UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF SUGAR PALM YARN AND WOVEN GLASS FIBRE-REINFORCED UNSATURATED POLYESTER COMPOSITES

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By

MOHD NURAZZI BIN NORIZAN

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Chairman : Associate Professor Khalina Abdan, PhD
Faculty : Engineering

This research was carried out to produce a composite material from sugar palm (*Arengga Pinnata*) yarn fibre as fibre reinforcement onto unsaturated polyester composites. Analysis of the curing characteristic of unsaturated polyester resin, the structural and morphological characteristics of sugar palm fibre, tensile strength of single sugar palm fibre and interfacial shear stress (IFSS) were studied. The gel time was decreased by up to 36% when 0.05% cobalt solution was used. Alkaline treatment using sodium hydroxide (NaOH) led to the enhancement of the single fibre tensile strength and the IFSS due to the internal morphological changes of the sugar palm fibres. The physical properties (water absorption, density and void content determination), mechanical properties (tensile, flexural, impact and compression), thermal (DMA and TGA) and morphological (SEM) properties of different sugar palm yarn fibre loadings from 10 to 50 wt.% composites was studied, followed by different orientations of fibres (45° and 90°) reinforced composites. From the experiment results, the percentage of water absorption increased as the sugar palm yarn fibre loading increased and reached the equilibrium of absorption on day 12. Increasing trends in the performance of tensile strength and flexural properties were shown for the yarn fibre loadings of up to 30 wt.%. However, a maximum impact strength and compression properties were achieved at 40 wt.% fibre loading. The thermal stability of the composites decreased as the fibre loading increased. The composites with 30 wt.% of fibre loading and 0° fibre orientation exhibited maximum tensile and flexural properties, respectively. While maximum impact and compression properties found at 40 wt.%, respectively. The experiment found the effect of treated sugar palm yarn fibre loading reinforced unsaturated polyester composites with 1% alkaline solutions decreased the physical, mechanical and thermal properties compared to the composites reinforced with untreated sugar palm yarn fibre. This is due to different interaction of single fibre and yarn fibre which affects the interfacial adhesion between the fibre with the matrix. The final 30 wt.% and 40 wt.% of fibres as reinforcement were chosen for

the hybridisation studies. Then, the ratio of reinforcement between the sugar palm yarn fibre and woven glass fibre was selected at 70/30, 60/40 and 50/50 wt.%, respectively, which equal to the volume of the mould. The physical, mechanical and thermal properties of the treated sugar palm yarn fibre hybrid composites were found to increase as the glass fibre loading increased up to 50/50 wt.% of fibre ratio for both 30 wt.% and 40 wt.% of fibre loadings. This is due to better interfacial bonding and better chemical interaction between the sugar palm fibre, glass fibre and unsaturated polyester resin as matrix. The improvement on the mechanical and thermal properties also contributed by the synergistic effect from the addition of glass fibre loadings. Hence, the study findings will pave the way towards a greater usage of sugar yarn fibre as a reinforcement for the structural applications such as anti-roll bar in automotive industry, bicycle frame and furniture.

