CHEMICAL MODIFICATION AND CHARACTERIZATION OF KENAF FIBER

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This report is submitted as partial fulfillment of the requirements for the award of the Degree of the Bachelor of Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
Universiti Malaysia Pahang

APRIL, 2010

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Author : SITI KHADIJAH BINTI GHAZALI

Date : 30 APRIL 2010

My lovely family members,
My kindly friends,
Thank you & love you all.

ACKNOWLEGEMENT

Assalamualaikum. At first, I like to thank to Allah Almighty for the help and guidance when I have to face a lot of hard time in order to finish this research. Then, I would like to thank also to my supervisor Dr. Mohammad Dalour Hossen Beg for his advices and kindness when I need help and opinion.

I also like to dedicated my thank to the entire lab person that always be patient to fulfill my requirements especially to Madam Intan Shafinas Mohammed because she always help me when I have a problem about the materials for my experiments. In addition, thanks also to other lab persons because always be patient when they need to teach me how to use some equipment that I did not know.

Besides, my sincere thanks to my parents and my lovely sister (Siti Aishah Ghazali) because of their understanding and cares. Their courage and word always make me strong to face all the obstacles to finish this project. Last but not least, thanks also to all my group members that always give their hands when I have any problem with my research. Thanks also to my lovely friends especially for my best friends, Fitri Yanti and Nor Aini that always help me when I need them.

ABSTRACT

Composites produced by natural fiber as a reinforcement material were environmentally friendly since natural fiber can be biodegradable but it has some limitation because of their surface impurities. In this study, alkali treatment was performed on kenaf bast fibers in order to remove surface impurities. Grinding and without grinding kenaf bast fibers has been used. Kenaf bast fibers were immersed into 3%, 6%, and 9% concentrated sodium hydroxide (NaOH) solutions for 1 h, 3 h and 6 h. The effects of solution concentrations and treatment times on the mechanical properties of kenaf bast fibers were evaluated. Kappa test was achieved by using titration process and Scanning Electron Microscope (SEM) analysis of untreated and alkali treated kenaf bast fibers were carried out. Those results for Kappa test showed that the kenaf bast fibers treated with 6% NaOH at 3 h has lower kappa number and it showed that the lignin content was lower as well. Besides, results of scanning electron microscope analysis showed that the surface of fiber became cleaner after alkali treatment. In addition, composites reinforced by untreated and alkali-treated kenaf bast fibers were fabricated by using hot press method. Tensile tests were carried out for produced composites. Results showed that tensile strength of the composite using treated and without grinding fibers increased in comparison with the composite using without grinding untreated fibers and grinding and without grinding fibers.

ABSTRAK

Komposit yang dihasilkan daripada fiber semulajadi tidak akan menyebabkan pencemaran alam sekitar memandangkan fiber semulajadi mudah untuk dilupuskan. Rawatan menggunakan larutan alkali telah dilakukan ke atas kenaf fiber untuk tujuan mengubah struktur permukaan kenaf fiber. Dalam projek ini juga, terdapat dua jenis kenaf fiber yang telah digunakan iaitu kenaf fiber yang panjang dan kenaf fiber yang telah dihancurkan. Rawatan ini telah dijalankan dengan merendam kenaf fiber di dalam larutan natrium hidroksid (NaOH) dengan kepekatan yang berbeza iaitu 3%, 6%, dan 9% untuk masa yang berlainan iaitu 1 jam, 3 jam dan 6 jam. Kesan kepekatan dan masa yang berbeza ke atas kenaf fiber telah dinilai. Ujian Kappa dilakukan dengan menngunakan kaedah ‘titration’ dan ‘Scanning Electron Microscope’ ataupun SEM analisis juga turut dijalankan. Berdasarkan semua keputusan untuk ujian Kappa, kenaf fiber yang dirawat dengan menggunakan 6% NaOH untuk masa 3 jam mempunyai nilai kappa yang lebih rendah. Selain itu, keputusan untuk SEM pula menunjukkan permukaan kenaf fiber yang dirawat dengan larutan alkali lebih licin berbanding dengan permukaan kenaf fiber yang tidak dirawat. Kemudian komposit telah dihasilkan dengan menggunakan kenaf fiber yang dirawat dan telah dirawat sebagai penyokong melalui alat yang dikenali sebagai ‘hot press’. Ujian kekuatan keatas komposit yang terhasil telah dijalankan. Keputusan untuk ujian tersebut menunjukkan komposit yang dihasilkan dengan menggunakan kenaf fiber yang panjang dan dirawat mempunyai kekuatan yang lebih besar.

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LIST OF ABBREVIATIONS

NaOH	Natrium hydroxide
NaOCl	Sodium hypochlorite
H_2O_2	Hydrogen peroxide
OCl^-	Hypochlorite ion
Na^+	Sodium ion
AOX	Adsorbable organically bond halogens
HOO^-	Perhydroxyl anion
SEM	Scanning Electron Microscope
BSE	Backscattered electrons
DBSE	Diffracted backscattered electrons
KFI	Kenaf Fiber Industry
CH_3COOH	Acetic acid

KI	Potassium iodide
H_2SO_4	Sulphuric acid
$KMnO_4$	Potassium permanganate

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"I hereby acknowledge that the scope and quality of this thesis is qualified for the award of the Bachelor Degree of Chemical Engineering"

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Date : ________________________

CHAPTER 1

INTRODUCTION

1.1 Background of The Study

The growing global environmental concern, high rate of depletion of petroleum resources, as well as new environmental regulations have forced the search for new fiber reinforced composite materials that are compatible with the environment (Gaceva, Dekanski, Panic, Poleti, Grodanov, Buzarovska, Aavella, and Gentile, 2006). The classical composite materials manufactured with fiberglass as reinforcement present environmental problems at the end of their useful life. The alternative could be the use of vegetables fibers as reinforcement (Corrales, Llop, Mutjie, Vilaseca, and Gandini, 2002). Natural fibers represent environmentally friendly alternatives to conventional reinforcing fibers, due to the following advantages: they are abundantly available renewable resources; they are nontoxic; natural fibers used in polymer composites can lead to materials with high specific strength because of their low density (Gaceva, et al., 2006).

On the contrary, the major limitations of using these fibers as reinforcements in such matrices include poor interfacial bonding between hydrophilic natural fibers and hydrophobic polymer matrix (Gaceva, et al., 2006) and the high moisture absorption, which could result in dimensional changes of the fibers that may lead to micro-cracking of the composite and degradation of mechanical properties (Mohd Edeerozey, Akil, Azhar and Zainal Ariffin, 2006) due to the degradation of cellulose of the fibers (Corrales, et al, 2002). All of these drawbacks are caused by the surface impurities of the fiber. Thus, fiber surface modification methods have been developed to modify the surface fiber because mechanical properties are controlled to large extent by the efficiency of bonding at the fiber-matrix interface boundary. Besides, good adhesion at the interface could facilitate stress transfer from fiber to fiber across matrix and hydrophilic properties of natural fibers versus hydrophobic properties of plastics.

Surface modification has two methods which is chemical and physical treatment. The chemical modifications do not affect the body of the fiber and do not alter its structure. Only the outer surface of the grafted natural fibers obtained using this process is modified.

1.2 Problem Statement

In recent years, composites made of natural fibers have received increasing attention in light of the growing environmental awareness because of its biodegradable nature. However, the problems by using natural fiber which is kenaf bast fiber are lack of good adhesion between kenaf fiber and polymer matrices and low resistance to the moisture due to the surface impurities and hydrophilic nature of the fibers. These problems make the used of natural fiber as reinforcement material in composite become ‘gloomy’.

1.3 Research Objectives

The objective of this research is to remove surface impurities such as lignin and hemicelluloses in order to make kenaf bast fiber more hydrophobic by using alkali treatment in order to produce high strength of thermoset compoite.

1.4 Scopes of Study

The scopes of this research were

a) Preparation of the kenaf fiber
b) Treatment conditions of kenaf fiber
c) Characterization of treated kenaf fiber
d) Composite fabrication method
e) Characterization of thermoset composite produced.

1.5 Significant of Study

This study is conducted to investigate the effects of chemical modification on kenaf bast fiber by using NaOH solution. Alkali treatment is one of the methods to modified fibers surface in order to improve their characteristics as reinforcement in making the composite. This treatment will make the use of natural fiber like kenaf more attractive and effective in their many applications. Natural fiber also is economically and environmental friendly because of its biodegradable nature.

1.6 Expected Results

The elimination of amorphous non-cellulosic constituents (lignin and hemicelluloses) will increase the crystallinity, molecular alignment of cellulose, surface roughness of the fiber. Alkali treatment also can make fiber more hydrophobic in order to get good interfacial adhesion between fiber and thermoset polymer matrix.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter includes the literature review on the chemical modification and characterization of the kenaf fiber and the production of thermoset composite. The non-cellulose constituents on the kenaf fiber surface such as lignin and hemicellulose will lead to the production of low-quality of thermoset composite when this kind of fiber is combined to the thermosetting materials. Alkali treatment, by using sodium hydroxide will clean the surface of the fiber and enhanced performance of the thermoset composite.

2.2 Types of Fiber

Fibers or fibers are a class of hair-like materials that are continuous filaments or are in discrete elongated pieces, similar to pieces of thread (Wikipedia, 2010). They can be spun into filaments, thread or rope. They can be used as a component of composite materials. Fiber can be classified into two main groups, which are man-made fiber and natural fiber. In general, natural fibers can be subdivided as to their origin such as plants, animals, or minerals; while man-made fibers can be subdivided to synthetic and natural polymers (Muhammad, 2008).

2.2.1 Natural Fibers

Natural plant and animal fibers have provided the raw materials to meet our fiber needs. No matter which climatic zone humans settled they were able to utilize the fibers of native species to make products such as clothes, cloths, buildings and cordage. The first composite material known was made with clay and straw to build walls in Egypt 3,000 years ago. Many of the ancient plant fibers are no longer in use. Fibers such as jute, sisal, coir and kapok only started to be imported into Europe from the nineteenth century (History of Natural Fiber, 2010).

The use of natural fiber for the reinforcement of the composites has received increasing attention both by the academic sector and the industry (Taj, Munawar, & Khan, 2007). Natural fibers (Amar, Manjusri, and Lawrence, 2005**)** are generally lignocellulosic in nature, consisting of helically wound cellulose microfibrils in a matrix of lignin and hemicellulose. Currently, many types of natural fibers have been investigated for use in plastics including flax, hemp, jute straw, wood, rice husk, wheat, barley, oats, rye, cane (sugar and bamboo), grass, reeds, kenaf, ramie, oil palm empty fruit bunch, sisal, coir, water, hyacinth, pennywort, kapok, paper mulberry, raphia, banana fiber, pineapple leaf fiber and papyrus (Bledzki et al., 2007).

2.2.2 Synthetic Fiber

Synthetic fibers are the result of extensive research by scientists to improve upon naturally occurring animal and plant fibers. In general, synthetic fibers are created by forcing, usually through extrusion, fiber forming materials through holes (called spinnerets) into the air, forming a thread. Before synthetic fibers were developed, artificially manufactured fibers were made from cellulose, which comes from plants. These fibers are called cellulose fibers. The first artificial fiber, known as artificial silk, became known as viscose around 1894, and finally rayon in 1924 (Wikipedia, 2010).

