



**Study of The Effect of Surface Treatment of Kenaf
Fiber On Mechanical and Water absorption of Kenaf
Filled Unsaturated Polyester Composite**

by

**ILYAS AMMER SAEID SALEM
(1631621964)**

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DECLARATION OF THESIS

Author's full name : ILYAS AMMER SAEID SALEM
Date of birth : 26/12/1987
Title : Study of The Effect of Surface Treatment of Kenaf Fiber On
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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

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Kajian Kesan Rawatan Permukaan Gentian Kenaf Pada Mekanikal Dan Penyerapan Air Untuk Komposit Kenaf Diisi Poliester Tak Tepu

ABSTRAK

Dalam kajian ini, komposit poliester tak tepu / serat kenaf (UP/KF) telah disediakan dengan menggunakan teknik “*hand lay-up*”. Kesan rawatan permukaan serat kenaf pada morfologi, struktur kimia, mekanikal dan penyerapan air komposit poliester tak tepu / serat kenaf telah dikaji. Asid stearik (SA) yang berbeza kepekatan iaitu 0, 0.4, dan 0.8 wt% telah digunakan. Spektrum Fourier Inframerah (FT-IR) serat kenaf selepas rawatan dengan asid stearik menunjukkan keamatan puncak sekitar 3300-3400 cm^{-1} , dimana ia adalah disebabkan oleh regangan O-H. Ini bagaimanapun serat kenaf terawat dengan stearik asid telah menunjukkan kehilangan kumpulan O-H dan menyebabkan puncak tersebut menghilang. telah digunakan. Kekuatan tegangan komposit poliester tak tepu / serat kenaf terawat didapati lebih tinggi untuk 40 wt% muatan serat kenaf. Nilai Kekuatan tegangan piling tinggi diperolehi adalah daripada rawatan dengan 0.4 % kepekatan asid stearik iaitu 56 MPa dan modulus tegangan 2409 MPa. Dari hasil kekuatan lenturan yang diperolehi, ia jelas dilihat bahawa 40 wt% muatan serat kenaf dan rawatan dengan kepekatan 0.4 wt% asid stearik memberi nilai tertinggi pada 72 MPa dan modulus lenturan pada 3929 MPa. Pemerhatian menggunakan Mikroskop Imbasan Elektron (SEM) mendedahkan bahawa permukaan serat kenaf selepas rawatan dengan SA menjadi lebih kasar. Selain itu, rawatan serat kenaf dengan asid stearik memberikan pengedaran yang lebih untuk kenaf dalam polimer matriks. Keputusan kajian penyerapan air mendedahkan bahawa peningkatan muatan KF dalam komposit akan menyebabkan peningkatan kecenderungan untuk menyerap air. Walau bagaimanapun, penyerapan telah menurun dengan ketara selepas rawatan dengan asid stearik dan juga tempoh masa untuk sampai ke keadaan keseimbangan.

Study of The Effect of Surface Treatment of Kenaf Fiber On Mechanical and Water absorption of Kenaf Filled Unsaturated Polyester Composite

ABSTRACT

In this research, unsaturated polyester/kenaf fiber (UP/KF) composites was prepared by using hand lay-up process. The effect of surface treatment of kenaf fiber on mechanical, morphology, chemical structure and water absorption of kenaf filled unsaturated polyester composites were studied. Different concentrations of stearic acid (SA) were applied, i.e. 0, 0.4, and 0.8 wt%. The Fourier transform infrared (FT-IR) spectra of kenaf fiber shows high intensity of the peak around $3300-3400\text{ cm}^{-1}$, which is attributed to the hydrogen bonded O-H stretching. However, the treated kenaf fiber with stearic acid shows the elimination of O-H group and this peak is vanished. This is due to the reaction of (-COOH) group of stearic with (-OH) group of kenaf fiber. Tensile strength of treated kenaf fiber/ unsaturated polyester composites was found to be higher for 40 wt% loading of kenaf fiber. The highest tensile strength value was obtained after treatment with 0.4 wt% concentration of stearic acid at 56 MPa and tensile modulus was at 2409 MPa. From the flexural strength result obtained, it is clearly seen that 40 wt% loading of kenaf fiber and treatment with 0.4 wt% concentration of stearic acid give the highest value at 72 MPa and flexural modulus at 3929 MPa. Scanning Electron Microscope (SEM) observation revealed that the surface of kenaf fiber after treatment with SA became rougher. Moreover, the treatment of kenaf fiber with stearic acid gives better fiber to matrix interaction of kenaf fiber inside the polymeric matrix. The results of water absorption study revealed that increasing the loading of KF in the composite will result is increasing the tendency to absorb water. However, the absorption was significantly decreased after treatment with stearic acid as well as the time to reach to the equilibrium state.

