INVESTIGATION ON THE MECHANICAL AND PHYSICAL PROPERTIES OF TREATED AND UNTREATED WOVEN SUGAR-PALM FIBRE REINFORCED COMPOSITE

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ABSTRACT

Natural fibres offer environmental benefits such as biodegradable and can be obtained from renewable resources. In term of strength, natural fibres are still inferior by the synthetic fibres. However, there is lack of investigations done to characterise and compare different types and orientation done on the natural fibres. The basic purpose of the study is to determine the mechanical and physical properties of sugar-palm fibres under various treatment processes, to analyse the performance of mechanical and physical properties of different type of orientations and to compare the obtained properties with other natural fibres. The study starts with preparation of the sugar-palm fibres, followed by surface treatments by alkali and acid solutions and weaving the fibre into a woven fibre before putting it into the mould. After dried, the composite board was cut into specimens and tested for 3-point flexural test and tensile test. Surface morphological analysis by using *SEM* machine was done before doing the data analysis. The outcome of the study proved that the chemical treated fibres has higher tensile and flexural properties compared to the untreated fibres where the value of tensile stress for alkali treated, acid treated and untreated fibres are 27.92 *MPa*, 26.41 *MPa* and 25.75 *MPa* respectively. In addition, the value of flexural stress for alkali treated, acid treated and untreated fibres are 81.16 *MPa*, 79.07 *MPa* and 41.34 *MPa* respectively. As a conclusion, woven sugar-palm fibre reinforced epoxy composite is appropriate to use as a replacement for the synthetic fibre such as glass and carbon fibre since it has good tensile and flexural properties. With these properties, it is proved that the treated sugar-palm fibre composite can withstand high stress load during operation compared to the untreated sugar-palm fibre composite.

ABSTRAK

Gentian asli menawarkan faedah alam sekitar seperti kebolehan untuk terbiodegradasi serta dapat diperolehi daripada sumber yang boleh diperbaharui. Dari segi kekuatan, gentian asli mempunyai nilai yang lebih rendah daripada gentian sintetik. Tujuan utama kajian ini adalah untuk menentukan sifat mekanikal dan fizikal gentian gula kabung untuk pelbagai jenis rawatan, untuk menganalisis sifat mekanikal dan fizikal gentian gula kabung bagi orientasi yang berbeza dan untuk membandingkan keputusan yang diperolehi dengan gentian semula jadi yang lain seperti kenaf, sisal dll. Proses kajian ini dimulakan dengan penyediaan gentian gula kabung, rawatan gentian menggunakan cecair alkali dan asid serta penganyaman gentian sebelum ia dimasukkan ke dalam acuan. Selepas dikeringkan, komposit tersebut dipotong kepada beberapa spesimen dan diuji untuk ujian 3-titik lenturan dan ujian tegangan. Permukaan komposit telah analisis dengan menggunakan mesin *SEM*. Hasil kajian membuktikan bahawa gentian yang telah dirawat dengan bahan kimia mempunyai sifat tegangan adan lenturan yang lebih tinggi berbanding dengan gentian yang tidak dirawat di mana nilai tegangan bagi gentian yang dirawat dengan alkali dan asid; serta gentian yang tidak dirawat adalah 27.92 *MPa*, 26.41 *MPa* dan 25.75 *MPa*. Di samping itu, nilai lenturan untuk gentian yang dirawat dengan alkali dan asid; serta gentian yang tidak dirawat adalah 81.16 *MPa*, 79.07 *MPa* dan 41.34 *MPa*. Kesimpulannya, komposit yang diperbuat daripada gentian gula kabung yang dianyam serta diperkukuh dengan epoksi adalah sesuai untuk digunakan untuk menggantikan gentian sintetik kerana ia mempunyai sifat tegangan dan lenturan yang baik. Ia terbukti bahawa komposit gentian gula kabung yang dirawat boleh menahan beban tekanan yang tinggi semasa digunakan berbanding dengan komposit gentian gula kabung yang tidak dirawat.

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LIST OF SYMBOLS AND ABBREVIATION

°C	Degree Celsius
%	Percentage
µm	Micrometre
15% wt.	15% weight
ASTM	American Standard Testing Machine
CO_2	Carbon Dioxide
g	Gram
g/cm^3	Gram per centimetre cubic
g/mol	Gram per mol
GPa	Giga Pascal
HCl	Hydrochloric Acid
kg	KiloGram
kg/L	Kilogram per litre
kN	KiloNewton
kV	KiloVolt
m	Mass
M	Molarity
mm	Millimeter
mm/min	Millimetre per minute
MPa	Mega Pascal
NaOH	Sodium Hydroxide
NFRC	Natural Fibre Reinforced Composite
PET	PolyEthylene Terephthalate
SEM	Scanning Electron Microscope

UN	United Nation
UTHM	Universiti Tun Hussein Onn Malaysia
UTS	Ultimate Tensile Strength
V	Volume

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The usage of natural fibre as a component of composite material has been a trend in the recent years. The use of natural fibres at the industrial level improves the environmental sustainability of the parts being constructed, especially in the automotive industry. In the building industry, the interest in natural fibres is mostly economical and technical. Natural fibres also allow higher insulation properties than the current materials offered. In addition, it is cheaper than the synthetic fibres since Malaysia has vast amount of natural fibres resources available.