A similar product known as cellulose acetate was discovered in 1865. Rayon and acetate are both artificial fibers, but not truly synthetic, being made from wood. Nylon, the first synthetic fiber, made its debut in the United States as a replacement for silk, just in time for World War II rationing. Its novel use as a material for women's stockings overshadowed more practical uses, such as a replacement for the silk in parachutes and other military uses (Wikipedia, 2010). Synthetic fibers are now available, ranging in properties from the highelongation and low-modulus elastomeric fibers, through the medium-elongation an medium-modulus fibers such as polyamides and polyesters, to the low-elongation, high modulus carbon, aramid and inorganic fibers (Hannant, 1989; Jusoh, 2008). The modern synthetic fiber that was made from older artificial materials and become the most common of all reinforcing fibers for polymer matrix composites is glass fiber (Agarwal and Broutman, 1990; Jusoh, 2008).

2.3 Main Component of Natural Fibers

Fiber is actually very complex. It is a combination of at least four major components which are distinctly different in chemical composition. These four major components are cellulose, hemicellulose, lignin, and pectin.

2.3.1 Cellulose

Cellulose is the substance that makes up most of a plant's cell walls. Since it is made by all plants, it is probably the most abundant organic compound on Earth. Aside from being the primary building material for plants, cellulose has many others uses. According to how it is treated, cellulose can be used to make paper, film, explosives, and plastics, in addition to having many other industrial uses. The paper in this book contains cellulose, as do some of the clothes you are wearing. For humans, cellulose is also a major source of needed fiber in our diet (Science, 2010).

Cellulose is usually described by chemists and biologists as a complex carbohydrate. Carbohydrates are organic compounds made up of carbon, hydrogen, and oxygen that function as sources of energy for living things. Plants are able to make their own carbohydrates that they use for energy and to build their cell wall. Since cellulose is the main building material out of which plants are made, and plants are the primary or first link in what is known as the food chain (which describes the feeding relationships of all living things), cellulose is a very important substance. It was first isolated in 1834 by the French chemist Anselme Payen (1795–1871), who earlier had isolated the first enzyme. While studying different types of wood, Payen obtained a substance that he knew was not starch (glucose or sugar in its stored form), but which still could be broken down into its basic units of glucose just as starch can. He named this new substance "cellulose" because he had obtained it from the cell walls of plants (Science, 2010).

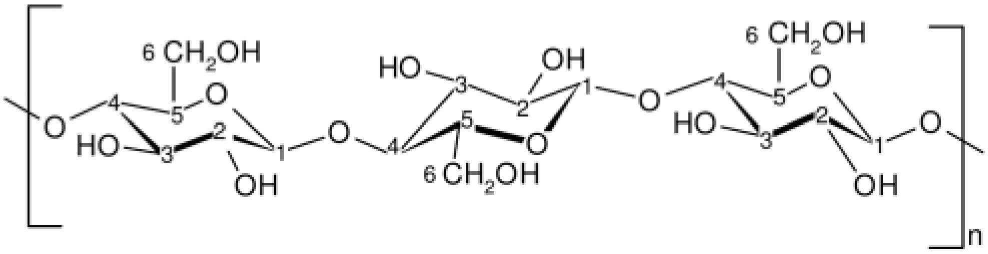


Figure 2.1: Cellulose structure (Cellulose, 2010)

Cellulose has a strong tendency to form intra- and inter-molecular hydrogen bonds by the hydroxyl groups on these linear cellulose chains, which stiffen the straight chain and promote aggregation into a crystalline structure and give cellulose a multitude of partially crystalline fiber structures and morphologies (Klemn, Heublein, Fink, and Bohn, 2005).

2.3.2 Hemicellulose

Hemicellulose is a polysaccharide composed of a variety of sugars including xylose, arabinose, mannose. Hemicellulose that is primarily xylose or arabinose are referred to as xyloglucans or arabinoglucans, respectively. Hemicellulose molecules are often branched. Like the pectic compounds, hemicellulose molecules are very hydrophilic. They become highly hydrated and form gels. Hemicellulose is abundant in primary walls but is also found in secondary walls (Plant Cell Walls, 2010).

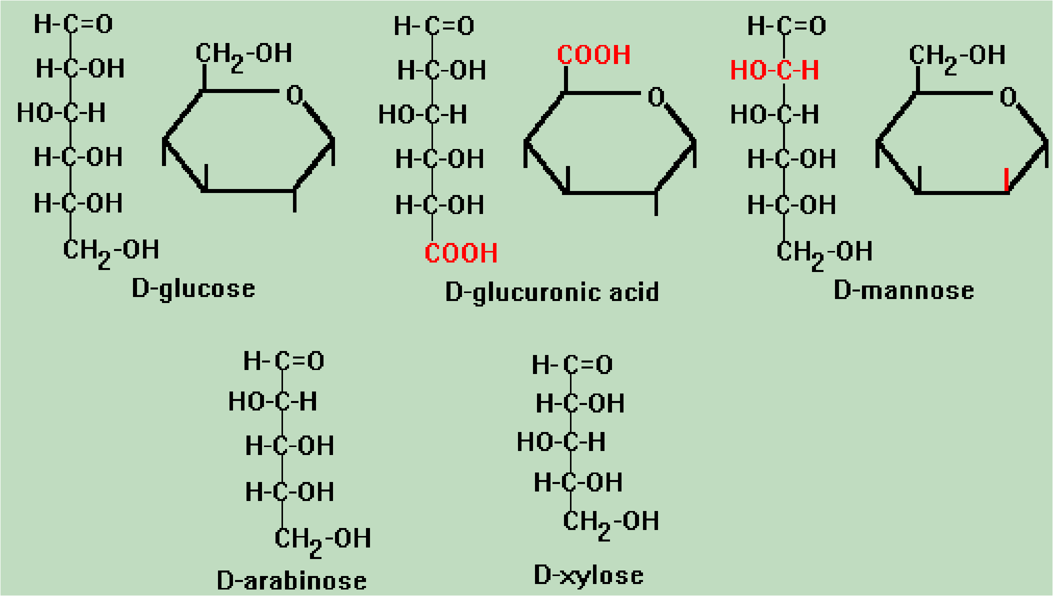


Figure 2.2: Some monomer of hemicelluloses (Hemicellulose, 2010).

Usually, all of the pentoses are present. There may even be small amounts of L- sugars. Note that there are hexoses as well as acids formed by oxidation of sugars. Mannose and mannuronic acid tend to be present, and there can be galactose and galacturonic acid. The groups that are different from glucose are shown in red on the six-carbon structures. The pentoses are also present in rings (not shown) that can be 5-membered or 6-membered. Xylose is always the sugar present in the largest amount (Hemicellulose, 2010).

In contrast to cellulose that is crystalline, strong, and resistant to hydrolysis, hemicellulose has a random, amorphous structure with little strength. It is easily hydrolyzed by dilute acid or base, but nature provides an arsenal of hemicellulase enzymes for its hydrolysis. These enzymes are commercially important because they open the structure of wood for easier bleaching, and older methods of bleaching consume larger amounts of chemicals such as chlorine that are bad for the environment (Hemicellulose, 2010).

2.3.3 Lignin

Lignin or lignen is a complex chemical compound most commonly derived from wood, and an integral part of the secondary cell walls of plants (Martone, Estevez, Lu, Ruel, Denny, Somerville, & Ralph, 2009; Wikipedia, 2010). The term was introduced in 1819 by de Candolle and is derived from the Latin word *lignum* (Sjöström, 1993).

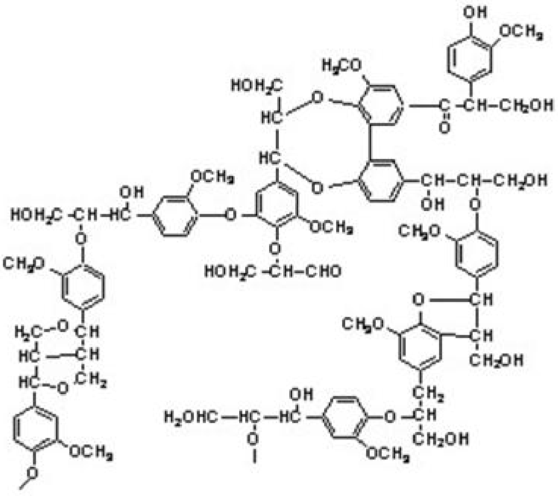


Figure 2.3: Structure of the lignin (Lignin, 2010).

Lignin can be classified in several ways but they are usually divided according to their structural elements. All plants lignin consist mainly of three basis building blocks of guaiacyl, syringyl, and p-hydroxyphenyl moieties, although other aromatic type units also exist in many different types of plants, which forms a randomized structure in a tri-dimensional network inside the cell walls (Xiao et al., 2001; James and Jeffrey, 1997; Jusoh, 2008). The function of the lignin in plants is as an encrusting agent in the cellulose/hemicelluloses matrix or called plant cell wall adhesive. Therefore, lignin acts as a structural support material in plants by filling the spaces between the polysaccharide fibers, which hold the natural structure of the plant cell walls together (Sain and Panthapulakkal, 2004; James and Jeffrey, 1997; Jusoh, 2008).

2.4 Bast and Core Fiber

Bast fibers exist in the inner bark or phloem of many dicotyledonous plants to provide structural rigidity to the stems. These fibers occur in bundles which run parallel to the stems between nodes. The fiber strands are composed of many smaller cells termed ultimate fibers. Just inside the phloem is a wood-like core material consisting of short and fine fibers (Timothy and Michael, 1997; Jusoh, 2008).

According to Alexander et al. (2005), the shape and size of the stem of various bast fiber crops are different but they all contain varying amount of fiber cells in the phloem. Long individual fiber or long fibers bundles can be obtained from many bast fiber crops at relatively low cost. However, bast fibers have a non-homogeneous cell structure than do the much shorter wood fibers which are uniform, readily available and inexpensive (Jusoh, 2008).

Bast fibers have been used for generations to produce textiles and are removed from the phloem by a controlled decay and separation process called retting. In general, the core material is unused after the outer bast fibers are removed (Timothy and Michael, 1997; Jusoh, 2008). Bast fiber crops have rigid herbaceous stalks containing nodes at regular intervals that are fluted or channeled. From the inside to outside the stalks have a hollow core, except at joints, followed by the pith and finally the protective layer epidermis (Alexander et al., 2005; Jusoh, 2008).

