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Mechanical Properties of Benzoylation Treated Sugar Palm Fiber and Its Composite

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Abstract: Studies on the effect of treated sugar palm fiber with alkaline treatment and benzoylation treatment on single fiber tensile strength and interfacial shear strength (IFSS) are shown in this paper. Also shown is the tensile strength of treated sugar palm fiber composite with variable fiber loading (10%, 20% and 30%). For alkaline treatment sodium hydroxide (NaOH) was used, while benzoyl chloride (C_7H_5ClO) was used in benzoylation treatment. Polymer matrix that was used in the process of making sugar palm fiber composite is epoxy resin with hardener. For the single fiber test and IFSS, the alkaline treatment was carried out using 1% concentration of sodium hydroxide for one hour soaking time while 5ml of benzoyl chloride was used to agitate with sodium hydroxide for benzoylation treatment with variable soaking time (10, 20 and 30 minutes). Treated sugar palm fiber showed higher single fiber strength and IFSS compared to untreated fiber due to the efficiency of both treatments which help rearrangement of fibrils along the tensile force direction. Tensile properties of sugar palm fiber composite show improvement in tensile stress and tensile modulus for treated sugar palm fiber composite while tensile strain show the opposite result. This is because the bonding strength between fiber and matrix increased by removing the outer layer together with impurities from the fiber during chemical treatment. Thus, with this treatment method, sugar palm fiber can be use as reinforcement material for composite and use them for commercial use such as for furniture and component inside vehicle.

Keywords: Sugar palm fiber, sodium hydroxide, benzoyl chloride, tensile, IFSS.

I. INTRODUCTION

Mechanical properties of a composite are greatly influenced by the relationship between fiber and its matrix. The relationship of fiber and its matrix makes a substantial contribution in characterizing composites properties. Poor mechanical properties of a composite caused by poor adhesion between surface fiber and matrix which resulting in relatively weak dispersion of force to occur [1]. Natural fiber's main properties are hydrophilic, this would hinder the effectiveness when reacting with its matrix. Hydrophilic properties caused by the hydroxyl groups present in fiber which causing several problems when mixing with matrix to make bio-composites. Other than that, impurities and waxy layer covering the fiber along with the reactive functional groups in the fiber impede it from bonding with its matrix. The resin used in manufacturing of composites has become one of the important elements to be considered in view of the issue of interface limitation between natural fiber and polymer matrix [2]. This leads to increasing interest in surface modification of natural fibers by researchers nowadays. Fiber surface entails modification with different surface treatments for instance physical treatment, chemical treatment, reactive additives and coupling agent to increase interfacial adhesion between fiber and matrix. Reactive groups on fiber surface can be exposed with chemical treatments. As a result, it promotes efficient bonding with matrix and improved mechanical and thermal properties for the composites [3-5]. Benzoylation treatment is one of the chemical treatment methods that was used to reduce the hydrophilicity of fiber at the same time enhance the interlocking between fiber and matrix which lead to increasing in composite strength and its thermal stability. Pre-treatment is used to activate the hydroxyl group during benzoylation treatment and the fiber soaked in benzoyl chloride solution. The fibers where then soaked in ethanol solution for an hour in order to remove benzoyl chloride solution that was attached to the fiber surface and later washed with tap/ distilled water followed by oven dried [6]. Benzoylation treatment on top of alkaline pre-treated sisal fiber was implied by Joseph et al. [6] and the fiber composite was reported to have higher thermal stability compared to the untreated fiber composite. This method later was used in this paper with some modifications to study its effects on sugar palm fiber and its composite.

Natural fibers contains several chemical composition. Generally, fibers contain cellulose, hemicellulose, lignin and pectin, which contribute to the overall properties of the fiber [7]. Naturally different fiber has different composition of the said components, however most of fibers contain 60–80% cellulose, 5–20% lignin, and moisture as much as 20% [8], thus making cellulose the main component.

