UNIVERSITI PUTRA MALAYSIA

EFFECTS OF FLAME-RETARDANT AGENTS ON MECHANICAL PROPERTIES AND FLAMMABILITY OF IMPREGNATED SUGAR PALM FIBRE-REINFORCED POLYMER COMPOSITES

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By

ABU HATIM BIN IBRAHIM

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

October 2013

DEDICATION

For all your advice and encouragement, this thesis is gratefully dedicated to:

My Beloved Father and Mother

Hj. Ibrahim Bin Ahmad

Hjh. Roshayati Binti Mohd. Said

and

My Family

Thank you very much for your continuous support and effort towards the publication of this thesis.

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

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October 2013

Chairman : Associate Professor Zulkiflle Leman, PhD

Faculty : Engineering

This research evaluated the effects of flame-retardant agents on mechanical properties and flammability of impregnated sugar palm fibre-reinforced polymer composites. The study was divided into three stages, where the first stage focused on the characterisation of tensile properties of sugar palm fibre (SPF) impregnated with thermosetting resins, such as unsaturated polyester, vinylester and epoxy resins were characterised. The results showed that the impregnation of thermosetting resins, which involved diffusing the resins into the lumen cells of sugar palm fibre (SPF), increased the stiffness. The tensile strengths of vinylester- (VE-)- impregnated, unsaturated polyester- (UP-)-impregnated and epoxy-impregnated fibres significantly increased to 158.31 MPa, 167.53 MPa and 188.06 MPa, respectively, compared with the tensile strength of the control specimen, 107.12 MPa. The maximum tensile modulus of SPF impregnated with epoxy, UP and VE were 21%, 12.7% and 8.4% compared with non-impregnated SPF.

The second stage of the study focussed on interfacial shear strength (IFSS) and the effects of embedded lengths of SPF reinforced with UP, VE and epoxy polymers by

single fibre pull-out test (SFPT). The test samples were fabricated by inserting a fibre into a mixture of UP-impregnated fibre/UP, VE-impregnated fibre/VE and epoxy-impregnated fibre/epoxy. SFPT were conducted to examine the effect of embedded length on the IFSS of the fibre in the thermosetting matrix. The embedded length for the optimum IFSS of UP/UP was 4 mm at 2.67 MPa. The embedded length for the optimum IFSS for VE/VE was 5 mm at 2.46 MPa. The embedded length for the optimum IFSS of epoxy/epoxy was 3 mm at 3.25 MPa. The results showed that the IFSS gradually decreased as the embedded length increased. It was concluded that the embedded length for the optimum IFSS of the thermosetting matrix was 3 mm.

The third stage of the study involved an investigation of the effect of impregnated SPF reinforced composites filled with aluminium trihydroxide (ATH) and magnesium hydroxide (MH) which served as flame retardant fillers. The SPF composites were impregnated with UP resin. The study investigated the effects of flame retardant fillers (FRF) with a loading range of 10% - 50%. The results showed that the tensile strength, tensile modulus and elongation at break of the composites filled with ATH were significantly higher than the tensile strength, tensile modulus and elongation at break of the composites filled with MH. The tensile strength decreased from 6.87 MPa to values ranging from 3.72 - 5.84 MPa and 3.16- 5.01 MPa for ATH- impregnated sugar palm fibre composites (ISPFC) and MH-ISPFC, respectively. The tensile modulus decreased from 1843.23 MPa to values ranging from 1179.62 - 1816.8 MPa and 1063.71 – 1522.99 MPa for ATH-ISPFC and MH-ISPFC, respectively. The elongation at break decreased from 8.17% to values ranging from 5.15 - 7.11 % and 3.61-7.41% for ATH-ISPFC and MH-ISPFC, respectively. The impact strength also decreased from 1.96 kJ/m^2 to values ranging from 1.03 - 1.91 kJ/m^2 and 0.97 - 1.54 kJ/m^2 for ATH-ISPFC and MH-ISPFC, respectively.

