UNIVERSITI PUTRA MALAYSIA

PROPERTIES OF RESIN-IMPREGNATED SUGAR PALM FIBRE- REINFORCED EPOXY COMPOSITES

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By

NUR SOFEANA ZULAIKA BINTI MOHD NOR MUNAWAR

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

May 2018

DEDICATION

This thesis is dedicated to my beloved parents;

Mohd Nor Munawar Bin Abd. Bari

and

Rosliza Binti Ibrahim

Thank you very much for all your love and support that contributed to the publication of this thesis.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Natural fibres are produced in abundance and readily available as compared to synthetic fibre which is expensive and takes a longer time to degrade. Sugar palm (*Arenga pinnata*) fibre is one of potential locally available natural fibre belongs to the Palmae family. This study focuses on the effect of vacuum resin impregnation on sugar palm fibre (SPF) using epoxy, vinyl ester (VE) and polyester (PE) as the impregnating agents. Initially, the SPF is impregnated with epoxy, VE and PE where the fibres are placed in a beaker filled with the specific impregnating agent and vacuum at a constant impregnation pressure of 600 mmHg for 5 minutes. Then, the fibres are drained to remove the excess resins and cured in an oven for 30 minutes at a temperature of 140°C before tested for its physical and tensile properties. The impregnated fibres are used to fabricate into a composite with epoxy as the matrix. The physical, mechanical and chemical composition of the composites is evaluated.

It is found that the single SPF impregnated with epoxy offered greater value of tensile strength and modulus followed by SPF/VE and SPF/PE. The scanning electron micrograph indicates that epoxy resin displays better impregnation on sugar palm fibre compared to the other thermosetting resins. FT-IR proves the existence of lignocellulosic materials in the fibres such as lignin, cellulose and hemicellulose.

Similar results are observed for the resin impregnated SPF reinforced epoxy composite, where their tensile properties showed a better performance. Meanwhile, under flexural testing, all the impregnated SPF reinforced epoxy composites showed a reduction in their values indicating that the composite is not suitable for bending application. SPF/VE reinforced epoxy composite has the best flexural strength while SPF/Epoxy reinforced epoxy composite has the best flexural modulus for impregnated SPF composites. SPF/PE reinforced epoxy composite resulted in the worst reading for both tensile and flexural testing.

The dimensional stability for resin impregnated SPF reinforced epoxy composites has significantly improved. Both water absorption (WA) and thickness swelling (TS) of all the impregnated fibre composite are reduced. SPF/Epoxy reinforced epoxy composite has the most reduction in value for both testings. On top of that, the density and specific gravity (SG) for the impregnated SPF reinforced epoxy also showed a reduction in their reading. SPF/VE reinforced epoxy composite has the highest reduction followed by SPF/PE reinforced epoxy composite and SPF/Epoxy reinforced epoxy composite.

Overall results showed that both fibre and composite part shows that VE is the best impregnating agent while SPF/PE gave the worst result. It can be concluded that treating the SPF with VE will enhance the properties of sugar palm fibre composite.

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

SIFAT-SIFAT GENTIAN ENAU DIIMPREGNASI DAMAR DIPERKUAT KOMPOSIT EPOKSI

Oleh

NUR SOFEANA ZULAIKA BINTI MOHD NOR MUNAWAR

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Gentian semulajadi dihasilkan dengan banyak dan mudah didapati berbanding gentian sintetik yang mahal dan mengambil masa yang lebih lama untuk lupus. Pokok Enau (*Arenga pinnata*) adalah sejenis tumbuhan yang berasal daripada keluarga Palmae dan mempunyai gentian secara semulajadi. Kajian ini menumpukan kepada kesan penyerapan damar pada gentian enau (SPF) menggunakan epoksi, vinil ester (VE) dan poliester (PE) sebagai ejen impregnasi. Pada mulanya, SPF diimpregnasi dengan epoksi, VE dan PE di mana gentian diletakkan di dalam bikar yang diisi dengan ejen impregnasi dan vakum pada tekanan 600 mmHg selama 5 minit. Kemudian, gentian yang telah diimpregnasi disalirkan untuk mengeluarkan damar yang berlebihan dan dikeringkan dalam ketuhar selama 30 minit pada suhu 140°C sebelum diuji untuk sifat fizikal dan mekanikal. Selepas itu, gentian yang diimpregnasi difabrikasi ke dalam plat komposit dengan menggunakan epoksi sebagai matrik. Sifat fizikal, mekanikal dan komposisi kimia dinilai.

