The Effect of Stearic Acid Treatment and Calcium Carbonate on properties of Kenaf Reinforced Polyester Composites.

by

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A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science (polymer engineering)

School of Material Engineering

UNIVERSITI MALAYSIA PERLIS

2017

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Academic Session : 2016/17

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ACKNOWLEDGEMENTS

All praise is due to Allah, The One Who makes it easy for me to complete this work. May His ceaseless blessing perpetually shower on the noble soul of our beloved prophet, Muhammad, his households, his companions and generality of those who follow their path until the day of Reckoning. My immense appreciation goes to my supervisor in person of DrRozyantybintRahman for her advice, guidance and valuable contributions towards the success of this work.

This work will not be complete without acknowledge my family especially my darling husband, Dr Muhammad-Bashir Yusuf for his financial, spiritual, and moral support, my kids for their sacrifice and prayers and my blood brothers and sisters and my in laws from Yusuf's family. I say JAZAAKUMULLAHU KHAYRAN to you all. I also extend my profound appreciation to my roommates, my laboratory friends, muslims and non-muslims friends in UniMAP, May Allah Almighty, ease your affairs. I am indebted to my late parents for their prayers and wishes during their lifetime for me to proceed on my studies and become someone valuable in my home and society. May Almighty Allah grant you Jannatualfridaus (Aameen).

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LIST OF ABBREVIATIONS

CO_2	Carbon dioxide
SA	Stearic acid
KF	Kenaf fiber
SEM	Scanning electron microscope
FT-IR	Fourier Transform infrared spectroscopy
MEKP	Methyl ethyl ketone peroxide
UP	Unsaturated polyester
CC	Calcium Carbonate
CT	Treated Calcium Carbonate

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Kesan Rawatan Asid Stearik dan Kalsium Karbonat pada Sifat Komposit Kenaf Poliester.

ABSTRAK

Dalam kajian ini, kenaf/poliester tak tepu dan komposit hibrid kenaf / $CaCO_3$ poliester telah disediakan menggunakan kaedah pelapisan taagar. Kesan rawatan permukaan kenaf dan $CaCO_3$ pada sifat mekanikal, morfologi, struktur kimia dan penyerapan air dikaji. Spektroskopi Fourier Transform Infrared (FT-IR) diperhatikan untuk gentian kenaf yang tidak dirawat dan dirawat, dan partikel $CaCO_3$ yang tidak dirawat dan dirawat. Gentian kenaf yang tidak dirawat dan dirawat menunjukkan puncak penyerapan yang sangat kuat dan luas di rantau 3200-3600cm-1 dari regangan getaran ikatan hidrogen O-H. Walau bagaimanapun, puncak 2918 cm-1 dan 2917 cm-1 daripada kenaf yang dirawat menunjukkan bahawa asid stearik telah berjaya menyalut permukaan gentian kenaf serta 2918 cm-1 dalam $CaCO_3$ yang dirawat. Kekuatan tegangan komposit poliester kenaf/$CaCO_3$ didapati mempunyai nilai tertinggi dengan 6% $CaCO_3$ (70.1MPa) berbanding dengan komposit serat kenaf terawat/poliester tak tepu (57.7MPa) dan komposit serat kenaf tidak dirawat/poliester tidak tepu (43.2MPa) pada 20% kandungan serat. Kekuatan lenturan dan modulus lenturan menunjukkan corak yang sama seperti kekuatan tegangan dan modulus tegangan (68.5MPa dan 3741.4MPa masing-masing). Pengimbasan Mikroskop Elektron (SEM) mendedahkan bahawa permukaan gentian yang dirawat menjadi kasar selepas rawatan keranadisaluti oleh SA permukaannya. Rawatan ini meningkatkan interaksi dan lekatan gentian dan matriks yang lebih baik. Hasil kajian penyerapan air menunjukkan bahawa peningkatan kandungan serat kenaf dalam komposit menyebabkan peningkatan penyerapan air, dan komposit hibrid mencapai keseimbangan lebih awal daripada komposit lain disebabkan oleh penggabungan $CaCO_3$ yang menghalang penembusan air diikuti oleh komposit yang dirawat dan tidak dirawat.

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The Effect of Stearic Acid Treatment and Calcium Carbonate on Properties of Kenaf Polyester Composite.