For example, Kenaf is famous for making furniture and other natural fibres like sisal, banana, jute, oil palm and coir has been used as reinforcement in thermoplastics composite for applications in consumer goods.

For these applications, the natural fibres came in with several types and shapes which are long, short, whiskers, random, woven, etc. Different types and orientations are expected to behave in different characteristics.

The main purpose of this study is to obtain the mechanical and physical performances of treated and untreated woven sugar-palm fibres. Here, the treated terminology defines as fibres undergo prescribed chemical treatment. In order to obtain the main objective, several sub-objectives are drawn as described in the next section.

There are two stages involved in this study, which are preparation and testing processes of the natural fibre. For the first stage, the treated woven sugar-palm fibre will be prepared. Two parameters will be used which are the solution used and soaking period. Two different types of solution will be used for the treatment, classified as acidic and alkali condition for a prescribed soaking period.

The second stages will involve the determination of mechanical and physical properties of the prepared samples. As for the mechanical testing, tensile test and flexural test will be used and for the physical test, porosity testing will be used. This will eventually defines the mechanical and physical behaviour of the composite.

Ticoalu, Aravinthan and Cardona (2011) studied gomuti fibre/polyester composites. The study was to investigate the tensile and flexural properties of the fibres. The test was done to four different forms of fibre orientation which are random original layout, chopped random, unidirectional and woven mat. During the experiment, the fibre was not chemically treated in order to determine the properties of untreated gomuti fibre/polyester composites.

Bachtiar et al. (2010a) studied the tensile properties of single sugar palm fibre. Retting process was done to the fibres in order to separate the stalks from its core. Single fibre tensile test was conducted based on *ASTM* D3379 for single fibre tensile test. During the test, a single fibre was attached to a cardboard frame.

Bachtiar et al. (2008) study the effect of alkali treatment on tensile properties of sugar palm fibre reinforced epoxy composites. The treatment was done using Sodium Hydroxide, *NaOH* solutions at two different concentrations of 0.25*M* and 0.5M and three different soaking times which are 1 hour, 4 hours and 8 hours. The sugar palm/epoxy composites were reinforced with 10*%* weight fraction of fibres and produced using hand layup technique. The composites then were tested for the determination of its tensile properties.

Bactiar et al. (2010b) in their other work studies the flexural properties of alkaline treated sugar palm fibre reinforced epoxy composites. The sugar palm/epoxy composites were reinforced with 10% weight fraction of fibres. The fibres were treated using sodium hydroxide, *NaOH* solution with concentration of 0.25*M* and 0.5*M* for 1 hour, 4 hours and 8 hours soaking time. The authors stated that the purpose of treating the fibre with alkali was to enhance the interfacial bonding between the matrix and the fibre surfaces.

Ishak et al. (2009) studied the effect of biological base treatment agent, sea water, to the flexural strength of sugar palm fibre composites. The sugar palm fibres were soaked in sea water for 30 days. In order to maintain the salinity of the sea water, the geographical location of sea water can affect the purity of the sea water. Therefore, the sample was taken 200 *m* away from the shore. The sugar palm epoxy composite that tested were made from 20*%* and 30*%* fibre content. The result of the test was compared with untreated sugar palm epoxy composite.

Sastra et al. (2005) studied the flexural properties of Arenga Pinnata fibres as a natural fibre and epoxy resin as a matrix. In their study, the fibres were mixed with epoxy resin at various fibre weight percentages of 10, 15 and 20*%* with 3 different fibres orientations such as long random, chopped random and woven roving. This research used hand layup technique to produce the specimen test with curing time for the composite plates is in room temperature of 25 to 30^{o}C.

Meatherall (2008) study the establishment of protocols for natural fibre density measurement. In this study, the researcher investigated the current method for the testing of natural fibre density by using oilseed flax fibre. A few methods have been tested such as diameter and linear density method; Helium Pycnometry method and Archimedes method.

Most of the porosity element in previous studies involved with model development that integrates the volumetric composition of the fibre composites with their mechanical properties. Madsen, Thygasen and Lilholt (2009) study a model, based on a modified rule of mixtures to determine the influence of porosity of the composite stiffness. The model that developed integrates the volumetric composition of the composites with their mechanical properties. The fibre weight fraction was used as an independent parameter to calculate the complete volumetric composition.

Another study made by Madsen and Lilholt (2003) investigates the physical and mechanical properties of unidirectional plant fibre composites. This study focused on the evaluation of the influence of porosity to the fibre composites. A theoretical model was fitted to the data in order to describe the composite volumetric interaction between contents of fibre, matrix and porosity by using the weight fraction and porosity fraction of the fibre composites.

1.2 Research Objective

This research was done to investigate several uncertainties regarding the sugar-palm fibre epoxy composite.

1. To determine the mechanical and physical properties of sugar-palm fibre under various treatment processes.
2. To analyse the performance of mechanical and physical properties between woven and non-woven type of sugar-palm fibre reinforced composite.
3. To compare the obtained properties with other natural fibres.

1.3 Problem Statement

Natural fibres offer environmental benefits such as biodegradable and can be obtained from renewable resources. In term of strength, natural fibres are still inferior by the synthetic fibres. The sugar-palm fibres come out with several types and shapes which are long, short, whiskers, random, woven, etc. Different types and orientations are expected to behave in different characteristics. However, there are less investigation done to characterise and compare the different types and orientation of the natural fibres. Therefore, it is necessary to investigate the mechanical and physical properties of the woven type and the possible improvement by chemical treatments.