2.5 Kenaf Bast Fiber

Kenaf, *Hibiscus Cannubinus* (Figure 1), is a plant from Malvaceae family. It is annual or biennial herbaceous plant (rarely a short-lived perennial) growing to 1.5-3.5 m tall with a woody base. The stems are 1-2 cm diameter, often but not always branched. Kenaf has a long history of cultivation for its fiber in India, Bangladesh, Thailand, parts of Africa, and to a small extent in southeast Europe. Kenaf fibers (Figure 2) have significant amount of amorphous lignin (15-19%) and amorphous hemicellulose (22-23%) along with crystalline cellulose (44-57%) (Sanadi & Caulfield, 2008). The fibers are made up of cellulose microfibrils bonded together by lignin. Hemicellulose is very hydrophilic, soluble in alkali, and easily hydrolyzed in acids while lignin is not hydrolyzed by acids, but soluble in hot alkali (John & Anandjiwala, 2009).

Kenaf fiber can be divided into two types: kenaf bast fiber and kenaf core fiber where kenaf bast fibers have thicker walls compared to the core fibers (Rowell and Stout, 2009). Kenaf bast fiber has high potential as a reinforcing fiber in thermoplastic composites because of its superior toughness and high aspect ratio in comparison with other fibers. The bast consists of a woody core surrounded by a stem. The stem consists of a number of fiber bundles, each containing individual fiber cells or filament like fibers (Rowell et al., 2009). Kenaf bast fiber is a composite made up of a crystalline, thermoset polymer matrix (lignin and the hemicellulose). Besides, kenaf, an annual hibiscus fiber plant, has been found to be an important source of fiber for composites and other industrial applications. A single fiber of kenaf can have a tensile strength and modulus as high as 11.9 GPa and 60 GPa respectively (Karnani, Krishnan, and Narayan, 2009). So, combining kenaf fiber with the thermoset material provides a strategy for producing advanced thermoset composite that take advantage of the properties of types of materials (Gaceva et al., 2009).

Figure 2.4: Kenaf plant (*Hibiscus Cannubinus*)

Figure 2.5: Kenaf bast fiber

2.6 Surface Treatment of Natural Fiber

2.6.1 Alkali Treatment

Chemical modification of the kenaf fiber can be done by using acid or alkali treatment. However, this study only focused on alkali treatment since kenaf fiber more sensitive in acidic solution and one of the objectives of this study is to remove hydroxyl group from the kenaf fiber. Roger M. Rowell reported that if hydroxyl reactivity is selected as the preferred modification site, the chemical must contain functional groups which will react with the hydroxyl groups of the lignocellulosic components. Alkali treatment is also called as mercerization treatment and can be achieved using sodium hydroxide or liquid ammonia. Sodium hydroxide treatment of cellulose fibers leads to the irreversible mercerization effect, which increases the amount of amorphous cellulose at the expense of crystalline cellulose (Beg, 2007). Mercerization improves adhesion, characteristics by removing surface impurities, thus exposing micro-fibrils, which then render the fiber topography with a rough texture. The rough and cleaned surface facilitates mechanical adhesion in addition to improved wetting ability of the resin (Beg, 2007).

This treatment is believed as an effective way to eliminate the impurities especially lignin and hemicelluloses. The treatment consists on dissolving lignin and hemicelluloses using aqueous solution in order to recover cellulose fibers. The process is less harmful and does not attack the fibers mechanically (Zbidi, Sghaier, Nejma and Zidi, 2009). The removal of surface impurities also gives advantageous for fiber-matrix adhesion as it facilitates both mechanical interlocking and the bonding reaction at the interface (Gaceva et al., 2006). Alkali treatment also makes the fiber more hydrophobic as well to improve the compatibility between the fiber and the matrix materials and it is prove by Rowell et al. The following reaction takes place as a result of alkali treatment (Rowell et al., 2009).

$$\text{Fiber} - \text{OH} + \text{NaOH} \longrightarrow \text{Fiber} - \text{O}^-\text{Na}^+ + \text{H}_2\text{O} \quad (2.1)$$

Natural fibers are amenable to chemical modification due to the presence of hydroxyl groups. The hydroxyl groups may be involved in the hydrogen bonding within cellulose molecules, thereby activating these groups or can introduce new moieties that form effective interlocks within the system (Rowell et al., 2009). The effect of alkali on cellulose fiber is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes (Rowell et al., 2009). So, this treatment results in freeing the hydrogen bonds making them more reactive and giving some porous fibers. It generates the increase of the void content in fibers, the improvement the wettability and the fiber-matrix contact (Jonoobi, Harun, Shakeri, Misra, & Oksman, 2009). Alkali treatment also can improve UV resistance by removing lignin, could also increase the number of reactive hydroxyl groups (-OH) on the fiber surface available for chemical bonding, thus improving composite strength (Zbidi, et al., 2009).

Based on Roger M. Rowell studied, agro-based composites exposed outdoors undergo photochemical degradation caused by ultraviolet light. This degradation takes place primarily in the lignin component, which is responsible for the characteristic color changes. The lignin acts as an adhesive in the cell walls, holding the cellulose fibers together. The surface becomes richer in cellulose content as the lignin degrades. In comparison to lignin, cellulose is much less susceptible to ultraviolet light degradation. After the lignin has been degraded, the poorly bonded carbohydrate-rich fibers erode easily from the surface, which exposes new lignin to further degradative reactions.

2.7 Bleaching

Natural fiber can be bleach and used for various types of composite fabrication. Bleaching of kenaf results in white fibers with a significant decrease in tenacity (Ting, 2003). Bleaching agents are compounds which are used to remove color from substances such as textiles. In earlier times textiles were bleached by exposure to the sun and air. Today most commercial bleaches are oxidizing agents, such as sodium hypochlorite (NaOCl) or hydrogen peroxide (H_2O_2) which is quite effective in "decolorizing" substances via oxidation.

2.7.1 Sodium Hypochlorite

Sodium hypochlorite solution, commonly known as bleach, is frequently used as a disinfectant or a bleaching agent. Sodium hypochlorite (NaOCl) is a chemical compound consisting of sodium, oxygen, and chlorine that has been used for centuries for bleaching and disinfecting. Today, sodium hypochlorite (commonly called chorine bleach) is mass produced by the chlorination of soda ash and is employed in many household products, including laundry bleaches, hard surface cleaners, mold and mildew removers, and drain cleaners. Sodium hypochlorite is the salt formed by a negatively charged hypochlorite ion (OCl^-) and a positively charged sodium ion (Na^+). Pure hypochlorite is highly reactive and unstable; therefore, it is usually supplied as a dilute aqueous solution. In solution, hypochlorite eventually decomposes to yield a variety of byproducts including oxygen, chlorine gas, and salt. One of these byproducts, hypochlorous acid, is a powerful oxidizing agent (meaning it can accept electrons from other materials) that lends hypochlorite excellent bleaching and disinfecting abilities. The term "available chlorine" is often used to describe the concentration of hypochlorous acid in solution (which provides a measure of the solution's oxidative ability).

2.7.2 Hydrogen Peroxide

Hydrogen peroxide is a relatively cheap and powerful oxidizing agent. Hydrogen peroxide is available in various concentrations, including 6 %, 30 % and 50 %. The 6% hydrogen peroxide is available in pharmacy stores, which is commonly used as a disinfectant. Hydrogen peroxide is a good oxidizing agent and it is environmentally friendly. Hydrogen peroxide is widely used as an oxidative bleaching agent in the pulp bleaching process. Recently, its use in pulp bleaching has increased in order to reduced AOX (adsorbable organically bond halogens) for environmental reasons. Hydrogen peroxide produces the perhydroxyl anion (HOO) through ionization or radical reaction mechanism (Byoung, Ryuichiro, Kokki, & Ki, 2001).

2.8 Fiber Characterization

After the treatment process, the treated kenaf fiber will be undergoing chemical analysis process in order to determine the lignin content extracted from the fiber by using alkali treatment. One of the analysis processes is Kappa Test in order to calculate the Kappa number. The effects of the alkali solution on the chemical compositions on the surface of kenaf fiber also can be done by using Scanning Electron Microscopy (SEM) (Jonoobi, et al., 2009).

2.8.1 Kappa Test

The Kappa number is an indication of the residual lignin content or bleachability of wood pulp by a standardized analysis method. The Kappa number is a measurement of how much a standard potassium permanganate solution that is consumed by the pulp.

2.8.2 Scanning Electron Microscope (SEM)

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties (Swapp, 2010).

Accelerated electrons in an SEM carry significant amounts of kinetic energy, and this energy is dissipated as a variety of signals produced by electron-sample interactions when the incident electrons are decelerated in the solid sample. These signals include secondary electrons (that produce SEM images), backscattered electrons (BSE), diffracted backscattered electrons (DBSE that are used to determine crystal structures and orientations of minerals), photons (characteristic X-rays that are used for elemental analysis and continuum X-rays), visible light (cathodoluminescence--CL), and heat. Secondary electrons and backscattered electrons are commonly used for imaging samples: secondary electrons are most valuable for showing morphology and topography on samples and backscattered electrons are most valuable for illustrating contrasts in composition in multiphase samples (for rapid phase discrimination) (Swapp, 2010).

2.9 Types of Composite

Composite materials (or composites for short) are engineered materials made from two or more constituent materials with significantly different physical or chemical properties which remain separate and distinct on a macroscopic level within the finished structure. Composites are made up of individual materials referred to as constituent materials. There are two categories of constituent materials: matrix and reinforcement. At least one portion of each type is required. The matrix material surrounds and supports the reinforcement materials by maintaining their relative positions. The reinforcements impart their special mechanical and physical properties to enhance the matrix properties. A synergism produces material properties unavailable from the individual constituent materials, while the wide variety of matrix and strengthening materials allows the designer of the product or structure to choose an optimum combination (Composite, 2010).

Engineered composite materials must be formed to shape. The matrix material can be introduced to the reinforcement before or after the reinforcement material is placed into the mold cavity or onto the mold surface. The matrix material experiences a melding event, after which the part shape is essentially set. Depending upon the nature of the matrix material, this melding event can occur in various ways such as chemical polymerization or solidification from the melted state (Composite, 2010).

2.9.1 Thermoset Composite

Thermoset composite materials have the advantage of having extremely high strength and stiffness at relatively low cost when compared with thermoplastics. Before this, each thermoset composite consists of a reinforcement material such as glass fiber or woven cotton that has been impregnated with a plastic resin such as phenolic or epoxy. The resulting composite has unique properties depending on the resin-reinforcement combination selected. Thermoset materials are generally stronger than thermoplastic materials and are also better suited to high-temperature applications up to the decomposition temperature. The common types of thermoset material are epoxy resin, polyester, and vinyl ester. However, epoxy resins are used as a matrix in a large number of polymer-matrix composites due to the large number of compounds that can react with the epoxy ring to form resin systems with a very wide range of properties (Kanchanomai, Rattananon, & Soni, 2005). Epoxy resins have performance advantages over polyester and vinyl esters such as better adhesive properties (the ability to bond to the reinforcement or core) and superior mechanical properties (particularly strength and stiffness).