ABSTRACT

In this research, kenaf/unsaturated polyester and kenaf fiber/$CaCO_3$ polyester hybrid composites were fabricated using hand lay-up method. The effect of surface treatment of kenaf and $CaCO_3$ on the mechanical properties, morphology, chemical structures and water absorption were studied. The Fourier Transform Infrared (FT-IR) spectroscopy was observed for untreated and treated kenaf fibers, and untreated and treated $CaCO_3$ particles. The untreated and treated kenaf fibers showed a very strong and broad absorption peak in the region 3200-3600cm^{-1} of hydrogen bonded O-H stretching vibration. However, the peaks 2918cm^{-1} and 2917cm^{-1} of the treated kenaf showed that stearic acid had successfully coated the surface of the kenaf fiber as well as 2918cm^{-1} in treated $CaCO_3$. The tensile strength of kenaf/ $CaCO_3$ polyester composites was found to have highest value with 6% $CaCO_3$ (70.1MPa) in comparison with treated kenaf fiber/unsaturated polyester composites (57.7MPa) and untreated kenaf fiber/unsaturated polyester composites (43.2MPa) at 20% fiber loading. The flexural strength and flexural modulus showed similar trend as tensile strength and tensile modulus (68.5MPa and 3741.4MPa respectively). The Scanning Electron Microscope (SEM) revealed that the surface of the treated fibers became rough after treatment due to the coated of SA onto the surface. The treatment enhances better interaction and adhesion of fiber and matrix. The results of water absorption study depicted that increasing the loading of kenaf fiber in the composites resulted in increasing water absorption, and hybrid composites attained equilibrium earlier than other composites due to the incorporation of $CaCO_3$ that prevent further water penetration followed by treated and untreated composites respectively.

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CHAPTER 1

INTRODUCTION

1.1 Overview

The worldwide availability of natural fibers encouraged their use in composite and paper industry. One of the natural fiber that is currently receiving much attention in polymer composites industry is kenaf. The properties of Kenaf can be compared to those of other known non-wood fibers such as bagasse, rice straw, bamboo and wheat (Ashori et al., 2006). The expanding consideration on the utilization of natural fibre composites is principally because of their accessibility from sustainable natural resources, light weight, low cost and biodegradability. Moreover, kenaf fibers have been used widely as reinforcement in polymeric composite materials and these composite materials have bright future due to its eco-friendly and renewability. However, this study focuses on how stearic acid treated kenaf fibers/$CaCO_3$ reinforced unsaturated polyester resin.

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1.2 Background of study

During the last few years, there is an high demand for fiber-reinforced composites as a result of their exclusive characteristics like high speci fic strength a

thermal expansion, good fatigue performance and processing advantages at low cost. Moreover, due to their properties such as lightweight, adequate mechanical strength and inexpensive, natural fibers have a growing usage as reinforcement materials (Mishra et al., 2013 & Joshi et al., 2004).

The plant fibers promotes healthy ecosystem because of their biodegradability while the economic interest of the industry is satisfy by their low cost and high performance. The CO_2 released into the atmosphere at the end of life-span of natural fiber-reinforced plastics when they are used in landfill or it undergoes combustion process, is neutral when compared to the quantity intake during their growth. Low abrasive nature of fiber is advantageous when talking about technical process and recycling process of the traditional composite materials. Decompose of natural fiber-reinforced plastics at the end of their life cycle make them to be the most environmental friendly materials. In non-structural applications, natural fiber composites are mostly used to replace glass fibre. These find applications in sporting goods, automotive components, such that electrical and construction appliances manufactured using glass fibre in the past are now using natural fibre which is more environmental friendly (Wallenberg & Weston, 2004). Nevertheless, their cheap cost and high performance satisfies the economic concern of producers (Hazwani et al., 2007).

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Instead of using natural fibers alone as reinforcement materials, hybridizing thesematerials with inorganic fillers leads to an improved mechanical property and cost effective composite materials. Hybrid composites consist of two or more reinforcing materials in a matrix. The mechanical performance of composites relies mainly on the properties of matrix, reinforcement materials, and their interaction. Therefore, fabrication of hybrid composites results in an improved mechanical properties and reduced material costs compared to the conventional composites (Sreekala et al., 2002 and Rajkumar 2015).

Calcium carbonate ($CaCO_3$) is one of the most favourite fillers used in some fields of rubber, plastics, building materials, coating, papermaking and so on (Izgin et al., 2012). Some factors such as cheap price, abundance, lightness, surface coating facility, easiness of manufacturing and ability to carry high load have caused extensive use of this material as reinforcement (Yang et al., 2008). Incompatibilty of hydrophilic and energetic surface of calcium carbornate with low energy surface of hydrophobic polymers, are problems that must be tackled before using it as functional filler (Maged et al., 2002). Surface treatment is carried out on the filler to eliminate this problem.