CHAPTER 1

INTRODUCTION

1.1 Overview

The demand of high performance materials has led to extensive research and development of new and improved materials, such as composite materials. In recent years, natural fibers seem to be a superior material which has emerged as an abundant and sustainable replacement for the nonrenewable and expensive synthetic fibers. Several natural fibers have been used as a reinforcement in composites materials for advanced applications such as banana, sisal, oil palm, jute, kenaf and coir. Among different types of fibers, kenaf fiber has been widely explored over the past several years (Hazizan et al., 2011). Kenaf fiber can be extracted from bast kenaf plants. Moreover, kenaf natural fibers have been used widely as reinforcement in polymeric composite materials. These composites have a sparkling future due to its eco-friendly and renewability. Hence this work is presenting reinforces of unsaturated polyester resin composite with treated kenaf fiber.

1.2 Background

The quest to develop high-performance materials prepared and mixed with natural fibers is increasing worldwide, especially the material with integrity to be used in various application. Due to natural large variation in properties under different conditions, it becomes a great challenge in using it as a plastic reinforcement (Omar et al, 2012). The properties of bio-composites are influenced by different variables such as, the type of fibers, source of fibers, preparation methods, and modification of fiber (if any). Recently

there has been a great interest in preparation of bio-composites comprising of natural fibers reinforced with polymers for industrial application (Bras et al, 2010).

Moreover, using of natural fibers as fillers and or reinforces in plastic composites is a growing trend and making them attractive to manufacturers. This is due to their flexibility during processing, highly specific stiffness, and low cost.

Kenaf fiber has several advantages over other traditional reinforcement materials, as such it has attracted the attention of researchers globally. The availability of kenaf fiber in Asia is more and it is abundantly available, environmental friendly, renewable, and cheap. Moreover, it has advantages over the synthetic fibers (glass, carbon and aramid fibers) such as light weight and full biodegradable. The biodegradability of kenaf fiber has a great contribution to a healthy ecosystem while their low cost and high performance satisfies the economic concern of manufacturers (Lei et al., 2007) (Hazwani et al., 2007).

However, using kenaf also gives some disadvantages which occur during the processing stage of adding kenaf fiber into a polymer matrix. The lack of good interfacial adhesion between the fiber and polymer matrix, which results in poor mechanical properties in the final products.

Due to the chemical structure of natural fibers (such as kenaf) which is mainly has hydrophilic moiety (mainly OH), which will cause high polar characteristic. Therefore, it is important to enhance compatibility between the fiber and the polymer matrix in order to obtain a uniform composites with good properties. Several chemical treatments can be done to natural fibers in order to obtain better compatibility (Colom et al., 2003).

There were several attempts in the past years to modify the surface of natural fibers in order to enhance adhesion with the polymer matrix. Various methods such as corona

treatment (Belgacem et al., 1994), plasma treatment (Felix et al., 1994), mercerization (Bisanda and Ansell., 1991), heat treatment (Sapieha et al., 1989), graft copolymerization (Felix and Gatenholm., 1991) (Maldas et al., 1989) and silane treatment (Mieck et al., 1995) (Bisanda and Ansell., 1991) have been reported to investigate the compatibility of natural fiber in polymeric composites, in most cases the results were positive.

