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Tensile Behaviour of Chemical Treatment for Bamboo Epoxy Composites

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Previous studies proved that mechanical properties of natural fibres reinforced polymer composites are excellent and competent to be utilised in high-tech applications. In contrast, the presence of chemical constituents such as cellulose, lignin, hemicellulose and wax substances in natural fibres preventing them from firmly bind with the polymeric resin and thus, resulted in poor mechanical properties of composites. To overcome this defect, modification of fibre surface was done by chemical treatment. In this study, the effect of 1 %, 2 % and 3 % concentration of sodium hydroxide solution on tensile behaviour of the bamboo reinforced epoxy composite was studied. The result showed that treatment with highest studied concentration significantly influenced the tensile properties of the composite. Thus, indicates that the treatment has done excellent work in modifying the surface of bamboo fibre for better adhesion with epoxy matrix.

1. Introduction

Bamboo has been said as a woody plant that possessing natural strength which makes it desirable to be used especially in-house construction in the last hundred years ago. Moreover, bamboo is starting to be applied in composite technology and with proper methods and has been seen as a potential fibre to replace synthetic fibre like carbon and glass (Sankar et al., 2013).

The main drawback of using natural fibres in polymeric composites is the incompatibility between natural fibre and polymer matrix. This is due to the presence of chemical components (i.e. cellulose, lignin, hemicellulose and wax substances) cause the natural fibres to exhibit hydrophilic properties, in which the polar molecules like water easily attracted to them and increase the moisture content. High moisture content in natural fibres makes them difficult to bind properly with the polymer matrix and thereby degrading the mechanical properties of fibre reinforced polymer composites. Thus, chemical treatment on natural fibre is promoted in order to increase the interfacial bonding compatibility between fibre and polymer matrix.

Recently, literatures on chemical treatment for natural fibres start to emerge widely. The common treatment that has been used is alkaline treatment. Alkaline treatment is a treatment where fibres are immersed in certain concentration of alkaline solution like sodium hydroxide solution (NaOH) for a determined period of time. It has been reported that this treatment will give effect mainly on the surface of fibre (Li et al., 2007). Following that, the treated natural fibres will undergo few mechanical testing such as tensile and bending in order to study the effect of chemical treatment on their mechanical properties. Phong et al. (2011) reported that 1 % of NaOH solution is sufficient to treat bamboo fibre as their experimental results showed the treated bamboo fibre reinforced epoxy composites exhibited higher mechanical properties compared to untreated.

On the other hand, numerous attempts of fibre treatment work conducted by Jacob et al. (2004) and Mishra et al. (2003), they used 4 % and 5 % of NaOH solution to treat sisal fibre reinforced composites. Mishra et al. (2003) also stated that 5 % of NaOH treated sisal fibre-reinforced polyester composites better than 10 % as the higher alkali concentration delighted natural fibre excessively and weaken the fibre.

Based on previous literatures, the main variables of chemical treatment that commonly mentioned are the concentration of a chemical solution, the duration for soaking of natural fibre in the chemical solution and the

drying hours for natural fibres to dry. In determining the suitable values for these parameters, the researchers have to repeat their experimental study in order to identify the optimized chemical treatment composition. Hence, a proper method, or tool has to be implemented to save time and to determine the best chemical treatment composition for these natural fibres.

Box-Behnken design (BBD) is one of the classes of response surface design. It is used for optimizing product performance and often has been used to set design of experiments (DOE). BBD helps in constructing maps of correlation between the determined parameters. In BBD, the level for low (-1), middle (0) and high (+1) values for each variable have to be determined first. In the end, it resulted to a total number of 17 experiments and each experimental trial derived, will consist of a combination of different level of those parameters.

2. Methods and materials

2.1 Alkaline treatment of bamboo

To determine the optimum chemical treatment condition, an experimental design composed of three variables was built by using Box-Behnken design (BBD). Initially, design points for low, middle and high levels of each condition must be determined. The design points for chosen variables is tabulated in Table 1.