1.3 Problem Statement

The most adequate way to increase the material efficiency without causing environmental unfriendly is to use natural fiber reinforced composite. The objectives are to improve the performance of the materials and also to search for the solution to lower the cost. The latest incorporation of natural materials develop plastic based material has led to higher material properties and more possibilities use in many applications.

The widely availability of natural fibers such as kenaf in Asia is becoming more and owing to its benefits above well-known reinforcement materials with regards to their density, recyclability, abrasiveness and decomposability, make it grow rapidly in fiber reinforced composites. However, most natural fibersmajor problem is their hydrophilicity nature due to their strong polar character, which results in low resistance to environmental conditions (i.e ability to absorb moisture in large amount). Another problem of natural fibers in composites fabrications is poor adhesion to polymer matrix. This is due to high cellulose content in them which brings about instability in weight, dimension, strength and stiffness.

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There are various attempts in the past years to modify the surface of natural fibers in order to promote good adhesion with polymeric matrix. Several methods have been employed to treat the surface of natural fibers, however, these treatments have many disadvantages such as the use of expensive equipment or expensive chemicals. A very easy and interesting treatment is to modifying the surface of the fibers with fatty acids such as stearic acid, though many researchers have worked on modifying with sodium hydroxide (mercerization). The principle of this treatment is that the carboxyl group of stearic acid reacts with the hydroxyl groups of the fiber to yield ester and water molecules in a process called esterification reaction. This process reduces the hydroxyl group numbers present in the fiber. Another advantage of treating fiber with stearic acid is that, it is not sensitive to oxidation during the processing temperature of the composites (Arnson et al., 1989). However, in this current study, kenaf fiber will be treated with stearic acid.

Despites the great advantages of natural fibers, high moisture absorption tendency, poor wettability and adhesion and low thermal stability limit their uses. Hybridization of natural fibers with synthetic fiber or fillers eradicates these limitations (Pothan et al., 2007). Natural fiber and synthetic filler can be combined in the same matrix to produce hybrid composites of better properties obtained from individual constituents.

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1.4 Objectives

1. To determine the effect of kenaf fiber loading and kenaf fiber treatment with stearic acid on the mechanical properties of kenaf/unsaturated polyester composites.

2. To determine the effect of $CaCO_3$ on the mechanical properties of kenaf/$CaCO_3$ polyester hybrid composites.
3. To characterize surface morphology, and determine the water absorption of untreated kenaf/polyester, treated kenaf/polyester composite and treated kenaf/$CaCO_3$ polyester hybrid composite.

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1.5 Scopes

The main scope of this research is to fabricate untreated kenaf/polyester composites, treated kenaf fiber polyester composite and treated kenaf/$CaCO_3$ polyester hybrid composites and to determine the effect of treating kenaf fiber with stearic acid by examining mechanical properties, structure and surface morphology of kenaf/polyester composites. Also the effect of $CaCO_3$ on the hybrid composites is to be determined. The tensile test were conducted according to ASTM D3039 standard and flexural test according to ASTM D7264 standard. FT-IR analysis was carried out to investigate the structure of the treated and untreated kenaf, and treated and untreated $CaCO_3$ while scanning electron microscope (SEM) analysis were conducted to observe the compatibility among pure kenaf/polyester composite, treated kenaf/polyester composite and kenaf/$CaCO_3$ polyester composites. Water absorption test was carried out by soaking the specimens of different composites in distilled water at room temperature overnight and the weight change is recorded.

CHAPTER 2

LITERATURE REVIEW

2.1 Natural Fibers

In recent years, natural fibers have gained popularity over synthetic fibers due to their low cost, light weight, abundance as natural and renewable resources, and versatile mechanical properties, among others (Yousif, et al., 2012 & Frank et al., 2012). Natural fibers with better properties than synthetic fibers are preferred by researchers and industries because of their numerous applications in many fields such as automotive, textile, fiber board, cushion, paper, mattress, door, wall panel, air cleaner, dashboard, and insulation mat manufacturing, as well as in the construction and transportation industries (Misha et al., 2015 & Lips et al., 2009).

Natural fibers consist of cellulose, hemicelluloses and lignin and each component varies in percentage in different types of fibers used.Cellulose is known as one of the most abundant cellulose is semicrystalline polysaccharide which is responsible for the hydrophilic state of all natural fibers. Hemicelluloses comprise of fully amorphous structures, causing it to be partially soluble in water and most alkaline solutions. Lignin is described as amorphous polymers that consist of aromatics while having slight effect on water absorption (Westman et al., 2010). Table 2.1 shows the chemical compositions of different types of natural fibers (Klemm et al., 2003).

