**UNIVERSITI MALAYSIA PAHANG**

# BORANG PENGESAHAN STATUS TESIS

**JUDUL**: MODIFICATION OF COIR FIBER AS POLYMER REINFORCEMENT

**SESI PENGAJIAN**: **2009/2010**

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Date : …………………………

# MODIFICATION OF COIR FIBER AS POLYMER REINFORCEMENT

**MOHD AIZUDDIN BIN AB RAZAK**

**A thesis submitted in fulfillment**
**of the requirements for the award of the degree of**
**Bachelor of Chemical Engineering**

**Faculty of Chemical & Natural Resources Engineering**
**Universiti Malaysia Pahang**

**APRIL 2010**

I declare that this thesis entitled *"Modification of Coir Fiber as Polymer Reinforcement"* is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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***To my beloved mother, father, families, and friends, who gave me everlasting inspiration, never ending encouragements and priceless support towards the success of this study.***

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# ABSTRACT

Studies on the use of natural fibers as replacement to man-made fiber in fiber-reinforced composites have increased and opened up further industrial possibilities. This research will enhance the usage of composite in industrial application and also in household product. The objectives of this research are to modify the surface property of coir fiber and to study the bonding mechanism between the coir fiber and the polymer. In order to achieve the objective, there are several steps have been done during the research that is the surface treatment of fiber by NaOH solution with manipulated concentration, composites production with epoxy resin and coir fiber as the reinforcement and also the characterization of the composites. The process flows of this research are the fiber preparation, stock solution preparation, fiber treatment, fiber testing, polymer preparation, and polymer testing. From the result, untreated coir fiber has Kappa number of 46.75, 20% NaOH treated fiber has Kappa number of 32.63, 25% NaOH treated fiber has Kappa number of 28.86, and 30% NaOH treated fiber has Kappa number of 20.09. The treatment have made the fiber more thermal resistant as approved by the increase in the decomposition temperature of coir fiber and also increase in weight percent remaining at specific temperature that is at 300°C. For untreated fiber, it have decomposition temperature of 336.64°C while for 20%NaOH treated fiber, the decomposition temperature is 366.13°C. From the results also, increasing in NaOH concentration has result in increasing tensile strength of the composite and the optimum concentration for the highest tensile strength is at 25% NaOH treated fiber. The conclusion that have been made is the alkali treatment have been successfully done and renewable fibers like coir can be used as reinforcing materials for low cost composites, due to the economic and environmental advantages of such materials.

# ABSTRAK

Kajian mengenai penggunaan serat alam sebagai pengganti serat buatan manusia dalam komposit serat-diperkuatkan telah meningkat dan membuka kemungkinan industri dengan lebih lanjut. Penelitian ini akan meningkatkan penggunaan komposit dalam aplikasi industri dan juga dalam produk rumah tangga. Tujuan dari penelitian ini adalah untuk mengubahsuai sifat permukaan serat sabut dan mempelajari mekanisme ikatan antara gentian sabut dan polimer. Untuk mencapai tujuan tersebut, ada beberapa langkah telah dilakukan selama penelitian iaitu merawat permukaan serat dengan larutan NaOH dengan kepekatan yang dimanipulasi, penghasilan komposit dengan resin epoksi dan serat sabut sebagai penguat dan juga ujian terhadap komposit. Proses yang terlibat dalam kajian ini adalah persiapan serat, persiapan larutan kimia, rawatan serat, ujian ke atas serat, persiapan polimer, dan ujian ke atas polimer. Dari hasil kajian, serat sabut tidak dirawat mempunyai nombor Kappa sebanyak 46.75, manakala rawatan 20% NaOH ke atas serat mempunyai nombor Kappa sebanyak 32.63, rawatan 25% NaOH ke atas serat pula mempunyai nombor Kappa sebanyak 28.86, dan rawatan 30% NaOH ke atas serat pula mempunyai nombor Kappa sebanyak 20.09. Rawatan NaOH telah membuat serat lebih tahan panas yang dibuktikan oleh peningkatan suhu penguraian serat sabut dan juga peningkatan peratus berat sisa pada suhu tertentu iaitu pada 300°C. Untuk serat tidak dirawat, ia memiliki suhu penguraian sebanyak 336.64°C, sedangkan untuk rawatan 20% NaOH ke atas serat, suhu penguraian ialah 366.13°C. Dari hasil kajian juga, peningkatan kepekatan NaOH telah menghasilkan peningkatan kekuatan ketegangan komposit dan kepekatan optimum untuk kekuatan ketegangan tertinggi adalah rawatan 25% NaOH ke atas serat. Kesimpulan yang telah dibuat adalah rawatan alkali telah berjaya dilakukan dan serat sabut boleh digunakan sebagai bahan penguat untuk komposit kos rendah, kerana keuntungan ekonomi dan kelebihan terhadap persekitaran dari bahan tersebut.

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# LIST OF ABBREVIATIONS & SYMBOLS

| | | |
|---|---|---|
| % | - | Percentage |
| wt% | - | Weight percent |
| cm | - | Centimeter |
| DP | - | Degree of polymerization |
| g | - | Gram |
| kg | - | Kilo Gram |
| kN | - | Kilo Newton |
| m | - | Meter |
| ml | - | Milliliter |
| mm | - | Millimeter |
| N | - | Normality |
| NaOH | - | Sodium Hydroxide |
| OH | - | Hydroxide |
| ºC | - | Degree Celsius |
| Pa | - | Pascal |
| TAPPI | - | Technical Association of the Pulp and Paper Industry |
| V | - | Volume |
| SEM | - | Scanning Electron Microscopy |

# CHAPTER 1

# INTRODUCTION

## 1.1 Research Background

From being an importer of petrochemicals, Malaysia is today an exporter of major petrochemical products. A wide range of petrochemicals are produced in Malaysia such as olefins, polyolefin, aromatics, ethylene oxides, glycols, oxo-alcohols, exthoxylates, acrylic acids, phthalic anhydride, acetic acid, styrene monomer, high impact polystyrene, ethyl benzene, vinyl chloride monomer and polyvinyl chloride and polybutylene terephthalate. These world scale plants have also contributed significantly to the development of local downstream plastic processing activities by providing a steady supply of feedstock material for the plastic industry.

These factors have led the plastic products industry to become one of the most dynamic industries in Malaysia's manufacturing sector. The plastic industry can be divided into four sub-sectors, namely packaging sub-sector, electrical & electronics and automotive components sub-sector, consumer and industrial products sub-sector and others. The packaging sub-sector, both flexible and rigid (including bags, films, bottles and containers), remains the largest market for the plastic industry. The main production processes involved in the plastic producers industry are injection molding, pipes and profiles extrusion and foam molding.

There are more than 1,550 companies in operation, producing products ranging from common household items, packaging materials and conveyance articles to parts and components for the electrical and electronics, automotive, office automation, computer and telecommunications industries.

Malaysia is currently a net exporter of plastic products. The total export of plastic products amounted to RM9.34 billion in 2008, an increased of 11.5 per cent compared with 2007. The main products exported were containers of plastics, plates, films, sheets, foils, strips and other articles of plastics. The main export destinations included the EU, the People's Republic of China, Hong Kong, Singapore, Japan and Thailand.

The Malaysian plastics industry has been rated as among the most competitive in Asia. Globalization poses both challenges and opportunities simultaneously for Malaysian plastic manufacturers. Therefore, it is imperative for Malaysian manufacturers to sustain their competitiveness, through improved technologies, enhanced skills and penetrating new markets in developed and developing economies.[1]

Fiber crops have accompanied human society since the start of our time. In early history, humans collected the raw materials for ropes and textiles from the wild. Later societies learned to cultivate such crops. Plant fiber crops are among the earliest known cultivated plants and humans continued the domestication of these crops over millennia. Fiber crop varieties have been extensively developed through breeding and selection, according to societies’ needs and values.

For instance, hemp and linen fragments were found in Neolithic sites in Syria, Turkey, Mesopotamia (present-day Iraq), and Persia (present-day Iran), and have been carbon dated back to 8000-6000 B.C. Fiber crops have been bred focusing on fiber quality, climatic adaptability, and yield factors. Ingenious fiber crops, such as flax, hemp, and nettle, possessed great agricultural importance for the production of textile fibers until the late 19th century. However, the mechanization of cotton harvest, processing, and development, and the growing demand for and production of cheap synthetic textile fibers destroyed the production of traditional fiber crops.

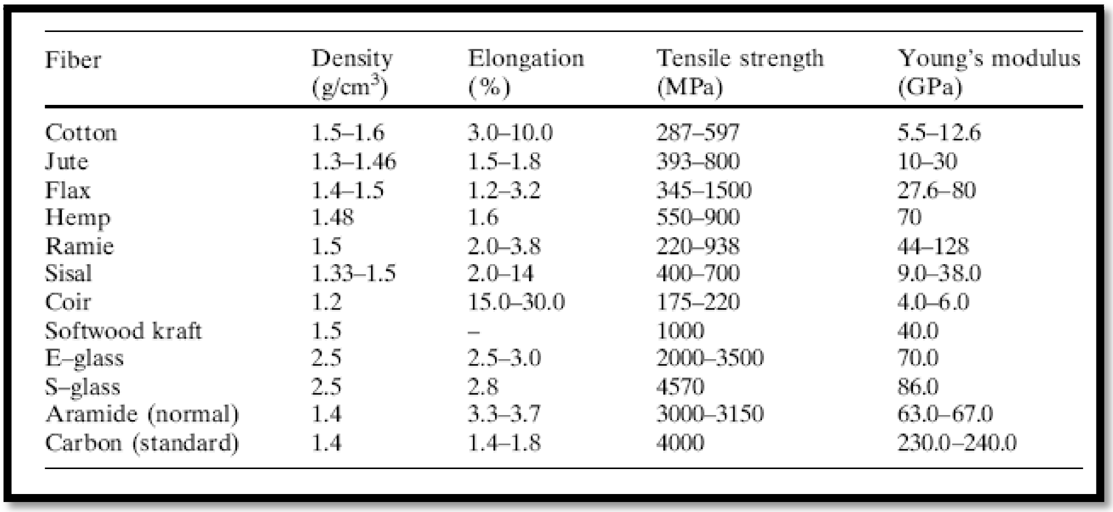
More recently, increasing environmental awareness, concern for environmental sustainability, and the growing global waste problem, initiation of ecological regulations and legislation such as the end-of-life vehicles regulation, the depletion of fossil fuels, and the increasingly higher price of crude oil have together created a groundswell of interest in renewable resources. Legislative pressure for greener technologies as well as customers' demands for more environmental friendly consumer goods are forcing materials suppliers and manufacturers to consider the environmental impact of their products at all stages of their life cycle, including materials selection processing, recycling, and final disposal. This and the worldwide availability of plant fibers and other abundantly accessible agro waste is responsible for this new research interest in the field of polymer science, engineering, and is responsible for a new interest in research in sustainable technology. Research has as its objective the development, processing, and manufacturing, recycling, and disposal of "green" plastics, adhesives, polymer composites, blends, and many other industrial products from renewable resources.