Table 1: The Initial Setting of Box-Behnken Design (BBL	D)
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Factors / Independent Variables	Symbols	Coded and Actual Levels		
		Low (-1)	Middle (0)	High (+1)
Concentration of NaOH (%)	X1	1	2	3
Soaking duration (h)	X2	3	6	9
Drying duration (h)	X3	2	48	72

The input data in the Table 1 then were randomized and modelled by BBD using the Design-expert (6.0.8) software. It gave a total number of 17 experimental runs (consists of 12 runs with 5 runs replications of centre points) as listed in Table 3. Following that, alkaline treatment of bamboo was carried out according to the conditions of variables stated in Table 3. The treatment work took place at room temperature. The percentage of chemical solution was prepared by weight percentage or weight per volume percent (w/v). In order to obtain 1 % of NaOH concentration, 1 gram of NaOH pellets was diluted in 100 ml of distilled water. The percentage of solution can be calculated according to the Eq(1).

Weight percentage (%) =
$$\frac{\text{Mass of solute (g)}}{\text{Volume of solution (ml)}} X 100 \%$$
 (1)

2.2 Specimen fabrication and tensile testing

The bamboo material tested was cut approximately 5 m above the bamboo root, which belonged to Bambusa vulgaris, (or also known as buluh minyak in Malay) and aged about 3–4 years. This type of bamboo was chosen as it is commercially utilised in Malaysia. Initial, the raw bamboo poles were cut into smaller parts. Those parts were sent to Omega's Timber factory to be extruded into much finer size via a high-quality cutting machine. The final form of raw bamboo materials were cylinder-shaped bamboo sticks which were about 3 mm in diameter and approximately 200 mm in length. Next, those bundles of cylinder bamboo sticks were inspected and treated with 1 %, 2 % and 3 % of NaOH solution for 3, 6 and 9 h. Then, they were dried for 24, 48 and 72 h at room temperature.

After drying, they were aligned on a waxed mould and was poured with the epoxy resin that mixed with the corresponding hardener in a ratio 1:3. The epoxy resin, then was spread over the aligned bamboo sticks by using a plastic scraper to ensure that the resin filled every corner. Once the bamboo sticks were totally soaked with epoxy resin, the composite was covered with a vacuum bag which already connected to a vacuum pump. Here, a vacuum was applied, acting as a mechanical pressure that pressed on the composite throughout the cure period. This technique is known as vacuum bagging where atmospheric pressure or vacuum is applied as to hold the resin-coated composite until the resin cures. It was chosen due to several advantages, such as reducing trapped air in composite, humidity and excess resin.

For tensile test, the specimens were cut by laser cutter into a dog-bone shape specimen according to the specification of ASTM D638 Type I (Standard, 2003) as shown in Figure 2. The tensile test is carried out on Shimazu Universal Testing Machine for at least three specimens for each treatment condition. The crosshead displacement rate of 1 mm/min was used and the output of the tensile test results was then recorded in the form of load versus displacement traces. The test is stopped when the specimen breaks off.

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Figure 2: Detailed dimensions of the dog-bone specimen for tensile test (mm)

3. Results and findings

3.1 Mechanical properties of bamboo/epoxy composite

Figure 3 presents typical stress-strain curves of untreated bamboo/epoxy composites under three different angles of bamboo orientations (i.e. 0°, 45° and 90°). These curves were plotted based on force and displacement results obtained from the tensile test. From the curves, it is clear that the 0° bamboo orientation offered higher tensile strength compared to 45° and 90°. Coutney (2000) explained that the failure of aligned fibre reinforced composites caused by the fibre fracture as the loading geometry leads to an equal strain condition. It means the fibres which are aligned parallel to the applied tensile force give an excellent resistance against breakage, while the fibre orientation that off-axis tensile load resistless. Thus, it can be summarised that the fibre reinforced composites offer higher tensile strength when the fibres are oriented parallel to the axis of acting force due to anisotropic behaviour.



Figure 3: Typical stress-strain curves of bamboo/epoxy composites with 0°, 45° and 90° of bamboo orientations

On the contrary, the failure of fibre orientations that are not parallel to force axis is often related to matrix tensile failure. Coutney also reported (Coutney, 2000) that when the fibre orientation is $\theta = 90^{\circ}$, the condition of a composite is said to be under equal stress. At this point, the fibres do carry and transfer the load, but not as efficient as in the equal strain condition. Therefore, the primary fracture mode for $\theta < 90^{\circ}$ is the matrix or transverse failure. Despite all the differences, the stress-strain curves exhibited by these orientations display no noticeable failure trend prior to breakage due to brittle properties (Beer et al., 2002). Thereby, the ultimate tensile strength of fibre reinforced composite is similar to their breaking strength.