2.9.2 Thermoplsatic Composite

Thermoplastic composite materials, like thermosetting polymer composites, frequently suffer from a lack of fiber-matrix adhesion. This is typically remedied using fiber surface modification. Chemical modification or the addition of a third, compatibilizing phase bridging the fiber and matrix phases has been successfully applied to improving the interfacial characteristics of many polymer composite systems. Another problem encountered during the production of thermoplastic composites is quality of consolidation. Hot pressing is typically employed to obtain a uniform distribution of matrix material around the reinforcing fiber, remove voids, as well as bring about sufficient contact between the fiber and matrix. Compared with thermosets, composites fabricated from thermoplastic materials typically have a longer shelf life, higher strain to failure, are faster to consolidate and retain the ability to be repaired, reshaped and reused as need arises (Thermoplastic composite materials, 2010).

2.10 Composite Fabrication

In general, the reinforcing and matrix materials are combined, compacted and processed to undergo a melding event. After the melding event, the part shape is essentially set, although it can deform under certain process conditions. For a thermoset polymeric matrix material, the melding event is a curing reaction that is initiated by the application of additional heat or chemical reactivity such as organic peroxide. For a thermoplastic polymeric matrix material, the melding event is a solidification from the melted state. For a metal matrix material such as titanium foil, the melding event is a fusing at high pressure and a temperature near the melt point.

For many molding methods, it is convenient to refer to one mold piece as a "lower" mold and another mold piece as an "upper" mold. Lower and upper refer to the different faces of the molded panel, not the mold's configuration in space. In this convention, there is always a lower mold, and sometimes an upper mold. Part construction begins by applying materials to the lower mold. Lower mold and upper mold are more generalized descriptors than more common and specific terms such as male side, female side, a-side, b-side, tool side, bowl, hat, mandrel, and so on (Moulding Methods, 2010).

2.10.1 Hot Press

Like RTM, two matching metal molds are heated. Instead of dry reinforcement, prepregs or pre-impregnated preforms are used. Prepregs are fabrics that are pre-impregnated with resin (sometimes resin and fillers) and treated with temperature in such ways that are partially cured (the so called “B stage” of cure). When reheated in hot press molding, the resin becomes liquid again, and finally cures (Composite, 2010). Hot pressing is a high-pressure, low-strain-rate powder metallurgy process for forming of a powder or powder compact at a temperature high enough to induce sintering and creep processes (Wikipedia, 2010). This is achieved by the simultaneous application of heat and pressure. Hot pressing is mainly used to fabricate hard and brittle materials (Hot pressing, 2010).

2.10.2 Compression Molding

A method of molding in which the molding material, generally preheated, is first placed in an open, heated mold cavity. The mold is closed with a top force or plug member, pressure is applied to force the material into contact with all mold areas, and heat and pressure are maintained until the molding material has cured. The process employs thermosetting resins in a partially cured stage, either in the form of granules, putty-like masses, or preforms. Compression molding is a high-volume, high-pressure method suitable for molding complex, high-strength fiberglass reinforcements. Advanced composite thermoplastics can also be compression molded with unidirectional tapes, woven fabrics, randomly orientated fiber mat or chopped strand. The advantage of compression molding is its ability to mold large, fairly intricate parts. Compression molding produces fewer knit lines and less fiber-length degradation than injection molding (Composite molding, 2010).

2.11 Composite Characterization

The ultimately most interesting properties are after all those of the finished composite. The relevant properties may be physical, mechanical, thermal, optical, electrical, and environmental in nature. Assuming that fibers and matrix have appropriately selected to ensure that the composite tolerates the general environment it is intended for, the most relevant properties for structural composites are the mechanical ones.

2.11.1 Tensile Testing

Tensile test are performed for several reasons. The results of tensile tests are used in selecting materials for engineering applications. Tensile properties frequently are included in material specifications to ensure quality. Tensile properties often are measured during development of new materials and processes, so that different materials and processes can be compared. Finally, tensile properties often are used to predict the behavior of a material under forms of loading than uniaxial tension. The strength of a material often is the primary concern (Davis, 2004). Strength refers to the ability of a structure to resist loads without failure because of excessive stress or deformation (Gedney, 2005).

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter include the methodology applied in order to carry out the experiment and characterization for determine the effect of alkali treatment on the kenaf bast fiber performance. This chapter also includes the procedure to produce thermoset composite and characterization. This experiment will be carried out at Basic Sciences Lab in Chemical Engineering Lab.

3.2 Materials and Equipments

The main materials that have been used in this experiment are long unbleached kenaf bast fiber as a reinforcement material to produce thermoset composite. The kenaf bast fiber was ordered from one company in Kelantan called Kenaf Fiber Industry (KFI) with 10 kg in quantity and the price was RM110.

Besides that, epoxy resin and its hardener ordered from Leco Company. Other chemicals that have been used for surface modification of kenaf bast fiber were sodium hydroxide (NaOH) and 30% of hydrogen peroxide (H_2O_2). For washing process, 1% of acetic acid (CH_3COOH) was used. In fiber characterization process, the chemicals that have been used were sodium thiosulphate ($Na_2S_2O_3$), potassium iodide (KI), sulphuric acid (H_2SO_4) and starch indicator. Tensile testing machine, Scanning Electron Microscope (SEM) machine, waterbath, heater and stirrer and grinder machine were among the equipment that has been used to finish this study.

3.3 Method

3.3.1 Preparation of Fiber

One of the scopes of this project is to study the effect of types of fiber which is grind and without grind fiber on the composite performance. So, for the grind fiber preparation process, 180 g of kenaf bast fiber was grinded to small size by using grinder. After the grinding process, the grinded fiber was sieved to separate undesired particles that can affect the performance of the thermoset composite. The sieving process used a lot of time since it was done manually. Besides, for the without grinding kenaf bast fiber the process was same with grinding sample which is I have to choose and separate the undesired particles for the same objective.

Figure 3.1: Grinding fibers

3.3.2 Alkali Treatment

In this study, the alkali treatment was done by using different concentration of sodium hydroxide (NaOH) solution which is 3%, 6% and 9%. In order to achieve that desired concentrations, I have to make some calculation. Based on the calculation, I got the weight of the NaOH that I need to prepare all the desired concentration of NaOH solution. The solutions were prepared in 500 ml conical flask. When the solution was prepared, I have to weight 20 g of sample (kenaf bast fiber) for each NaOH solution and the waterbath was heated until the temperature reached 70 °C at the same time. Since all the materials were done, the alkali treatment process started. The alkali treatment was carried out by immersed the samples into the 250 ml 3%, 6%, and 9% NaOH solution at the same time. The conical flasks were put into the heated waterbath for 1 hour. After that period, the conical flasks were taken out from the waterbath and the samples were washed by using deionized water and the washed samples were let to be dry at room temperature. The experiment was repeated for without grinding samples and at different time which is 3 hour and 6 hour.

3.3.3 Bleaching process

Bleaching process was done by using 2% of hydrogen peroxide (H_2O_2) solution for 6 hour. Since our lab only has 30% of hydrogen peroxide, I have made dilution process of the solution in order to get the desired concentration. The 2% hydrogen peroxide solution was prepared in 250 ml volumetric flask. After all the materials prepared, bleaching process started with the grind samples. Same with alkali treatment process, samples were immersed in the 250 ml hydrogen peroxide solution in the 500 ml conical flask. The experiment was carried out in waterbath at temperature 50 °C. The sample was taken out from the waterbath after 6 hour and followed by washing process by using deionized water. The washed samples were let to dry at room temperature for further process. The experiment is repeated for the without grinding samples.

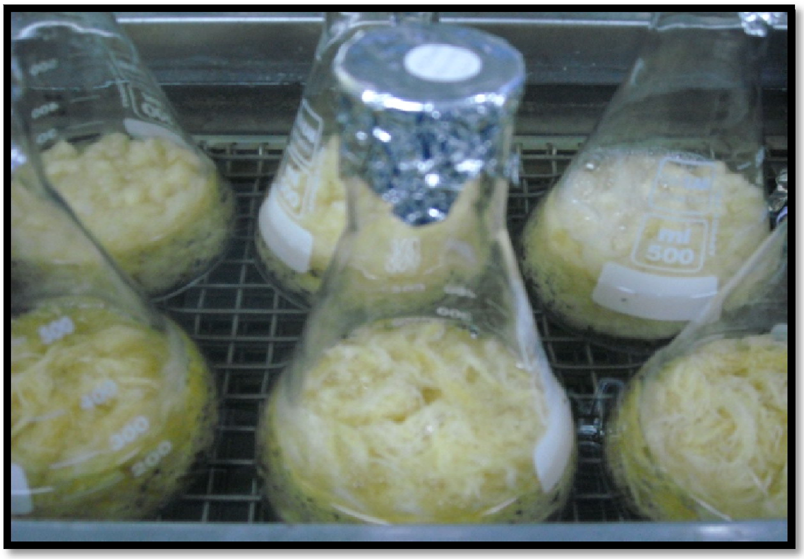


Figure 3.2: Bleaching process

3.3.4 Washing process

In the fiber washing process after the alkali treatment and bleaching process, 1% of acetic acid (CH_3COOH) was used. Our lab has acetic acid with 98% in purity. So, to get 1% of acetic acid, dilution process was carried out in the 250 ml of volumetric flask. In order to wash the samples, each sample was immersed in the 250 ml of acetic acid solution for almost 5 minutes in 500 ml of conical flask. After 5 minutes, the samples were taken out from the conical flask and washed it again by using deionized water and let it dried at room temperature. This process was repeated with without grinding samples.

Figure 3.3: Washing and drying process

3.4 Fiber Characterization

The treated kenaf bast fiber was undergoing some characterization process in order to evaluate the effects of alkali treatment on the fiber. The untreated sample also goes through the same characterization process for comparison.

3.4.1 Kappa Test

The content of lignin in the fiber before and after the treatment was achieved by determining the Kappa number by using titration method. In order to determine the kappa number, I have to prepare some solution which is 0.1 N potassium permanganate ($KMnO_4$), 4.0 N sulphuric acids (H_2SO_4), 1.0 N potassium iodide (KI), 0.2 N sodium thiosulphate ($Na_2S_2O_3$) and 0.2 N of starch indicator. Some calculation was made in order to know the required weight of the solid chemicals to prepare the needed normality of the materials. The solution of the solid chemicals was carried out in the 500 ml of conical flask. Besides, the dilution process of sulphuric acids was carried out in the 250 ml of volumetric flask. In the 100 ml and 0.2% of starch indicator preparation, 1 g of soluble starch was weighted before poured it in 100 ml of boiled deionized water.

The prepared starch indicator was cooled at room temperature before the experiment started. Then, weighted 0.5 g of each sample and put it into the 500 ml beaker before poured it with 300 ml of deionized water. The magnetic stirrer was put into the same beaker (reaction beaker) and the stirrer process started. At the same time, 50 ml of 0.1 N potassium permanganate and 4.0 N sulphuric acid solutions was measured. After that, both solutions were mixed together into the same beaker before transferred them into the reaction beaker. The beaker of the mixing solution was rinsed with 100 ml deionized water and then the rinsed water was poured into the reaction beaker. The temperature of the reaction solution was measured. After 10 minutes of reaction, 10 ml 1.0 N potassium iodide solution was added into the reaction beaker in order to stop the reaction.