Renewable resources from agricultural or forestry products form a basis for new industrial products or alternative energy sources. Plant-based fibers are already used in a wide range of products. Plant fibers find applications as textiles and geotextiles, twines and ropes, special pups, insulating and padding materials, fleece, felts and nonwoven materials, and increasingly as reinforcement for polymers.

The mechanical properties of plant fibers are much lower when compared to those of the most widely used competing reinforcing glass fibers. However, because of their low density, the specific properties, (property-to-density ratio), strength, and stiffness of plant fibers are comparable to the values of glass fibers. The public generally regards products of renewable raw materials as environmental friendly. [2]

Traditional fiber-reinforced composites use various types of glass, carbon, aluminum oxide, and many others as reinforcing component. Natural fibers, especially bast (bark) fibers, such as flax, hemp, jute, henequen and many others were applied by some researchers as fiber reinforcement for composites in recent years. Advantages of natural fibers over man-made fibers include low density, low cost, recyclability and biodegradability. These advantages make natural fibers potential replacement for glass fibers in composite materials. Mechanical properties of natural fibers, especially flax, hemp, jute and sisal, are very good and may compete with glass fiber in specific strength and modulus. Figure 1.1 lists the mechanical properties of some natural and man-made fibers.

**Table 1.1:** Mechanical properties of natural fibers as compared to conventional reinforcing fibers.



| Fiber | Density ($g/cm^3$) | Elongation (%) | Tensile strength (MPa) | Young's modulus (GPa) |
|---|---|---|---|---|
| Cotton | 1.5–1.6 | 3.0–10.0 | 287–597 | 5.5–12.6 |
| Jute | 1.3–1.46 | 1.5–1.8 | 393–800 | 10–30 |
| Flax | 1.4–1.5 | 1.2–3.2 | 345–1500 | 27.6–80 |
| Hemp | 1.48 | 1.6 | 550–900 | 70 |
| Ramie | 1.5 | 2.0–3.8 | 220–938 | 44–128 |
| Sisal | 1.33–1.5 | 2.0–14 | 400–700 | 9.0–38.0 |
| Coir | 1.2 | 15.0–30.0 | 175–220 | 4.0–6.0 |
| Softwood kraft | 1.5 | – | 1000 | 40.0 |
| E–glass | 2.5 | 2.5–3.0 | 2000–3500 | 70.0 |
| S–glass | 2.5 | 2.8 | 4570 | 86.0 |
| Aramide (normal) | 1.4 | 3.3–3.7 | 3000–3150 | 63.0–67.0 |
| Carbon (standard) | 1.4 | 1.4–1.8 | 4000 | 230.0–240.0 |

Unfortunately, the performance of coir as a reinforcement in polymer composites is unsatisfactory and not comparable even with other natural fibers due to its low cellulose content (36–43%), high lignin content (41–45%) and high microfibrillar angle. Morphological studies of coir fibers show the outer sheath of lignin that develops the cellulose ultimate. The removal of this surface layer of lignin usually results in a better and more stable bond. [3]

The coir fibers are usually treated in order to improve resin-fiber interfacial bonding. Among the methods in improving the adhesive character of the coir fiber, alkaline treatment may be considered to be the most economical technique, the main disadvantage is the deterioration in the fiber strength during the treatment. [4]

Because of the above facts, alkali treatment and bleaching has been chosen as surface treatments for coir fibers in order to obtain better adhesion of the coir fibers with the epoxy resin in my research. The chemical and thermal property of coir fiber is determined by Kappa test and thermo gravimetric analysis respectively. Besides that, the mechanical property of the polymer which is tensile strength is determined. The improved fiber-matrix adhesion has been examined by scanning electron microscopy (SEM).

## 1.2 Problem Statement

Coir fiber have been reported to be as substitute for synthetic fiber reinforcements such as glass in plastics due to their low cost, low density, acceptable specific strength, good thermal insulation properties, reduced tool wear, reduced thermal and respiratory irritation and renewable resources. However, natural fibers are generally hydrophilic and are inherently incompatible with hydrophobic thermoplastics such as polypropylene (PP). Major problem of using natural fibers with thermoplastics is the poor interfacial bonding between the fiber and thermoplastic. [5] This is due to presence of a layer of lignin and a small amount of oily layer at the outer surface of coir. [6]

This research is attempted to find the way how to improve the interfacial bonding between the coir fiber and the polymer itself and also to remove the layer of lignin and oily the layer through chemical modification or treatment in order to get a high quality fiber.

## 1.3 Objectives

The objectives of this study are:

(i) To modify the surface property of coir fiber.

(ii) Study the bonding mechanism between the coir fiber and the polymer.

## 1.4 Scope of Study

In order to achieve the objectives stated above, the following scopes of study have been planned:

(i) Surface treatment of fiber by NaOH solution with manipulated concentration.

(ii) Making composites with epoxy resin and characterizing of the composites.

## 1.5 Rationale and Significance

The ecologically relevant parameters provide some distinct advantages to the use of plant fiber crops. One of them is the plant fibers are of less concern to occupational health and safety during handling than other fibrous materials. The favorable aspect ratios and high specific properties at low cost make them an ecologically-friendly alternative to conventional reinforcing fibers in composite materials. The ecological character, the biodegradability, and the price of the plant fibers are very important for their acceptance in large volume engineering markets such as the automotive and construction industry. Innovative plant fiber-reinforced polymers are rapidly finding more and more applications in secondary structural applications especially for various applications in the automotive industry.

Plant fibers are currently used in considerable quantities in various applications in the automotive industry only in the interior of passenger cars and truck cabins. For example, fiber crops, such as flax and hemp, currently grown in the European Union (EU) and in North America provide an alternative to the overproduction of food crops and land division. The most important end user of natural fiber products in Europe is the automobile industry. Plant fibers are used as trim parts in door panels or cabin linings. Coir fibers bonded with natural latex are used as seat cushions. Plant fibers are increasingly used for thermo acoustic insulation purposes. These insulating materials are mainly based on recycled textiles and have high fiber content exceeding 80 wt%. At the present time, there are very few exterior parts made from plant fiber composites.

An increased demand for renewable resources, including plant fibers, as well as sources for energy or raw materials (reactants) for paints, lacquer, varnishes, adhesives, and polymers can have an important socioeconomic impact. It will generate a nonfood crop source for the economic development of farming and agricultural-based areas of the world.

Natural fiber-reinforced composites can be applied in the plastics, automobile and packaging industries to cut down on material cost. A better understanding of the chemical composition and surface adhesive bonding of natural fiber is necessary for developing natural fiber-reinforced composites. The components of natural fibers include cellulose, hemicellulose, lignin, pectin, waxes and water soluble substances. [7]

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 History of Biopolymer

Composite materials are attractive because they combine material properties in ways not found in nature. Such materials often result in lightweight structures having high stiffness and tailored properties for specific applications, thereby saving weight and reducing energy needs. Fiber-reinforced plastic composites began with cellulose fiber in phenolics in 1908, later extending to urea and melamine, and reaching commodity status in the 1940s with glass fiber in unsaturated polyesters. From guitars, tennis racquets, and cars to microlight aircrafts, electronic components, and artificial joints, composites are finding use in diverse fields.

The fiber-reinforced composites market is a multibillion-dollar business. Glass fiber is the dominant fiber and is used in 95% of cases to reinforced thermoplastic and thermoset composites. Current research findings show that in certain composite applications, natural fibers demonstrate competitive performance to glass fibers.

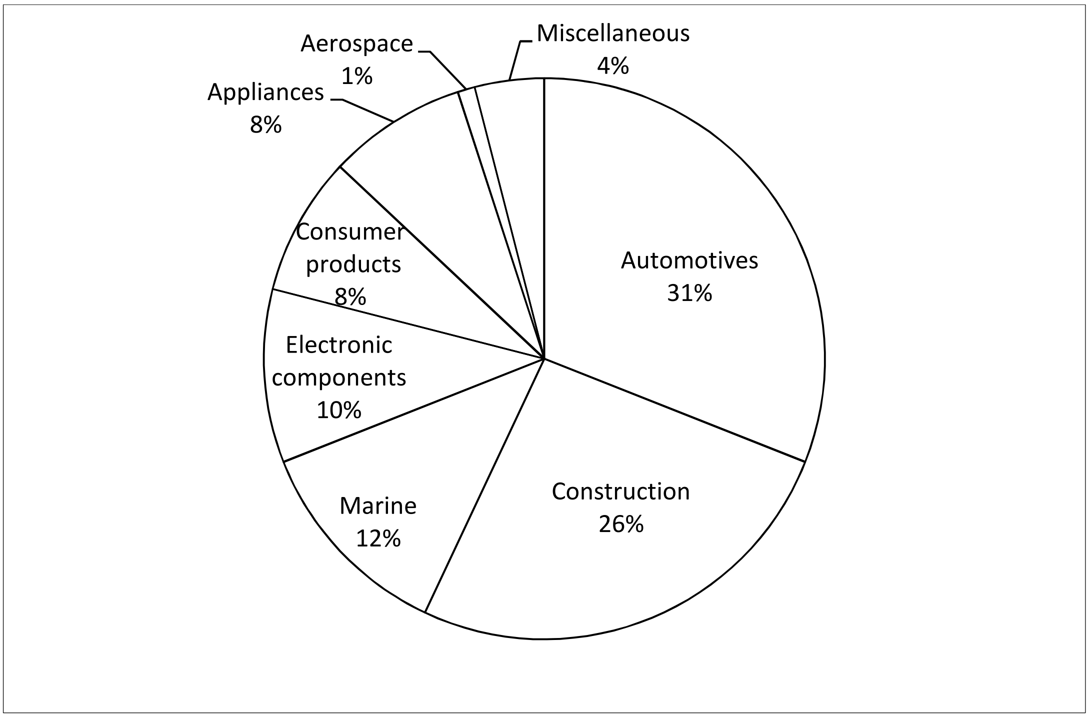


**Figure 2.1:** Fiber-reinforced plastic composites used in 2002. (Adapt from *Plast. News.* August 26, 2002)

After decades of development of high-performance artificial fibers like carbon, aramid, and glass, natural fibers have gained renewed interest, especially as a glass fiber substitute in automotive industries. Advantages of natural fibers over synthetic or man-made fibers such as glass and carbon are as follows: low cost, low density, acceptable specific strength properties, ease of separation, carbon dioxide sequestration, and biodegradability. Natural fiber composites are now emerging as realistic alternative to wood-filled and glass-reinforced plastics. Ecofreindly biocomposites have the potential to be the new material of the 21st century and be a partial solution to many global environmental problems. [8]