1.4 Scope of study

The study will be focused on scopes as listed below :

1. The natural fibre that will be used in this study is sugar-palm fibre and will use the treated woven sugar-palm fibre as the main material reinforced with epoxy matrix.
2. The thickness of the samples will be limited to only a layer (approximation within 5 to 8 *mm*) of woven sugar-palm fibre in the composite.
3. In the preparation of the treated sugar-palm fibre, the type of the solution will be categorised according to Hydrochloric acid, *HCl* (acidic condition) and Sodium Hydroxide, *NaOH* (alkali condition).

4. For mechanical and physical properties, the samples will be tested for tensile and flexural testing to evaluate the mechanical properties; whereas the density tests and *SEM* analysis to evaluate the physical property of the treated woven sugar-palm fibre.

1.5 Significant of study

The significance of this study is to explore the potential of the abundant natural resources from forests for use as a reinforcement fibre in composites. The use of sugar-palm fibre also has an economical advantage if the glass or carbon fibre can be replaced by sugar-palm fibre.

CHAPTER 2

LITERATURE REVIEW

United Nation (*UN*) has declared that the year 2009 will be the international year of natural fibres (Food and Agriculture Organization of the United Nations, 2008). The natural fibre industries had employ millions of people all over the world, especially in the developing countries. Since this industry is a non-food commodity, the products are processed in many small and large industries and used by consumers all over the world. Generally, by using the natural fibres that are CO_2 neutral resources, it had been that believed it will contribute to a greener planet. Not only limited to that, by using the natural fibres in other application such as building or automotive industries will also give some advantages to the world's economy and this advantage will be discussed in later section.

2.1 Type of fibres

In this world, the fibres that are used to make products of everyday use such as tables, chairs, cars, clothes, etc. can be divided into two types, namely synthetic fibre and natural fibre. These two fibres are generally differentiated with the type of materials as they are made with.

2.1.1 Synthetic Fibre

According to Excellup (2009), synthetic comes from the word synthesis which means make while the word synthetic means manmade. Therefore, the manmade fibres are called synthetic fibres. A synthetic fibre consists of a chain of small units joined together and each small unit consists of chemical substances. Then, the small units combine to form a large single unit called polymer. The word '*polymer*' actually comes from Greek words of *poly* and *mer* that each of them means many and part or unit. In simpler word, polymer is made from many repeating units.

Synthetic fibre can be divided into three types which are rayon, nylon and; polyester and acrylic. Rayon fibre was found at the end of nineteenth century and during that time, the fibre was made from a chemical treated wood pulp. At the end of the research, the scientists had produced a fibre that has similar properties to silk and because of that, rayon fibre was also called artificial silk.

Although the rayon fibre was obtained from natural sources such as wood pulp, yet it is still a manmade fibre. The advantages of this fibre are that the price is much cheaper and can be woven like the original silk fibres. This type of fibre can also be dyed in a wide variety of colours. Rayon fibres, if it is mixed with cotton, can produce bed sheets and if it is mixed with wool can produce carpets.

Figure 2.1 : Rayon scarf (Proex Trading Private Limited, 2011)

Another type of fibre that falls under the same category as rayon is nylon. The production of fibre nylon started in 1931, where at the start of production, the fibre was made without using any raw material like from plant or animal. Since this fibre is a manmade fibre, it was produced from coal, water and air; and hence created the first fully synthetic fibre.

The advantage of this synthetic fibre is that it is strong, light in weight and elastic. The fibre is glossy and easy to wash. That's why this type of fibre is famous and usually used for making clothes. From the nylon fibre, products like socks, ropes, tents, toothbrushes, car seat belts, sleeping bags, curtains, etc. was produced. Since nylon thread is actually stronger than a steel wire, it is also used for making parachutes and ropes for rock climbing activities (Excellup, 2009).

Figure 2.2 : Nylon tent (Focus Technology Co., Ltd., 2012)

The last type of synthetic fibres is polyester and acrylic. Fabrics that are made from polyester fibre do not wrinkled easily. The fabrics will remain crisp and easy to wash. Therefore, most of the application of polyester is to make clothes and dresses. The most popular polyester fibre is Terylene and it can be drawn into very fine fibres that can be woven like any other yarn. Another form of polyester that always be used is PolyEthylene Terephthalate (*PET*), where it usually used for making bottles, utensils, films, wires, etc.

In four season countries, polyester usually applied in winter season where sweaters, shawls and blankets are mostly used. Many of these fabrics are not made from natural wool, although it has similarities with the original wool. Most of the fabrics are manufactured from acrylic, another type of synthetic fibre. Apparently, the original wool obtained from the natural sources is quite expensive while the fabrics and clothes that are made from acrylic are fairly cheap. The synthetic fibres are more durable and affordable since it is reasonably cheap which makes them more popular than the natural fibres.