Then, the free iodide was titrated with 0.2 N sodium thiosulphate solutions. A few drops starch indicator is added into the reaction beaker when the color of the solution was changed to light yellow. The titration process was stopped when the color of the solution in the reaction beaker becomes colorless. The volume of 0.2 N sodium thiosulphate solutions used for the titration process was measured. This experiment was repeated for blank solution with the same method but without using kenaf fiber.

3.4.2 Scanning Electron Microscope

Scanning electron microscope (SEM) analysis was conducted in order to evaluate the surface morphology of the kenaf bast fiber after and before alkali treatment. The characterization process was achieved by using SEM equipment (ZEISS, EVO 50 with excitation energy of 8.00 kV)

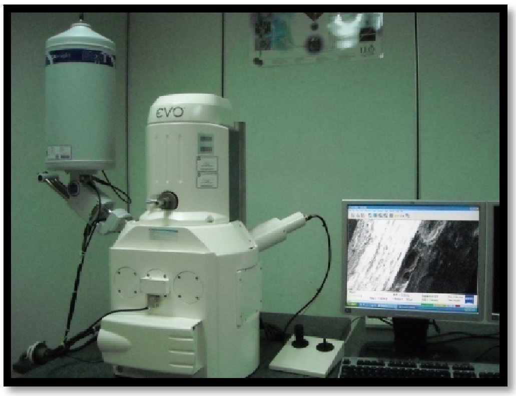


Figure 3.4: Scanning Electron Microscope

3.5 Composite Fabrication

Thermoset composite was fabricated by using hydraulic hot press machine. The temperature of the hot press was set at 90 °C. The fabrication process was started by arranged the fiber like a mat and then put the mat of fiber into the mold. After the mat of fiber was prepared, the volume of required epoxy resin and its hardener was measured based on the volume ratio which is 4:1. When the mixture of epoxy resin and its hardener was ready, poured it onto the mat of fiber on the mold. Then, the mold was bringing into the hot press machine and the fabrication process started. The mold was let in the machine for about half an hour. After that period of time, stop the hot press machine before took the mold from it and the process to take out the composite from the mold was started.

3.6 Composite Characterization

3.6.1 Tensile Testing

Characterization of thermoset composites produced was performed by using tensile testing equipment in Chemical Engineering Lab. In order to achieve the result for this process, the thermoset composite that was taken out from the mold was cut into dumbbell shape. Then, the width and the thickness of the sample were determined at some points by using electronic digital caliper. Width was measured as the distance between the cutting edges of the die at the narrow section while the thickness of the specimen was measured at different parts of the specimen before the average value was taken.

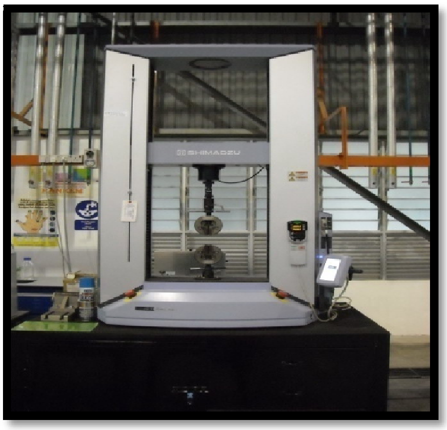
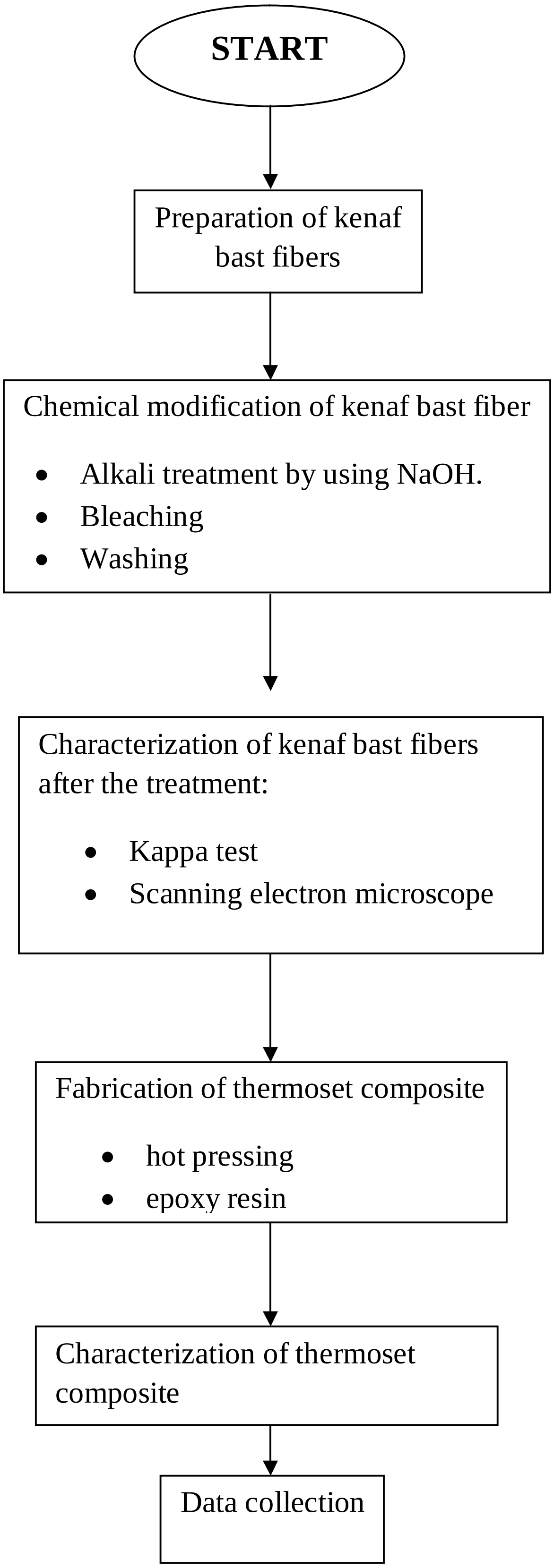


Figure 3.5: Tensile testing equipment

3.7 Experimental Procedures



CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Introduction

This chapter consists of the results and discussions for the alkali treatment effect on the kenaf bast fiber. The experiment was conducted in order to determine the effect of chemical treatment on the surface of the kenaf bast fiber. Then, the thermoset composite was produced by using treated and untreated kenaf bast fiber the strength of the composites were evaluated in order to know the effect of the treated fiber on the performance of composites produces. Besides that, the tensile testing also conducted to evaluate the effect of grinding and without grinding kenaf bast fiber on the thermoset composites.

4.2 Fiber characterization

Fiber characterization process was conducted by through Kappa test (titration method) and Scanning Electron Microscope (SEM).

4.2.1 Kappa Test

Kappa number was determined by using titration process which the volume of sodium thiosulphate solution used to titrate the potassium permanganate solution in the reaction beaker was measured.

i. For blank solution
   T_1 = 29 °C
   V_1 = 0 mL
   V_2 = 64.7 mL

ii. For untreated sample
   T_1 = 29 °C
   V_1 = 0 mL
   V_2 = 26.0 mL

The table below showed the volume of sodium thiosulphate used by the specimen during the titration process for treated samples:

i. Grinding samples

Table 4.1: Volume of 0.2 N $Na_2S_2O_3$ (ml) used in titration process on treated fiber

Concentration (%)	**Time (hr)**	**Volume of 0.2 N $Na_2S_2O_3$ (ml)**
3	1	32.3
	3	35.9
	6	33.7
6	1	35.4
	3	37.6
	6	36.1
9	1	35.3
	3	35.0
	6	35.6

ii. Without grinding samples

Table 4.2: Volume of 0.2 N $Na_2S_2O_3$ (ml) used in titration process on treated fiber

Concentration (%)	**Time (hr)**	**Volume of 0.2 N $Na_2S_2O_3$ (ml)**
3	1	37.1
	3	30.3
	6	36.8
6	1	33.6
	3	36.6
	6	33.8
9	1	38.9
	3	39.9
	6	35.3

Based on the volume of the sodium thiosulphate in both tables, the value of kappa number was calculated by using some equation. Those equations are:

$$k = \frac{p \, x \, f}{w} \tag{4.1}$$

$$p = \frac{(b-a)N}{0.1} \tag{4.2}$$

$$k = \frac{p \, x \, f}{w} \, [1 + 0.013(25 - t)] \tag{4.3}$$

From the calculation by using those equations, the kappa numbers were:

i. Grinding samples

Table 4.3: Kappa number values for grinding samples

Concentration (%)	**Time (hr)**	**Volume of 0.2N $Na_2S_2O_3$ used (ml)**	**Kappa number**
3	1	32.3	128.7
	3	35.9	111.1
	6	33.7	117.3
6	1	35.4	114.8
	3	37.6	102.3
	6	36.1	107.1
9	1	35.3	108.9
	3	35.0	110.1
	6	35.6	109.1

ii. Without grinding samples

Table 4.4: Kappa number values for without grinding samples

Concentration (%)	**Time (hr)**	**Volume of 0.2N $Na_2S_2O_3$ used (ml)**	**Kappa number**
3	1	37.1	105.8
	3	30.3	134.1
	6	36.8	104.2
6	1	33.6	122.0
	3	36.6	105.1
	6	33.8	116.9
9	1	38.9	98.2
	3	39.9	92.8
	6	35.3	110.5

Kappa test was conducted by using titration method which is the amount of sodium thiosulphte solution used to titrate the potassium permanganate solution was measured. In this titration process, firstly 0.5 g of treated and untreated of kenaf bast fiber was put into the beaker that contains 300 ml of deionized water. Then, the mixture of potassium permanganate and sulphuric acids solution were transferred into the same beaker. The solution in the beaker was stirred by using magnetic stirrer and reaction was let to occur for almost 10 minutes. After 10 minutes passed, 10 ml of potassium iodide was added to the beaker in order to stop the reaction. A few drops of prepared starch indicator were added when the color of the solution in the beaker changed to light yellow. The titration process was stopped when the color of the solution became colorless. Then, the sodium thiosulphate used was measured and the results showed in the Table 4.1 and Table 4.2. Based on both tables, the volume was supposed to increase since the concentration of NaOH used for alkali treatment higher. However most of the results were not constant. This condition was occurred may be because of Kappa test by using titration process was not suitable for soft fiber liked kenaf and not efficient enough but the results still can be accepted.

After that, by using that volume, the kappa number was calculated by using equation (4.1), (4.2), and (4.3) and the results were showed in Table 4.3 and Table 4.4. The results for grinding samples showed that the treated sample with 6% NaOH at 3 h has lower kappa number and it was chosen for further characterization process. In addition, based on the results for without grinding samples, the treated samples with 3% NaOH at 6 h, 9% at 1 h and 9 % NaOH at 3 h has lower kappa number compared to the 6% NaOH at 3 h treated samples. However, the sample treated with 6% NaOH at 3 h was chosen for further characterization process since the literature showed that 3% NaOH was not enough to treat the kenaf fiber while 9% NaOH made the structure of kenaf fiber damage.