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There are abundance of cellulosic fibers available in nature from variety of places and plant species. Bamboo, coir, cotton, flax, hemp, kenaf, pineapple leaf fiber, sisal and sugar palm are among the most prominent natural fibers used commercially and in research due to their fiber strength [9]. Thermosets are the polymers that widely used in composites. It cured by thermal or chemical which act as a catalyst. It is an irreversible process once it is cured.

Epoxy resins is/ are among the most common and popular thermosets polymer due to its good overall properties as a composite. Instead of using catalyst to cure like polyester resins, epoxies are cured with hardener which also called a curing agent. Epoxy resins and its hardener are mixed according to a fixed ratio. Which, in order to ensure a complete reaction between resin and hardener, the mix ration must be correct or else the resin will not cure properly to achieve its full properties.

The interphase of fiber is a main consideration to ease the stress transferred along the composite [9]. Therefore, the tensile test is performed to assess/analyze the forces transferred between fibers and its matrix. The join connection of fiber and its matrix is the main factor in finding the interfacial strength under stress. Thus, to analyze and identify the interfacial bonding behaviour and the IFSS, a micromechanical technique is used [9]. Sugar palm tree (*Arenga pinnata*), easily found throughout Southeast Asia regions in abundant quantity. Treatment of sugar palm fiber with different concentrations of sodium hydroxide (NaOH) and various soaking time were recorded by Bachtiar et al. [10] and the study reported that fiber treated with one hour soaking time in 0.25M NaOH solution to be optimum for obtaining high tensile strength and high flexural modulus in fiber. For this study, strength of untreated and chemically treated single sugar palm fiber is focused, and experiments of interfacial strength of sugar palm fiber mounted with epoxy resin were conducted. The tensile strength of untreated and treated sugar palm fiber reinforced epoxy composite with different fiber loading were also investigated.

II. MATERIALS AND METHODS

A. Materials

Sugar palm fibers used were acquired from Kampung Kuala Jempol, Negeri Sembilan, Malaysia. Epoxy (density of 1.2128 g/cm^3) and hardener (density of 0.988 g/cm^3) were used to make fiber composite with ratio of 2:1. Other materials that were used were sodium hydroxide, benzoyl chloride and ethanol.

B. Alkaline treatment

Fibers were soaked in 0.25M (1%) of NaOH alkaline solutions for 1hour. Then, fibers were washed multiple times with distilled water until pH level become neutral and oven dried overnight at temperature 60ºC.

C. Benzoylation treatment

The pre-treated fibers were divided into 3 batches, with each batch soaked in the mixture solution of 1% NaOH and 5ml of C_7H_5ClO with respective soaking time (10, 20 and 30 minutes). After soaked, fibers were washed with distilled water and soaked in absolute ethanol for 1 hour to remove residue of benzoyl chloride that attached to the fibers surface. Lastly, the fibers were again washed multiple times with distilled water until the pH level become neutral and oven dried in overnight at 50^{0}C.

D. Fabrication of composite

Composites of untreated and chemically treated sugar palm fiber with different fiber loading (20 wt%, 30 wt% and 40 wt%) were constructed by using hand-layup process. Long sugar palm fibers with the length of approximately 150 mm were used and placed in unidirectional position in a closed steel mould with measurements of 150 mm length x 150 mm width x 3mm depth. A mixture of resin and hardener with 2:1 ratio was stirred thoroughly and left for 5 minutes for the bubbles in the mixture to disappear before it can be poured into the mould. Before pouring, the metal mould was sprayed with mould releasing agent in order to avoid the composite from sticking to the mould after hardened. Two 150 N load (Fig. 1) bars were used to compressed the mould and left for 24 hours to cure.

Fig. 1. Two 150 N load used in the experiment.

