UNIVERSITI PUTRA MALAYSIA

PREPARATION AND CHARACTERIZATION OF RUBBERWOOD FIBER-POLYPROPYLENE COMPOSITES

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By

AKRAMSADAT TAYEFEH MORSAL

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

August 2007

DEDICATION

TO MY PARENTS, MY LOVELY MOTHER AND DAD

Abstract of thesis presented to the Senate of University Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

PREPARATION AND CHARACTERIZATION OF RUBBERWOOD FIBER-POLYPROPYLENE COMPOSITES

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August 2007

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In this study, blending of rubberwood fiber with polypropylene and the effects of EBNR (ethylene-butene copolymer) as the compatibilizer, on the mechanical and thermal properties of rubber wood fiber, polypropylene composites were investigated by using FTIR, TGA, DMA and SEM. Composites were prepared at 10, 20, 30, 40, and 50 % by weight of fiber. Different fiber loadings, durations, temperatures, and rotation speeds process were tested to determine the optimum condition of blending. Consequently, the composite with 40% fiber loading, temperature at 175 °C, for 15 min and rotation speed of 40 rpm were selected to be the best composite.

Mechanical tests including tensile, flexural and impact strength (notched and unnotched) were performed. In addition, water absorption study was carried out. The properties of composite without elastomer showed the reduction in tensile strength and impact strength (notched and unnotched) with increase in fiber loading, however the increase in tensile modulus, flexural strength and modulus of the composite was observed.

The presence of EBNR in all loadings led to reduction in tensile and flexural strength and increased the impact strength. With attention to all aspects, the composite with 10% EBNR loading showed the best mechanical and physical properties. With the increased of fiber loading, the water absorption was also increased, and the addition of EBNR led to less water absorption, and the lowest absorption was observed for 10% EBNR loading.

The result from FTIR analysis indicated that the interaction is only physical between components of the composite. The presence of EBNR on the composite made a weak improvement in thermal stability as shown by thermogravimetric analysis (TGA). DMA studies established that EBNR led to reduction of stiffness and enhancing mobility of the resulting composite. SEM micrographs gave clear indication of the effect of EBNR in reduction of void sizes and numbers, and close interaction of PP and fiber was clearly demonstrated for composite with elastomer.

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

PENYEDIAAN DAN PENCIRIAN KOMPOSIT GENTIAN KAYA GETAH-POLIPROPILENA

Oleh

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Dalam kajian ini, adunan gentian kayu getah bersama polipropilena dan pengaruh EBNR (kopolimer etilena-butena) sebagai perserasi terhadap sifat mekanik dan terma komposit gentian kayu getah dan polipropilena telah dikaji menggunakan berbagai teknik seperti FTIR, TGA, DMA dan SEM. Komposit telah dihasilkan pada 10, 20, 30, 40 dan 50% berdasarkan berat gentian. Amaun gentian yang digunakan, jangka masa, suhu dan laju putaran yang berbeza telah digunakan untuk mendapatkan keadaan adunan yang optimum. Akhirnya komposit dengan 40% kandungan gentian, suhu 175°C, jangka masa selama 15 min dan kelajuan putaran 40 rpm telah dipilih untuk menghasilkan komposit yang terbaik.

Ujian mekanikal termasuk regangan kelenturan dan kekuatan impak (takuk dan tak takuk). Disamping itu ujian serapan air turut dijalankan. Komposit tanpa elastomer menunjukkan penurunan dari segi kekuatan tensil dan impak (takuk dan tak takuk). Walau bagaimana pun, kekuatan tensil, lenturan dan modulus komposit meningkat dengan bertambahnya kuantiti gentian.

Kehadiran EBNR dalam semua penambahan menjurus kepada penurunan kekuatan tensil dan lenturan tetapi meningkatkan kekuatan impak. Berdasarkan semua aspek, komposit dengan 10% kandungan EBNR menunjukkan sifat mekanik dan fizikal yang terbaik. Dengan peningkatan kandungan gentian, serapan air meningkat dan penambahan EBNR menjurus kepada pengurangan serapan. Oleh itu serapan terendah dikesan pada 10% kadungan EBNR.

Analisis menggunakan FTIR menunjukkan terdapat hanya interaksi fizikal antara komponen dalam komposit. Kehadiran EBNR dalam komposit tidak memperbaiki kestabilan terma seperti ditunjukkan dalam analisis termogravimetri (TGA). Kajian DMA menunjukkan EBNR menjurus kepada penurunan kekerasan dan menambahkan mobiliti komposit.

mikrograf SEM jelas menunjukkan kesan penambahan EBNR dapat mengurangkan saiz dan bilangan rongga dan interaksi antara PP dan gentian telah ditunjukkan dalam komposit dengan elastomer.