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Oleh

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Penyelidikan ini dijalankan untuk menghasilkan bahan komposit daripada gentian serabut enau (*Arengga Pinnata*) sebagai tetulang gentian kepada komposit poliester tak tepu. Analisis ciri-ciri pengerasan resin poliester tak tepu, ciri-ciri struktur dan morfologi gentian enau, kekuatan tegangan serat enau, dan tegasan ricih antara muka (IFSS) telah dikaji. Tempoh pengegelan telah berkurang sehingga 36% apabila larutan kobalt 0.05% digunakan. Rawatan alkali dengan menggunakan natrium hidroksida (NaOH) telah membawa kepada peningkatan kekuatan tegangan serat dan IFSS yang disebabkan oleh perubahan morfologi dalaman gentian enau. Sifat-sifat fizikal (penyerapan air, ketumpatan, penentuan kandungan lowong), sifat-sifat mekanikal (kekuatan tegangan, lenturan, hentaman dan mampatan), sifat-sifat terma (DMA dan TGA) dan sifat-sifat morfologi (SEM) pada gentian serabut enau yang berbeza muatan daripada 10 hingga 50 peratusan berat (% berat) komposit telah dikaji, diikuti dengan orientasi komposit bertetulang gentian yang berbeza (45° dan 90°). Daripada hasil eksperimen, peratusan penyerapan air meningkat apabila muatan gentian serabut enau meningkat dan mencapai keseimbangan penyerapan pada hari ke-12. Peningkatan trend dalam prestasi kekuatan tegangan dan sifat lenturan telah dicerap bagi muatan gentian serabut sehingga 30 % berat. Namun, kekuatan hentaman dan mampatan yang maksimum telah dicapai pada muatan gentian 40 % berat. Kestabilan terma komposit telah berkurang apabila muatan gentian meningkat. Komposit dengan muatan gentian sebanyak 30 % berat dan orientasi gentian 0° telah menunjukkan sifat tegangan dan lenturan yang maksimum. Manakala kekuatan hentaman dan mampatan yang maksimum didapati berada pada 40 % berat. Eksperimen mendapati kesan rawatan komposit poliester tak tepu bertetulang gentian muatan serabut enau dengan 1% larutan beralkali mengurangkan sifat-sifat fizikal, mekanikal dan terma berbanding komposit yang bertetulang gentian serabut enau tak terawat. Ini adalah disebabkan perbezaan interaksi oleh gentian tunggal dan tetulang gentian dimana memberi kesan terhadap tegasan ricih antara muka di antara gentian dan matriks. Peratusan berat yang terakhir sebanyak 30 % berat dan 40 % berat telah dipilih untuk kajian penghibridan. Nisbah tetulang antara gentian enau dan gentian kaca tenunan yang dipilih adalah 70/30, 60/40 dan 50/50 % berat, yang bersamaan dengan isipadu acuan pembentuk. Sifat-sifat fizikal, mekanikal dan terma komposit hibrid gentian enau terawat telah didapati meningkat apabila muatan gentian kaca ditingkatkan sehingga 50/50 % berat daripada nisbah gentian untuk kedua-dua muatan gentian sebanyak 30 % berat dan 40 % berat. Ini disebabkan pengikatan antara muka dan saling tindak balas kimia yang lebih baik antara gentian enau, gentian kaca, dan resin poliester tak tepu yang bertindak sebagai matriks. Penambahbaikan sifat-sifat mekanikal dan terma juga disumbangkan oleh kesan bersinergi daripada penambahan muatan gentian kaca. Oleh itu, hasil kajian akan dapat membuka jalan ke arah penggunaan gentian enau yang lebih meluas sebagai tetulang untuk aplikasi struktur sebagai contoh kit bar antigelong didalam industri automotif, kerangka basikal dan juga perabot.

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“Bring me blocks of iron.” At length, when he had filled up the space between the two steep mountain-sides, He said, “Blow (with your bellows)” Then, when he had made it (red) as fire, he said; “Bring me, that I may pour over it, molten lead. Thus were they made powerless to scale it or to dig through it.” (AlKahf:18:96-97)

This thesis dedicated for my mother, Mariyam,
For your patience and spirit.

My beloved wife, Nadzirah,
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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
CBS	Cassava bagasse
CO_2	Carbon dioxide
CS	Cassava starch
DMA	Dynamic mechanical analysis
DSC	Differential scanning calorimetry
DTG	Derivative thermogravimetric
E*	Complex modulus
E'	Storage modulus
E"	Loss modulus
EU	European union
FRP	Fibre reinforced polymer
FTIR	Fourier transform infrared
H_2O	Water
IR	Infrared
IFSS	Interfacial shear stress
kBr	Potassium bromide
LDPE	Low density polyethylene
MEKP	Methyl ethyl ketone peroxide
NaOH	Sodium hydroxide
NFC	Natural fibre reinforced composites
OH	Hydroxyl group
PE	Polyethylene

pc	Piece
POM	Polarize optical microscope
PP	Polypropylene
PS	Polystyrene
PU	Polyurethane
PVC	Polyvinylchloride
RTM	Resin transfer moulding
SEM	Scanning electron microscopy
SPF	Sugar palm fibre
Tan δ	Tan delta
TDI	Toluene diisocyanate
Tg	Glass transition temperature
TGA	Thermogravimetric analysis
vol	Volume
vol.%	Volume percent
wt.%	Weight percent
WVP	Water vapor permeability

LIST OF UNITS

°C	Degree celcius
°C/min	Degree celcius per minute
°	Degree
GPa	Gigapascal
gsm	gram square meter
tex	gram per 1000 meters
g/cm^3	gram per cubic centimeter
J/g	Joule per gram
J/m	Joule per meter
kJ	kilo Joule
kN	kilo Newtons
Kg/m^3	Kilogram per cubic meter
kJ/m^2	kilojoule per square meter
MPa	Megapascal
$MPa\sqrt{m}$	Megapascal square root meter
ms^{-1}	meter per second
µm	micrometer
mm	millimeter
ml/min	milliliter per minute
min	minute
M	Molar
%	Percentage
cm^{-1}	per centimeter
rpm	rotation per minute

CHAPTER 1

INTRODUCTION

1.1 Overview

A composite is the combination of two or more elements in any form and for a variety of uses. The concept of composite materials is based on combining different materials for a new material with superior performance beyond that exhibited by their individual constituents. As a result, their high strength-to-weight and high stiffness-to-weight ratios can be tailored towards specific applications. Applications for composite materials have grown steadily throughout the years, penetrating and conquering newer markets.