The fire propagation performance of core particle board of ISPFC filled with ATH and MH fillers were also investigated. The fire propagation test was evaluated using a performance index (I), which indicated the heat released from the tested particle boards. The result showed that, Both ATH-ISPFC and MH-ISPFC boards reduced the average performance index from 35.5 to values ranging from 22.0 - 25.6 and 21.5 – 31.5 for ATH-ISPFC and MH-ISPFC, respectively. In general, ATH-ISPFC exhibited better fire retardant performances for FRF loadings in range of 10wt%-40wt% FRF loadings but the fire performance for 50wt% FRF loading revealed that MH exhibits the optimal fire retardant performance. An increase in flame retardant fillers resulted in a negative effect on the mechanical properties of composite materials but yielded better fire retardant performances of the composites.

Abstrak tesis yang di kemukan kepada Senat Univeriti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KESAN AGEN TAHAN-API TERHADAP SIFAT MEKANIKAL DAN SIFAT KEMUDAHBAKARAN TERHADAP KOMPOSIT POLIMER DEPERKUATAN-GENTIAN IJUK YANG DIIMPREGNASI

Oleh

ABU HATIM BIN IBRAHIM

Oktober 2013

Pengerusi : Professor Madya Zulkiflle Leman, PhD

Fakulti : Kejuruteraan

Kajian ini di jalan untuk mengkaji kesan agen tahan-api terhadap sifat mekanikal dan sifat kemudahbakaran terhadap komposit polimer diperkuatan-gentian ijuk yang diimpregnasi. Kajian ini dibahagikan kepada tiga peringkat , di mana peringkat pertama tertumpu kepada pencirian sifat tegangan gentian ijuk (SPF) dimpregnasi dengan resin termoset, seperti poliester tak tepu, vinylester dan resin epoksi. Hasil kajian menunjukkan bahawa impregnasi resin termoset , yang terlibat meresap resin ke dalam sel lumen gentian ijuk (SPF), meningkat kekukuhan. Kekuatan tegangan vinylester -(VE-)–berimpregnasi , poliester tidak tepu -(UP -)–berimpregnasi dan epoksi –berimpregnasi meningkat dengan ketara kepada 158,31 MPa, 167,53 dan 188,06 MPa, masing-masing , berbanding dengan kekuatan tegangan spesimen kawalan , 107,12 MPa. Modulusi tegangan maksimum SPF diimpregnasi dengan epoksi, UP dan VE adalah 21%, 12.7% dan 8.4 % berbanding dengan bukan SPF diimpregnasi.

Peringkat kedua kajian ini memberi tumpuan kepada kekuatan ricih antara muka (IFSS) dan kesan panjang tertanam SPF diperkukuhkan dengan UP, VE dan epoksi

polimer oleh ujian tarik keluar gentian tunggal (SFPT). Sampel ujian telah dibuat dengan memasukkan serat ke dalam campuran UP-gentian impregnasi / UP, VE – gentian impregnasi / VE dan epoxy - gentian impregnasi / epoksi. SFPT telah dijalankan untuk mengkaji kesan panjang tertanam di IFSS gentian dalam matrik termoset. Panjang optimum tertanam untuk IFSS UP / UP adalah 4 mm pada 2.67 MPa. Panjang optimum tertanam untuk IFSS untuk VE / VE adalah 5 mm pada 2.46 MPa. Panjang optimum tertanam untuk IFSS epoksi / epoksi adalah 3 mm pada 3.25 MPa. Hasil kajian menunjukkan bahawa IFSS secara beransur-ansur menurun apabila panjang terbenam meningkat. Kesimpulan telah dibuat bahawa panjang tertanam untuk IFSS optimum matrik termoset adalah 3 mm.