Figure 2.3 : *PET* bottle (Japan Trekking Circle, 2007)

2.1.2 Natural Fibre

Generally, natural fibres can be defined as bio-based fibres or carbon fibres that are normally made from vegetables and animals. This well-defined natural fibres are consists of all natural cellulosic fibres and protein based fibres. Fibres that are taken from trees such as cotton, jute, sisal, flax, hemp, abaca and ramie fall under cellulosic fibres category. Fibres other than these falls under protein based fibre category such as wool and silk (Food and Agriculture Organization of the United Nations, 2008).

Westman (2010) stated in his report that the natural fibres are primarily consist of cellulose, hemicelluloses, pectin and lignin. The individual percentage of these components varies with the different types of fibres. This variation can also be effected by growing and harvesting conditions. Cellulose is a semicrystalline polysaccharide and is responsible for the hydrophilic nature of natural fibres. Hemicellulose is a fully amorphous polysaccharide with a lower molecular weight compared to cellulose. The amorphous nature of hemicelluloses results in it being partially soluble in water and alkaline solutions. Pectin, whose function is to hold the fibre together, is a polysaccharide like cellulose and hemicellulose. Lignin is an amorphous polymer but unlike hemicellulose, lignin is comprised mainly of aromatics and has little effect on water absorption.

Figure 2.4 : Asbestos cement-roof (Chiesa, 2010)

There are also mineral fibres that falls under natural fibre category such as asbestos. As we all know, asbestos occur naturally in the soil on earth is not a bio based material. Products that contain asbestos are not sustainable since it will bring harm to human health and therefore, these products were prohibited to use in any countries.

On the other hand, fibres such as viscose-rayon and cellulose acetate are manmade cellulose fibres where they were produced by using chemical procedures from pulped wood or other sources such as cotton and bamboo. Furthermore, regenerated protein like soybean, polymer fibre such as bio-polyester and chitosan fibres are examples of semi-synthetic products that made from renewable resources.

Table 2.1 : Comparison between natural and synthetic fibres (Food and Agriculture Organization of the United Nations, 2008)

Type of waste	Approximate time taken to degenerate	Nature of material
Peels of vegetable and fruits, leftover foodstuff, etc.	1 to 2 weeks	Biodegradable
Paper	10 to 30 days	Biodegradable
Cotton cloth	2 to 5 months	Biodegradable
Wood	10 to 15 years	Biodegradable
Woollen clothes	About a year	Biodegradable
Tin, aluminium and other metal cans	100 to 500 years	Non-biodegradable
Plastic bags	Several years	Non-biodegradable

A material which gets decomposed through natural processes such as action by bacteria is called biodegradable while a material which is not easily decomposed by natural processes is called non-biodegradable. As can be seen from Table 2.1, natural fibres such as waste and leftover foodstuff and paper take about 1 to 4 weeks to degenerate since they are biodegradable materials. Synthetic fibres such as plastic bags will take longer time to degenerate since it is a non-biodegradable whereas cans that made from tin, aluminium and other metals will take even greater time to degenerate.

Since plastic bags are non-biodegradable materials, it will take several years to decompose and hence it is not environment friendly. It will also cause environmental pollution. Besides, the burning process in the synthetic materials is quite slow and it does not get burnt easily. During the process, the material releases lots of poisonous fumes into the atmosphere causing air pollution and harm human health.

2.2 Natural Fibre as a replacement to Synthetic Fibre

In consideration of material issues of high strength fibres, such as lack of material resources and reducing the dependency of glass fibre, lots of researches, scientists and engineers have been made to develop environmental friendly materials that are made from natural fibres. In addition, new environmental regulations and uncertainties about petroleum and timber resources have triggered much interest in developing composite materials from natural fibres (Leman et al., 2008).

Natural fibres are usually low in density, have low costs since it is a renewable sources, nonabrasive nature to machine, and high filling levels possible. Furthermore, natural fibres have low energy consumption, high specific property, and biodegradable and also have acceptable mechanical properties (Bactiar et al., 2010b). Natural fibre also offers environmental advantage such as reduced dependence on non-renewable energy or material sources, lower pollutant emissions and lower the greenhouse gas emission. It also has advantages of enhanced energy recovery and life biodegradable components.

In addition, Misha et al. (2001), believed that natural fibres have wide variety of fibres available throughout the world, where in some part of the world it has become natural or agricultural-based economy income. There has been growing interest in the use of natural fibres as reinforcement materials. Natural fibre can be applied to many industries such as building construction, furniture, automotive and packaging due to the advantages they offer that mentioned earlier. As stated earlier, automotive is one of the major industries that used the natural fibres in manufacturing their product (Mueller, 2005).

Researcher Weinheim that was cited in Ishak et al. (2009) mention in his paper that the latest model of Mercedes S was using 27 components that made from natural fibre composites. Furthermore, in household application, furniture were engineered to composite panels that made from chopped natural fibres that produces wood replacement products in order to sustain in a home green situation.

Natural fibres such as hemp, flax, sisal, jute, abaca, kenaf, ramie, henequen, pineapple leaf, oil palm, sugarcane bagasse, sugar palm, rice husk, coir, bamboo, wood, chicken feather, cotton and silk have been reported as being used as fibres in polymer composites (Herrera-Franco & Valadez-Gonzalez, 2004) (Wool, 2005).