4.2.2 Scanning Electron Microscope (SEM)

The objective of this kind of analysis was to evaluate the surface of the kenaf bast fiber before and after the conducted alkali treatment for grinding and without grinding samples. This analysis was done by using scanning electron microscope machine located in Chemical Analysis Centralize Lab (Makmal Analisis Kimia Berpusat) conducted by Mr. Azinuddin Zulfahmi Megat. The figures below showed the results for the analysis:

i. Grinding samples

a) Untreated samples

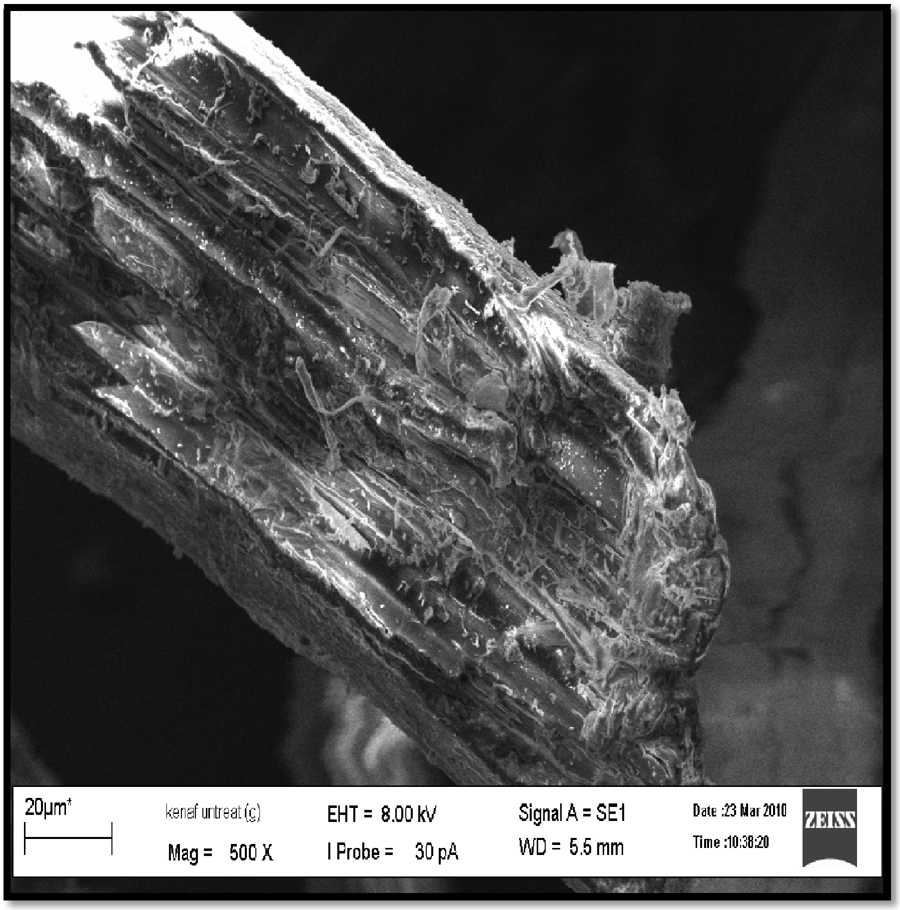


Figure 4.1: SEM image of grinding and untreated kenaf bast fiber

b) Treated samples

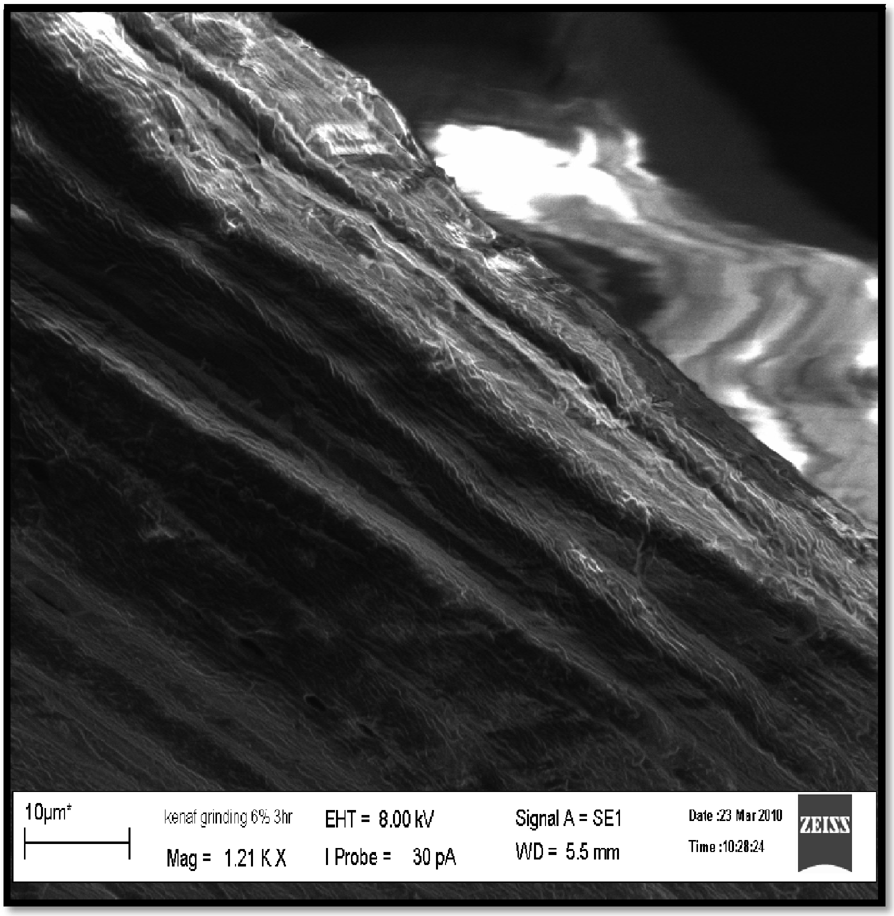


Figure 4.2: SEM image of grinding and treated kenaf bast fiber

ii. Without grinding samples

a) Untreated samples

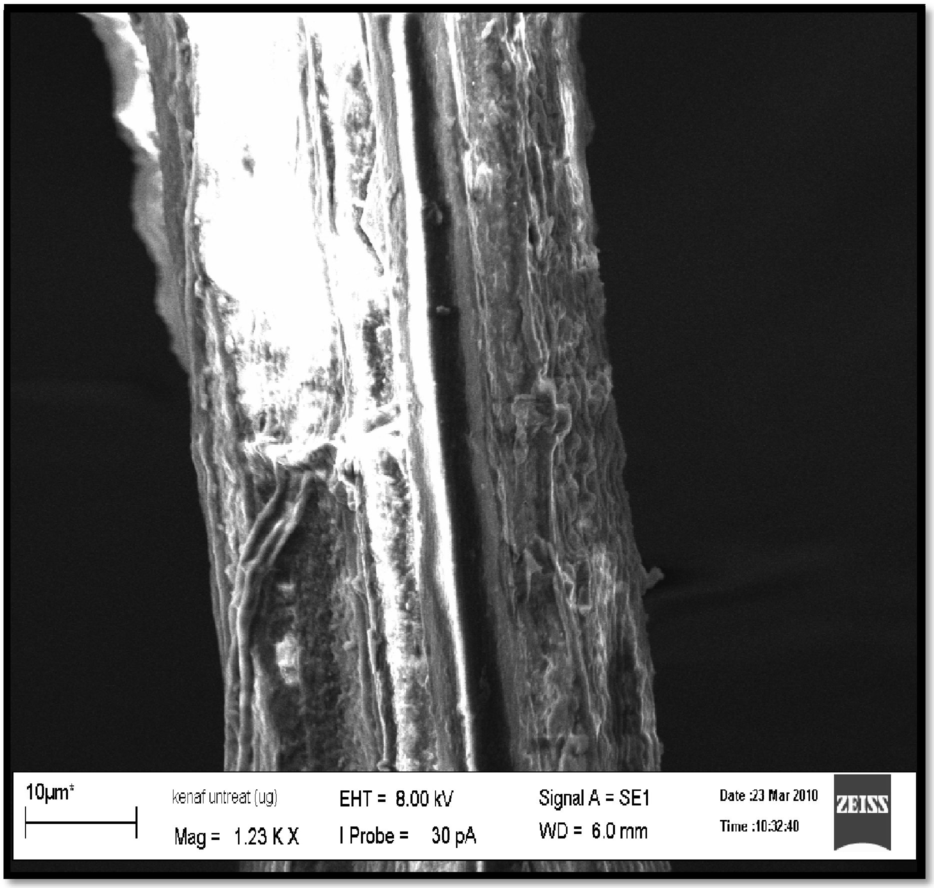


Figure 4.3: SEM image of without grinding and untreated kenaf bast fiber

b) Treated samples

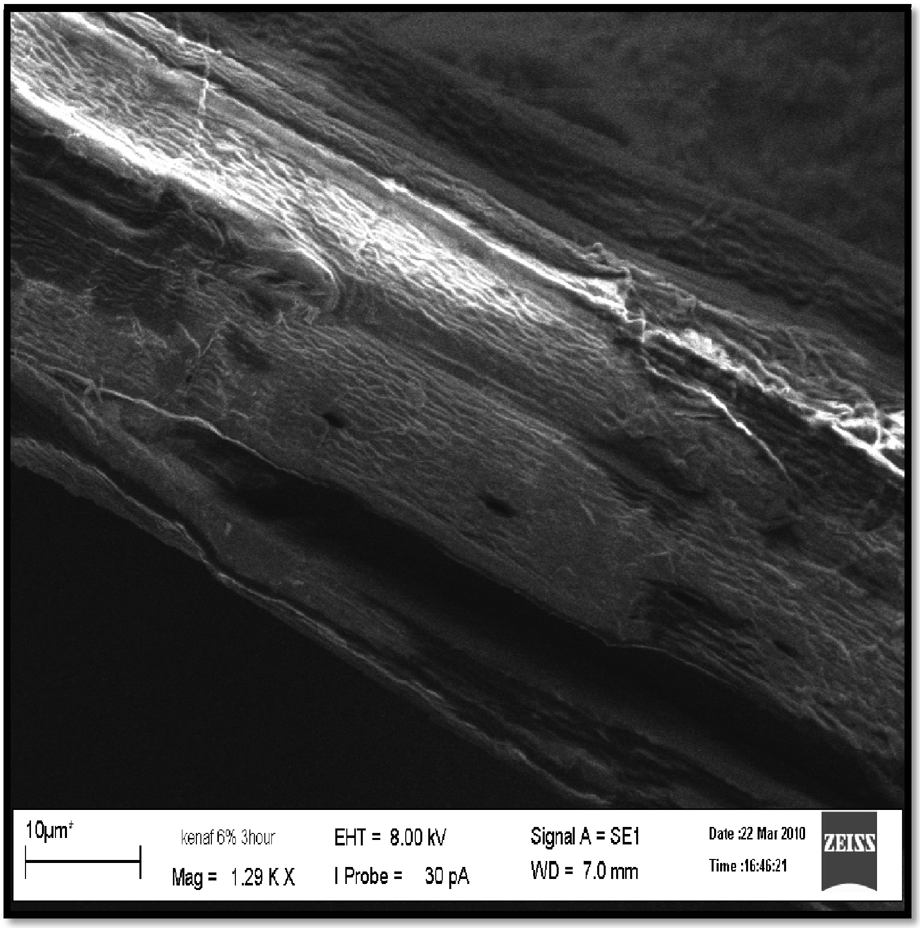


Figure 4.4: SEM image of grinding and untreated kenaf bast fiber

Based on the results of kappa test, samples treated with 6% NaOH at 3 h was selected for further characterization process by using Scanning Electron Microscope (SEM) equipment in order to evaluate the surface morphology after the alkali treatment. The characterization process also conducted for both grinding and without grinding untreated samples as a comparison. Figure 4.1 showed the surface of untreated and grinding kenaf bast fiber with magnification of 500. From the figure, there were a lot of impurities on the fiber's surface. The same condition can be seen from Figure 4.3 with magnification of 1230 which was the SEM analysis result for untreated and without grinding kenaf bast fiber. The impurities may be consists of some chemical compound of the fiber itself such as hemicelluloses, lignin and pectin compound. The lignin content made the fiber more hydrophilic. The hydrophilic nature of fiber due to the lignin content caused the poor interfacial bonding between fiber and resin used in making composites. In addition, the hydrophilic nature of fiber also made the fiber not applicable to be used because of high moisture absorption. The lignin content also made the color of the green composites produced change if they were exposed to the ultraviolet light.

