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EFFECT OF IMPREGNATION OF SILICA INTO KENAF FIBRE-REINFORCED EPOXY COMPOSITES

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EFFECT OF IMPREGNATION OF SILICA INTO KENAF FIBRE-REINFORCED EPOXY COMPOSITES

By

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

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

EFFECT OF IMPREGNATION OF SILICA INTO KENAF FIBRE-REINFORCED EPOXY COMPOSITES

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April 2018

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Natural fibers are mechanically and thermally weaker than synthetic fibre due to its high water intake. To overcome this problem, addition of hydrophilic silica is expected to penetrate into the lumen and cell walls of fibre thus serving as blocking agent to inhibit water absorption. Two methods were adopted for the fabrication of hybrid epoxy reinforced with kenaf/silica: (1) Vacuum Infusion (VAC) and (2) Hand Lay-up/Hot Press (HP). For VAC process, the silica/epoxy solution was infused into fibre using vacuum pressure while for HP composites, silica/epoxy solution was spread onto fibre before hot pressed at 85˚C for 20 mins and cold pressed subsequently for another 5 mins. Another method was applied for HP which involves impregnation (IMP) of silica/epoxy solution into fibre. To achieve this, fibre was first degassed before submerged into silica/epoxy solution overnight and then compressed to remove excess resin prior to hot pressing the composite. Overall, the highest flexural strength and flexural modulus was achieved by IMP specimen of 40 vol% kenaf and 5 vol% silicas, with 78.8MPa and 5.11 GPa respectively. For HP specimens, the increment in kenaf volume leads to increase in flexural properties. In addition to that, the flexural strength and flexural modulus of HP specimens with 0, 1 and 5 vol% silica were found to be higher than VAC specimens. Despite that, water uptakes of VAC specimens were lower (lower water absorption rate is more desirable), ranging from 8-18.4% while HP specimens absorbs as much as 13-29%. Increment of kenaf volume was found to increase water uptakes considerably. On the other hand, inclusion of silicas also increased the water uptakes but only slightly. Interestingly, IMP specimens with 40, 50 and 60 vol% kenaf and 5 vol% silica absorbed 30.5, 36.7 and 54.4% less water respectively than their HP counterparts, indicating that silica has successfully impregnated the fiber walls and lumen thus blocking water from entering the fibres. Finally, the thermal stability of silica infused specimens was better due to the higher degradation temperature. Generally, composites with 40 vol% kenaf and 5 vol% silica had the highest degradation temperature at 335.43˚C for HP specimen while 60 vol% kenaf and 0 vol% silica HP composite possessed the lowest degradation temperature with 318.79°C.