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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.

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AKRAMSADAT TAYEFEH MORSAL

Date: 22 September 2007

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LIST OF ABBREVIATIONS

ASTM	American society for testing and material
DMA	Dynamic mechanical analysis
DTG	Negative first derivatives of the thermogravimetry
E’	Storage modulus
E”	Loss modulus
EBNR	Ethylene-butene copolymer
EPDM	Ethylene propylene diene terpolymer
FTIR	Fourier transformed infrared
GPa	Giga pascal
J/m	Joule per meter
LDPE	Low density polyethylene
MA-PE	Maleated polyethylene
MA-PP	Maleated polypropylene
MPa	Mega pascal
PP	Polypropylene
PE	Polyethylene
RWF	Rubberwood fiber
RWPC	Rubberwood plastic composite
SBS	Styrene butylene styrene copolymer
SEM	Scanning electron microscpy
TGA	Thermogravimetric analysis
TPEs	Thermoplastic elastomers
WPC	Wood-plastic composite

CHAPTER 1

Introduction

1.1 General Review

In the recent years, there is a rapid growth in the study and use of composite materials. The major thrust in the area of composite material has been directed towards the development and study of high performance reinforcing materials like nylon, polyester, Kevlar, glass, and carbon fiber in appropriate polymer composites. Nevertheless, these materials are expensive and non-renewable resources. Because of the uncertainties prevailing in the supply and price of petroleum-based products, there is every need to develop naturally occurring alternatives. Recently, natural fibers such as sisal, kenaf, jute, bamboo, coir, henequen, pineapple and rubber wood fiber have attracted the attention of scientists and technologists for applications in consumer goods, low-cost housing and civil structures (George et al., 1993).

The use of natural fibers for composites offers many potential advantages like low costs, low density, low energy consumption, high specific properties, biodegradability, flexibility, wide variety of fibers available throughout the world, generation of a rural/agriculture-based economy, reduced wear of processing machinery and no health problem. The accomplished researched by

Sanadi et al. (1994) are recognized the below advantages about adding of natural fiber to polypropylene;

1) Low cost manufacturing, 2) Indefinite property stability, 3) Flat sheet stoke thermoforming, 4) Reprocess able to correct flaws, 5) Fast processing cycle, 6) Storage flexibility, 7) High fracture toughness, 8) Good damage tolerance with high impact resistance 9) Good resistance to micro-cracking 10) Better water absorption properties compared to other wood based composite.

Wood has been used as building and engineering material since early times and offers the advantages of not just being aesthetically pleasing but also renewable, recyclable and biodegradable. (Zadorecki et al., 1989).

Wood based composites with a continuous thermoplastic phase also give the opportunity to process the composite using conventional thermoplastic processing equipment (Bengtsson et al., 2005). More over, the processing of wood compared to the inorganic fillers used in a great amount before. (Dalavag et al., 1985).

In spite of all the advantages mentioned above, there are also some drawbacks in using wood fillers as reinforcement in thermoplastics. The main draw backs are the difficulties of achieving good dispersion and strong interfacial adhesion between the hydrophilic wood and the hydrophobic polymers which leads to composites with rather poor durability and mechanical properties (Dalavag et

al., 1985; Park et al., 1996; Kokta et al., 1989; Lai et al., 2003; Maldas et al., 1994). And also high moisture absorption of fiber and composites and low processing temperature is permissible (Mishra et al., 2001).

1.2 Rubber wood Fiber

In the utilization of rubber wood, the study of its wood structure and characteristics is important as it establishes the variations and properties of the wood. The parameters for its optimal utilization can then be adjusted to accommodate the various inherent properties of the timber.

The fiber morphology of rubber wood compared to other fibers is given in Table1.1.

Table 1.1: Morphology of rubber wood fibers compared to other fibers

Type of fiber	Fiber length (mm)	Fiber Width (***μ***)	Fiber wall thickness (***μ***)
Rubber wood	1.25	25.75	5.70
Rubber wood	1.40	31.30	5.00
Douglas fir	3.40	40.00	-
Oil palm trunk	1.22	35.30	4.50
Kenaf core(180_{dap})	0.36	31.60	6.40
Kenaf bast (180_{dap})	2.42	13.60	3.70

dap – day after planting Source: (Liew, 1993; Mohd et al., 1985; Rowell et al., 1997)

The fiber length of rubber wood was comparable more to oil palm and kenaf core fibers and less than Douglas fir or Kenaf bast. The longer the fiber the higher is the probability of interaction with the polymer. The simplest known fiber is spherical filler of particulate. With wood and agro-fiber, the theoretical analysis of composite behavior became complicated, as its fiber length and thickness, as well as cell voids have to be considered. As the width and length of fiber does not have a 1:1 ratio, fiber orientation effect has to be taken into consideration when dealing with wood composite properties.