Peringkat ketiga kajian ini melibatkan penyiasatan kesan aluminium trihydroxide (ATH) dan magnesium hidroksida (MH) yang berkhidmat sebagai pengisi kalis api bertetulang komposit SPF berimpregnasi. Komposit SPF telah diimpregnasi dengan resin UP. Kajian ini disiasat mengenai kesan pengisi kalis api (FRF) dengan pelbagai nisbah muatan sebanyak 10% - 50%. Hasil kajian menunjukkan bahawa kekuatan tegangan , modulus tegangan dan pemajangan pada takat putus bagi komposit dipenuhi dengan ATH adalah jauh lebih tinggi daripada kekuatan tegangan , modulus tegangan dan pemajangan pada takat putus bagi komposit dipenuhi dengan MH. Kekuatan tegangan berkurangan daripada 6.87 MPa kepada nilai-nilai antara 3,72-5,84 MPa dan 3,16-5,01 MPa untuk ATH –komposit ijuk diimpregnasi (ISPFC) dan MH- ISPFC. Modulus tegangan menurun dari 1843,23 MPa kepada nilai-nilai antara 1179,62-1816,8 MPa dan 1063,71-1.522,99 MPa untuk ATH - ISPFC dan MH- ISPFC. Pemajangan pada takat putus menurun daripada 8.17% kepada nilai-nilai antara 5,15-7,11 % dan 3,61-7,41 % untuk ATH - ISPFC dan MH- ISPFC. Impak kekuatan juga menurun daripada 1.96 kJ/m2 kepada nilai-nilai antara 1,03-1,91 kJ/m2 dan 0,97-1,54 kJ/m2 untuk ATH - ISPFC dan MH- ISPFC.

Prestasi penyebaran api papan partikel teras ISPFC dipenuhi dengan pengisi ATH dan MH juga dikaji. Ujian penyebaran api telah dinilai dengan menggunakan indeks prestasi (I), yang menunjukkan haba yang dikeluarkan dari papan partikel diuji. Hasil menunjukkan bahawa, Kedua-dua ATH - ISPFC dan papan MH- ISPFC mengurangkan indeks prestasi purata daripada 35.5 kepada nilai-nilai antara 22,0-25,6 dan 21,5-31,5 untuk ATH- ISPFC dan MH- ISPFC. Secara umum, ATH - ISPFC menunjukkan persembahan kalis api lebih baik untuk nisbah FRF dalam bebanan 10wt % - 40wt % FRF tetapi prestasi api untuk bebanan 50wt % FRF mendedahkan bahawa MH mempamerkan prestasi kalis api yang optimum. Peningkatan dalam pengisi kalis api menyebabkan kesan negatif ke atas sifat-sifat mekanikal ba

han komposit tetapi memberikan persembahan kalis api lebih baik terhadap komposit.

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I certify that a Thesis Examination Committee has met on 3th October 2013 to conduct the final examination of Abu Hatim Bin Ibrahim on his thesis entitled " Effects of Flame-Retardant Agents on Mechanical Properties and Flammability of Impregnated Sugar Palm Fibre-Reinforced Polymer Composites" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. 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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DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institutions.

ABU HATIM BIN IBRAHIM

Date: 3 October 2013

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LIST OF ABBREVIATIONS

API	Average performance index
ASTM	American Society for Testing and Materials
ATH	Aluminium trihydroxide
ATH- ISPFC	Impregnated sugar palm fibre composites filled with aluminium trihydroxide
BS	British Standard
FE	Front end
FRF	Flame retardant filler
FRIM	Forest Research Institute Malaysia
FR-ISPFC	Impregnated sugar palm fibre composite filled with flame retardant filler
FRP	Fibre reinforced polymer
IFSS	Interfacial shear strength
ISPFC	Impregnated sugar palm fibre composites
ISPFRTP	Impregnated sugar palm fibre reinforced thermosetting polymer
ISPFRTPC	impregnated sugar palm fibre reinforced thermosetting polymer composites
JIS	Japanese Industrial Standard

MEKP	Methyl ethyl ketone peroxide
MH	Magnesium hydroxide
MH-ISPFC	Impregnated sugar palm fibre composites filled magnesium hydroxide
OH	Hydroxyl
PE	Unsaturated polyester
SEM	Scanning electron microscope
SFPT	Single fibre pull-out test
SPB	Sugar palm bunch
SPF	Sugar palm fibre
SPT	Sugar palm trunk
UP	Unsaturated polyester
UTM	Universal testing machine
VE	Vinylester