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

KESAN PENGISITEPUAN SILIKA KE DALAM KOMPOSIT EPOKSI YANG DIPERKUKUH GENTIAN KENAF

Oleh

FARID BIN BAJURI

April 2018

Pengerusi: Norkhairunnisa Mazlan, PhD
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Gentian semula jadi adalah lebih lemah berbanding gentian sintetik secara mekanikal dan termal,, disebabkan oleh penyerapan air yang tinggi. Bagi mengatasi kelemahan ini, penambahan silika hidrofilik dijangka dapat menembusi lumen dan dinding sel gentian untuk bertindak sebagai agen penghalang penyerapan air. Dua kaedah digunakan untuk memfabrikasi epoksi yang diperkuatkan dengan kenaf/nanosilika hibrid: (1) Infusi Vakum (VAC) dan (2) susun-atur tangan/tekanan panas (HP). Untuk kaedah VAC, campuran nanosilika/epoksi diinfusikan ke dalam gentian menggunakan tekanan vakum manakala untuk komposit HP, campuran nanosilika/epoksi disapu ke atas gentian sebelum dimampatkan menggunakan tekanan panas pada suhu 85°C selama 20 min dan seterusnya tekanan sejuk selama 5 min. Kaedah lain yang menggunakan HP melibatkan infusi (IMP) campuran nanosilika/epoksi ke dalam gentian. Untuk kaedah ini, gentian akan divakumkan terlebih dahulu sebelum direndamkan de dalam campuran nanosilika/epoksi semalaman dan ditekan sebelum dimampatkan bawah tekanan panas. Secara keseluruhannya, kekuatan lenturan dan modulus lenturan tertinggi ditunjukkan oleh spesimen IMP yang difabrikasi dengan 40 vol% gentian kenaf dan 5 vol% nanosilika, masing masing dengan 78.8MPa dan 5.11GPa. Bagi spesimen HP, peningkatan dalam isipadu kenaf meningkatkan sifat lenturan. Selain itu, kekuatan lenturan dan modulus lenturan spesimen HP dengan 0, 1 dan 5 vol% didapati lebih tinggi daripada spesimen VAC. Selain itu, penyerapan air spesimen VAC adalah lebih rendah (penyerapan air yang rendah adalah lebih wajar), dalam linkungan 8-18.4% manakala spesimen HP menyerap lebih kurang 13-29%. Peningkatan isi padu kenaf didapati meningkatkan kadar penyerapan air dengan ketara. Sebaliknya, kadar penyerapan air yang disebabkab oleh penambahan nanosilika adalah kurang ketara. Yang menariknya, spesimen IMP yang difabrikasi dengan 40, 40 dan 60 vol% kenaf dan 5 vol% nanosilika menyerap 30.5, 36.7 dan 54.4% kurang air berbanding dengan spesimen HP, membuktikan bahawa infuse nanosilika berjaya menghalang air daripada memasuki lumen dan dinding sel gentian.Akhir sekali, kestabilan terma spesimen yang diinfusi dengan nanosilika adalah lebih tinggi disebabkan oleh suhu degradasi yang lebih tinggi. Secara keseluruhannya, komposit

HP dengan 40 vol% kenaf dan 5 vol% silika memiliki suhu degradasi yang tertinggi pada 335.43˚C manakala komposit HP dengan 60 vol% kenaf dan 0 vol% silika memiliki suhu degradasi terendah spada 318.79°C.

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I certify that a Thesis Examination Committee has met on 4th April 2018 to conduct the final examination of Farid bin Bajuri on his thesis entitled "Effect of Impregnation of Silica into Kenaf Fibre Reinforced Epoxy 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 Sciences.

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LIST OF ABBREVIATIONS

VAC	Vacuum Infusion
HP	Hot Press
IMP	Impregnation
FRP	Fibre Reinforced Polymer
KFRE	Kenaf Fibre Reinforced Epoxy
UP	Unsaturated Polyester
RTM	Resin Transfer Moulding
VE	Vinyl Ester
KFRUP	Kenaf Fibre Reinforced Unsaturated Polyester
PMMA	Polymethyl Methacrylate
PVC	Polyvinyl Chloride
EVA	Ethylene Vinyl Acetate
RCF	Recycled Cellulose Fibre
RH	Rice Husk
BB	Beech Bark
PP	Polypropylene
MAPP	Maleated Propylene
MEKP	Methyl Ethyl Ketone Peroxide
BJR	Banana And Jute Short And Random Orientation
WJWB	Warp Jute Weft Banana
WBWJ	Warp Banana Weft Jute
WAWBJ	Warp And Weft Banana And Jute
CNT	Carbon Nanotube
SWNT	Single Walled Nanotubes
MWNT	Multi Walled Nanotubes
TGA	Thermogravimetric Analysis
SEM	Scanning Electron Microscopy
DTG	Derivative Thermogravimetric
IDT	Initial Decomposition Temperature
TDT	Thermal Decomposition Temperature
Tmax	Temperature With The Highest Weight Loss

CHAPTER 1

INTRODUCTION

1.1 Overview

In this chapter, the research background, research problem, research objectives and thesis structure is discussed to introduce the main motivation behind the work. For section 1.2, composites that are reinforced with natural fibres and nanoparticles are introduced alongside with methodology to fabricate the composites. In section 1.3, problems encountered for composites reinforced with natural fibres and nanoparticles were discussed. In section 1.4, research objectives were discussed in details. Finally in section 1.5, the structure of this thesis is discussed.