1.3 Problem Statement

It is highly of interest recently to use natural fibers in polymeric matrices as resulting composites characteristics in term of the strength and toughness are much better than these of unreinforced polymers. In addition, natural fibers are light in weight, strong, renewable, very cheap and abundant. Natural fibers were found over the last decade, as a potential resource for producing low-cost composite materials.

However, even though natural fibers have numerous advantages, they are not totally problems free. The major issue of these natural fibers is their strong polar character, which results low resistance to environmental conditions due to their capability to absorb moisture in big amount because cellulose is hydroscopic in nature. This absorption of moisture leads to instability in weight, dimensional, strength and stiffness. Moreover, this problem creates incompatibility with most thermoplastic polymers.

Kenaf is one of the natural (plant) fibers used as reinforcement in polymer matrix composites. Kenaf (*Hibiscus cannabinus*, L. family Malvacea) has been found to be an important source of fiber for composites, and other industrial applications. Kenaf is well known as a cellulosic source with both economic and ecological advantages; in 3 months (after sowing the seeds), it is able to grow under a wide range of weather conditions, to a height of more than 3 m and a base diameter of 3–5 cm. Kenaf was priced at \$400 per tonne in 1995 and from \$278 to \$302 per tonne in 2000. From the viewpoint of energy

consumption, it takes to obtain 1 kg of kenaf 4 times higher energy of the energy to obtain 1 kg of glass fiber. There were several attempts in the past years to modify the surface of natural fibers in order to enhance adhesion with the polymeric matrix. Various methods have been applied to treat the surface of natural fibers, however, these treatments have many disadvantages such as the use of expensive equipment or the use of expensive chemicals. A very attractive treatment is to modify the surface of natural fibers with fatty acids such as stearic acid. The principle of this treatment is the carboxyl group of stearic acid reacts with the hydroxyl groups of the fiber through an esterification reaction and, thus, reduces the hydroxyl groups number available to bonded with water molecules. One more advantage of treatment with stearic acid is that it is not sensitive to oxidation during the processing temperatures of natural fiber/polymer composites (Arnson et al., 1989)

With this in view, this current research was conducted.

1.4 Objectives

1. To investigate the effect of kenaf fiber loading on mechanical properties of kenaf/unsaturated polyester composites.
2. To determine the effect of treatment of kenaf fiber with stearic acid by applying different concentrations of stearic acid on mechanical properties of kenaf/unsaturated polyester composites.
3. To characterize structure, morphology, determine the water absorption of treated and untreated kenaf /unsaturated polyester composites.

1.5 Scopes

The main scope of this research is to study the effect of treatment of kenaf fiber with stearic acid on kenaf/unsaturated polyester composites to enhance compatibility between the fiber and the polymer by examining mechanical, structure and surface

morphology. Kenaf /unsaturated polyester composites were prepared via hand layup method and with different kenaf fiber loadings, i.e. 0%, 10, 20, 30 and 40 wt%. Different concentrations of stearic acid were applied to treat kenaf fiber, i.e. 0, 0.4, and 0.8 wt%. The tensile test was conducted according to ASTM D3039 standard, while flexural test was conducted according to ASTM D7264 standard. The structure was investigated by using FT-IR analysis. Scanning electron microscope (SEM) analysis was conducted to observe the comparability between pure and treated kenaf fiber with unsaturated polyester. Water absorption was determined by soaking the specimens of different composites in distilled water. The specimens were obtained by cutting according ASTM D5229. The specimens will be immersed in distilled water at room temperature and the weight change will be recorded.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview of Polymer Composite Materials

The researchers across the world have started to focus on increasing demand for environmentally friendly materials. Natural fibers exhibit many advantages, besides ecological considerations, which encourage to replace using of synthetic fibers in polymer composites, which offer a very high potential for lightweight structures applications as an outstanding reinforcement (Aji et al., 2009). The most significant advantages of using natural fibers are that they are obtained from a renewable resources so only minimum energy is required for processing, biodegradable in nature, and very low cost in comparison with synthetic fibers (Yuhazri et al., 2012).

