UNIVERSITI PUTRA MALAYSIA

# PREPARATION AND PHYSICO-CHEMICAL PROPERTIES OF POLYPROPYLENE AND OIL PALM EMPTY FRUIT BUNCH FIBRE COMPOSITES

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**By**

**NORAZLINA BINTI HASHIM**

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**JANUARY 2008**

**Chairman : Associate Professor Dr. Mohamad Zaki bin Ab Rahman, PhD**

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Composites of polypropylene (PP) and oil palm empty fruit bunch (OPEFB) were prepared by melt blending, and the effects of ethylene-butene copolymer (EBNR) as an impact modifier, on the mechanical and thermal properties of OPEFB:PP composites were investigated by using FTIR, DSC, TGA and SEM. Composites were prepared at 10, 20, 30, 40, 50 and 60% by weight of fibre. Different fibre loading, temperatures, rotation time and rotation speeds were tested to determine the optimum condition of blending. Two range sizes of fibre were used in this study and there are below 200 µm and between 200-315 µm of length (known as 200 µm and 315 µm). The study was mainly conducted on composite with 20% and 40% fibre loading. The optimum temperature and reaction period for blending OPEFB with PP were 170$^{o}$C and 10 minutes with rotation speed at 50 rpm, respectively.

The result from FTIR analysis indicated that the interaction is only physical between the components of the composite. Tensile tests on the composites showed that EBNR had a positive effect on the elongation at break and negative effect on tensile modulus, but no improvement on tensile strength. This impact modifier was found to decrease the stiffness of the composites. The impact strength was improved in addition of EBNR, but for flexural test, no significant effect was observed. The increasing in impact strength was shown for both notched and unnotched samples starting from 1% of EBNR loading but they reduced when it reached at optimum volume. Smaller fibre size, 200 µm gave higher impact strength compared to 315 µm.

The presence of EBNR on the composite made a weak improvement in thermal stability as shown by thermogravimetric analysis (TGA). DMA studies established that the EBNR led to reduction of stiffness and enhancing mobility of the resulting composite. The surface morphology showed less void in EBNR composites where OPEFB fibre seems to be covered with a smooth layer of EBNR.

The environmental degradation behaviour on the physical and mechanical properties of OPEFB:PP composites has been studied with special reference to the influence of ageing conditions like treatment with water, soil degradation, and chemical resistance. With the increased of fibre loading, the water absorption was also increased, and the addition of EBNR led to less water absorption, and the lowest absorption was observed for 8% EBNR loading. Tensile properties decrease with water uptake and time of immersion. The effect of alkaline (0.5M NaOH) and acidic (0.5M HCl) solutions on the composites were investigated in a period of one month.

The results showed the decreasing in tensile strength and mass. Temperature dependence studies were also conducted at different temperature, 25$^{o}$C, 45$^{o}$C and 75$^{o}$C, and found that the degradation was faster at higher temperature. The biodegradability of composites was evaluated by the soil-burial test in six months. The mass of composites slowly showed the decreasing after that time as expected.

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

**PENYEDIAAN DAN CIRI-CIRI FIZIK-KIMIA KOMPOSIT POLIPROPILENA DAN FIBER TANDAN KOSONG BUAH KELAPA SAWIT**

Oleh

**NORAZLINA BINTI HASHIM**

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Komposit polipropilena (PP) dan tandan kosong buah kelapa sawit (OPEFB) telah disediakan melalui kaedah adunan dan kesan menggunakan pengubah impak, kopolimer etilena-butilena (EBNR) kepada sifat mekanik dan terma komposit OPEFB:PP telah dikaji dengan menggunakan pelbagai teknik seperti FTIR, DSC, TGA dan SEM. Komposit disediakan pada 10, 20, 30, 40, 50 dan 60% berdasarkan berat fiber. Amaun fiber, suhu, jangka masa dan laju putaran yang berbeza telah dikaji untuk mendapatkan keadaan adunan yang optimum. Dua skala saiz fiber digunakan iaitu lebih kecil daripada 200 μm dan antara 200-315 μm panjang (dikenali sebagai 200 μm dan 315 μm). Akhirnya, penambahan OPEFB sebanyak 20% dan 40% digunakan dalam kajian ini. Suhu optimum dan jangka masa untuk adunan OPEFB dan PP adalah 170$^{o}$C, 10 minit adunan dengan 50 rpm kelajuan putaran.

Analisis FTIR menunjukkan hanya terdapat interaksi fizikal antara komponen dalam komposit. Ujian tensil bagi komposit menunjukkan EBNR memberi kesan positif ke atas pemanjangan pada takat putus dan merendahkan modulus tensil tetapi tiada perubahan dalam kekuatan tensil dapat dilihat. Pengubah impak ini telah merendahkan kesan kekakuan komposit. Kekuatan impak ditingkatkan dengan penambahan EBNR di dalam komposit, tetapi tiada perubahan lenturan dikesan. Peningkatan dalam kekuatan impak dapat dilihat untuk kedua-dua sampel yg ditakuk dan tak ditakuk bermula daripada 1% EBNR tetapi berkurang setelah penambahan EBNR mencapai tahap optimum. Saiz fiber yang lebih kecil, 200 µm memberi kekuatan impak yang lebih tinggi berbanding saiz 315 µm.

Kehadiran EBNR dalam komposit tidak memperbaiki kestabilan terma seperti yang ditunjukkan dalam analisis termogravimetri (TGA). Kajian DMA menunjukkan EBNR menjurus kepada penurunan kekerasan dan menambahkan mobiliti komposit. Kajian permukaan sampel menunjukkan kurang ruang kosong terbentuk di dalam komposit EBNR di mana OPEFB diselaputi lapisan licin EBNR.