Figure 4.2 with magnification of 1210 and Figure 4.4 with magnification of 1290 showed the SEM results for 6% NaOH at 3 h treated grinding and without grinding fiber respectively. Both figures showed that the surface condition of kenaf bast fiber after alkali treatment. The surface became more cleanly compared to the untreated surface of untreated fiber. The cleaned surface explained that most of the surface impurities such as lignin compound had been removed by using alkali treatment. After the impurities removed, the fiber became more hydrophobic compared to the untreated kenaf bast fiber. The surfaces of treated fibers also have more porosity compared to the untreated fiber. The hydrophobic nature and porosity of the modified fibers can provide better bonding between that fibers and hydrophobic resin. Better bonding between reinforcement and matrix material always important in order to produce high performance of thermoset composites. The removal of lignin content also can avoid the composite from ultraviolet degradation if they were exposed to the ultraviolet light. The different values of magnification used for all the SEM results because some of the fiber too small and it needs high value of magnification in order to make the surface condition of the fibers clearer.

4.3 Composite Characterization

In the composite characterization process, tensile testing analysis was conducted for 6% NaOH treated fiber at 3 h for grinding and without grinding kenaf bast fiber. The tables and figures below showed the results for both types of samples:

Table 4.5: Results for tensile testing

Sample	**Condition**	**Strain, ε**	**Stress, σ (MPa)**
Grinding	Untreated	18.24	7.67
	Treated	17.04	7.20
Without grinding	Untreated	6.77	2.63
	Treated	8.80	7.72

Thermoset composites were fabricated by using hot press equipment from untreated and treated kenaf bast fiber for both grinding and without grinding samples so that the results can be compared. After the fabrication process, the characterization process was performed by using tensile testing for all the thermoset composites produced. The tensile testing was conducted in order to evaluate the amount of stress that thermoset composites can be withstands without break. Table 4.5 showed that the results of the tensile testing. Based on the results, the stress that thermoset composite produced from without grinding samples can withstand was increased after the alkali treatment compared to the results before treatment. However, the result was decreased for thermoset composite produced from grinding samples after the alkali treatment compared to the results before treatment. The result after the treatment supposed to be increased compared to the result before treatment. This condition was occurred may be because of the fiber orientation not well development. Orientation of fibers related to one another plays an important role in composites (Beg, 2007). Besides, this condition also occurred because of the structure of the composite already damage because of the high force given during the process to take it out from the mold.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusions

The chemical treatment for surface modification of natural fiber likes kenaf bast fiber can be done by using alkali solution which is sodium hydroxide solution. Alkali solution can modify the surface of the fiber by removing all the impurities on the fiber's surface especially lignin compound. The impurities removal can eliminate most of the major drawbacks especially poor interfacial bonding between the fiber and resin, ultraviolet degradation and high moisture absorption of the natural fiber used to produce composites product. Based on the results from the this study, without grinding and treated kenaf bast fiber can provide stronger thermoset composite compared to the grinding and treated kenaf bast fiber. It is because of long fiber quite strong compared to the small size of fiber.

5.2 Recommendations

The future study can be done by using other types of chemical in order to produce better natural fiber and can make it more applicable to produce high quality composites. Besides, the UV-Vis spectrophotometer can be used to determine the Kappa number instead of used titration process because it is more efficient and accurate. Then, for the fiber characterization process by using SEM equipment can be done with same magnification value so that the results can be compared with each other. More fiber characterization process can be done especially by using Thermogravimtric Analyzer (TGA) equipment. Furthermore, impact test can be done for the characterization process of thermoset composite produced.

REFERENCES

Âström, B. T. (2010). Manufacturing of Polymer Composites. Retrieved from http://books.google.com.my

Byoung, A. J., Ryuichiro, K., Kokki, S. & Ki P. H. (2001). Effect of Activator on Hydrogen Peroxide Bleaching of White Ledger. Kyushu University of Japan. Vol 7(5).

Beg, M. D. H. (2007). *The Improvement of Interfacial Bondong, Weathering and Recycling of Wood Fibre Reinforced Polypropylene Composites*. Ph.D Thesis. University of Waikato, Hamilton, New Zealand.

Corrales, F., Llop, M., Mutjie, P., Vilaseca, and Gandini F. Y. A. (2002). Jute Fibers Esterification to Improve The Adhesion With The Matrix in Reinforced Polymers. University of Girona, Spain.

Chemical Composition of Wood (2010). Retrieved from http://www.ipst.gatech.edu/faculty

Composite material (Wikipedia) (2010). Retrieved from http://en.wikipedia.org/wiki/Composite_material

Carbohydrates-Chemical Structure (2010). Retrieved from http://www.scientificpsychic.com/fitness/carbohydrates2.html

Chemical Composition of Wood (2010). Retrieved from http://www.ipst.gatech.edu/faculty

Dimeski, D., Manov, Z., Srebrenkoska, V., Grozdanov, A., Gaceva, G. B., Avella, M., and Zucchini, V. Kenaf Fiber/Propylene Composites as Potential Material for Partitioning Panels in Buildings.

Davis, J. R. (2010). Tensile Testing. 2nd edition. Retrieved from http://books.google.com.my

Fiber (2010). Retrieved from http://www.testdiet.com/Fiber.htm

Geochemical Instrumentation and Analysis. (2010). Retrieved from http://serc.carleton.edu/research_education/geochemsheets/techniques/SME.html

Gaceva, G. B., Dekanski, A., Panic, V., Poleti, D., Grodanov, A., Buzarovska, A., Aavella, M., and Gentile, G. (2006). Natural Fibers in Polymer Composite Materials.

Gedney, R. (2010). Tensile Testing Basics, Tips and Trends. Retrieved from http://www.admet.com/assets/Tensile_Testing_Basics_Quality_Magazine.

History of Natural Fiber (2010). Retrieved from http://www.binhaitimes.com/history.html

Hemicellulose (2010). Retrieved from http://www.rpi.edu/dept/chem-eng/Biotech Environ/FUNDAMNT/hemicel.htm

Jusoh, M. J. (2008). Study on the Properties of Woven Natural Fibers Reinforced Unsaturated Polyester Composites.

Jonoobi, M., Harun, J., Shakeri, A., Misra, M., and Oksman, K. Chemical Composition, Crystallinity, and Thermal Degrdation of Bleached and Unbleached Kenaf Bast Fiber (Hibiscus cannabinus) Pulp and Nanofibers.

John, M. J. and Anandjiwala, R. D. Recent Developments in Chemical Modification and Characterization of Natural Fiber - Reinforced Composites.

Kanchanomai, C., Rattananon, S. and Soni, M. (2005). *Material Behaviour*. Effects of Loading Rate on Fracture Behaviour and Mechanism of Themoset Epoxy Resin. 24(2005), 886-892.

Karnani, R., Krishnan, M., and Narayan, R. (1997). Biofiber-Reinforced Polypropylene Composites. Department of Chemical Engineering Michigan State University.

Lignin (2010). Retrieved from http://www.palaeos.com/Plants/Lists/Glossary/GlossaryL.html

Mohd Edeerozey, A. M., Akil, H. M., Azhar, A. B. and Zainal Ariffin M. I. (2006). Chemical Modification of Kenaf Fiber, 61(10), 2023-2025. doi: 10.1016/j.matlet. Retrieved from http://www.sciecedirect.

McCrady, E. (Nov 1991). The Nature of Lignin. Vol 4(4). Retrieved from http://cool.conservation-us.org/byorg//abbey/ap/ap04/ap04-4/ap04-402.html

Manufacturing techniques for Composite Fabrication. (2010).Retrieved from http://www.fibermaxcomposites.com/index_files/manufacturingtechniques

Rowell, R. M. and Stout, H. P. Jute and Kenaf. Retrieved on 2009.

Rowell, R. M. (1995). A New Generation of Composite Materials from Agro-Based Fiber. Department of Forestry, University of Wisconsin.

Sanadi, A. R., and Caulfield, D. (2008). Thermoplastic Polyolefins as Formaldehyde Free Binders in Highly Filled Lignocellulosic Panel Boards: Using Glycerine as a Processing Aid in Kenaf Fiber Polypropylene Boards. Denmark. 11(4), 487-492.

Taj, S., Munawar, M. A. and Khan, S. (2007). Natural Fiber-Reinforced Polymer Composite. Lahore, Pakistan.

Ting, Z. (2003). Improvement of Kenaf Yarn for Apparel Applications. Louisiana State University.

Zbidi, F., Sghaier, S., Nejma, M. B. and Zidi, M. (2009). *Journal Applied Sciences*. Influence of Alkali Treatment and Enzymatic Treatments on The Properties of Doum Palm Fibers and Composite. 9(2), 366-371.