The chemical constituent of rubber wood is summarized in Table 1.2.

Table 1.2: Chemical composition of rubberwood and several other cellulosic fibers

Composition	Rubberwood	Oil palm trunk	Kenaf	American Beech
Holocellulose	67.0	45.7	47-57	77
Alpha-cellulose	41.5	29.2	31-39	49
Lignin	26.0	18.8	15-19	22
Ash	1.5	2.3	2-5	0.4
Alkali soluble	19.2	40.2	28-33	14

Source: (Peel, 1960; Mohd et al., 1985)

Rubberwood, which has equivalent lignin content to hardwood such as American Beech or softwood including fir was expected to give acceptable WPC strength. On the other hand, the holocellulose would have greater tendency to be reactive, as there were more amorphous region for actual chemical interactions. These amorphous regions are potential crosslinking and grafting area.

In the utilization of rubberwood, the density and bulk density are to be taken into consideration. The average density rubberwood is 530 Kg/cm^3 which are very similar to dark red meranti (540 Kg/m^3). The density differential for stem and branch wood was found to be about 3% (Lim, 1996). Compared to other fiber materials, rubberwood has relatively lower bulk density of 100-106 g/l than the industrial wood waste (180 g/l). However its density is not as low as *Acacia mangium* (70 g/l) or oil palm fibers (76 g/l) (Chew et al., 1991).

1.2.1 Application and Importance of Rubberwood Fiber

With the structure and characteristic of RWF, the use of lignocellulosic or natural fibers, such as wood and cellulose, in the production of thermoplastic composites is becoming more attractive, at least judging from the increasing numbers of literature and convention in that area. This development has been observed because reinforcement by natural fiber or lignocellulosic filler already offer several advantages over their inorganic counter parts and also over traditional reinforcing materials such as glass fiber, talc and mica. The advantages are low cost, acceptable specific strength properties, low density, high toughness, good thermal properties and biodegradability. It has been demonstrated that wood fiber reinforced polypropylene composites have properties similar to traditional fiber reinforced polypropylene composites (Narayan, 1992). Therefore, these materials have been subjected in making conventional panel products or producing plastics composites.

1.3 Plastics

Over the last half decade, plastics are commercially used materials that are based on polymers or pre-polymers, and the volume usage of plastics in industry now is still expanding at twice the rate economy as a whole. The rapid growth of their production is caused by three factors such as growth of world population, average increase of living standards and replacement of older materials by plastics (Elias, 1993). The motivation for the rapid growth is the suitability of plastics for mass production, which depends mainly on easy and reproducible shaping, low volume cost and some attractive properties (Birly et al., 1988).

Plastics are from the formation of molecules composed of many units with high molar masses or commonly known as polymers. The term polymer carries with it the connotation of polymer molecules composed of many equal mers. Most of the plastics are made from large molecules that are constructed by a chain-like attachment of certain building block molecules (Beck, 1980). The nature of the repeat unit in polymer and the constrains imposed by the chain or network structure can result in a regularity of structure which is termed as crystallinity may have important consequence. The occurrence of crystallinity in a polymer affects profoundly both processing and properties. This, as the crystallinity increases then the polymer molecules pack closer together and properties such as density, hardness and shrinkage increase (Whelan, 1982).

1.3.1 Properties of Plastics

The use of thermoplastic as matrix for composites is increasingly growing and becoming popular products in manufacturing industry. Thermoplastic is a family of polymer materials composed of long – chain molecules with weak cross – links to adjacent links. When fully polymerized, they remain capable of being repeatedly softened or turned into mobile viscous liquids when exposed to heat and hardened or re-hardened when cooling (Richardson, 1989). Thermoplastics are corrosion resistant, relatively cheap, light weight and with better flexibility compared to metal and steel which are being replaced in many application, but their poor toughness stress relaxation behavior and low modulus limit their commercial applications (Oksman, 1996).

The use of thermoplastic polypropylene (PP) in demanding engineering application has increased rapidly in recent years (Gloria, 2001). With its attractive properties of high melting point, low density and good chemical inertness, it becomes one of the most important choices for fiber, filler compounding and blending works. Through such work, it is possible to prepare composites with enhanced properties and which can be tailor-made base on-end-use requirements. Such composites can be also remelted and reused, thus PP composites are profitable, recyclable products and more acceptable from the ecological point of view. Besides, PP is at the moment one of the cheapest thermoplastic in the market.