2.3 Natural Fibre drawbacks

Although the natural fibre has been proved to be practical to replace the synthetic fibre, there are still some disadvantages that make a pulls back to the synthetic fibres replacement. One of the most significant drawbacks in the natural fibre reinforced composites is that the natural fibre has poor compatibility of the fibre and the matrix due to the hydrophilic characteristic of the cellulose and the hydrophobic nature of matrix material. This leads to poor interfacial adhesion between the fibre and the matrix as well as poor fibre dispersion (Ishak et al., 2009).

Since the purpose of using matrix is to transfer the load to the stiff fibres through shear stresses at the interface and to achieve this, sufficient bonding between the matrix and the fibres are needed critically. But, due to the insufficient bonding between the interfaces, the advantages of using natural fibres as reinforcement composites cannot be fully utilised and it implies poor properties for the composites. Other than that, limitations of using natural fibres such as sugar-palm is that it has low fire-resistant, higher water content and higher water absorption.

Furthermore, Bachtiar et al. (2010b) state that the natural fibres can vary a lot in its properties due to the influence of their growing conditions, the fibres processing technique, the fineness of the fibre and the sample test-length, which can make accurate predictions of the respective composite properties difficult. Moreover, the natural fibres have a complex structure of elementary fibres which consists of cellulose, hemicelluloses, pectin, lignin, etc. and thus they are not considered in the same, straightforward way as monofilament fibres (Saheb & Jog, 1999).

2.4 Arenga Pinnata, sugar-palm tree

Sugar-palm, with scientific name of Arenga Pinnata or Arenga Saccharifera is a member of Palma family. This tree is usually found in tropical countries such as India, Indonesia, Guam, Malaysia, Myanmar, Papua New Guinea and Philipines (Bactiar et al., 2010a) (Bactiar et al., 2010b) (Ticoalu, Aravinthan, & Cardona, 2011). In Malaysia, sugar-palm tree can be found in state of Negeri Sembilan and Sarawak. Sugar-palm is a tree that is known for its sugar-palm by-product, other than vinegar, distilled alcoholic drink and its sweet sap.

Figure 2.5 : Sugar palm tree (Bachtiar, Sapuan, & Hamdan , 2008)

The sugar-palm fibre that will be used in this research is the hairy black fibres that covered the trunk of the sugar palm tree. The fibre is knows with several names such as sugar-palm fibre, ijok, gomuti and gomutu. The fibres can be varies in its colour from black or dark brown to light brown. It appears to be stiff, layered and not oriented in any particular direction.

In certain countries such as Indonesia, for traditional application, sugar-palm fibre is used for handcraft like kupiah (Acehnese typical headgear used in prayer) and roofing for traditional houses (Bactiar et al., 2010b). For other application, the fibres are usually used for brooms, paint brushes, septic tank base filters, clear water filters, door mats, carpets, ropes, chair and sofa cushions, and for fish nests to hatch eggs.

Ticoalu et al. (2011) mention in their paper that the diameter of the single sugar-palm fibre varies from 50 μm to 800 μm while Bachtiar et al. (2010a) reported that the range of the fibre in his research is in range of 99 μm to 311 μm.

2.5 Surface treatments

Cao et al. (2005) described that the untreated fibre, the fibres were packed together but it got split after treatments. This process is called fibrillation, where the treatment breaks the untreated fibre bundle down into smaller ones by dissolution of the hemicellulose. This process will increase the effective surface area available for contact with the matrix, and hence the interfacial was improved.

Fibres can be treated with several chemical solutions that suit the purpose of to change the surface tension which can help to improve their interfacial properties in the polymer matrix and to reduce the moisture uptake. The improvement on the interfacial properties of the fibre can improve the mechanical and physical properties of the composites (Li et al., 2000; Li et al., 2007; Ishak et al., 2009; Bactiar et al., 2010 and; Sawpan et al., 2011).

Generally, there are two methods to modify the surface of the natural fibres which are physical and chemical methods (Herrera-Franco & Valadez-Gonzalez, 2005). Physical treatments can be applied by using cold plasma treatment, corona treatment, etc. while chemical treatment that are always used are maleic anhydride, organosilanes, isocyanates, sodium hydroxide, hydrochloric acid, permanganate and peroxide.

It has been conveyed by Bledzki and Gassan (1999) in their previous study that the usage of alkali treatment on a natural fibre is the cause of removal of lignin and hemicellulose. When the hemicellulose were removed, the interfibrillar region is likely to be less dense and less rigid. Therefore, it will make the fibrils more capable of rearranging themselves along the direction of stress loading. When fibres are stretched, such arrangements amongst the fibrils would result in better load sharing by them and hence in higher stress development in the fibre.

2.6 Manufacturing of sugar-palm fibre reinforced epoxy composite

The variations in the properties of the natural fibre reinforced composites (*NFRC*) as those of synthetic composite materials are affected by the type of fibre and resin that are used, the composition of fibre and *NRFC*; and the manufacturing methods. Furthermore, the *NFRC* is also affected by the moisture content of the fibres, treatments, cultivation and retting process of natural fibres (Ticoalu et al., 2011).

The manufacturing process of sugar-palm fibre reinforced epoxy composite starts with collecting the sugar-palm fibre from its tree. The trees can be easily found in tropical countries as mention in the previous section. After that, retting process was done to clean the fibres. Generally, this process was done in order to separate the stalk from the core fibre and this process was done in most of the previous research.