## 2.2 General Review of Coir Fiber

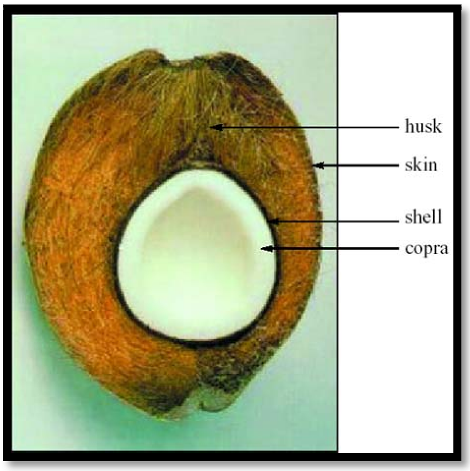


**Figure 2.2:** Cross section of a coconut palm

Coir (*Cocos nucifera,* Palmae) fibers are obtained from the fibrous husk (mesocarp) encasing the fruit of the coconut palm (Figure 2.3), which is a by-product of the copra extraction process. The term *coir* is derived from kayar, a rope or cord, and from kayaru, meaning to be twisted. Coconut palms are cultivated throughout tropical countries mostly for the high oil content of the endosperm (copra). The oil is widely used in both the food and nonfood industries. Large production areas are found along the coastal regions in the wet tropical area of Asia in the Philippines, Indonesia, India, Sri Lanka, and Malaysia. [2]

**Figure 2.3:** Photograph of a *Cocos nucifera* palm with coconuts.

In these countries millions of people make a living from coconut palm and its many products. However, India and Sri Lanka are the main producing countries of coir fiber. On average, from 100 coconuts 7.5 to 8.2 kg of coir fibers can be obtained. After separation of the nut manually from the husk, the fibrous husk can be processed by variety of retting techniques, but traditionally this takes place in ponds of brackish water (for 3 to 6 months), in salt backwaters, or in lagoons. Saltwater retting requires 10 to 12 months. Traditional water-retting procedures result in highly polluted waters and the accumulation of large chucks of coir pith. During the retting of the husks, the coir fibers are softened and can be easily decorticated and extracted by beating. After hackling, washing, and drying, the fibers are loosened manually and further cleaned. Afterwards the fibers are separated into bristle fibers (~20 to 40 cm long), by combing, and mattress fibers, which are random fibers having a length of 2 to 10 cm. The diameter of coir fibers varies between 0.1 and 1.5mm. [2]

Better grade coir fibers are light in color, but the color ranges from golden yellow for fibers obtained from not completely ripened nuts to reddish-brown from ripe nuts. Individual coir fibers are quite short, about 500 μm in length. The cells are thick walled, having an irregular lumen. The fiber surface is covered with cavities arising from dried out sieve cells. [9]

Coir fibers have high lignin but low cellulose content, as a result of which the fibers are resilient, strong, and highly durable. Coir is one of the toughest plant fibers available. It does not pill and is highly abrasion and rot (fungal and bacterial) resistant. Furthermore, coir is naturally insulating and sound absorbing, antistatic, and difficult to ignite. Due to the ability of coir fibers to tolerate water immersion for months without disintegrating, they find many applications as horticultural and erosion control products (geotextiles). Coir fibers are the only fruit fiber that is used in the textile industry. They are widely used as seat cushions in the automotive industry because of their high rate of moisture absorption. [10]

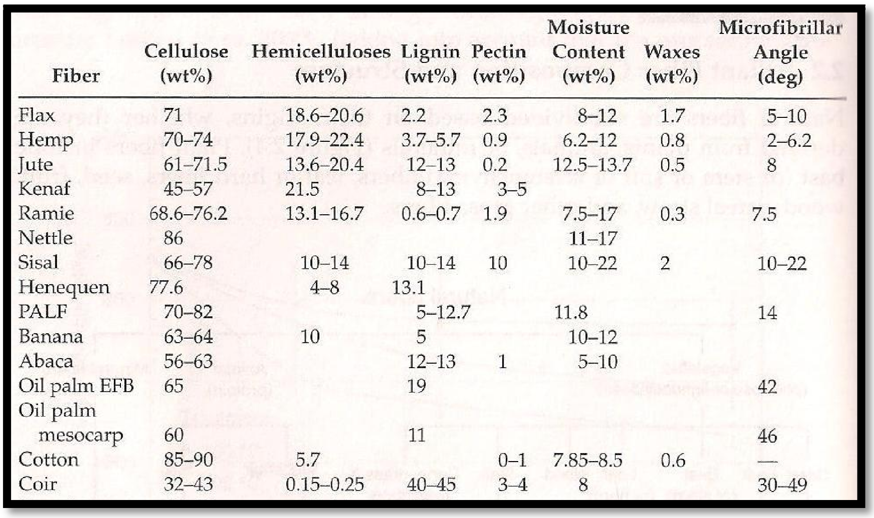
## 2.3 Introduction to Fiber Chemistry

All natural fibers, whether wood or nonwood types, are cellulosic in nature. The major constituents of natural biofibers are cellulose and lignin. The amount of cellulose, in lignocellulosic systems, can vary depending on the species and the age of the plant. [8]

The chemical composition as well as the structure of plant fibers is fairly complicated. Plant fibers are a composite material designed by nature. The fibers are basically a rigid, crystalline cellulose microfibril-reinforced amorphous lignin and/or hemicelluloses matrix. Most plant fibers, except for cotton, are composed of cellulose, hemicelluloses, lignin, waxes, and some water-soluble compounds. [2]

Lignocellulosic is a hygroscopic material that absorb water when expose to wet condition. Besides that, lignocellulosic can be recycled naturally with biological degradation, heat, aqueous, photochemical, chemical and mechanical. In other words, lignocellulosic is generated naturally from carbon dioxide, water, and contain compound that allowing them to be recycled to its basic compound. [11]

**Table 2.1:** Chemical Composition, and Moisture Content of Vegetable Fibers



| Fiber | Cellulose (wt%) | Hemicelluloses (wt%) | Lignin (wt%) | Pectin (wt%) | Moisture Content (wt%) | Waxes (wt%) | Microfibrillar Angle (deg) |
|---|---|---|---|---|---|---|---|
| Flax | 71 | 18.6–20.6 | 2.2 | 2.3 | 8–12 | 1.7 | 5–10 |
| Hemp | 70–74 | 17.9–22.4 | 3.7–5.7 | 0.9 | 6.2–12 | 0.8 | 2–6.2 |
| Jute | 61–71.5 | 13.6–20.4 | 12–13 | 0.2 | 12.5–13.7 | 0.5 | 8 |
| Kenaf | 45–57 | 21.5 | 8–13 | 3–5 | | | |
| Ramie | 68.6–76.2 | 13.1–16.7 | 0.6–0.7 | 1.9 | 7.5–17 | 0.3 | 7.5 |
| Nettle | 86 | | | | 11–17 | | |
| Sisal | 66–78 | 10–14 | 10–14 | 10 | 10–22 | 2 | 10–22 |
| Henequen | 77.6 | 4–8 | 13.1 | | | | |
| PALF | 70–82 | | 5–12.7 | | 11.8 | | 14 |
| Banana | 63–64 | 10 | 5 | | 10–12 | | |
| Abaca | 56–63 | | 12–13 | 1 | 5–10 | | |
| Oil palm EFB | 65 | | 19 | | | | 42 |
| Oil palm mesocarp | 60 | | 11 | | | | 46 |
| Cotton | 85–90 | 5.7 | | 0–1 | 7.85–8.5 | 0.6 | — |
| Coir | 32–43 | 0.15–0.25 | 40–45 | 3–4 | 8 | | 30–49 |

*Source:* (Bismarck A. et al., 2005)

### 2.3.1 Cellulose

Cellulose is a hydrophilic glucan polymer consisting of a linear chain of 1,4-β anhydroglucose units, which contain alcoholic hydroxyl groups (Figure 2.3). These hydroxyl groups form intermolecular and intramolecular hydrogen bonds with the macromolecule itself and also with other cellulose macromolecules or polar molecules. Therefore, all natural fibers are hydrophilic in nature. Although the chemical structure of cellulose from different natural fibers is the same, the degree of polymerization (DP) varies. The mechanical properties of a fiber are significantly dependent on the DP. [8]

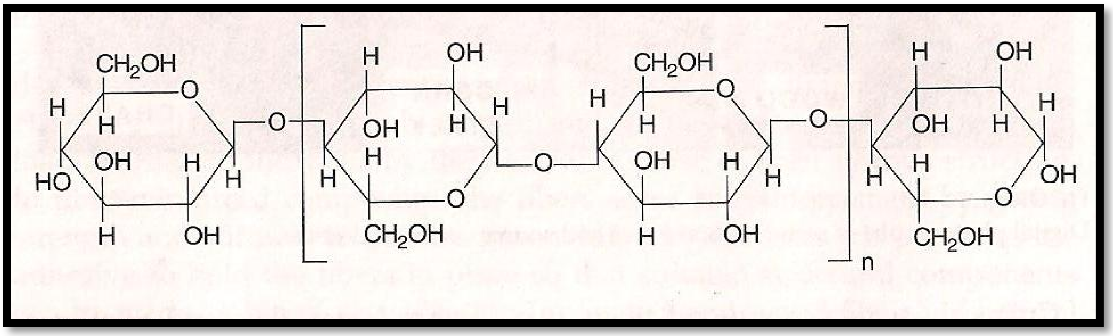


**Figure 2.3:** Molecular structure of cellulose

*Source:* (Mohanty A. K. et al., 2005)

Because of its chemical structure, many hydroxyl groups are available for interaction with water molecules by hydrogen bonding. In contrast to glass fibers where water adsorption is important only at the surface, cellulosic fibers interact with water not only at the surface, but also in bulk. The quantity of sorbed water depends on the relative humidity of the confined atmosphere with which the fiber is in equilibrium. The sorption isotherm of cellulosic material (water sorbed vs. water partial pressure) depends on the following:

1) The purity of cellulose: raw cellulosic material such as nonwashed sisal fibers absorb at least twice as much water as washed fibers.