III. CHARACTERISATIONS

A. Determination of Single Fiber Strength of Sugar Palm Fiber

ASTM D3379 standard for single fibers was used for this test. A piece of paper was folded and cut into 50 mm length x 45 mm width (Fig. 2) to make a frame for attaching a single sugar palm fiber for testing. The fiber then glued to the paper frame and let dry for 24 hours. Optical microscope (Olympus SZX12) was used to measure the diameter of each fiber used for testing before performing tensile test. By using universal testing machine with 5 kN load capacity, the specimens were tested with the crosshead speed of 1mm/min. Average was taken for five repetitions of each specimen.

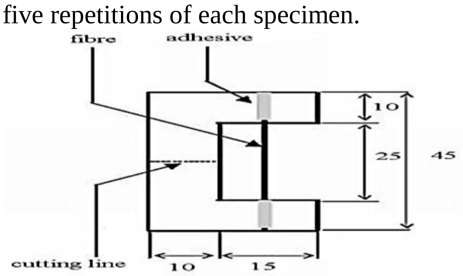

Fig. 2. Schematic diagram of paper frame attached with single sugar palm fiber [16].

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B. Determination of Sugar Palm Fiber Interfacial Shear Strength

To carry out a microdroplet test, a drop of epoxy resin with hardener to a ratio of 2:1 was dripped onto each sample of treated and untreated sugar palm fiber. Function of this experiment was to determine the interfacial bonding or IFSS characteristics. Each fiber was then glued to a piece of paper with dimensions of 50 mm length x 45 mm width, exactly like the one used in tensile test. Specific gauge (Fig. 3) was used to mount the paper with fiber attached for undergoing microdroplet test. Measurement for single sugar palm fiber diameters and the epoxy droplet length were taken using the same optical microscope used to measure fiber diameter. Universal testing machine (Instron 3366) was used in this test with 5 kN load with a 1mm/min crosshead speed by applying shearing force to pull the single fiber from resin droplet around the fiber. The result from the test can determine the interfacial shear strength. Average was taken for five repetitions of each specimen.

Fig. 3. Gauge used for microdroplet test.

C. Determination of Tensile Strength of Sugar Palm Fiber Composite

Instron 3365 test machine was used for tensile test based on ASTM D3039. Composite samples were cut into 150mm length x 15mm width x 3mm depth. The gauge length used for the test was 60mm, and with a 5mm/min of crosshead speed. Average was taken for five repetitions of each specimen.

IV. RESULT AND DISCUSSION

A. Single Fiber Strength of Sugar Palm Fiber

The effect of two chemical treatments of sugar palm fiber on tensile stress, tensile modulus and tensile strain are presented in Table 1. The tensile strength of the fiber varied from one another due to the effect of chemical treatments. From the experiment it is noticed that chemical treatments improved the tensile strength of sugar palm fiber. The greatest enhancement in tensile strength was observed for fiber undergone benzoylation with 30 minutes soaking time with 221.585 MPa which is 47.5% more than untreated fiber. The second highest was alkaline treated fiber with 202.940 MPa followed by benzoylation with 20 minutes of soaking time 163.224 MPa. While for benzoylation with 10 minutes soaking time show slight decrement in tensile strength with 12.4% lesser compare to untreated fiber of 150.190 MPa. The enhancement of tensile strength may be attributed from the elimination of lignin and hemicellulose, easing the fibrils to rearrange along the tensile direction [11]. Thus, causing higher tensile strength.

Table. 1 Single fiber strength of sugar palm fiber.