LIST OF SYMBOLS

α	alpha
%wt	Weight loading
ρ	Density (g/cm^3)
ε	Strain (%)
σ	Stress (MPa)
A	Cross section area (mm^2)
b	Width (mm)
d	Thickness (mm)
E	Young's modulus (MPa)
F	Force (kN)
L	Length (mm)
P	Load (N)
S_τ	Tensile strength (MPa)
W	Weight (g)

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Increasing global environmental concerns and new environmental regulations have initiated the development of new materials that enhance the optical utilisation of natural resources and renewable resources. Natural fibres such as coir, sisal, jute and sugar palm (*Arenga pinnata*), are renewable and low cost natural resources. Natural fibres are potential substitutes to synthetic fibres due to significant advantages. These composites materials are applicable in the aerospace, leisure, construction, sport, packaging and automotive industries. One significant advantage of plant fibres is their optimised strength to weight ratios. Other advantages include their enhanced workability due to an optimum fibre length and cell wall thickness, their high anisotropic qualities and their superior ion exchange capacity. Natural products are also readily biodegradable and renewable.

Natural fibre reinforced composites comprise a new area in polymer science. As an alternative reinforcement in fibre reinforced polymer (FRF) composites, natural fibres have recently become appealing to scientists, engineers and researchers. Due to their fair mechanical properties, low cost, high specific strength, eco-friendliness, and non-abrasiveness and bio-degradability, they are exploited as a replacement for conventional fibres, such as carbon, aramid and glass. The tensile properties of natural fibre reinforced polymers (thermoplastics and thermosets) are primarily influenced by the interfacial adhesion between the matrix and the fibres. Several chemical modifications are employed to improve the interfacial matrix fibre bonding, which enhances tensile properties of the composites. (Ku et al., 2011).

Malaysia is an agricultural country with abundant sources of agriculture crop waste such as banana oil palm, pineapple leaf, straw rice and sugar cane; the processing of these sources is challenging. The application of this abundant resource as a reinforcement in polymer composites is the best method for addressing this problem. This alternative can also contribute to economic improvements in the country.

However, a significant drawback of natural fibre reinforced composites is the poor compatibility of the fibre with the matrix due to hydrophobic materials and the hydrophilic characteristics of cellulose. Physical and chemical modifications of natural fibres are commonly performed to correct deficiencies in the materials, especially to improve the wettability and adhesion and bonding properties. Another treatment, which involves surface modification of the natural fibres, can be applied to optimise the properties interface and modify the hydrophilic and hydrophobic properties. (Leman, 2009).

1.2 Problem Statements

Numerous types of natural fibres are grown in Malaysia. One type of fibre is sugar palm fibre which has multiple purposes and benefits. Sugar palm exhibits high tensile strength and a long life prior to degradation (Tomlinson, 1962); thus it is a suitable material for the fabrication of ropes. Another advantage of sugar palm is that it does not require a secondary process for yielding, such as water retting. Traditionally, ropes were used for ship cordages due to their superior properties in wet environments (Bachtiar, 2008; Leman et al., 2008b). Due to advantages such as resistance to sea water and high durability. Sugar palm fibre has become at highly potential candidate as a substitute for glass fibre in the marine industry.

Numerous studies of the properties of sugar palm fibre reinforced polymer composites have been conducted due to the various advantages of this plant. However, minimal research on the enhancement of the properties of sugar palm fibre after exposure to wet environments is available. Water will react with lignocellulose materials; natural fibres are hygroscopic materials as they contain hydroxyls group in the cell walls of the fibres (Hill, 2006). When natural fibre composites are exposed to high humidity environments, they absorb moisture until they achieve equilibrium with the relative humidity of the environment. However, the mechanical properties of the composites significantly decrease once the fibres begin to absorb moisture (John and Thomas, 2008; Mishra et al., 2001; Chen et al., 2009).