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Table 2.1: The chemical compositions of different types of natural fibers (Klemm et al., 2003).

Fiber types	Cellulose	Hemicelluloses	Lignin
Harwood	43-47	25-35	16-24
Softwood	40-44	25-29	25-31
Bagasse	40	30	20
Coir	32-43	10-20	43-49
Corn cobs	45	35	15
Corn stalks	35	25	35
Cotton	95	2	2
Flax (retted)	71	21	2
Flax (untreated)	63	12	3
Hemp	70	22	6
Henequen	78	4-8	13
Istle	73	4-8	17
Jute	71	14	13
Kenaf	60	21	18
Ramie	76	17	1
Sisal	73	14	11
Sunn	80	10	6
Wheat straw	30	50	15

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Since some properties of natural fibers show good potential to be used even not entirely as a substitute with the traditional synthetic fibers, it is considered a positive steps to promote a friendly environment especially by using or extracting more agriculture residue that can be turn into useful applications or goods rather than being left as piles of waste that can pollute the environment. Agricultural crop wastes or residues in the form of oil palm, sugar palm, banana, kenaf and pineapple leaf and so on, are usually produced in billions of tons all over the world. Usually, these leftovers are types of renewable resources of biomass, are economical and are abundantly available. Therefore, it is of benefit to make use of these “good wastes” especially as reinforcements in composite to produce more environmental friendly applications (Sahari & Sapuan, 2011).Natural fibers can be classified into three stages namely vegetable fibers, animal fibers and mineral fibers. Figure 2.1 shows the classification of natural fibers (Ichhaporia, 2008).

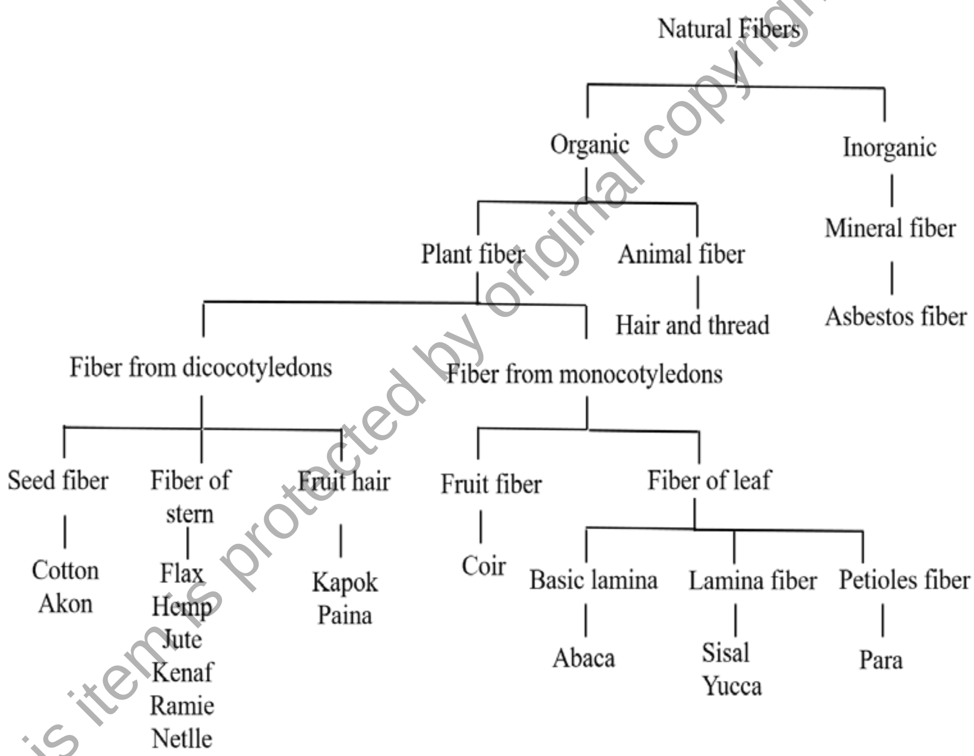

Figure 2.1: Classification of natural fibers (Ichhaporia, 2008)

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2.1.1 Properties and Characteristics of Natural Fibers

Natural fibers are non-abrasive towards mixing and molding equipment, which can contribute to significant equipment maintenance cost reductions. They are also safe in handling and working conditions in comparison to synthetic reinforcements, such as glass fibers. They are environmental friendly in terms of processing, hence, a reduction in in risk of dermal or respiratory problems. The most interesting aspect of natural fibers is their positive impact on the environment. They are renewable resources, where they are biodegradable and their production requires little energy.