2) The degree of crystallinity: all OH groups in the amorphous phase are accessible to water whereas only a small amount of water interacts with the surface OH groups of the crystalline phase.

All these characteristics of the cellulosic fiber play an important role when the fiber is to be incorporated in a matrix.

During the biological synthesis of plant cell walls, polysaccharides such as cellulose and hemicelluloses are produced, and simultaneously lignin fills the spaces between the polysaccharide fibers, cementing them together. This lignifications process causes a stiffening of cell walls, and the carbohydrate is protected from chemical and physical damage. [8]

### 2.3.2 Hemicelluloses

Hemicelluloses are polysaccharides composed of a combination of 5- and 6-ring carbon ring sugars. The polymer chains are much shorter (DP around 50 to 300) and branched, containing pendant side groups giving rise to its noncrystalline nature. Hemicelluloses form the supportive matrix for cellulose microfibrils. Hemicellulose is very hydrophilic and soluble in alkali and easily hydrolyzed in acids.

Depending on the plant species, these sugar along with uronic acids form various polymeric structure; some are associated with the cellulosic portion of the plant, while others are more closely associated with lignin.

By contrast to cellulose which is a polymer only of glucose, the hemicelluloses are polymers of five different sugars:

- Hexoses : glucose, mannose, galactose

- Pentoses : xylose, arabinose [12]

There are different kinds of hemicelluloses. Different wood species contain hemicelluloses of slightly different composition. The most common hemicelluloses in softwoods are galactoglucomannan. Hemicelluloses accumulate to 20-35% of wood cell material. [13]

Hemicelluloses chains are much shorter than cellulose chains because they have a lower degree of polymerization. A hemicelluloses molecule contains up to 300 sugar units. Hemicellulose is not linear and cannot form crystalline structures and microfibrils as cellulose does. [14] Hemicelluloses are distinguished from cellulose by the greater ease of hydrolysis in hot, acid solution and by solubility in the aqueous alkaline solution. [15]

### 2.3.3 Lignin

Lignin is a biochemical polymer that functions as a structural support material in plants. Lignin is a high molecular-weight phenolic compound, generally resistant to microbial degradation. The exact chemical nature of lignin still remains obscure. Although the exact structural formula of lignin has not yet been established, most of the functional groups and units, which make up the molecule, have been identified. The high carbon and low hydrogen content of lignin suggest that it is highly unsaturated or aromatic in nature. Lignin is characterized by its associated hydroxyl and methoxy groups.

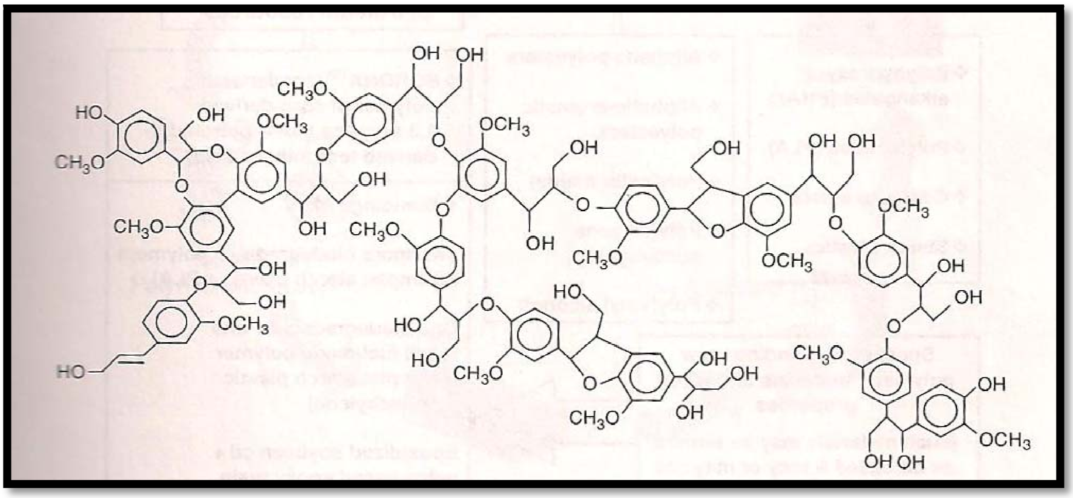


**Figure 2.4:** Molecular structure of lignin

*Source:* (Mohanty A. K. et al., 2005)

The topology of lignin from different sources may be different but it has the same basic composition. Although the exact mode of linkages of lignin with cellulose in lignocellulosic natural fiber is not well known, lignin is believed to be linked with the carbohydrate moiety through two types of linkages, one alkali-sensitive and the other alkali-resistant. The alkali-sensitive linkage forms an ester-type combination between lignin hydroxyls and carboxyls of hemicelluloses uronic acid. The ether-type linkage occurs through the lignin hydroxyls combining with the hydroxyls of cellulose. The lignin, being polyfunctional, exists in combination with more than one neighboring chain molecule of cellulose and/ or hemicelluloses, making a cross-linked structure. [8]

## 2.4 Fiber Treatment and Modification

It is well known that the performance of composites depends on the properties of the individual components and their interfacial compatibility. For numerous applications, plant fibers have to be prepared or modified with the following consideration in mind:

- Homogenization of the properties of the fibers
- Degree of elementarization and degumming
- Degree of polymerization and crystallization
- Good adhesion between fiber and matrix
- Moisture repellence
- Flame-retardant properties

These properties can be partly influenced by different fiber separation procedures, but subsequent fiber treatment processes are more influential. Several noncellulose components have to be removed to assure the compatibility of the plant fibers to the surrounding polymer matrix. Alkalization, washing, or boiling of the plant fibers in 2 to 10% sodium, potassium, or lithium hydroxide solutions leads to the removal of unwanted fiber components, which dissolve during the process.

Alkalization, depending on the concentration of the alkali and the process temperature, can significantly improve the fiber’s mechanical and surface properties. After naturalizing and thoroughly washing and drying the fibers, the following changes as compared to the raw fiber material can be revealed.

- Fibers are purified
- Unwanted fiber ingredients are largely removed
- Fiber separation ability is increased

During the alkalization processes the wax components are saponified and, thereby removed from the fiber surface. The waxes sticking to the fiber surfaces strongly influence the surface properties of cellulose-based materials. Most raw plant fibers are covered with hydrophobic noncellulose compounds (like waxes). It is, therefore, necessary to treat original fibers to remove these noncellulose components. Such a treatment increases the accessibility of surface groups (but not necessarily the specific fiber surface). These uncovered, reactive surface groups can be used in further chemical modification steps to increase the compatibility of natural fibers to nonpolar polymer matrices. [2]

### 2.4.1 Alkaline Treatment

Alkaline treatment or mercerization is one of the most used chemical treatments of natural fibers when used to reinforce thermoplastics and thermosets. The important modification done by alkaline treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax and oils covering the external surface of the fiber cell wall, depolymerizes cellulose and exposes the short length crystallites. [16] Addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide [17]:

$$\mathrm{Fiber} - \mathrm{OH} + \mathrm{NaOH} \rightarrow \mathrm{Fiber} - \mathrm{O} - \mathrm{Na} + \mathrm{H_2O}$$

Thus, alkaline processing directly influences the cellulosic fibril, the degree of polymerization and the extraction of lignin and hemicellulosic compounds. In alkaline treatment, fibers are immersed in NaOH solution for a given period of time. Ray et al. and Mishra et al. treated jute and sisal fibers with 5% aqueous NaOH solution for 2 h up to 72 h at room temperature. [18][19] Similar treatments were attempted by Morrison et al. to treat flax fiber. [20] Garcia et al. reported that 2% alkali solution for 90 s at 200°C and 1.5 MPa pressure was suitable for degumming and defibrillation to individual fibers. [21] These researchers observed that alkali led to an increase in amorphous cellulose content at the expense of crystalline cellulose. It is reported that alkaline treatment has two effects on the fiber which is increases surface roughness resulting in better mechanical interlocking and increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites. [22]

Consequently, alkaline treatment has a lasting effect on the mechanical behavior of flax fibers, especially on fiber strength and stiffness. [23] Van de Weyenberg et al. reported that alkaline treatment gave up to a 30% increase in tensile properties (both strength and modulus) for flax fiber–epoxy composites and coincided with the removal of pectin. Alkaline treatment also significantly improved the mechanical, impact fatigue and dynamic mechanical behaviors of fiber-reinforced composites. [24] Jacob et al. examined the effect of NaOH concentration (0.5, 1, 2, 4 and 10%) for treating sisal fiber-reinforced composites and concluded that maximum tensile strength resulted from the 4% NaOH treatment at room temperature. [25] Mishra et al. reported that 5% NaOH treated sisal fiber-reinforced polyester composite had better tensile strength than 10% NaOH treated composites. This is because at higher alkali concentration, excess delignification of natural fiber occurs resulting in a weaker or damaged fiber. The tensile strength of the composite decreased drastically after certain optimum NaOH concentration. [7]

## 2.5 Epoxy Resin

Epoxy or polyepoxide is a thermosetting polymer formed from reaction of an epoxide "resin" with polyamine "hardener". Epoxy has a wide range of applications, including fiber-reinforced plastic materials and general purpose adhesives. Epoxy is a copolymer; that is, it is formed from two different chemicals. These are referred to as the "resin" and the "hardener". The resin consists of monomers or short chain polymers with an epoxide group at either end. Most common epoxy resins are produced from a reaction between epichlorohydrin and bisphenol-A, though the latter may be replaced by similar chemicals. The hardener consists of polyamine monomers, for example Triethylenetetramine (TETA). When these compounds are mixed together, the amine groups react with the epoxide groups to form a covalent bond. Each NH group can react with an epoxide group, so that the resulting polymer is heavily crosslinked, and is thus rigid and strong. The process of polymerization is called "curing", and can be controlled through temperature and choice of resin and hardener compounds; the process can take minutes to hours. Some formulations benefit from heating during the cure period, whereas others simply require time, and ambient temperatures.