Kesan degradasi persekitaran kepada ciri-ciri fizikal dan mekanikal bagi komposit OPEFB:PP dikaji dengan melibatkan ujian sampel ke atas penyerapan air, degradasi tanah dan ketahanan ke atas bahan kimia. Penyerapan air meningkat dengan pertambahan fiber dan penambahan EBNR membawa kepada pengurangan kadar penyerapan air dengan serapan terendah pada 8% kandungan EBNR. Ciri-ciri kekuatan tensil berkurang dengan peningkatan penyerapan air dan waktu rendaman

sampel. Kesan larutan alkali (0.5M NaOH) dan asid (0.5M HCl) ke atas komposit dikaji selama satu bulan.

Keputusan menunjukkan pengurangan jisim dan kekuatan tensil. Kesan suhu ke atas sampel dilakukan pada tiga keadaan suhu yang berbeza iaitu pada 25$^{o}$C, 45$^{o}$C and 75$^{o}$C dan ia menunjukkan kadar degradasi bertambah dengan peningkatan suhu.

Proses biodegradasi komposit telah ditentukan dengan menanam sampel di dalam tanah selama enam bulan. Jisim komposit telah berkurang secara perlahan-lahan seperti yang dianggarkan.

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## DECLARATION

I hereby declare that the thesis is based on my original work except 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.

**NORAZLINA BINTI HASHIM**

Date: 29 February 2008

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# LIST OF ABBREVIATIONS/NOTATION/GLOSSARY OF TERMS

| | |
|---|---|
| ABS | Acrylonitrile-butadiene-styrene |
| AS | (3-aminopropyl)-triethoxysilane |
| ASTM | American Standard for Testing and Materials |
| BGRP | Bamboo fibre reinforced polypropylene composite |
| CPE | Chlorinated polyethylene |
| DMA | Dynamic mechanical analysis |
| DSC | Differential scanning calorimetry |
| DTG | Differential thermogravimetry |
| EBNR | Ethylene-butene copolymer |
| EFB | Empty fruit bunch |
| EPDM | Ethylene/propylene/diene copolymer |
| EPDM-MAH | Maleated ethylene/propylene/diene copolymer |
| EPM | Ethylene/propylene copolymer |
| EP-MAH | Maleated ethylene-propylene |
| EVA | Ethylene/vinyl acetate |
| FFB | Fresh fruit bunch |
| FTIR | Fourier transform infrared |
| GF | Glass fibre |
| GMA | Glycidyl metacrylate |
| GP/PP | Glass fibre-polypropylene composite |
| HCl | Hydrochloric acid |
| HDPE | High density polyethylene |
| LDPE | Low density polyethylene |

| | |
|---|---|
| LF/PP | Luffa fibre-polypropylene composite |
| MAPP | Maleic anhydride-polypropylene |
| MMA | Methyl metacrylate |
| MPOB | Malaysian Palm Oil Bard |
| MS | 3-(trimethoxysilyl)-1-propanethiol |
| NaOH | Sodium hydroxide |
| NBR | Acrylonitrile/butadiene elastomer |
| OPEFB | Oil palm empty fruit bunch |
| OPEFB:PP | Oil palm empty fruit bunch-polypropylene composite |
| PE | Polyethylene |
| PE/WF | Polyethylene-wood fibre composite |
| PET | Polyester |
| PIB | Polyisobutylene |
| PLA | Poly-lactic acid |
| PMMA | Polymethyl metacrylate |
| PORIM | Palm Oil Research and Development Board |
| PP | Polypropylene |
| PP/RNFC | Polypropylene-recycled newspaper cellulose fibre |
| PP/WF | Polypropylene-wood fibre composite |
| PPG | Polypropylene glycol |
| PP-g-GMA | Polypropylene grafted glycidyl metacrylate |
| PS | Polystyrene |
| PVC | Polyvinyl chloride |
| R&D | Research and development |
| RNFC | Recycled newspaper cellulose fibre |

| | |
|---|---|
| SBS | Styrene-butadiene-styrene |
| SEBS-MAH | Maleated styrene-ethylene/butadiene-styrene |
| TDI | Toluene diisocyanate |
| TDI/PPG | Toluene-2,4-diisocyanate/polypropylene glycol |
| $T_g$ | Glass transition |
| TG | Thermal gravimetric |
| TGA | Thermogravimetric analysis |
| $T_m$ | Melting temperature |
| TPO | Ethylene/propane thermoplastic elastomer |
| UV | Ultra-violet |
| WPC | Wood-plastic composite |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Advanced in science and technology pose new challenges in relation to certain environmental issues, such as biodegradability, recyclability, eco-friendliness etc., that needs to be addressed to help preserve and protect our environment. Composite materials from the annually renewable natural fibres and biodegradable matrices have been developed in the past decades in an attempt to find alternatives to the fossil fuel-based polymeric materials in the automotive and packaging industries.

The benefits offered by lignocellulosic materials include making the final product lighter and decreasing the wear of the machinery used in the production process. Moreover, lignocellulosic materials have the advantages of low cost, biodegradability and the absence of residues or toxic byproducts, whereas inorganic materials such as glass fibre, carbon fibre, talc, clay, synthetic fibre, etc. do not have these benefits (Yang *et al*., 2004; Lee *et al*., 2004). Yet another attraction of using these materials is the fact that it would allow various agro-wastes to be appropriately recycled.

The performance and properties of composite materials depend on the properties of the individual components and their interfacial compatibility. To ensure appropriate interfacial interactions their surface properties must be modified accordingly.