The development of fire retardant materials for environmental and health safety is crucial. Attention has become focused on inorganic compounds, such as metallic hydroxide additives, which provide effective flame retarding effects (Pearce et al., 1981; Hornsby et al.,1989). Flame retardant fillers have been used for hundreds of years to reduce the flammability of combustible materials; they were initially used in timber and clothing fabric and have recently been applied to polymers and polymer composites.

Many flame retardant fillers decrease the mechanical properties of polymers, which is concern regarding their use in structural composites. Although they decrease the flammability of materials, some filler materials increase the amounts of smoke and toxic fumes released by the decomposing materials. The adverse effects of fillers can be mitigated by surface treating the particles to promote a chemical interaction with the polymer matrix. Therefore, this research was conducted to investigate the incorporation of flame retardants in polymer composite, which are commonly used to minimise adverse effects on the mechanical properties, toxicity and production of smoke and maximise the resistance to flammability (Mouritz, 2006).

1.3 Research Objectives

The aim of this study is to determine the mechanical properties and flammability behaviour of impregnated sugar palm fibre reinforced thermosetting polymer composites (ISPFRTPC).

The specific objectives of this research are as follows:

1. To determine the tensile properties and interfacial shear strength (IFSS) of the sugar palm fibre impregnated with thermosetting polymer (UP, VE and epoxy).
2. To determine the mechanical properties of ISPFRTPC with flame retardant fillers. (ATH and MH)
3. To determine the flammability behaviour of ISPFRTPC with flame retardant fillers. (ATH and MH)

Significance of Study

The significance of this study is to develop a new resource of natural fibre, which can be extracted from the sugar palm tree in abundance. A variety of biocomposite products can be constructed using natural fibre resources, such as interior parts of automobiles, building and structural materials, and furniture. These biocomposite product are renewable, low cost, partially biodegradable and environmentally friendly. The use of natural fibres will reduce the demand for timber, which is currently encountering deforestation problems. This development may also mitigate the problem of handling residue from the sugar palm tree. In terms of fire safety regulations, applications of the sugar palm tree can be adopted into building materials to eliminate or reduce the need for synthetic materials in the construction of ceiling, roofing and ceiling frames.

1.4 Scope and Limitation of Study

This study focuses on the evaluation of the mechanical properties and the flammability behaviour of flame retardant impregnated sugar palm fibre reinforced unsaturated polyester polymer composites. Flame retardant fillers, such as aluminium hydroxide (ATH) and magnesium hydroxide were used to develop the flame retardant composites. The flame retardant filler loadings consisted of 10%, 20%, 30%, 40% and 50% loadings. The results from the mechanical testing (ASTM D5083, ASTM D790 and ASTM D256-00) and the fire testing (BS 476: part 6: 1989) were recorded. However, initial studies were performed to determine the tensile properties and interfacial shear strength (IFSS) of impregnated sugar palm fibre reinforced thermosetting polymers, which were impregnated with UP, VE and epoxy. These tests were performed according to JIS R 7601 and the pull-out test tests was performed in accordance with methods by Valadez-Gonzalez et al. (1999) and Park et al.(2006). Observations of the surface morphology of the fibres and fractured surfaces of the composite failure test specimens were performed using scanning electron microscopy (SEM).

1.5 Structure of Thesis

Chapter 1 presents the background of the study, problem statements, objectives of the research, significance of the study and structure of the thesis. In chapter 2, a literature review of relevant research is presented. The chapter begins with a comprehensive literature survey of natural fibre and sugar palm fibre. A review of the mechanical and flammability properties of the fibres and its composites are also included in this chapter. The methodology of the study is described in chapter 3. This chapter presents the evaluation of tensile properties and standard IFSS testing of single fibre ISPFRTP. This chapter also discusses techniques for preparation of flame retardant composites includes an analysis of the mechanical properties (tensile, flexural and impact properties) and details the flammability test of impregnated sugar palm fibre reinforced unsaturated polyester composites, which contain flame retardant fillers. Chapter 4 presents the results and a discussion of the mechanical and flammability properties of the specimens. The surface morphologies of the fibres and fractured specimen using scanning electron microscopy (SEM) are also evaluated in this chapter. Chapter 5 presents conclusions and recommendations for future studies.

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