Keputusan mendapati bahawa SPF tunggal yang dirawat dengan epoksi menawarkan nilai kekuatan tegangan dan modulus yang lebih besar diikuti oleh SPF/VE dan SPF/PE. Imej-imej pengimbasan elektron menunjukkan bahawa damar epoksi menawarkan impregnasi yang lebih baik pada gentian enau derbanding damar termoset lain. Spectra FT-IR membuktikan kewujudan bahan – bahan lignoselulosa dalam gentian enau seperti lignin, selulosa and hemiselulosa.

Hasil yang sama diperhatikan untuk komposit SPF yang diperkuat dengan epoksi, di mana sifat tegangannya menunjukkan prestasi yang lebih baik. Sementara itu di bawah ujian lenturan, semua komposit SPF diperkuat epoksi yang diimpregnasi menunjukkan penurunan dalam bacaan mereka yang menunjukkan bahawa komposit itu tidak sesuai untuk aplikasi lenturan. Komposit SPF/VE diperkuat epoksi menunjukkan bacaan tertinggi untuk kekuatan lenturannya manakala komposit SPF/Epoxy diperkuat epoksi menunjukkan bacaan tertinggi untuk modulus lenturan bagi komposit gentian enau yang

dirawat. Komposit SPF/PE diperkuat epoksi menghasilkan bacaan terendah untuk kedua-dua ujian tegangan dan lenturan.

Kestabilan dimensi untuk komposit epoksi diperkuat dengan SPF yang diimpregnasi bertambah baik. Kedua-dua penyerapan air (WA) dan ketebalan bengkak (TS) daripada semua komposit gentian berkurangan. Komposit SPF/Epoxy diperkuat epoksi mempunyai pengurangan nilai yang paling tinggi untuk kedua-dua ujian. Di samping itu, ketumpatan dan graviti spesifik (SG) untuk SPF yang diperkuatkan epoksi juga menunjukkan penurunan dalam bacaan. Komposit SPF/VE diperkuat epoksi mempunyai pengurangan tertinggi diikuti oleh komposit SPF/PE diperkuat epoksi dan komposit SPF/Epoxy diperkuat epoksi.

Keputusan keseluruhan untuk kedua-dua bahagian gentian dan komposit menunjukkan bahawa VE adalah agen impregnasi yang terbaik manakala SPF/PE memberikan hasil yang paling rendah. Oleh itu dapat disimpulkan bahawa impregnasi SPF dengan VE akan menambah baik sifat komposit gentian enau.

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This thesis was 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 the Supervisory Committee were as follows:

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This is to confirm that:

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LIST OF ABBREVIATIONS

ASE	Anti-Swelling Efficiency
ASTM	American Society for Testing and Material
B.O.D.	Biochemical Oxygen Demand
BMC	Bulk Moulding Compounds
CB	Cassava Bagasse
CSIR	Council of Scientific Industrial Research
DMA	Dynamic Mechanical Analysis
DMDHEU	1,3-dimethylol-4,5-dihydroxyethyleneurea
DSC	Differential Scanning Calorimetry
D-VTKA	Desmodur-Vinyl Trie Ketonol Acetate
E’	Storage Modulus
E’’	Loss Modulus
EDX	Energy Dispersive X-Ray Spectroscopy
FESEM	Field Emission Scanning Electron Microscope
FRIM	Forest Research Institute of Malaysia
FT-IR	Fourier Transform Infrared
G	Glycerol
GA	Glutaraldehyde
GMA	Glycidyl Methacrylate
GS	Glycerol-Sorbitol
IFSS	Interfacial Shear Strength
IR	Infrared
ISO	International Organization for Standardization
LCM	Liquid Composite Moulding
LE	Linear Expansion
LMwPF	Low Molecular Weight Phenol Formaldehyde
MC	Moisture Content
MEE	Moisture Excluding Efficiency
MEKP	Methyl Ethyl Ketone Peroxide
MF	Melamine Formaldehyde
MOE	Modulus of Elasticity
MOR	Modulus of Rupture
MUF	Melamine Urea Formalehyde
NAL	National Aerospace Laboratories
OPT	Oil Palm Trunk
OPTL	Oil Palm Trunk Lumber
PAW	Pressure Application Window
PE	Polyester
PEEK	Polyether Ether Ketone
PF	Phenol Formaldehyde
PLA	Polylactic Acid
PP	Polypropylene
PS	Polystyrene
PVAc	Polyvinyl Acetate
PVC	Polyvinyl Chloride
RF	Resorcinol Formaldehyde
RTM	Resin Transfer Moulding