Recently, natural fiber composites have been utilized in several engineering applications due to natural properties that they offer such a variety of lightweight, reform, cheap and environmentally friendly. Natural fiber composites are used in numerous industries for instance construction, marine, automotive, electrical, household appliances, and sporting goods (Wallenberg & Weston, 2004). Kenaf, sisal, coir, banana, hemp, pulp, wood flour, palm oil, pineapple leaf and coir is the main natural fiber used as reinforcement (Rowell et al., 1997).

Kenaf fiber provides very high values of the hardness and strength. Moreover, it has a higher aspect ratio comparing to other types of fibers, so the using of it as a polymer composites reinforcements is favorable (Sanadi et al., 1995). Kenaf (*Hibiscus cannabinus*,

family *Malvaceae L.*) is a perennial plant herb and a plant heat-season annual lane. The attractive feature of kenaf fiber is reform, inexpensive, lightweight, high mechanical property and biodegradability. The main compounds in kenaf bast fiber are 15% lignin and 75% cellulose, which using of it is totally environmental friendly (Mansur & Aziz, 1983). In addition, Kenaf fiber has excellent flexural strength and tensile strength that sorts it a favorable candidate for wide range of applications (Aji et al., 2009).

However, the main drawbacks of kenaf fiber is the high moisture absorption, which have a negative impact on kenaf fiber's mechanical properties for composite materials (Dhakal et al., 2007). Moreover, compatibility issue between the hydrophilic kenaf fibers and the hydrophobic polymer matrices requires proper preparation and modification steps to increase and enhance the bonding between the fiber and the matrix (Gassan & Cutowski, 2000; Dhakal et al., 2007).

2.2 Natural Fibers

Natural fibers are obtained from several resources and they are categorized into many groups based on their origins i.e., from minerals, animals or plants, which is shown in Figure 2.1 (Ichhaporia, 2008). Searching for sustainable resources, makes natural fiber is the best alternative materials; especially they show no health hazards, cheap materials and solve a big environmental pollution issues by using these waste materials in new industrial applications. Furthermore, using natural fibers as a reinforced material in polymer composites provides a solution for substitute issue in order to make a new class of materials with a good potential in the future in structural applications.