APPENDIX A

A.1 Determination of Kappa Number

Calculation of kappa number as follows:

$$k = \frac{p \ x \ f}{w}$$

where,

k = kappa number

p = amount of 0.1N permanganate actually consumed by the test specimen, mL

f = factor for correction to a 50% permanganate consumption, dependent on the value of p (refer to table)

w = weight of moisture-free pulp in the specimen, g

p = $\frac{(b-a)N}{0.1}$

where

p = amount of 0.1N permanganate actually consumed by the test specimen, mL

b = amount of the thiosulfate consume in the blank determination, mL

a = amount of the thiosulfate consume by the test specimen, mL

N = normality of the thiosulfate

Factor of temperature correction if the temperature not at 25 °C

k = $\frac{p\ x\ f}{w}\ [1 + 0.013(25 - t)]$

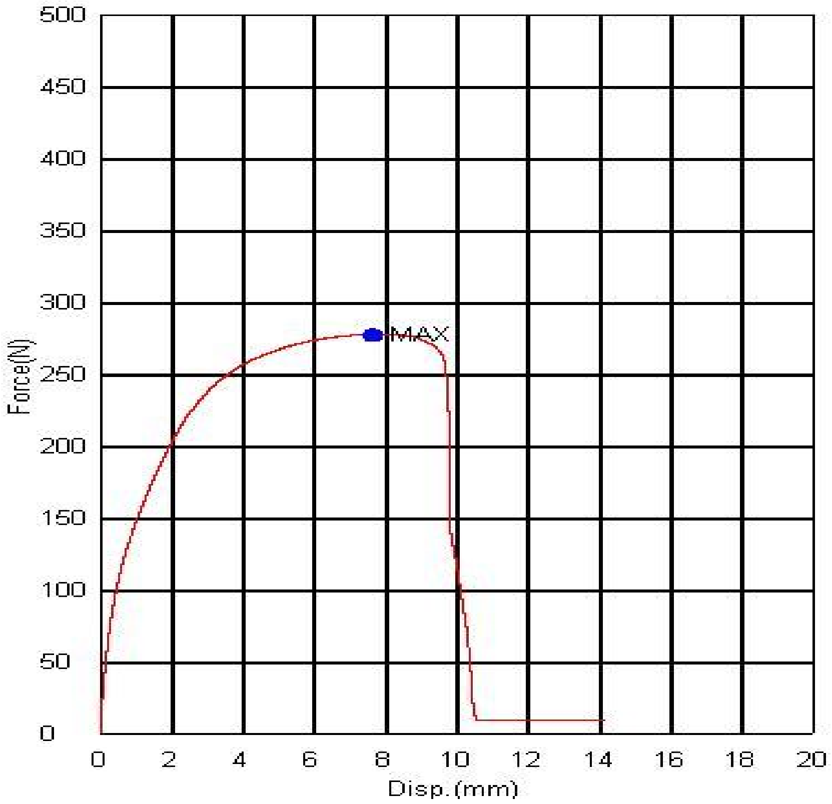
where

t = current temperature during experiment

A.2 Graph for Tensile Testing

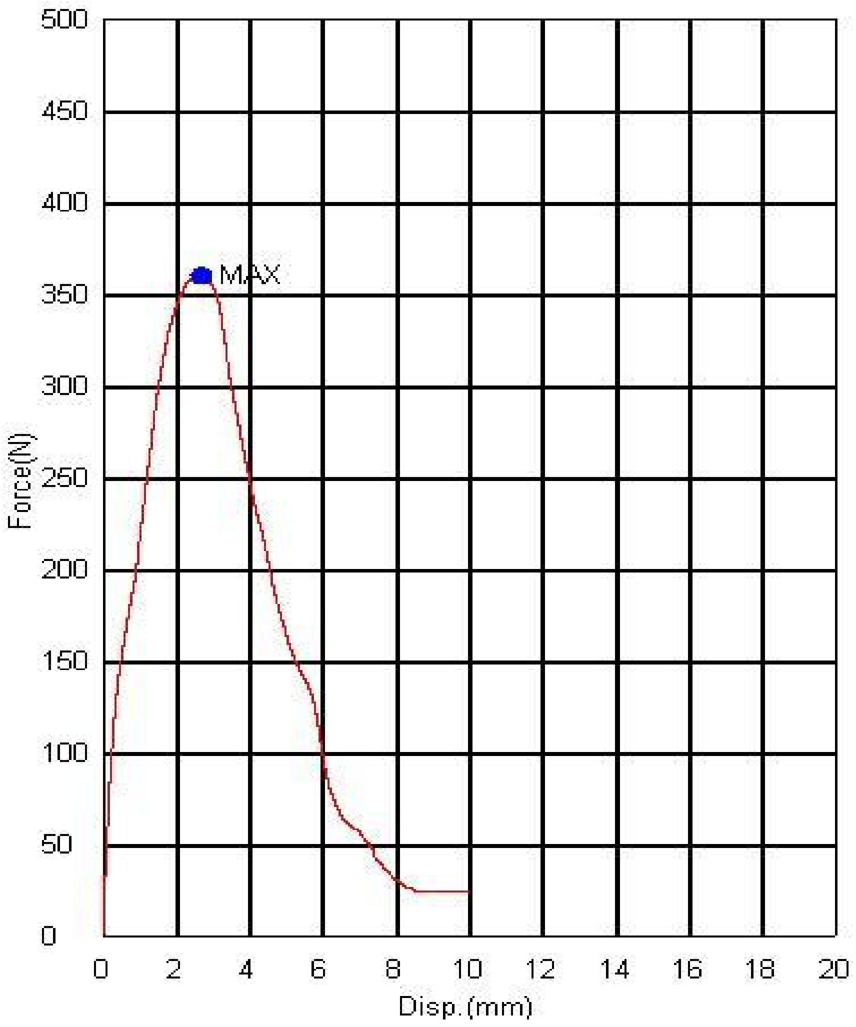
i. Grinding samples

a) Untreated kenaf bast fiber



Graph Force (N) versus Displacement (mm) for untreated fiber

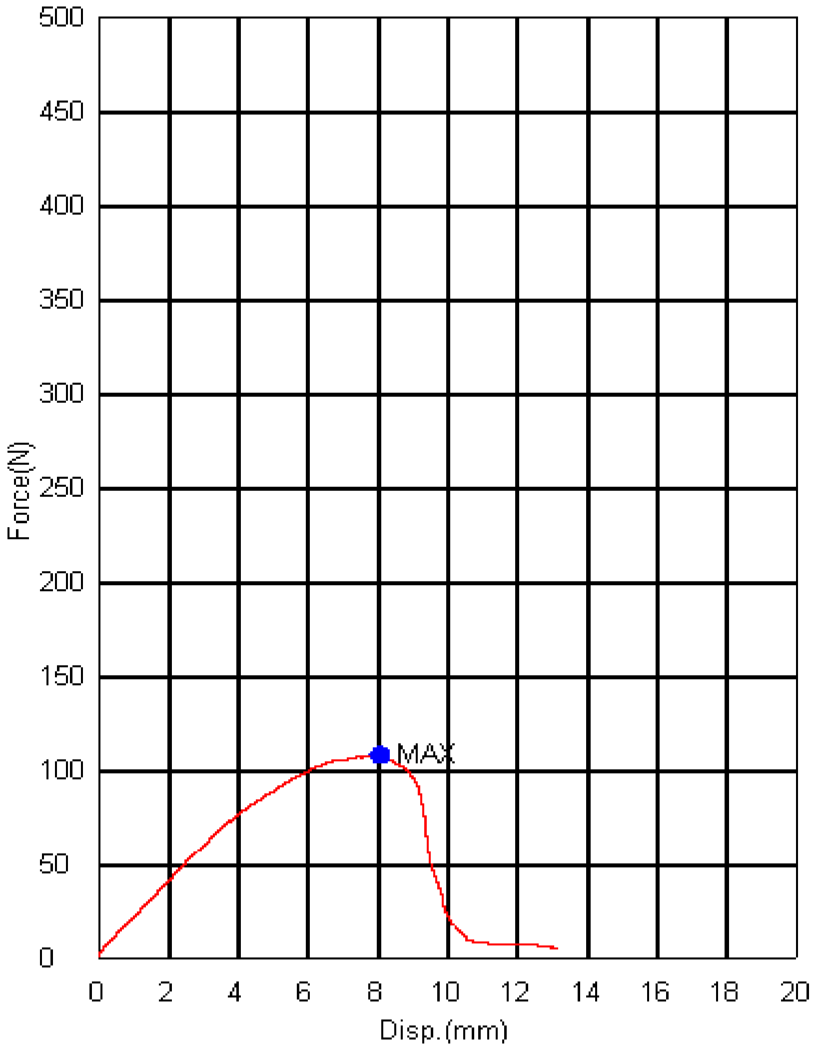
b) Treated kenaf bast fiber



Graph Force (N) versus Displacement (mm) for treated fiber

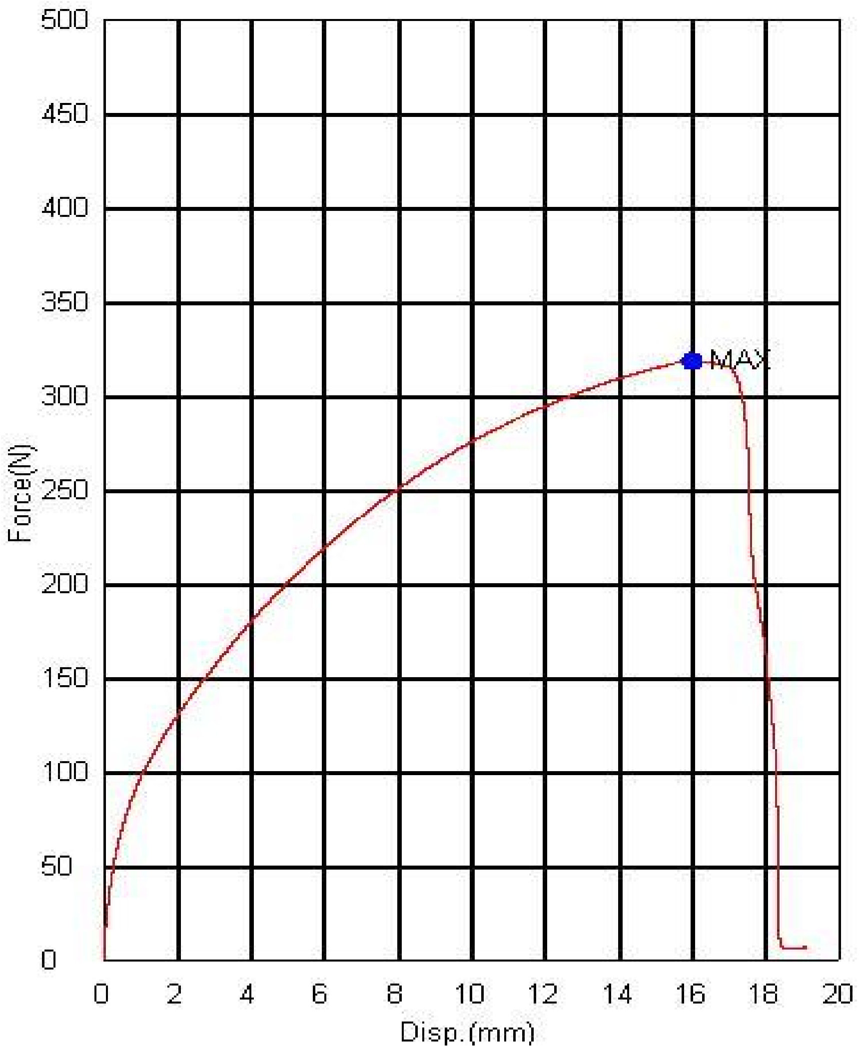
ii. Without grinding samples

a) Untreated kenaf bast fiber



Graph Force (N) versus Displacement (mm) for untreated fiber

b) Treated kenaf bast fiber



Graph Force (N) versus Displacement (mm) for treated fiber