The retting process starts with soaking the fibres in a water tank until the dirt vanished from the core section and the stalk was separated from the core. It is important to stirs the fibres occasionally to facilitate the separation process. The water in the tank will be changed several times in order to reduce the dirt from the separation process. After that, the fibres were left to dry at room temperature for a period of time, two weeks (Bactiar et al., 2010) (Sastra et al., 2009). Ishak et al. (2011) used air dried method for drying the fibres for 24 hours before dried for another 24 hours in an oven at $85^{o}C$.

Chemical treatments were done to the fibres in order to improve its interfacial properties. Bachtiar et al. (2010b) used alkali as a chemical treatment method and Sodium Hydroxide, $NaOH$ is the treatment solution. In this paper, the concentration that used was $0.25M$ and $0.5M$ of $NaOH$ with soaking time of 1 hour, 4 hours and 8 hours. Meanwhile, Ishak et al. (2009) used sea water as a treatment solution in his research and the fibres were left soaked for duration of 30 days.

After the treatment, the fibres were rinsed with distilled water until the rinsed solution reached $pH7$ (neutral). The treated fibres then were left to dry at room temperature for a few days. Bachtiar et al. (2010b) dried his fibres in duration of 4 days while Sastra et al. (2005) dried his fibres for three days.

The process continues with weaving the fibre into woven mat form, chopped the fibres for the chopped random form and arrange the fibres into unidirectional form. The composites were produced using hand lay-up method for practically (Sastra et al., 2009) (Ticoalu et al., 2011).

As for the next process, the mould for the composites was made of composite boards and other equipment such as glass and transparent plastic for the bottom layer. The mould was made by 3 layers of double-sided tape to form a square shape. The fibres then cut based on the mould size and placed over the transparent plastic in the bottom of the mould (Bactiar et al., 2010b) (Sastra et al., 2009). Sapuan et al. (2006) built the mould for the fibre composites by using plywood and coated with plastic while Bledzki et al. (2001) used steel frame as a mould for his research.

The matrix that used in this research is in epoxy group. The epoxy and hardener were mixed together based on the weight percentage to form a matrix. After that, the matrix was poured over the fibre and compressed, and distributed evenly until it achieved the specified thickness (Jawaid, Abdul Khalil, & Abu Bakar, 2006) (Bactiar et al., 2010b) (Ishak et al., 2009) (Sastra et al., 2009). Ticoalu et al. (2011) used polyester as main element of their matrix while Bledzki et al. (2001) used polyurethane as their main element in the matrix. The composites then left for 20 to 24 hours for curing process in room temperature of 25 to 30^{o}C until it dried evenly. After that, the composite board was cut into test samples and the cutting process used handsaw and other equipment such as jigsaw and ready for testing.

2.7 Porosity of fibre composite materials

So far, most study focused on the determination of fibres characteristics involves mechanical properties namely, tensile and flexural stress, impact strength, etc where it bring significant changes to the properties and ignored the existence of a minor component, porosity. Porosity can be defined as air-filled cavities inside a material. This component is unavoidable since it always occurs in all composite materials due to the mixing and consolidation of two different materials, the fibres and the matrix (Madsen & Lilholt, 2003).

In the case of natural fibre composites, the porosity makes a noteworthy contribution to the overall composite volume with porosity volume fractions above 0.05. In another work of Madsen et al. (2009), the writers had summarised that the porosity that happen in the composites was caused by a few factors which are (i) the existence of luminal cavities in natural fibres, (ii) the complex surface chemistry of natural fibres which complicates the fibre/matrix bonding and (iii) the irregular form and dimensions of the natural fibre. Other than that, the porosity also happen due to (iv) the low packing ability of natural fibres which limits the maximum obtainable fibre volume fraction and lastly (v) the applied processing techniques.

2.8 Review of existing studies

Ticoalu et al. (2011) studied gomuti fibre/polyester composites. The study was to investigate the tensile and flexural properties of the fibres. The test was done to four different forms of fibre orientation which are random original layout, chopped random, unidirectional and woven mat. During the experiment, the fibre was not chemically treated in order to determine the properties of untreated gomuti fibre/polyester composites.

Bachtiar et al. (2010a) studied the tensile properties of single sugar palm fibre. Retting process was done to the fibres in order to separate the stalks from its core. Single fibre tensile test was conducted based on *ASTM* D3379 for single fibre tensile test. During the test, a single fibre was attached to a cardboard frame.

Bachtiar et al. (2008) study the effect of alkali treatment on tensile properties of sugar palm fibre reinforced epoxy composites. The treatment was done using Sodium Hydroxide, *NaOH* solutions at two different concentrations of 0.25*M* and 0.5*M* and three different soaking times which are 1 hour, 4 hours and 8 hours. The sugar palm/epoxy composites were reinforced with 10% weight fraction of fibres and produced using hand layup technique. The composites then were tested for the determination of its tensile properties.

Bactiar et al. (2010b) in their other work studies the flexural properties of alkaline treated sugar palm fibre reinforced epoxy composites. The sugar palm/epoxy composites were reinforced with 10% weight fraction of fibres. The fibres were treated using sodium hydroxide, *NaOH* solution with concentration of 0.25*M* and 0.5*M* for 1 hour, 4 hours and 8 hours soaking time. The writer stated that the purpose of treating the fibre with alkali was to enhance the interfacial bonding between the matrix and the fibre surfaces.

