan sifat tegang, lenturan kekuatan, dan kekuatan hentaman bergerigis dan tidak bergerisis dan pengurangan dalam modulus bengkok. Pada keseluruhannya, komposit dengan 4% silana menunjukkan ciri mekanikal dan fizikal yang terbaik.

FTIR analisis menunjukkan bahawa terdapat interaksi antara komponen bahan komposit dan ikatan baru terbentuk selepas dimasukkan bahan tambahan. Kestabilan haba bagi komposit berkurangan dengan pertambahan kandungan serat. Kehadiran silana dalam bahan komposit itu tidak menunjukkan sebarang peningkatan kestabilan haba yang ketara seperti ditunjukkan oleh Analisis Thermogravimetri. Mikrograf SEM bagi permukaan retakan menunjukkan dengan jelas kesan silana dalam pengurangan saiz lubang, dan interaksi PBAT dan serat adalah dengan jelas ditunjukkan oleh komposit dengan bahan tambah yang menyebabkan peningkatan sifat mekanikal komposit. Keputusan DMA menunjukkan storan modulus dan kehilangan modulus bahan komposit bertambah dengan penambahan kandungan serat. Tambahan pula, T_g PBAT secara positif beralih selepas pertambahan serat itu, menunjukkan terdapat interaksi antara PBAT dan OPEFB. Komposit yang bertaut silang menunjukkan T_g yang lebih tinggi daripada komposit yang tidak bertaut silang. Dengan pertambahan kandungan serat, penyerapan air juga bertambah, dan kemasukan bahan tambah itu membawa kepada pengurangan penyerapan air. Kebolehan biodegradasi bahan komposit dikaji dengan ujian penanaman dalam tanah selama tiga bulan. Keputusan ujian menunjukkan bahawa penguraian bahan komposit adalah lebih cepat berbanding matriks tulen kerana bahan berselulosa lebih mudah diserang oleh mikroorganisma. Ini juga menunjukkan pertambahan silana serta pemuatan serat menggalakkan kadar penguraian bahan komposit. Walau bagaimanapun kadar biodegradasi PBAT adalah lebih perlahan daripada yang dijangkakan.

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Special thanks also to all the faculty staff members that have helped me throughout this period. It is a pleasure to acknowledge my husband Farokh Abazari and my parents for their kind support, encouragements, understanding.

I certify that an Examination Committee has met on 7 January 2010 to conduct the final examination of Zahra Nozari on her thesis entitled “Effect of Fiber Loading and Silane Treatment on Properties of Poly (Butylene Adipate-Co-Terephthalate)/Oil Palm Empty Fruit Bunch Fiber Biocomposites”, in accordance with Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1981.The Committee recommends that the student be awarded the Master of Science. Members of the Thesis Examination Committee were as follows:

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Date: 15 January 2010

This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirements for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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Date: 8 April 2010

DECLARATION

I hereby declare that the thesis is based on my original work expect for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

ZAHRA NOZARI

Date: 4 March 2010

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LIST OF ABBREVIATIONS

AS	(3-aminopropyl)-triethoxysilane
ASTM	American Society for Testing Materials
BDO	1, 4-butanediol
BGRP	Bamboo-glass fiber reinforced polypropylene hybrid composite
CCMs	Carbon-Carbon Composites
CMCs	Ceramic Matrix Composites
DMA	Dynamic Mechanical Analysis
DMI	Diphenyl methane diisocyanate
DMT	Dimethylterephthalate
DTG	Differential Thermogravimetry
E'	Storage Modulus
E"	Loss Modulus
EFB	Empty Fruit Bunch
FTIR	Fourier Transform Infrared
HDPE	High-Density Polyethylene
HMDI	Hexamethylene diisocyanate
LLDPE	linear low-Density Polyethylene
LDPE	Low Density Polyethylene
LF	Luffa Fiber
MA	Maleic Anhydride
MAPP	Maleic Anhydride Maleated Polypropylene

MMCs	Metalic Matrix Composites
MPa	Mega Pascal
NF	Natural Fiber
OPEFB	Oil Palm Empty Fruit Bunch
OPEFB/PBAT	Oil Palm Empty Fruit Bunch- Poly (butylene adipate co- terephthalate) composite
OPWF	Oil Palm Wood Flour
PBAT	Poly (butylene adipate co- terephthalate)
PHBV	Poly (hydroxybutyrate-co-valerate)
PCL	Poly (ε-caprolactone)
PCL-*g*-MA	Poly (ε-caprolactone)-*g*-maleic anhydride
PE	Polyethylene
PEFB	Phenolated Empty Fruit Bunch
PEG	Polyethylene Glycol
PFRR	Polymers from renewable resources
PHB	Polyhydroxybutyrate
PLA	Poly (lactic acid)
PMCs	Polymer Matrix Composites
PU	Polyurethane
PU/EFB	Polyurethane-Empty Fruit Bunch composite
PP	Polypropylene
RH	Rice Husk
Silane A-172	Vinyl tris(2methoxy ethoxy)silane
SEM	Scanning Electron Microscopy

Tan δ	Tangent Delta
TBOT	Tetrabutylorthotitanate
TDI	Toluene Diisocyanate
T_g	Glass Transition
TGA	Thermogravimetric Analysis
T_m	Melting Tempareture
TPS	Thermoplastic Starch
UV	Ultra-Violet

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Nowadays, our whole world seems to be encapsulated by plastics. Almost every thing we use come encased in plastic. One great disadvantage of plastics is the problem of disposal. Many environmental problems have been made because of the non- biodegradability of most plastics. As a result, they become new environmental pollutants because of their resistance to microbial degradation or deterioration. Although recycling is an environmentally attractive solution (Abdel-Rehim *et al*., 2004), the used of material components such as natural fibers, biodegradable polymers which have been considered to be environmentally safe are interesting alternatives for the development of new biodegradable composites (Averous *et al*., 2006).

Within this group of new and innovative polymer, polyesters play a superior role. This is due to the presence of the ester bonds in polyesters which are potentially hydrolysable. Even though aromatic polyesters such as poly (ethylene terephthalate) show excellent material properties while proving to be almost resistant to microbial attack, several aliphatic polyesters produced to be biodegradable but are deficient in properties, which are important for application. Therefore, aliphatic–aromatic copolyesters have been developed as biodegradable polymers for many years, to combine good material properties with biodegradability (Muller *et al.*, 2001).

However, even though the use of fully biodegradable polymers as a substitution to the traditional non-biodegradable polymers could contribute to the solution of the waste problem, but their use is limited due to their relatively high cost. So the use of low cost fillers as a way of reducing the cost of the end product is necessary. Lignocellulosic materials due to their attractive properties such as, low density, low cost, abundance, renewability and biodegradability, can be suitable filler for biodegradable matrices (Tserki *et al.,* 2006).

Oil palm empty fruit bunch fibers can be used as environmentally-friendly alternatives to conventional reinforcing fibers in composites. The main shortcomings of the use of natural fibers for reinforcement are the weak adhesion between polar-hydrophilic OPEFB and non polar-hydrophobic plastics, and poor resistance to moisture absorption. Nowadays there are several methods to improve the interfacial interaction between lignocellulosic materials and polymers which consist of: the chemical modification of natural fiber, the use of a modified polymer that able to reacting or interacting with fibers, use of coupling agent or compatibilizer with the ability to interact simultaneously at the interface with the polymer and the fiber.