Epoxy resins are a large family of resins that represent some of the high performance resins available in the market. Epoxies generally outperform most other resin types in terms of mechanical properties and resistance to environmental degradation, which leads to their almost exclusive use in the aircraft industry. As a laminating resin their increased adhesive properties and resistance to water degradation make these resins ideal for use in applications such as boat building. Here epoxies are widely used as a primary construction material for high performance boats or as a secondary application to sheath a hull or replace water-degraded polyester resins and gel coats.

The advantages of using epoxy resin are high mechanical and thermal properties, high water resistance, long working times available, temperature resistance can be up to 140°C wet/220°C dry and also low cure shrinkage. [26]

# CHAPTER 3

# METHODOLOGY

## 3.1 Introduction

The purpose of this study is to modify the surface property of coir fiber and to study the bonding mechanism between the coir fiber and the polymer. The experiment was conducted start from the preparation of the coir fiber, where the coconut husk was randomly collect locally, wash thoroughly with water for several times, dried, cut and grind. Next step was the preparation of the stock solution for the alkali treatment and also for the Kappa number test. The experiment was then proceed with the treatment of coir fiber with alkali treatment and bleaching process. After the treatment, the coir fiber was tested for lignin content by Kappa test and tested for surface morphology study using scanning electron microscopy and also thermal characterization of the fiber by using thermo gravimetric analysis. The next step was making the composite by using hot press method and the composite was tested for tensile strength. The experimental flow was summarized in figure 3.1.

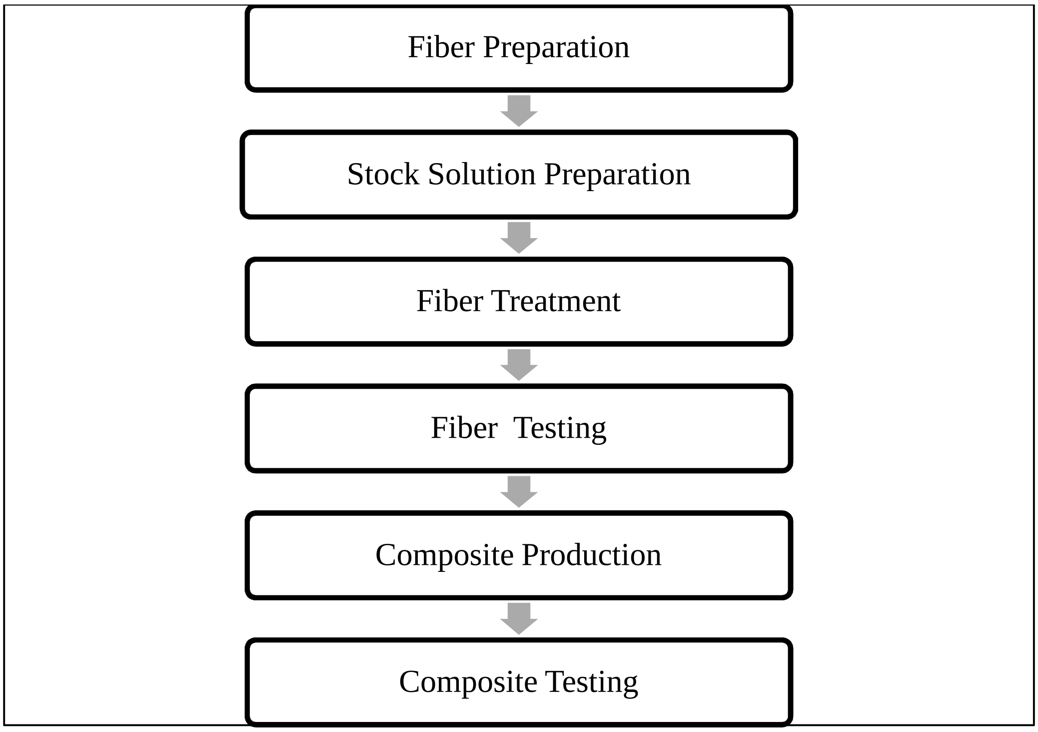


**Figure 3.1:** Experimental flow

## 3.2 Equipments/ Apparatus

In completing the study, some equipment was used. There are:

1. Oven
2. Electric Balance Shimadzu (A W220)
3. Conical Flask
4. Volumetric flask
5. Beaker
6. Pipette
7. Funnel
8. Hot Press
9. Hot Water Bath
10. Universal Tensile tester Shimadzu
11. Thermo gravimetric analyzer TGAQ500
12. Scanning electron microscopy EVO

## 3.3 Reagents

In completing the study, some reagents or chemical was used. There are:

1. Coir Fiber
2. Distilled water
3. Natrium Hydroxide ($NaOH$) (20%, 25%, 30%)
4. 0.1N Potassium Permanganate ($KMnO_4$)
5. 4.0N Sulphuric Acid ($H_2SO_4$)
6. 1.0N Potassium Iodide ($KI$)
7. 0.2N Sodium Thiosulfate ($Na_2S_2O_3$)
8. 2% Starch Indicator Solution
9. 2% Hydrogen Peroxide Solution ($H_2O_2$)
10. 1% Acetic Acid Solution ($CH_3COOH$)
11. Epoxy Resin & Hardener

## 3.4 Preparation of Coir Fiber

**Figure 3.2:** Dried coconut husk

The coir fiber used in this research was obtained from coconut tree that grow in village area of Negeri Sembilan. Coir fibers were extracted from the dried husk

(Figure 3.2) without any prior retting but after soaking in water for a few hours. The extracted coir fibers were then wash with water for several times to remove all the dirt particles. Then, the coir fibers were sun dried for 6 hours before grinding sieving to get an average fiber length of (0.015±0.005m) (Figure 3.3).

**Figure 3.3:** Coir fiber after grinding and dried

## 3.5 Fiber Treatment

### 3.5.1 Alkali Treatment

The alkali treatment of the coir fiber was done by using various concentration of Sodium Hydroxide (NaOH) which is 20%, 25%, and 30%. A specimen about 100g of coir fiber with approximate length of 2cm was taken. Four set of fiber with equal weight which is 10g was prepared using electric balance. The fiber was then treated with 100ml of NaOH solution of varying concentrations (20%, 25%, and 30%) by fully immersed the fiber in NaOH solution in 250ml conical flask and another one in distilled water for control purposes in a water bath with temperature of 70°C for 6 hours. The treated fibers was then rinsed several times with distilled water and dried under sun for 3 hours.

### 3.5.2 Bleaching Process

After alkali treatment process, the fiber was then continued to bleaching process. The purpose of the bleaching is to differentiate the color of untreated and treated coir fiber. The coir fiber was immersed to a 100ml 2% Hydrogen Peroxide solution in 250ml conical flask in a water bath with temperature of 70°C for 6 hours. The coir fibers was then rinsed several times with distilled water and dried under sun for 3 hours. Subsequently, the coir fiber were wash with Acetic Acid solution to neutralize the fiber by immersing the fiber in 100ml of 1% Acetic Acid solution for 15 minutes and then wash thoroughly with distilled water. [27]

## 3.6 Fiber Characterization

### 3.6.1 Kappa Test

The coir fiber was weighted from (2.0 $\underline{+}$ 0.1) g as it suggested only for first trial. The target was to get enough pulp which will consume approximately 50% of the potassium permanganate ($KMnO_4$) solution. The weighed fiber then disintegrated with using blender with 500ml of distilled water for 3 minutes. The fiber then transfer into the 2,000ml beaker (known as a reaction beaker). The blender was rinsed with 295 ml of distilled water and discharged it into the same beaker. The magnetic bar was put into the beaker and started to stir the fiber gently. 100ml of 0.1N $KMnO_4$ solutions was pipette into the 250ml beaker. 100ml of 4.0N sulphuric acids ($H_2SO_4$) was measured by using 100ml graduate cylinder and it was mixed with $KMnO_4$ solution. Then, the mixture solution was transferred into the fiber. And, the stop watch started at the same time. 250ml of beaker was then rinsed with 5ml of distilled water and it was poured into the reaction beaker (total volume of the reaction beaker becomes 1,000ml). The temperature of the fiber stock was measured.

After 10 minutes, the chemical reaction in the reaction beaker was stopped by adding of 20ml 1.0N potassium iodide (KI) solution. Then, free iodide was titrated with 0.2 N sodium thiosulfate ($Na_2S_2O_3$) solutions. When the color of the fiber stock changed to light yellow, a few drops of 2% starch indicator solution was titrated. The titration was stopped when the fiber color becomes white (the solution becomes colorless). The volume of 0.2 N $Na_2S_2O_3$ solution was that has been used for titration was measured. This experiment then duplicated. With the same methods, a blank test was carried out without using fiber. The corrected average kappa number was calculated at 25˚C ± 0.2˚C. [28]

### 3.6.2 Thermo Gravimetric Analysis

Thermal characterization of fibers was studied by thermo gravimetric analysis (TGA). The analysis was carried out in a TGAQ500 instrument (Figure 3.4), using 10 ± 0.5 mg of the fiber in a temperature range of 30 °C and 600°C, in a nitrogen and oxygen atmosphere (15 ml/min) and at a heating rate of 10°C/min.

**Figure 3.4:** Thermo gravimetric analyzer

### 3.6.3 Fiber Surface Morphology

In order to evaluate changes in the surface morphology, all investigated natural fibers were analyzed by scanning electron microscopy (SEM) using an Evo S2700 scanning electron microscope (figure 3.5). The excitation energy was 10 keV with a beam current of 0.5 nA.

**Figure 3.5:** Scanning electron microscopy

## 3.7 Composite Production

The mold was first prepared with a sufficient degree of finish. A release agent was then applied to the mold. The epoxy resin was prepared by mixing the resin with the hardener by 5:1 ratio. The epoxy resin was then applied with just a thin layer to the mold to get a resin-rich top surface. The coir fiber was then spread evenly on the resin with 50wt% of fiber. After that, the remaining resin was poured evenly on top of the spread fiber to make sure the fiber is fully wet by the resin. The mold was then closed and proceeds to the hot press machine (Figure 3.6). The polymer was then cured at 90°C for 45minutes. After that, the polymer was taken out and allowed for cooling at room temperature for 1 day before proceed with tensile test.