Ishak et al. (2009) studied the effect of biological base treatment agent, sea water to the flexural strength of sugar palm fibre composites. The sugar palm fibres were soaked in sea water for 30 days. In order to maintain the salinity of the sea water, the geographical location of sea water can affect the purity of the sea water. Therefore, the sample was taken 200 *m* away from the shore. The sugar palm epoxy composite that tested were made from 20% and 30% fibre content. The result of the test was compared with untreated sugar palm epoxy composite.

Sastra et al. (2005) studied the flexural properties of Arenga Pinnata fibres as a natural fibre and epoxy resin as a matrix. In this research, the fibres were mixed with epoxy resin at various fibre weight percentages of 10, 15 and 20% with 3 different fibres orientations such as long random, chopped random and woven roving. This research used hand layup technique to produce the specimen test with curing time for the composite plates is in room temperature of 25 to 30^{o}C.

Meatherall (2008) study the establishment of protocols for natural fibre density measurement. In this study, the researcher investigated the current method for the testing of natural fibre density by using oilseed flax fibre. A few methods have been tested such as diameter and linear density method; Helium Pycnometry method and Archimedes method.

Most of the porosity element in previous studies involved with model development that integrates the volumetric composition of the fibre composites with their mechanical properties. Madsen et al. (2009) study a model, based on a modified rule of mixtures to determine the influence of porosity of the composite stiffness. The model that developed integrates the volumetric composition of the composites with their mechanical properties. The fibre weight fraction was used as an independent parameter to calculate the complete volumetric composition.

Another study made by Madsen and Lilholt (2003) investigates the physical and mechanical properties of unidirectional plant fibre composites. This study focused on the evaluation of the influence of porosity to the fibre composites. A theoretical model was fitted to the data in order to describe the composite volumetric interaction between contents of fibre, matrix and porosity by using the weight fraction and porosity fraction of the fibre composites.

2.9 Mechanical properties of Arenga Pinnata fibre reinforced epoxy composite

Table 2.2 below shows comparison between tensile and flexural properties of several natural fibre reinforced composites with glass fibre and neat resin summarised by Ticoalu et al. (2011). As shown in the table, the properties of the natural fibre composites are almost half of the properties of the glass fibre.

Table 2.2 : Comparison values of the tensile and flexural properties of several natural fibre/polyester reinforced composites summarised by Ticoalu et al. (2011)

Fibre	Tensile stress *MPa*	Tensile modulus *MPa*	Flexural stress *MPa*	Flexural modulus *MPa*	Reference
Glass	76 – 160	5600 – 12000	140 – 260	6900 – 14000	Blaga (1978)
Neat resin	68.95	3930	118	3333	O’Dell (n.d)
Jute	45.82	3700	61.65	3050	O’Dell (nd)
Sisal	47.10	12900	80.43	9370	Bisanda (1991) in Mwaikambo (2006)
Coir	20.40	-	41.54	-	Singh & Gupta, in Mohanty et al. (2005)
Hemp	32.90	1421	54.00	5020	Rouison et al. (2006)
Flax	61.00	6300	91.00	4800	Rodriguez et al. (2005)

Table 2.3 below shows the summary of the properties of sugar-palm/epoxy and sugar-palm/polyester composites with different fibre orientations from several literatures.

Table 2.3 : Summary of sugar-palm / epoxy and sugar-palm / polyester composites properties from several literatures

Sugar-palm fibre	Tensile Stress *MPa*	Flexural Stress *MPa*	Flexural Modulus *MPa*	Reference
Random original / Polyester	15.4	43.67	2912	Ticoalu, Aravinthan and Cardona (2011)
Woven mat / Polyester	9.24	48.43	3098	Ticoalu, Aravinthan and Cardona (2011)
Chopped / Polyester	14.52	42.82	3538	Ticoalu, Aravinthan and Cardona (2011)
Unidirectional / Polyester	24.49	47.82	3396	Ticoalu, Aravinthan and Cardona (2011)
Chopped 15% wt untreated / Epoxy	13.78	-	-	Leman et al. (2008)
Chopped 15% wt 30d freshwater / Epoxy	21.27	-	-	Leman et al. (2008)
Chopped 15% wt 30d seawater / Epoxy	23.04	-	-	Leman et al. (2008)
Chopped random 20% wt / Epoxy	30.49	64.71	3145.8	Sastra et al. (2005), Sastra et al. (2006)
Long random 15% wt / Epoxy	49.61	92.65	3997.3	Sastra et al. (2005), Sastra et al. (2006)
Woven roving 10% wt / Epoxy	51.73	108.15	4421.8	Sastra et al. (2005), Sastra et al. (2006)

2.9.1 Tensile properties

Gowda, Naidu and Chhaya (1999) outlined that tensile properties of a composite material are mainly depending on fibre strength, modulus, fillers, fibre length and orientation, fibre/matrix interfacial bonding and fibre content. The tensile strength of the composite is influenced by the strength and modulus of fibres.

From the tensile test conducted by Bachtiar et al. (2010a) for investigating the tensile properties of a single sugar palm fibre, it can be concluded that the sugar palm had a good tensile properties. This statement was supported with the result of the test, where the average tensile properties such as Young's Modulus are 3.69 *GPa* with tensile stress of 190.29 *MPa* and strain at failure of 19.6%. This study proved that Arenga Pinnata or sugar palm fibres are suitable as reinforcing agents in composite materials.