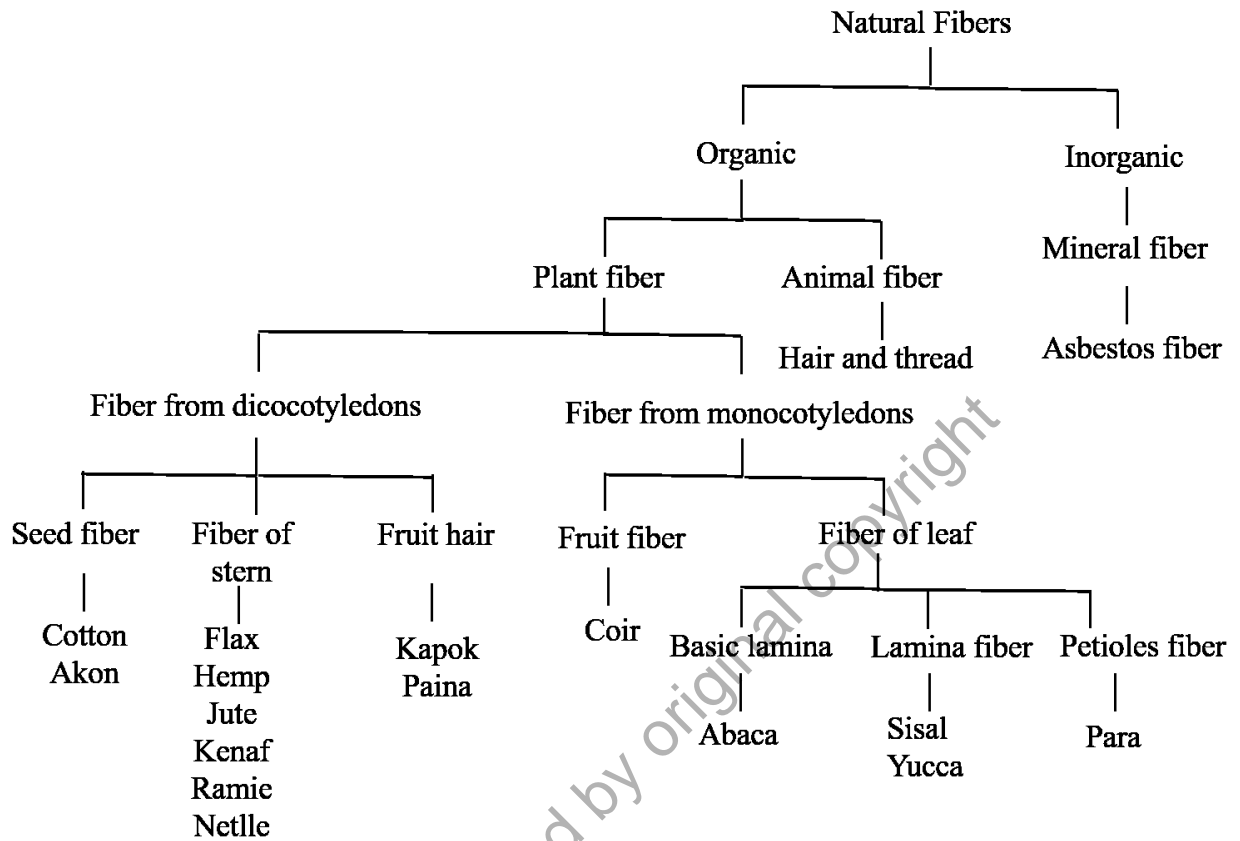


Figure 2.1: Classification of natural fibers (Ichhaporia, 2008)

Several fibers obtained from different types of plants such as banana, jute, sisal, coir, pineapple palmyra, hemp, talipot, etc, is being used as alternative sources for industrial material applications (Arun et al., 2010). Natural fibers can be extracted from different parts of the plants. Fibers are categorized into three main groups based the plant part that they are obtained. First group is the bast or stem fibers which can be extracted from different plants such as banana, mesta and jute. Second group is the leaf fibers which can be extracted from different plants such as screw pine, pineapple and sisal. Third group

is the fruit fibers which can be extracted from different fruits such as oil palm, coir and cotton.

Natural fibers properties rely generally on the plant nature, the plant's age, the location and area that it is grown in, and the method that is used to extract the fibers (Threepopnatkul et al., 2009).

2.2.1 Characteristics and Properties of Natural Fibers

Natural fibers, in general, is sensitive to mixing and forming tools, that can add a major reducing costs of equipment maintenance. As compared to synthesis fibers, natural fibers are safer to handle and process. Processing them is completely environmental friendly, which offers a better working environments and, therefore, minimizing the skin or respiratory problems.

The most remarkable advantage of the natural fiber is a positive environmental effect of them. Natural fibers are a sustainable resource, wherein they are biodegradable in nature. The main disadvantage of natural fibers as compared to that of synthetic fibers, is the achievement of uniformity, multi-dimensional, and their mechanical characteristics.

Fibers extracted from oil palm and coir are similar in term of their cellular structure and properties especially the hardness and toughness. The primary unit of a cellulose (in macromolecule structure) is anhydro-d-glucose, that has three alcohol hydroxyls (-OH) (Mohamed et al., 2013). The hydroxyls can be either form the hydroxyls between other cellulose molecules (intermolecular) or from the hydrogen bonds inside the molecule itself (intramolecular).

Thus, most of natural fibers are hydrophilic naturally; and they have moisture content about 8-13% (Amandine et al., 2013). However, cellulose and moisture is not the

only substances in natural fibers, they contain different other natural substances, i.e. lignin. The main roll of lignin is to bond the distinguished cells in the hard plant together, and to act as a cementing material. The structure, morphology and properties of plant fibers are influenced by lignin content.

Degree of polymerization (DP) is one of the main characteristic in natural fibers. DP differs in each cellulose molecules in the fiber, thus, the fiber is a mixture of a complex polymer chain of $(C_6H_{10}O_5)_n$. Among all the natural fibers, Bast fibers normally show the highest DP rate ($\sim 10,000$). Traditionally bast fibers have been used for different application such as ropes, packaging material, making twines, cords, as carpet-backing and as a geotextile material (Threepopnatkul et al., 2009).