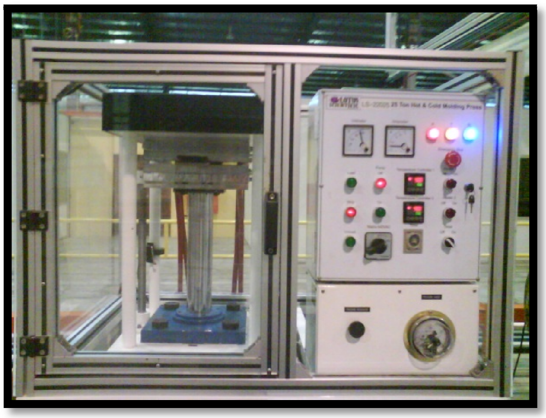
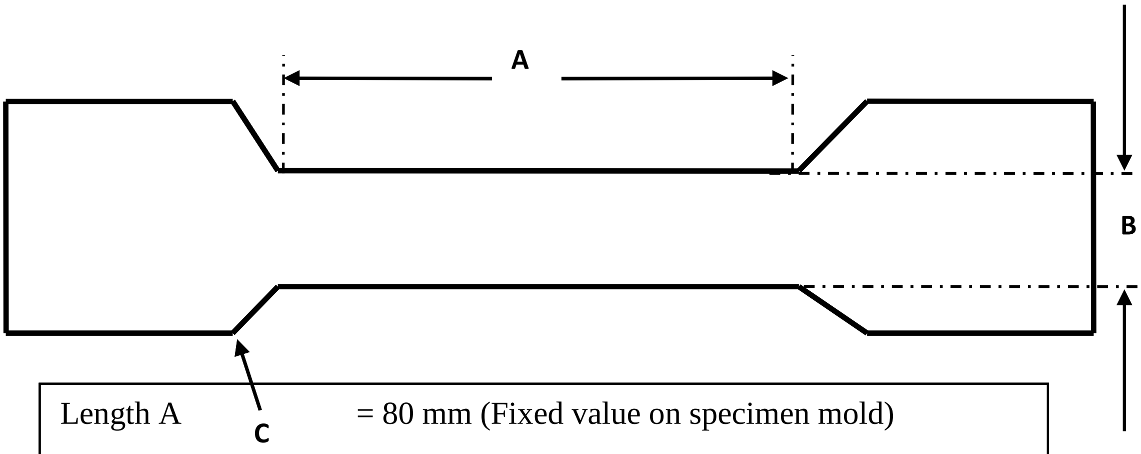


**Figure 3.6:** Hot press machine

## 3.8 Composite Testing

The tensile strength tests for the polymer were performed with Universal Tensile tester Shimadzu instrument (Figure 3.8). Polymer specimen for tensile testing is according to ASTM D638-58T. First, the polymer specimens were cut into dumbbell shape. Electronic digital caliper (Mitutoyo-Absolute Digimatic) was used to measure the width and thickness of the specimens at several points along their narrow section. Width of the specimens was measured as the distance between the cutting edges of the die in the narrow section. Thickness of the specimen was measured at different parts of the specimen and average value was taken. The dimensions of the specimens are shown in Figure 3.7.



Length A = 80 mm (Fixed value on specimen mold)

Length B = 12 mm (Fixed value on specimen mold)

**Figure 3.7:** Dimensions of dumb bell specimen (ASTM D638-58T, type v).

Each end of the polymer specimens were taped with masking tape to prevent it from being torn when it was placed in the grips of the testing instrument. Once polymer specimen was properly place in the testing instrument, the grips were tightened evenly and firmly to prevent slippage of the specimen during test. The crosshead speed of the instrument was fixed to 5mm/min. The minimum number of specimens needed for each sample was five.

**Figure 3.8:** Universal tensile tester

# CHAPTER 4

# RESULTS AND DISCUSSION

## 4.1 Kappa Number

The kappa test is an indirect method for determining lignin by the consumption of permanganate ion by lignin. The kappa number is the number of milliliters of 0.1N $KMnO_4$ consumed by 1 gram of pulp in 0.5N sulfuric acid after 10 minutes reaction time at 25°C under conditions such that 50% of the permanganate remains unreacted. Kappa no. used to show the lignin content in the pulp. The higher the kappa number, the higher the lignin content found in the pulp and vice versa.

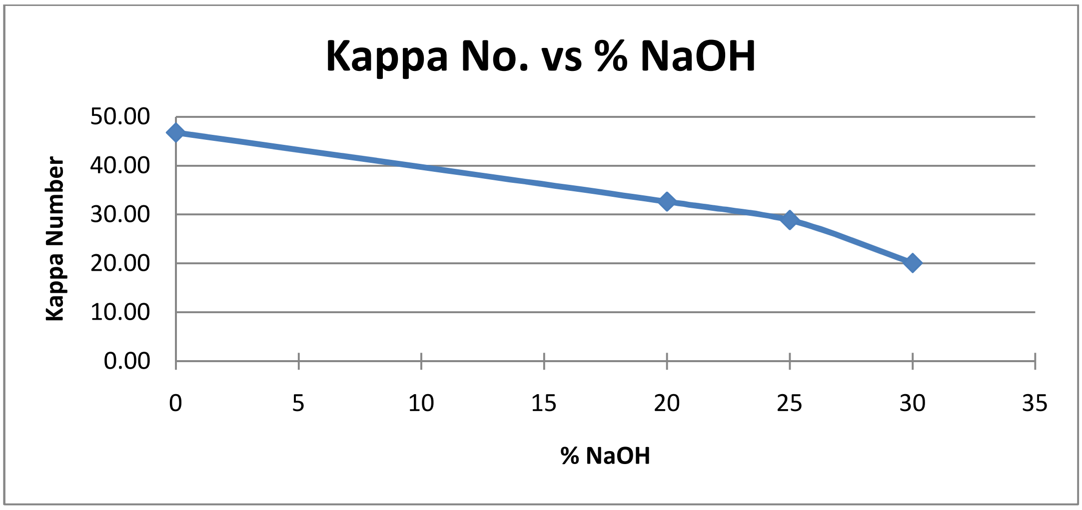


**Figure 4.1:** Graph of Kappa Number versus Concentration of Alkali used

The graph shows (Figure 4.1**)** untreated coir fiber have higher kappa number than 20%, 25%, and 30% NaOH treated fiber. We can see a trend that is when the concentration of alkali used is increased, it reduced the Kappa number. Higher kappa number means that the fiber contains high lignin content. Lignin is insoluble in acidic medium. Higher percentages of NaOH used can soluble more lignin than lower percentage of NaOH pulp. So, lignin content in high percentage of NaOH fiber become lower and the kappa no. also become lower.

## 4.2 Thermal Effects

The results of thermo gravimetric analysis of coir fibers in nitrogen and oxygen atmospheres studied here are shown in Fig. 4.2 and 4.3. These curves reveal the decomposition profiles of coir fibers, which are characterized by mass loss between room temperature and 150 °C, followed by double oxidation peaks, representing the decomposition processes of the fiber constituents.

In general, there were three stages in the decomposition profile of the coir fiber. The first stage was the loss of material at low temperatures (between room temperature and 200°C) and this may be attributed by the presence of absorbed or combined water in the coir fiber. The second stage was the degradation period of the fiber. In this stage the fibers start to lose their properties and become more unstable. While the third stage was the decomposition of fiber that is the fiber’s molecular chain started to break down (above 400 °C) and also carbonization of fiber occurs with higher mass loss, showing a residual mass of approximately 30% at the end of the process. This residual mass may be associated with the presence of silicon, also present in other natural fibers.

The TGA measurement gives information about the composition and thermal stability of coir fibers. For specific thermal analysis of the fiber, the analysis was taken for untreated fiber and 20% NaOH treated fiber. For the sake of comparison, the absorb moisture content, weight percent remaining at 300°C, and decomposition temperature were compared.

### 4.2.1 Untreated Fiber

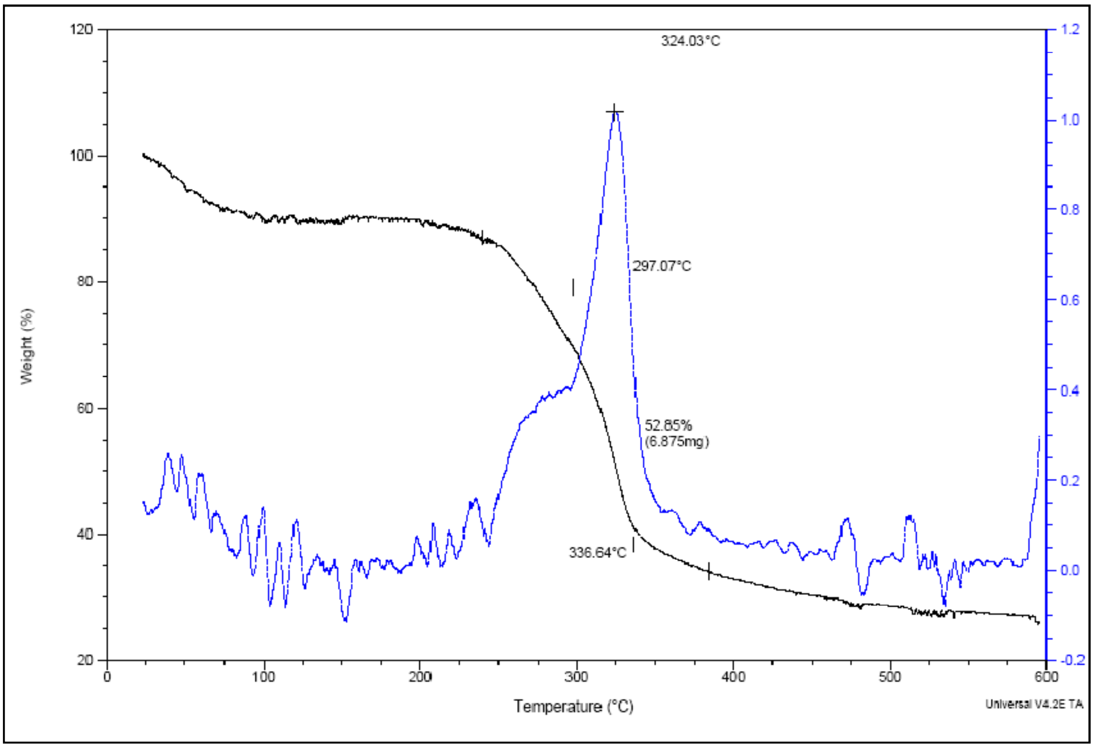


**Figure 4.2:** Decomposition profile of untreated coir fiber

From Figure 4.2, the absorb moisture content for the untreated coir fiber is 12.5%. While the weight percent remaining at 300°C is 70%. The decomposition temperature for untreated fiber is 336.64°C