The major drawback of natural fibers compared to synthetic fibers, is their non-uniformity, variety of dimensions, and their mechanical properties (even individual natural fiber in the same cultivation) (Bismarck et al., 2005). Therefore the task needs to be solved in order to boost their acceptance as a quality alternative to conventional reinforcing fibers (John et al., 2008 & Nishino et al., 2004).

Although, natural fibers gives some good impact most especially to the environment, however, it also has it owns limitations attached to it. The disadvantages that can be observed with natural fibers are their low impact strength properties despite of variation in quality whereby depending on the unpredictable influences including weather, moisture swelling of the fibers due to absorption of moisture, restricted or confined maximum processing temperature, lower durability and poor fire resistance (Brouwer, 2000; Abilash & Sivapragash, 2013; Yimer, 2013).

The weaker interfacial or adhesion bonds between highly hydrophilic natural fibers and hydrophobic, non-polar organophilic polymer matrix, leads to considerable reduction in the properties of the composites and, thus, significantly obstructs their industrial utilization and production. However, many approaches and schemes have

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been carried to nullify this deficiency in compatibility. These include the introduction of coupling agents and/or various surface modification techniques. The surface of the natural fibers can be modified and this can be achieved by physical, mechanical, and/or chemical means. For any composite, the circumstances for substantial reinforcement and virtuous properties are the homogeneous distribution and alignment of the reinforcing component.

Of recent, the researchers all over the world are taking into considering, the nano-sized particles as a higher potential filler to improve the physical and mechanical properties of polymer composite materials. In general, nano-sized particles are free of defects, therefore their usage in polymer composites can give a new prospect to overcome the problem faced when using traditional micrometer sized particle materials.

2.2 Plant Fibers

Plant fibers often referred to as vegetable fibres, plays a beneficial role in our daily life. Human being has been strongly dependent on plant fibres for all kinds of purposes. For example, fibrous materials such as wood and bamboo have found particular application in construction. Fibres from banana, coir, jute, pineapple and sisal have been used in aerospace, automotive, buildingand packaging industries (O'Donnel et al., 2004 & Munoz et al., 2015).The most interesting aspect of plant fibres is their positive environmental impact. A wide variety of fibres has also been used for production of textiles, paper and fibre boards. The mechanical properties of plant fibres depend on their physical, chemical and morphological properties such as the fibre orientation, crystal structure and diameter/cross-sectional area of the fibre (Bledzki et al., 1999). The strength of plant fibres is attributed to the rigidity and high molecular weight of

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cellulose chains, intermolecular and intramolecular hydrogen bonding, fibrillar and the crystalline structure of the fibres.

Plant fibres played an essential role in the composite industry and they can be classified based on their origin. The plant fibre types include bast, seed and leaf fibres. Bast fibres are obtained from the inner bark or bast surrounding the stem of the plant. These fibres have higher tensile strength than other fibres. Therefore, these fibres are used for durable yarn, fabric, packaging and paper industries. e.g. banana, flax, hemp, jute, kenaf, ramie, etc. Leaf fibres are collected from leaves, e.g. sisal, banana, abaca, etc. Seed fibres are collected from seeds or seed cases. e.g. cotton, coir, oil palm, kapok, etc. The major advantages of plant fibres over traditional materials such as glass fibres, talc and mica are: acceptable specific strength properties, light in weight, serving as an excellent reinforcing agent for plastics, less damage to processing equipment, less expensive, lower energy consumption, carbon dioxide sequestration, environmental friendly in nature, good relative mechanical properties and good thermal properties (Mohanty et al., 2000).

Fibers are mostly composed of cellulose, hemicelluloses, lignin, waxes, and several water soluble compounds, are the major constituents (Akil et al., 2011 & Chawla et al., 1998). They contained mostly 65-70% cellulose which is composed of three elements, C, H, O_2, with a general formula of $C_6H_6O_5$, that crystalline (Chawla et al., 1998). The lignin and other non-cellulosic substances are associated with the cell walls and their presence determines the final properties of the fiber. An essential attribute of plant fibers is their ability to absorb moisture from the atmosphere in comparatively large quantities. In addition, plant fibre is exposed to biological decay and most plant fibers darken and weaken with age and exposure to light. They are all easily considered as renewable resources and do not exacerbate the carbon dioxide emissions problem (Akil

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