Sugar Palm Fiber	*Tensile Stress (MPa)*	*Tensile Modulus (GPa)*	*Tensile Strain*
Untreated	150.190	4.460	10.244
NaOH	202.940	9.059	6.338
C_7H_5ClO (10* min)	131.636	8.581	4.580
C_7H_5ClO (20* min)	163.224	5.262	9.730
C_7H_5ClO (30* min)	221.585	7.569	7.738

*Indicate the soaking time of fiber during benzoylation treatment

The tensile strain also known as elongation at break of single sugar palm fiber varied due to chemical treatments. It is observed that the tensile strain decreases for the chemically treated fiber. As expected, the tensile strain value will be lower as there is improvement of the tensile stress properties. During chemical treatment, wax layer and impurities were removed and cause the fiber become thinner and stiffer [9]. Removal of lignin and hemicellulose resulted from chemical treatment also contribute to the decrease value of the tensile strain. Tensile modulus of treated sugar palm fiber shows significant improvement compared to untreated fiber. Alkaline treated fiber show the highest value with 9.058 GPa which is 103% more than the untreated fiber, followed by benzoylation with 10 minutes soaking time, 30 minutes and lastly 20 minutes. The improvement attributed by the removal of the lignin and hemicellulose may increase the cellulose crystallinity index. With removal of lignin and hemicellulose, it causes the spiral angle to decrease which later increase in the degree of molecular smooth surface microfibrillar structures. This make the fiber become more ductile and stiffer than before due to increase of crystallinity index of cellulose [12, 13].

Generally, the tensile strength of treated sugar palm fiber showed moderate value. This can be seen by comparing the value with other fibers shown in Table 2. Same thing can be said with tensile modulus of treated sugar palm fiber which share the same value as some of the other fibers. The significant improvement shown in tensile modulus for treated sugar palm fiber from untreated fiber indicate that the fiber become more ductile after chemical treatments due to the removal of lignin and hemicellulose. Even though the tensile strain of treated sugar palm fiber decreases after chemical treatment, they still show quite high value compared to other fibers. The decrease in tensile strain value after chemical treatments due to the removal of impurities and waxy layers on fiber surface help the fiber become relatively stiffer.

Table. 2 Tensile strength of other fibers [9].

Natural Fibers	*Tensile stress (MPa)*	*Tensile Modulus (GPa)*	*Tensile Strain*
Aramid	3000-3150	63-67	3.3-3.7
Bamboo	140-230	11-17	-
Bagasse	290	17	-
Coir	138.7	4-6	30
Flax	345-1035	27.6	2.7-3.2

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Natural Fibers	*Tensile stress (MPa)*	*Tensile Modulus (GPa)*	*Tensile Strain*
Hemp	690	70	1.6-4
Jute	393-773	26.5	1.5-1.8
Kenaf	215.4	53	1.6
Pineapple	400-627	1.44	14.5
Sisal	511-535	9.4-22	2.0-2.5
S-Glass	4570	86	2.8
E-Glass	2000-3500	70	0.5

B. IFSS of Sugar Palm Fiber with Epoxy Resin

Fig. 4 shows the results of interfacial strength of treated and untreated sugar palm fiber. The interfacial strength gained high improvement after the benzoylation treatment with various soaking time while for the 1% alkaline treatment it shows slight decrement. Recent studies showed that most untreated natural fiber will improve its mechanical properties after undergo chemical treatments. The improved strength of the single fiber from benzoylation treatment was due to the removal of waxy substance and impurities from the fiber surface. By removing the waxy layer, it creates a rough surface which improved adhesion between fiber and matrix. Benzoylation treatment cleansed the surface of the fiber from impurities and increased the interface quality by disrupting the moisture absorption process. After benzoylation treatment, the treated fiber shows significant improvement in IFSS value due to fibrillation phenomenon. This phenomenon happens when the untreated fiber packed together in a bundle and broken into smaller alignments through chemical treatment by dissolution of hemicellulose. Fibrillation increase the surface area availability for contact with matrix substance for penetration and bonding with fiber cell thus enhance the interfacial adhesion [14]. For alkaline treatment, in this experiment it shows slight decrement compared to untreated fibers to almost no difference. However, Mohd Nurrazi et al. [9] reported that alkaline treated sugar palm fiber shows higher IFSS value than untreated fiber. The decrease of IFSS value for alkaline treated fiber might be cause the nature of natural fiber itself. Sugar palm fiber that were used for this experiment were collected from Kampung Kuala Jempul which the sugar palm trees grow in the wild. This would cause the fiber to grow with inconsistent size and chemical composition due to the weather and soil which would affect the strength of each fiber.