### 4.2.2 20% NaOH Treated Fiber

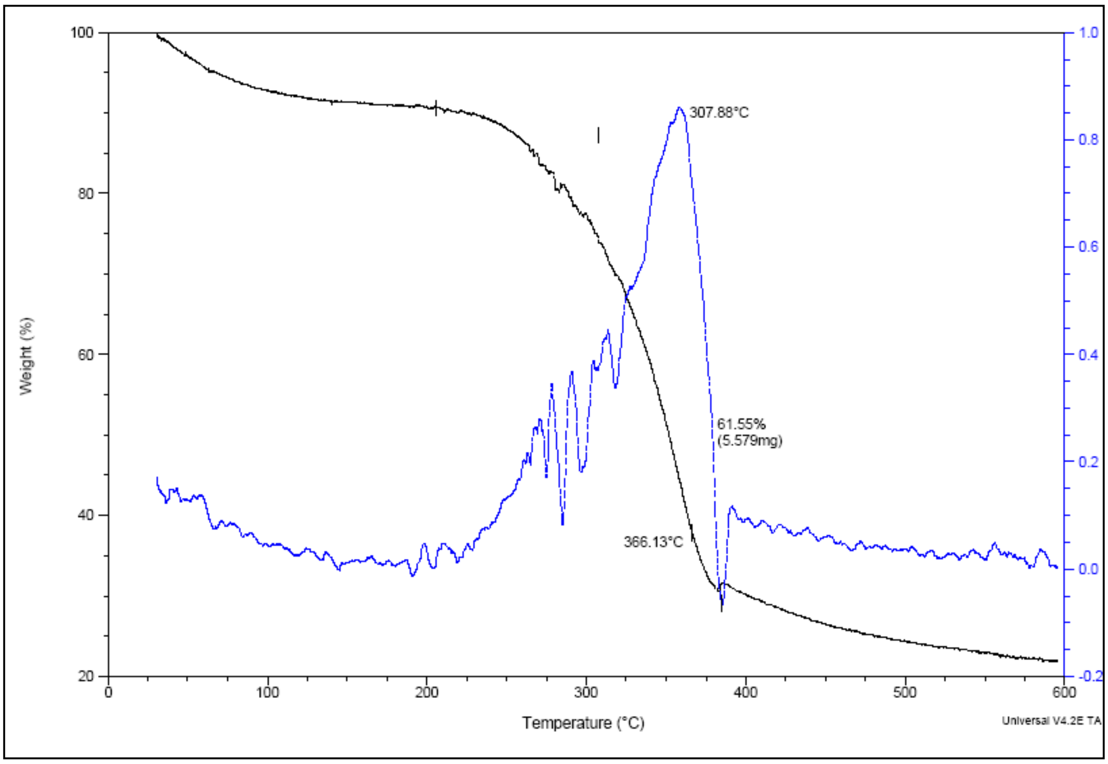


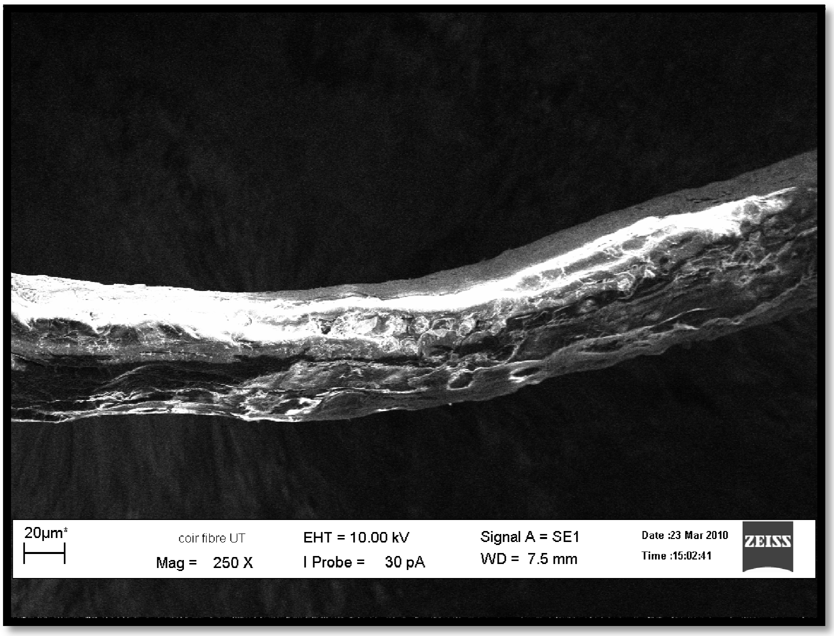
**Figure 4.3:** Decomposition profile of 20% NaOH treated coir fiber

From Figure 4.3, the absorb moisture content for the 20% NaOH treated coir fiber is 9%. While the weight percent remaining at 300°C is 78%. The decomposition temperature for 20% NaOH treated fiber is 366.13°C.

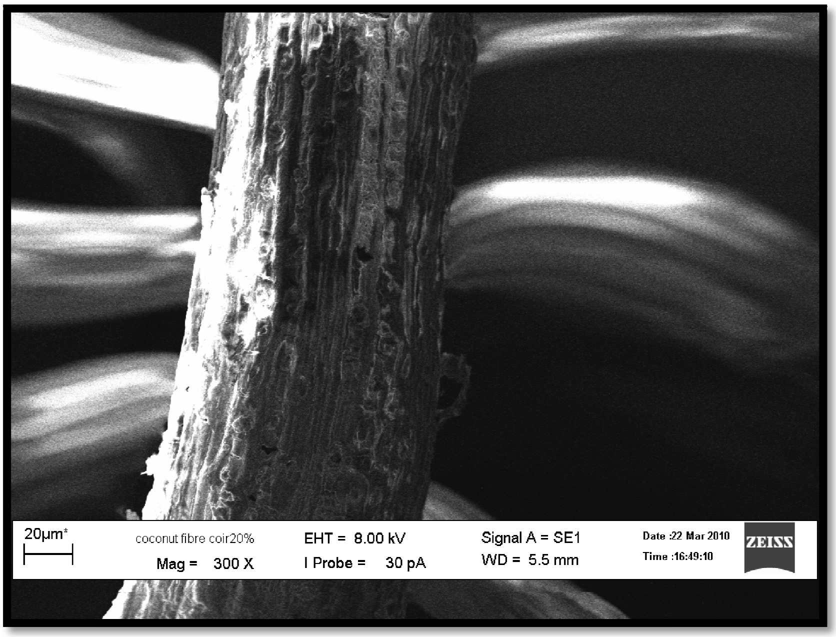
From the comparison that have been made above, we can understand that alkali treatment have improved the thermal stability of the coir fiber by making the fiber more thermal resistant as approved by the increase in the decomposition temperature of coir fiber and also increase in weight percent remaining at specific temperature that is at 300°C.

## 4.3 Fiber Surface Morphology

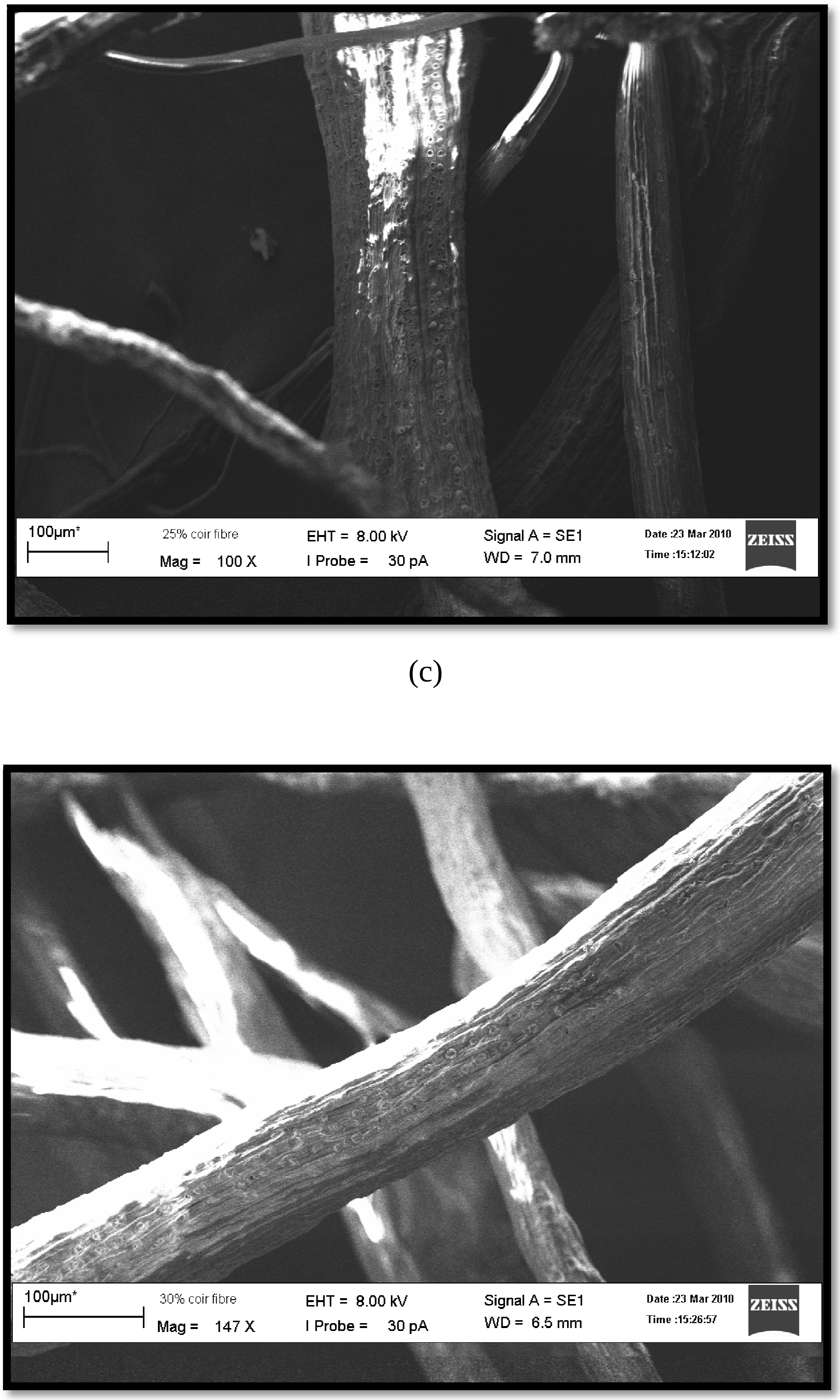
Scanning electron microscopic analysis examined the surface topology of untreated and treated fibers. The removal of surface impurities on plant fibers is advantageous for fiber–matrix adhesion as it facilitates both mechanical interlocking and the bonding reaction due to the exposure of the hydroxyl groups to the chemicals used in treatment.