2.2.2 Properties of Natural Fibers as a Filler in Polymer Composites

Bio-composites comprising of natural fibers indicate that the composites are comparatively poor water resistance, fiber/matrix interactions, and low durability. The main issue is the weaker adhesion bonds between natural fibers and polymer matrix, which are highly hydrophilic and hydrophobic, respectively. This issue is responsible for a major reduction in the composites properties and, thus, it limits their production and utilization in industrial applications. However, many attempts and trails have been done to overcome this drawback of compatibility, this includes using of coupling agents and/or numerous surface modification techniques. The surface treatment of the natural fibers can be achieved by different ways and means such as mechanical, physical and/or chemical techniques. The main key factor is to homogeneously distribute the fillers in the matrix and the alignment of these fillers in order to have substantial reinforcement and efficient properties.

Recently, nano-sized particles are taking in consideration by researchers all over the world as a high-potential filler material to improve the physical and mechanical properties of polymer composite materials. In general, nano-sized particle materials are defects free, therefore, the utilization of them as a filler in polymer can give a new prospect to overcome the restrictions usage of traditional micrometer sized particle materials. As a result of uniform and homogeneous distributions of nano-sized particle materials in the polymer matrices, enhanced properties of the polymer composites are expected, i.e. thermal properties, molecular mobility, and the mechanical properties. Nano-sized particle fillers exist in the minor zone while the micro particles in the plastic zone deformation. This can provide a solution for the matrix that has a brittle property issue by using nano-sized fillers to enhance and improve fracture and mechanical properties (Lee et al., 2009).

In order to improve the mechanical and physical properties of polymer composites, reinforcing the polymer matrices by a suitable filler is required. The most common and utilized reinforcing filler used in polymer composite materials is carbon blacks. Several other reinforcing fillers are used in polymer composites, including non-blacks such as calcium sulphate, aluminum silicate and silica. Several parameters generally influence the interaction between a given polymer matrices and the fillers, that have been given an extensive attention and studied extensively. These factors are the chemical composition of a given filler, particle size density of the filler and specific surface area. It has been found that using fine particles will give higher reinforcement properties. Moreover, the diluents of the fillers or impurities can cause a decrease in the mechanical and physical properties. The particle sizes also play a very important role, while it is generally ranging between 600 Å and above.

2.3 Challenge of Natural Fiber as a Reinforcement in Polymer Composite

The essential challenge happens during dealing with natural fibers, including kenaf fibers is the following: moisture absorption and interfacial adhesion. All natural fibers absorb moisture when immersed in water and in humid atmosphere especially in tropical countries. Absorption of moisture by natural fibers in composites can cause degradation and decomposition of fiber interface region resulting with poor stress relocation effectiveness and reduction of dimensional and mechanical properties. The major concerns of using natural fibers to reinforce polymer composites is to reduce their tendency to absorb moisture and the result of this moisture absorption on thermal, mechanical, and physical properties of the polymer composite materials. (Colom et al., 2003)

Therefore, it is necessary in order to consider natural fibers as an applicable reinforcement filler in composite materials to address and overcome the water absorption issue. Several studies have reported utilizing of natural fiber reinforced polymeric composites, the results indicate that using of coupling agents to treat the surface of natural fibers can reduce moisture absorption as well as enhancing certain thermal and mechanical properties for natural fiber and composite materials in general. Water and moisture diffuse into polymeric composites reinforced natural fibers in three various mechanisms. The first mechanism encompasses of penetration of water molecules ($\text{-H}_2\text{O}$) into the micro voids in between polymer molecules. The second mechanism encompasses a pathway movement inside the voids and rifts at the interfaces between natural fiber and polymer matrix. The third mechanism encompasses of penetration into micro cracks in the polymer matrix emerging from the swelling of fibers. (Papanicolaou et al., 2008) (Zainab Raheem. 2009).