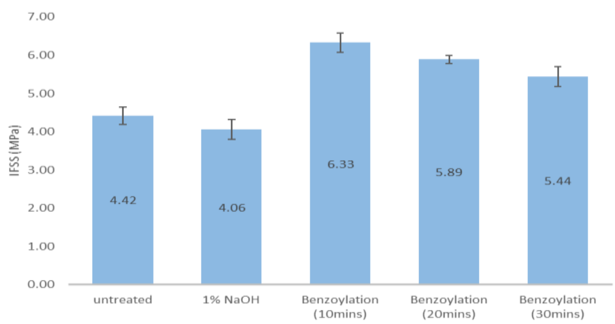

Fig. 4. IFSS of untreated and treated sugar palm fiber.

C. Tensile Strength of Sugar Palm Fiber Composite

From IFSS result shown in Figure 4, benzoylation treated sugar palm fiber with 10 minutes soaking time was chosen for fabrication of fiber composite due to its highest value among benzoylation treated fibers. The tensile properties of benzoylation treated fiber composite with different fiber loading are compared with untreated and alkaline treated fiber composites. The results are shown in Table 3.

Table. 3 Tensile strength of sugar palm fiber composite.

Treatment	*Tensile Stress (MPa)*	*Tensile Modulus (GPa)*	*Tensile Strain*
20 wt% Untreated	41.5022	4.023	.01493
30 wt% Untreated	38.3270	3.847	.02514
40 wt% Untreated	32.9825	4.385	.02574
20 wt% NaOH	38.3001	4.175	.01137
30 wt% NaOH	54.8017	5.320	.01452
40 wt% NaOH	58.6860	5.473	.02005
20 wt% C_7H_5ClO	44.1985	4.996	.01150
30 wt% C_7H_5ClO	53.9907	5.035	.01536
40 wt% C_7H_5ClO	38.7749	4.328	.02158

The tensile properties shown in Table 3 indicate that there is significant improvement among treated and untreated sugar palm fiber composites for tensile strength and tensile modulus value. The chemically treated fiber composites have better tensile strength and tensile modulus because of better interfacial bonding which helps stress transfer from matrix to fiber smoothly [15]. However, for tensile strain it shows the opposite trend. The low value on tensile strain of treated fiber composite due to the removal of impurities and wax layer during treatment process which cause the fiber to become stiffer. While there are improvements in the tensile strength and tensile modulus value of treated composites, the benzoylation treated fiber composite show decrement at 40 wt%. This might cause by the voids present in the composite which would affect the strength of the composite. The voids in composite occurred resulted from during fabrication process when epoxy resin was not poured properly onto the mould with fiber in it, it might accidently form voids during curing process. The present of voids inside resin would disturb the stress transfer along the composite which leading to failure at lower load [16].

V. CONCLUSION

In this study, both alkaline and benzoylation treated sugar palm fibers show improvement in tensile strength. Chemical treatment alter the structure of sugar palm fiber which help improving the properties. There shows no significant difference in the tensile strength of alkaline and benzoylation treated fiber which suggested that benzoylation treatment can be used for improving the fiber properties. This can be proved further with the high IFSS value showed by benzoylation treated fiber which show that benzoylation treated sugar palm fiber has a better potential to be used as reinforcement to polymer composites.

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The tensile strength of benzoylation treated sugar palm composite show almost similar strength as alkaline treated composite which is very encouraging. This proved that benzoylation treatment can be use on sugar palm fiber for surface modification method. Further studies and testing need to be done for benzoylation process on sugar palm fiber because it is known that benzoylation process also improved flexural strength and the thermal properties of natural fiber.

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