(a)



(b)



(d)

**Figure 4.4:** Scanning electron microscopic image of coir fiber at different alkali concentration: (a) Untreated fiber; (b) 20%NaOH; (c) 25%NaOH; (d) 30%NaOH

Figure 4.4 present the images of the appearance of the coir fiber before and after the alkali treatment, respectively. From figure 4.4(a) one may see that the surface of the coir fiber is covered with a layer of substances, which may include pectin, lignin and other impurities. The surface is not smooth, spread with nodes and irregular stripes. After the NaOH treatment (figure 4.4(b,c,d)), most of the lignin and pectin are removed resulting in a rougher surface. One may also notice that there are rows of pits on the surface. These would increase the mechanical bonding between the matrix and the coir fibers in the composite fabrication.

## 4.4 Tensile Strength

The volume fraction of the coir fiber in the composites was 50%. Tensile strength testing results are summarized in Table 4.1.

**Table 4.1:** Tensile strength and elongation at break of coir fiber at different alkali concentration

| Concentration of Alkali | Tensile Strength (MPa) | Elongation at Break (%) |
|---|---|---|
| 0 | 7.32 | 12.80 |
| 20 | 7.97 | 16.42 |
| 25 | 13.93 | 18.21 |
| 30 | 4.66 | 12.86 |

Significant difference is noticed in the tensile strength between the alkali treated and untreated coir fiber composites. Tensile strength of the composites when using the alkali treated coir fibers is usually greater. This implies that after alkali treatment, most of the fats, lignin and pectin covered the fiber surface are removed, which improved the fiber adhesive character in the combination with the matrix.

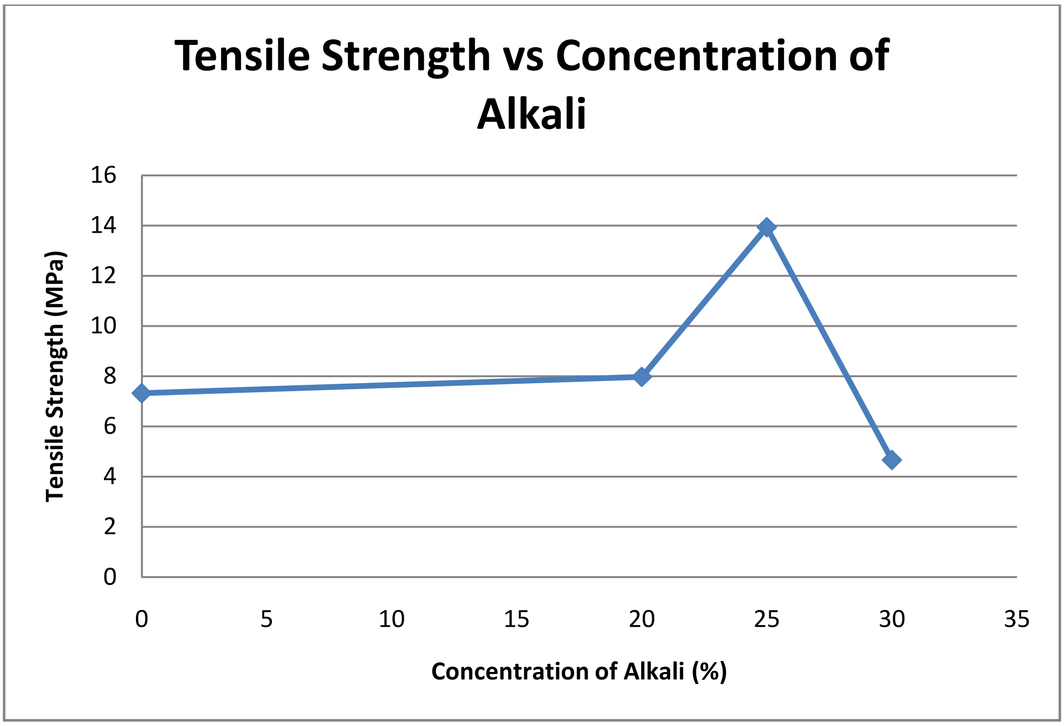


**Figure 4.5:** Graph of coir-epoxy composite's tensile strength versus concentration of alkali

The smaller tensile strength in the NaOH 30% indicates that the deterioration of the fiber during the alkali treatment is serious, resulting in a smaller tensile strength. In these 2 cases, higher alkali concentration may induce greater reduction of the coir fiber strength, but better adhesion caused by the denser NaOH between the fiber and the matrix may be beneficial to the tensile strength of the composites. To achieve a composite with better strength, alkali concentration of 25% may be recommended for economical consideration. Significant increase is noticed for the elongation at break values between the composites made by using the alkali treated and untreated coir fibers. The higher elongation at break for the alkali treated coir fiber composites indicates that the removal of the lignin and pectin improved the elasticity of the coir fiber.

# CHAPTER 5

# CONCLUSION AND RECOMMENDATIONS

## 5.1 Conclusions

This study on the title of “Modification of coir fiber as polymer reinforcement” has been successfully done. From the result obtained and analysis that have been done, it can be concluded that the purpose of this study has been achieved. This study is to modify the surface property of coir fiber. Other purpose of this study is to study the bonding mechanism between the coir fiber and the composite.

The different percentage of NaOH used does affect fiber properties such as lignin content, thermal stability, surface morphology and also tensile strength of the resulting coir-epoxy composite.

High kappa number means high lignin content. Increase percentages of NaOH decrease the kappa number. It is due to lignin which is resistance to acid medium. Untreated coir fiber has Kappa number of 46.75, 20% NaOH treated fiber has Kappa number of 32.63, 25% NaOH treated fiber has Kappa number of 28.86, and 30% NaOH treated fiber has Kappa number of 20.09.

Surface treatments of coir fiber have also improved the thermal stability of the coir fiber. The treatment have made the fiber more thermal resistant as approved by the increase in the decomposition temperature of coir fiber and also increase in weight percent remaining at specific temperature that is at 300°C. For untreated fiber, it have decomposition temperature of 336.64°C while for 20%NaOH treated fiber, the decomposition temperature is 366.13°C.

Removal of surface impurities such as lignin and oily layer is also result from surface treatment producing modifications on the surface and this result is supported by surface morphology study done by using SEM.

Tensile strength properties of coir-epoxy composite have also improved by the surface treatment by improving the adhesive ability of the coir fiber with the matrix in the fabricated composites, resulting in a greater tensile strength of the material. From the results, increasing in NaOH concentration has result in increasing tensile strength of composite and the optimum concentration for the highest tensile strength is at 25% NaOH treated fiber. Alkali concentration of 25% may be recommended for economical consideration. Significant increase is also noticed for the elongation at break values between the composites made by using the alkali treated and untreated coir fibers.

Because of the above conclusion, we can conclude that renewable fibers like coir can be used as reinforcing materials for low cost composites, due to the economic and environmental advantages of such materials.

## 5.2 Recommendations

As for the recommendations, we can further study the quality of the coir-epoxy polymer that has been made by characterize the composite by doing impact test and density test. With these test, we can definitely confirm that we have made a high quality polymer and can be commercialized. Besides that, we can optimize the strength of the composite by choosing the 25%NaOH alkali treatment and manipulate the fiber loading to get a right configuration between the coir and the epoxy resin.

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# APPENDICES

The following items are including in this Appendices.

- Appendix A : Stock Solution Preparation
- Appendix B : Calculation of Kappa number

## Appendix A: NaOH Solution Preparation

1. Determination of weight of NaOH used

a) Calculate volume percent of NaOH from solution

V% = (Volume NaOH) / ((Volume NaOH) + (Volume of water))

b) Calculate volume of NaOH needed to produce 450ml of solution

Volume of NaOH for 450 ml = V% X 450

c) Calculate mass of NaOH needed

Mass of NaOH = (Volume of NaOH for 450 ml) X density of NaOH

d) Calculate volume of water to be added

Volume of water = 450ml – (Volume of NaOH for 450 ml)

- Example of calculation for 20% NaOH (taking a basis of 100g)

a) V% = (20/2.13) / ((20/2.13) + (80/1))
   = 0.10504

b) Volume of NaOH for 450 ml = 0.10504 X 450
   = 47.2689 ml

c) Mass of NaOH = 47.2689 ml X 2.13g/cm$^3$
   = 100.68g

d) Volume of water = 450ml - 47.2689 ml
   = 402.73 ml

- Summary of mass of NaOH & volume of water used

| **Concentration of NaOH (%)** | **Mass of NaOH (g)** | **Volume of water (ml)** |
|---|---|---|
| 20 | 100.68 | 402.73 |
| 25 | 129.69 | 389.12 |
| 30 | 160.55 | 374.62 |
| **Total** | **390.92** | **1166.47** |

## Appendix B: Calculation of Kappa Number

Calculation of kappa number as follows:

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

k = kappa number
p = amount of 0.1 N permanganate actually consumed by the test specimen, mL
w = weight of moisture-free pulp in the specimen, g

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

p = amount of 0.1 N permanganate 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 25ºC :

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

t = current temperature during experiment

**Example of calculation**

**20% NaOH**

| NaOH (%) | Mass of fiber (g) | $Na_2S_2O_3$ (ml) | Temp (ºC) | Kappa No |
|---|---|---|---|---|
| 0 | 2 | 10.0 | 30 | 46.75 |
| 20 | 2 | 24.1 | 32 | 32.63 |
| 25 | 2 | 28.7 | 31 | 28.86 |
| 30 | 2 | 37.9 | 32 | 20.09 |

Blank Test:

Volume $Na_2S_2O_3$ = 60 ml

**20% NaOH**

Temperature = 32 ºC

Mass of fiber = 2g

Volume $Na_2S_2O_3$ = 24.1 ml

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

$$p = \frac{(60 - 24.1)(0.2)}{(0.1)}$$

$$= 71.8 \text{ ml}$$

Correct kappa number :

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

$$= \frac{71.8}{2}\,[1 + 0.013\,(25-32)]$$

$$= 32.63$$