Modern established composite materials constitute a significant proportion of these engineered material markets, ranging from daily products to sophisticated niche applications. These advanced materials see use in construction, military, automotive and aerospace industries. Their technologies are especially attractive due to their advantages over legacy materials such as metal, as composites offer high specific strength, low density, light weight, enhanced corrosion and temperature capability. An added advantage would be their biodegradability (Ishak *et al*., 2013; Nair *et al*., 1996).

In recent years, natural fibres have attracted interest for the reinforcement of polymer composites in low-cost engineering materials. The use of natural fibres as a potential replacement for synthetic fibres such as glass and carbon fibres in composites materials has intensified research recently due to environmental and economic factors. The primary advantages of natural fibres over synthetic fibres include abundance and relatively low cost, low mass and specific density, high specific strength and renewability and biodegradability (Mohanty *et al*., 2002). In the meantime, the present use of the term 'biodegradable' in natural fibre composites actually refers to the utilization of natural sources in the polymer industry, which could reduce dependence on petroleum resources and decrease industrial carbon dioxide (CO_2) emissions (Sahari *et al*., 2013a).

Natural fibres, when compared to synthetic fibres, generally have lower mechanical properties. These low mechanical properties are a major inhibitory when trying to develop high performance products. A few methods for increasing mechanical performance is to modify the interfacial adhesion, chemical structure of natural fibres and hybridise the natural fibres with synthetic fibres. One benefit of hybridisation is that the advantage of one type of fibre can overcome the disadvantages of the other one type of fibre.

A viable compromise between the higher material properties of synthetic fibres and the environmental benefits of natural fibres is found by utilizing both synthetic and natural fibres to create a hybrid fibre reinforced composite system. Material properties are also improved by the efficient arrangement of the structural numbers. The use of hybrid natural and synthetic fibre reinforced composites for structural applications has been shown to be feasible alternative to traditional synthetic structural materials. (Musch, 2008). As a result, a balance in cost, performance and sustainability may be reached through a proper composite material design.

Natural fibre composites are essentially a plant fibre embedded within a thermoplastic or thermoset polymer. The density of these natural fibres is similar as their plastic counterparts, which are usually 40 to 50% lower than the density of glass fibre (Rajan and Curtin, 2015; Saba *et al*., 2014). Therefore, polymeric materials could be reinforced or filled without having significant effects on their density. Several types of natural fibres, such as kenaf fibre, oil palm fibre, sugar palm fibre, pineapple leaf fibre, banana fibre, flax, hemp, sisal, coir and jute fibre, have attracted the attention of scientists and technologists for their widespread applications.

These fibres are used to reinforce thermoplastic polymer matrices such as polystyrene (PS), polypropylene (PP), polyethylene (PE), polyvinyl chloride (PVC), polyurethane (PU) and so on. Phenolic, unsaturated polyester, vinyl ester and epoxy resin are for thermosetting polymer matrices.

1.2 Problem statement

Unsaturated polyester resins are an important class of high-performance engineering polymers used in numerous structural applications, primarily compression moulding (sheet moulding compounds), injection moulding (bulk moulding compounds), resin transfer moulding (RTM), pultrusion, filament winding and hand lay-up process (Vilas *et al*., 2001). To produce unsaturated polyester composite product, the determination of gel and curing time is a very important stage in the processing of unsaturated polyester resins, as in order to achieve a good quality of product, the curing reaction should occur in a controllable way (Jansen and Kraeger 2018). This is especially vital when using RTM or another complex shape is to be produced, as resin must be in working fluidic flow and unwanted fast cure must be observed in the equipment. Other than that, it is also important to take precautionary steps, especially in the estimation of mixing time, from the start of the initial mix until the resin is fully injected into the mould. These crucial steps include determining the right formulation mix of initiator and accelerator required to optimize the time needed before the resin starts to gel and harden within the required time.

Furthermore, since the structural performance of natural fibre reinforced composites is dependent on the performance of fibres itself to maximize the efficiencies of stress transfer mechanism from the matrix to the fibres. The challenge of raw sugar palm fibres is that it is randomly wrapped along with palm leaf ribs. If directly used, this may affect interaction with the chain of the matrix of composites. The tangling nature of sugar palm fibre itself becoming harder to be ignored. When the long fibres are in a tangling form, the possibility of the fibres to become randomly oriented is high especially after the infusion of resin into the mould and during the lay-up process. According to Miao and Shan (2011), short and randomly oriented fibre reinforced composite structures possess much lower mechanical properties than those made with oriented structures. Therefore, they are not suitable for use in structural applications in which mechanical performance is of primary importance.