1.2 Research Background

Composite is a structure that is made of by combination of two or more materials. Polymeric composite is usually made of polymer resin that is reinforced by different types of materials to improve the composite's properties depending on the applications (Kaw, 2006). While polymer resin is tough, it is relatively weak and requires reinforcement to produce a usable final product. In this work, resin reinforcement is focused on two types of reinforcing materials which are long fibre and particulates filler. Addition of fibres will increase the mechanical properties of a composite by dissipating energy received upon straining along its length. Fibres are the strongest when the force is exerted parallel to its length and the weakest when force is exerted perpendicular to its length (Alberto, 2013). It is important in a composite to have a good adhesion between fibre and resin to provide better energy dissipation (Andrea et al., 2017). Particulate filler are small particles that are introduced into resin for multiple purposes. One of them is to reduce the consumption of more expensive resin. For other purposes, it depends on the filler materials used (Tanahashi, 2010).

Synthetic fibres such as carbon fibre and glass fibre are usually used to reinforce resin. However these materials are expensive and unsustainable which made the usage of natural plant fibres being promoted to replace the synthetic fibres. Apart of the sustainability, natural fibre such as kenaf provide ecological sustenance through the reduction of carbon dioxide (CO_2) and fertilization of soil. Furthermore, the abrasive nature of synthetic fibres gives hurdle for handler to fabricate the composites. Without proper protection, glass fibres may accidentally be inhaled by the handler and causing lung complications. For reason discuss above, natural fibres are more user friendly to be used. In addition, natural fibres also have lower density compared to synthetic fibres meaning that the fabricated composites will have lower mass. Another valuable natural fibre properties is having high specific strength and stiffness (Akil et al., 2011; Pickering et al., 2016). Natural fibres with contain high celluloseand aligned cellulose microfibrils in the fibre direction offer better mechanical performance (Pickering et al., 2016). Bast fibres such as kenaf is example of natural fibre that possess this properties.

While reinforcing fibres will improve the mechanical properties of a composite, there is another kind of filler material used to improve various aspects of composite properties such as particulates. Particulates are materials with extremely high surface area and large pore volume. It is able to improve performance of polymer composites even at low particles concentration (Zhang et al., 2003). For example, carbon nanotube has been used to enhance the dielectric performance in polymer film while silica aerogels have been used to improve the thermal resistance of a polymeric material (Kim et al., 2014). Furthermore, filler have been used to reduce material costs by reducing the amount of more expensive matrix needed. Silica has a good chemical stability and good thermal stability making it suitable for different controlled release applications (Slowing et al., 2008) . The high thermal stability of silica makes it favourable material to improve the thermal stability of composites. Even at less than 3 vol%, particulates can adequately increase the composites' modulus, strength toughness and thermal deformation temperature (Zhang et al., 2003). Further research was found that silica is able to improve the hardness and scratch resistance of a coating while keeping the coating clear with its inclusion (Chen et al., 2003).

VAC (Vacuum Infusion) is one of a method of fabricating composites by introducing vacuum pressure to impregnate resin into the fibres. The space occupied by air in fibre is removed to be replaced by resin. Since the empty spaces are filled with resin, it is expected that the resin in fibre structure will have better adhesion with each other. Hand-lay-up is the most common method of fabricating composites due to its easy and cheap nature. Hand-lay-up followed by hot press (HP) is an extension of the process. Pressing give the composite the desired shape while heat is applied to increase the crosslink between resin and hardener. Impregnation (IMP) is an extension of HP process where, prior to hot pressing, the fibres are soaked with resin under vacuum condition with purpose to remove existence of voids in the resin and promote better penetration of resin into the fibres' lumen.