From Table 2.3, it has been found by Ticoalu et al. (2011) in his research that the unidirectional orientation form has the highest tensile stress with 24.5 *MPa*. This value is slightly higher than the tensile stress of coir fibre reinforced polyester composites in Table 2.2 which is 20.40 *MPa*. Furthermore, this value also slightly higher than the sea water and fresh water treated chopped random sugar-palm of 15*%* wt fibre reinforced epoxy composite, with values of 23.04 *MPa* and 21.37 *MPa* respectively. Woven mat has the lowest average tensile stress which is 9.2 *MPa* if compared to other type and orientation of fibres from Table 2.2 and Table 2.3.

Result from tensile test of sugar-palm fibre reinforced epoxy composite in Bachtiar et al. (2008) shows that there's significant increase in tensile stress in fibre that was treated with 0.25*M NaOH* concentration with soaking time of 1 hour. The tensile stress improved about 16.4*%* where it increased to 49.88 *MPa* compared to the untreated composite. As for the tensile modulus, there's a significant increase with increasing the alkali solution concentration and soaking time.

However, in the research done by Sastra et al. (2005), it is appeared that the woven roving sugar-palm 10*% wt*/epoxy composite has the highest value of tensile stress among all orientation of sugar-palm fibre composites, with 51.73 *MPa*. Long random 15*% wt*/epoxy was second with 49.61 *MPa* and followed by chopped random 20*% wt*/epoxy with 30.49 *MPa*.

2.9.2 Flexural properties

In a research done by Ticoalu et al. (2011), the woven mat has the highest average flexural stress of 48.4 *MPa* with lowest standard deviation of 3.65. Follows behind the woven mat is the unidirectional form with average flexural stress of 47.82 *MPa* with highest standard deviation of 15.22. This shows that the result flexural test of unidirectional form is more varied as compared with others. Random original and chopped fibre composites have values of flexural stress about the same which are 43.67 *MPa* and 42.82 *MPa* respectively. However, the chopped fibre composite has the highest flexural modulus with 3538 *MPa* followed by unidirectional fibre, woven mat fibre and random original fibre with 3396 *MPa*, 3098 *MPa* and 2912 *MPa* respectively.

The outcome of the research done by Bactiar et al. (2010b) was that the concentrations of the solution cause significant change to the flexural stress of the fibre composites. The maximum flexural stress occurs at 0.25*M NaOH* solution with 1 hour soaking time which is 96.71 *MPa*. This value had improved about 24% from the untreated fibre composites. As for its flexural modulus, the maximum value occurs at concentration of 0.5*M NaOH* solution with 4 hours soaking time which is 6948 *MPa*. This value had improved greatly about 148% compared with untreated fibre composite.

Ishak et al. (2009) reported that the flexural stress of 30% fibre content is highest among the rest of fibre content that were tested with 53.87 *MPa* where this value had shown improvement about 7.35% if compared with the untreated fibre composite. However, reverse trend was obtained with 20% fibre content where the value had decreased about 8.12% compared to the untreated fibre composites. The writer concluded that the biological base treatment by using sea water had significantly improved the surface characteristic of the fibres.

Result from the flexural test of Arenga Pinnata fibre reinforced epoxy composite in Sastra et al. (2005) shows that the 10% fibre weight percentage of woven roving Arenga Pinnata showed the highest value for flexural properties. The result shows that the flexural stress and flexural modulus are 108.157 *MPa* and 4421.782 *MPa* respectively. The writer conclude that the woven roving Arenga Pinnata fibre has a better bonding between its fibre and matrix compared with other fibre orientations, long random and chopped random.

2.10 Physical properties of sugar-palm fibre reinforced epoxy composite

In this section, the physical properties that will be looked upon are the porosity and density tests. These tests will determine the compatibility between the fibres and the matrix mix.

2.10.1 Density

The density test will take place to determine the density of both the matrix and the fibres of the composite. The density of the fibre composite will be determined by using the Archimedes principle of buoyancy method where the dry/wet weights of composite were measured.

From the studies done by Meatherall (2008), it was found that the Archimedes method using canola oil as an immersion fluid was simple and effective method for general use in measuring flax fibre density. Moreover, the diameter and linear density method appeared to have poor performance in accuracy, repeatability and speed, though it has low cost, where it is a good thing and excellent in ease of use and safety category.

However, the Helium pycnometry method might provide more reliable results and the researcher would recommended this test if a large amount of samples need to be tested. On the other hand, this method would make the user to spend a big amount of money since this test required a higher cost compared to other density method.

2.10.2 Porosity

The porosity test will take place to determine the volume of voids inside of the fibre. The porosity of the specimen in this research will be determined by using loss of weight in water method.

From the studies done by Madsen et al. (2009), an analysis of the volumetric composition and the mechanical properties had been performed for the fibre reinforced composites. The outcome of this study was that a model, based on a modified rule of mixtures has been developed by relating the stiffness to parameters that described the fibre composites. The model was validated with experimental data for volumetric composition and stiffness for several fibre composites. The writer concludes that the agreement between the model and the experimental data is good.

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