Composite materials using natural fibres which are environmentally friendly, biodegradable, reasonably strong, lightweight, hazard-free and abundant have potential for use in reinforcement materials. Despite the advantages listed, the main limitation of natural fibres, their hydrophilicity properties, potentially leads to deterioration of the physical, mechanical and thermal properties of the composites due to worst in the interfacial adhesion of natural fibres with the hydrophobic nature of synthetic fibres and matrix. Hydrophilic properties are a major drawback in terms of incompatibility between synthetic fibres and polymer matrices, which are hydrophobic in nature. Incompatibility will substantially affect the efficiency of applied stress between matrix and fibres.

Synthetic fibres like glass, aramid and carbon fibre are widely used as reinforcement fibres in composites, which have been proven to be a good mechanical strength enhancer. The desired tensile strengths and modulus of glass fibres are visibly much higher than natural fibres. The disadvantages of synthetic fibre are that without proper handling, it may cause skin irritation, causing it to be dangerous to human health, and its lack of biodegradability (Van de Velde and Kiekens, 2001). However, differences in the characteristics of glass and natural fibres are important when their applications and costs are taken into account (Bledzki and Gassan, 1999). Synthetic and natural fibres have been used in reinforcing polymers in such a way that fibres with a promising strength and matrix will convey the applied stress to the fibre structures. This combination is called hybridisation of composite materials, which refers to two materials with different properties being combined to produce new materials with novel properties.

1.3 Significance of Study

1- Development of novel composites from sugar palm yarn fibre.
2- A novel study on the interaction of untreated and treated sugar palm yarn fibre reinforced unsaturated polyester composites.
3- A novel study on the interaction of the hybridisation mechanism involved with sugar palm yarn fibre with woven glass fibre reinforced unsaturated polyester composites.

1.4 Objectives

The specific objectives of this research are:

1- To determine the curing characteristic of unsaturated polyester resin, chemical compositions and structural changes of untreated and treated sugar palm fibre, single fibre tensile and interfacial shear stress properties of untreated and treated single sugar palm fibre properties.
2- To determine the effects of sugar palm yarn fibre loading reinforced unsaturated polyester composites on its physical, mechanical, thermal, and morphological properties with different fibre orientations (0°, 45° and 90°).
3- To determine the effects of alkaline treatment of sugar palm yarn fibre reinforced unsaturated polyester composites on its physical, mechanical, thermal, and morphological properties.
4- To determine the effects of hybridisation of sugar palm yarn/woven glass fibres reinforced unsaturated polyester composites on its physical, mechanical, thermal, and morphological properties.

1.5 Scope and limitations

The fibre used is sugar palm fibre purchased from Hafiz Adha Enterprise at Kampung Kuala Jempol, Negeri Sembilan, and the thermosetting polymer used is unsaturated polyester resin purchased from CCP Composites Resins Malaysia Sdn. Bhd. The sugar palm fibre was yarned using a manual hand spinning machine with 2500 tex of twisting. The limitation of the machine on its capability to produce other than 2500 tex because this machine was design specifically for 2500 tex due to physical characteristic of sugar palm fibre that has spike and very coarse to be twisted in lower tex. Unsaturated polyester was reinforced with sugar palm yarn fibre with various fibre loadings. Different fibre loadings that were prepared are 10 wt.%, 20 wt.%, 30 wt.%, 40 wt.% and 50 wt.%. Sugar palm yarn fibre at 2500 tex needs better penetration of matrix in composite systems. Hence in this study was limited on using low viscosity of unsaturated polyester resin due to its ability to penetrate yarn's internal structure.

1.6 Thesis outline

This thesis is structured into five chapters. The first chapter contains an overview of natural and synthetic fibres composites, the significance of research, highlights of the research problems, and finally the objectives, scope, and limitations of the research. Chapter two is an overview of the literature on natural fibres reinforced composites with focusing on sugar palm composites. Chapter three presents the overall research methodology for the overall structure of the research work. This chapter describes the materials, a specific approach in designing and planning the experimental design, experimental test procedures and standards. The following chapter four presents the results and discussion of the research works. Chapter four is divided into four sections, covering the analysis on the properties of unsaturated polyester resin and sugar palm fibre, the effects of untreated sugar palm yarn fibre reinforced unsaturated polyester composites, the effects of fibre orientation, the effects of alkaline treatment to the fibre reinforced composites, and lastly the effects of hybridisation of sugar palm yarn fibre and woven glass fibre reinforced unsaturated polyester composites on the physical, mechanical, thermal and morphological properties. Finally, chapter five presents a summary of the research findings and recommendations for future works.

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