1.3 Problem Statement

Natural fibres possess many desirable properties. Nevertheless it is inevitable that the mechanical property of natural fibres is lower than synthetic fibres. The properties of natural fibres are variable depending on the chemical composition and structure of the fibres, growing conditions, harvesting time, extraction method, treatment and storage procedures (Pickering et al., 2016). While presence of cellulose contributes to the natural fibres mechanical properties, hygroscopic and hydrophilic nature (Akil et al., 2011). Cellulose structure inside natural fibres and water surface tension cause water to be absorbed by natural fibres while the hydroxyl group of the cellulose bond with hydroxyl group from water to hold the water inside natural fibres. These properties cause high water absorption rate and swelling of natural fibres. The swelling of fibre due to water absorption then damage the surrounding resin. This, as a whole will cause the composite structure to lose its integrity. Furthermore, hydrophilic nature of natural fibre is incompatible with hydrophobic resin causing larger gap between fibre and resin interface leading to poor interfacial bonding which ultimately limit the mechanical performance (Nabi and Jyoti, 2015; Pickering et al., 2016). Cellulosic materials have a primary thermal decomposition between 200 ˚C and 400 ˚C (Fisher et al., 2002; Jonoobi et al., 2009). Weight loss of approximate 5.5% of 48 vol% kenaf reinforced epoxy as compared to 3.5% weight loss of neat epoxy exposed for 3 hrs at 150 ˚C has been observed with both kenaf reinforced epoxy and neat epoxy (Azwa and Yousif, 2013). Even though cellulosic materials have a thermal decomposition temperature of higher than 150 ˚C, prolonged exposure to heat will trigger faster rate of decomposition. Natural fibre has high cellulose content which contributes to the high flammability of the fibres and high lignin content which contributes to lower thermal degradation temperature and higher char formation.

To address the problems stated above, fabrication method plays important role. Both VAC and IMP processes use vacuum to eliminate the spaces between fibres and resin to promote better interfacial bonding of both matters. In IMP process silica is added to impregnate into the fibres' lumen with purpose to block further water molecules movement into the fibres. This can reduce water absorption into natural fibres which subsequently reduce the fibre swelling. The high thermal stability of silica can be introduced into the system to improve the thermal stability of the overall composites.

1.4 Research Objective

The main objective of this work is to investigate the material properties of hybrid silica/kenaf reinforced epoxy composites in terms of flexural properties, compressive properties, water absorption properties, thermal stability analysis and modal analysis. The specific objectives of this research are:

1) To investigate the above properties by fabricating composites with 20, 30, 40, 50 and 60 vol% kenaf.
2) To determine the above properties with the inclusion of silica at 1 and 5 vol%.
3) To determine the best fabrication method between vacuum infusion (VAC), hand-lay-up proceeded by hot pressing (HP) and impregnation proceeded by hot pressing (IMP).

1.5 Thesis Outline

In Chapter 1, the research background, research problems and research objectives have been discussed.

In Chapter 2, literature review of kenaf fibres, epoxy resin and silica that are used in this work is discussed in details. Further literature reviews regarding flexural properties, compressive properties, thermal stability analysis, water absorption properties and modal analysis of similar composites are discussed too. Final section of Chapter 2 discuss the fabrication technic used in this work.

In Chapter 3, method of fabrications and test condition for each experiments conducted to fabricated specimens are discussed.

In Chapter 4, results and discussion of experiments conducted on fabricated specimens are discussed.

In Chapter 5, the whole research will be summarised and suggestions for future work are made.

This research focuses on fabricating hybrid epoxy reinforced with kenaf/silica. The effect of adding silica into composite was studied at 0, 1, 5 vol%. The type of silica used is hydrophilic. The reinforcing kenaf used was random orientation type and untreated and its volume was fixed at 20, 30, 40, 50 and 60 vol%. Epoxy and silica were mixed using homogenizer at 3000rpm for 10mins. All the composites produced are studied with flexural test, compressive test and water absorption test while selective composites are studied with thermal stability analysis, morphological analysis and modal analysis. The results obtained are only limited to the materials and fabrication parameters mentioned above. In addition to that, data from thermal stability analysis, morphological analysis and modal analysis are not comprehensive on all types of composites.

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LIST OF PUBLICATIONS

Farid Bajuri, Norkhairunnisa Mazlan, Mohamad Ridzwan Ishak and Junichiro Imatomi. 2016. Flexural and Compressive Properties of Hybrid Kenaf/Silica Nanoparticles in Epoxy Composite. *Procedia Chemistry, 19*, 955-960.

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