RW	Rubberwood
S	Sorbitol
SCRIMP	Seeman Composite Resin Infusion Moulding Process
SEM	Scanning Electron Microscope
SG	Specific Gravity
SPB	Sugar Palm Bunch
SPF	Sugar Palm Fibre
SPFREC	Sugar Palm Fibre Reinforced Epoxy Composite
SPFrond	Sugar Palm Frond
SPT	Sugar Palm Trunk
TAPPI	Technical Association of the Pulp and Paper Industry
T_g	Glass Transition
TGA	Thermogravimetric Analysis
T_m	Melting Point
TS	Thickness Swelling
UP	Unsaturated Polyester
UPM	Universiti Putra Malaysia
UTM	Universal Testing Machine
UV	Ultraviolet
VARI	Vacuum Assisted Resin Infusion
VARTM	Vacuum Assisted Resin Transfer Moulding
VE	Vinyl Ester
VERITy	Vacuum Enhance Resin Infusion Technology
VIP	Vacuum Infusion Process
WA	Water Absorption
WPG	Weight Percentage Gain

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Natural fibres are currently being used as a replacement for woody materials in the industry. Woody material takes a longer time to grow compared to natural fibre. It is also a substitute for the conventional synthetic fibre and petroleum-based product used in the industry which is expensive and harmful to the environment. Examples of synthetic fibres are glass fibre and carbon fibre The environmental pollution caused by these synthetic fibres has made scientist and developers turn to natural fibres which are more environmentally friendly [1].

Natural fibre composites are used in the area that requires strength not to be the most critical factor [2]. This is the reason why these fibres are likely to be commonly used in the near future. Some of the examples of natural fibres are kenaf, hemp, flax, coir, pineapple leaf, bamboo, oil palm, sugarcane bagasse, rice straw, banana, rice husk and sugar palm [3–6]. The availability, low cost, reduce health problem due to processing and handling, environmentally friendly, low density, renewability, less abrasive to equipment has made natural fibre an interesting field to be explored and use in the industry [7,8].

Natural fibres are hydrophilic while the polymers are hydrophobic [6,8–11]. The problem arises with interfacial bonding between the fibre and resin. The function of the resin in the natural fibre reinforced composite is to transfer the load to the stiff fibre via shear stress at the interface [5,8]. The process needs good interfacial bonding between fibre and matrix. Other disadvantages of natural fibres are the low modulus of elasticity, poor durability, high water absorption and weak fire resistance [2,12,13]. There are some ways to improve the compatibility between the matrix and fibre such as physical treatment and chemical treatment. These methods will be further discussed in chapter 2.

1.2 Problem Statements

Why is sugar palm fibre chosen? The capabilities of sugar palm fibre reinforced composite are yet to be explored deeply. There are still many things need to be discovered regarding the properties either mechanically, physically or chemically. Sugar palm trees are most likely to be cut down and disposed of as most people in Malaysia do not know the benefit of sugar palm tree. So far, all parts of sugar palm tree can be fully utilised just like how parts of the banana tree have a multifunction use. Sugar palm fibres have high durability and are resistance towards seawater [7]. This is proven by studies that show these fibres were used as ropes for ship cordages [14]. These fibres also do not require any secondary processing and are in a natural woven form [15].

The hydrophilic nature of sugar palm has made it prone to absorb moisture from surrounding and thus not suitable for outdoor application [15–18]. This leads to poor interfacial bonding and thus affects the mechanical properties of resulting composite [16]. Sugar palm fibres have to undergo physical treatment in order to improve the dimensional stability of fibre [17]. The physical treatment use in this study is by vacuum resin impregnation with thermosetting resins. Once cured, the resin will be embedded in the cell lumen and cell wall of fibre thus reducing hydrophilic sites for the bonding of hydroxyl group [19].

To date, there are only two studies done on the impregnation of sugar palm fibres namely by Ishak et al. [3] and Ibrahim et al. [20]. No recent studies have been done and the limitation of this has made the author refer to impregnation on other lignocellulosic materials.

In this study, physical treatment via vacuum resin impregnation is applied to the sugar palm fibre with three type of thermosetting resin which are epoxy, vinyl ester and polyester before the fibres are used for composite panel fabrication. The function of impregnation process is to fill/impregnate the cell lumen and cell wall of fibre as well as covering the surface of fibre with hydrophobic thermosetting resin. This will reduce and prevent moisture absorption of fibre as well as improving the properties of resulting composite.

The purpose of turning all impregnated fibres into a composite plate is to see which impregnated fibre has the best improvement. The best impregnated fibre may not be the highest result for composite panel.

1.3 Objectives of Study

The main objective of the research is to study the effect of resin impregnation on sugar palm fibre reinforced epoxy composites.

The specific objectives of the study are as follows:

1. To determine the effect of using different types of thermosetting resins (epoxy, vinyl ester, polyester) on the properties of sugar palm fibre done via vacuum impregnation.

2. To characterise the mechanical, physical properties and chemical composition of resin impregnated sugar palm fibre reinforced epoxy composites.

1.4 Scope of Study

Here, sugar palm tree at a flowering stage is chosen. Fibres from this tree have good properties as the tree is at its mature stage [21,22]. This study does not cover the detailed aspect of the sugar palm biological characteristics such as species, location, age or tree. The study focuses on the properties of resin impregnated sugar palm fibre reinforced epoxy composite. Three different thermosetting resins these being epoxy, vinyl ester and polyester are used as the impregnating agent on the sugar palm fibre. The properties determined are mechanical, physical and chemical.

Firstly, the control and resin impregnated sugar palm fibres are tested for its surface morphology and mechanical property. Next is the fabrication of composite using the resin impregnated sugar palm fibres using epoxy as the matrix. The impregnated fibres are in a natural woven formed. Hand layup process along with compression machine is used to fabricate composite panel with a thickness of about 3.2 mm. 30% fibre loading is set for the fabrication of composite as it is found to be the most optimum fibre loading for best results [10]. The properties investigated for the composite are tensile properties, flexural properties, SEM, FT-IR, water absorption, thickness swelling, density, specific gravity and chemical composition.

1.5 Significant Contribution to New Knowledge

The significance of this study is to establish data on vacuum resin impregnation on sugar palm fibres. A study done by the author found that there are limited data available on the topic of vacuum resin impregnation of sugar palm fibres. Another significant contribution is to develop a new biocomposites material that can be obtained from the sugar palm tree. Plenty of natural fibre composite products can be fabricated using natural fibres such as furniture, interiors of automobile, buildings and aerospace applications. The benefits of using these natural fibre composite products are that they are biodegradable, renewable, low cost and eco-friendly. The use of natural fibres will reduce the need and demand for synthetic materials. This physical treatment method may also improve the properties of sugar palm fibres to be used in the aerospace industry.

1.6 Thesis Layout

In Chapter 1, the background of the study, problem statement, objectives of research, scope and significance of study as well as thesis layout are presented. Chapter 2 discussed the literature review or relevant researchs. The chapter started with sugar palm tree, sugar palm fibres, sugar palm composites, impregnation of sugar palm, impregnation of lignocellulosic materials, different impregnation mediums and thermosetting resins. Chapter 3 presents the materials and methodology used in the study. This chapter describes the techniques of material preparation and standards used for mechanical and physical testings. Results and discussions are discussed in Chapter 4. The physical, mechanical, FT-IR and surface morphology of sugar palm fibre and its impregnated composites are further evaluated in this chapter. Chapter 5 presents the conclusions and recommendation for future studies.

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LIST OF PUBLICATIONS

Chapter in Book

Munawar, N. S. Z., Ishak, M. R., Jawaid, M., Zuhri, M. Y. M. 2018. A Review on the Impregnation of Sugar Palm Fiber and Other Lignocellulosic Materials. In Sugar Palm Fibre and Composites (1 – 54). CRC Press. (Published)

Journals

Munawar, N. S. Z., Ishak, M. R., Jawaid, M., Zuhri, M. Y. M. 2018. Modification of Single Sugar Palm Fibre via Vacuum Resin Impregnation. BioResources; 1 – 8. (Submitted)

Munawar, N. S. Z., Ishak, M. R., Jawaid, M., Zuhri, M. Y. M. 2018. Tensile and Flexural Properties of Impregnated Sugar Palm Fibre Reinforced Epoxy Composite. Journal of Reinforced Plastics and Composites; 1-9. (In Progress)

Munawar, N. S. Z., Ishak, M. R., Jawaid, M., Zuhri, M. Y. M. 2018. Physical Properties and FT-IR of Impregnated Sugar Palm Fibre Reinforced Epoxy Composite. Journal of Natural Fibers; 1-7. (In Progress)

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