Table 2 summarizes the mechanical properties of bamboo/epoxy composites with corresponding bamboo orientations. The 0° bamboo orientation clearly outperformed 45° and 90° by offering a tensile strength of 138.88 MPa, that is approximately 90 % higher than both of them. However, the 45° bamboo orientation exhibited a tensile strength of 8.42 MPa which is about 18 % greater than 90° bamboo orientation and thus, make 90° bamboo orientation has the lowest tensile strength among all. A similar pattern is observed for the

strain at break (ϵ) and Young's modulus (E) for each bamboo orientation where the 0° bamboo orientation shows the highest values while 90° bamboo orientation exhibits the lowest values. For the next tensile test of treated composites, the bamboo fibres are arranged in 0° as this orientation degree displays excellent mechanical properties.

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Bamboo	Tensile strength	Strain at break	Young's modulus
orientation (°)	(MPa)	(%)	(GPa)
0	138.88	2.70	4.96
45	8.42	0.62	1.37
90	5.89	0.54	1.09

Table 2: Mechanical properties of unidirectional bamboo composite

No.	Conditions	Tensile strength (MPa)	Strain at break (%)	Young's modulus (GPa)
0	Untreated bamboo fibre	138.88	2.70	4.96
1	1 wt.% NaOH + 3h soak + 48h dry	242.25	6.17	4.39
2	1 wt.% NaOH + 6h soak + 24h dry	251.55	5.68	4.50
3	1 wt.% NaOH + 6h soak + 72h dry	226.85	7.36	3.43
4	1 wt.% NaOH + 9h soak + 48h dry	263.74	5.90	3.78
5	2 wt.% NaOH + 3h soak + 24h dry	258.13	7.48	3.36
6	2 wt.% NaOH + 3h soak + 72h dry	253.09	7.25	3.66
7	2 wt.% NaOH + 6h soak + 48h dry	301.52	8.45	3.00
8	2 wt.% NaOH + 9h soak + 24h dry	270.82	6.67	3.74
9	2 wt.% NaOH + 9h soak + 72h dry	296.19	7.59	3.25
10	3 wt.% NaOH + 3h soak + 48h dry	284.91	7.14	3.20
11	3 wt.% NaOH + 6h soak + 24h dry	276.98	7.68	3.05
12	3 wt.% NaOH + 6h soak + 72h dry	312.29	8.49	3.14
13	3 wt.% NaOH + 9h soak + 48h dry	339.27	8.33	3.70

Table 3: Alkaline treatment conditions and test results for bamboo/epoxy composites

The results from the tensile test conducted on alkali-treated bamboo/epoxy composites are tarbulated Table 3. The bamboo orientation in all treated composites is aligned at 0° as this orientation has offered excellent properties. Based on the results, all treated bamboo/epoxy composites surpassed untreated composites as for the tensile strength (σ), strain at break (ϵ) and Young's modulus, (E). The cellulose chains in bamboo fibre as reported by Ray et al. (2001) were broken when reacted with higher alkaline concentration. As pointed out by Wong et al. (2010), a higher NaOH concentration could have cleaned the fibre efficiently. Thereby, the exposed cellulose or bamboo fibre easily bonded with epoxy matrix. These results have confirmed that the alkali treatment able to improve the compatibility between bamboo fibre and the polymer matrix, thereby improve the strength of the composite. The highest average tensile strength of treated composites was demonstrated by the treatment conditions No.13, that is 339.27 MPa.

Figure 4 shows the typical stress-strain curves of bamboo/epoxy composites that treated with 1 wt.% of NaOH concentration. It can be seen that the composite in which the bamboo fibres were treated for 9 h and dried for 48 h (1 wt.%-9hr-48hr) exhibited the highest tensile strength. It can be suggested that a high NaOH concentration increases the surface roughness of fibre. It provides a wider contact area between the fibre and polymer matrix and eventually improves the interlocking between them.

Nevertheless, the treatment conditions No.3 displayed the lowest tensile strength, that is roughly 20 % less than the treatment conditions No.13. In brief, the tensile strength of treated composites increased from 1 wt.% to 3 wt.% of NaOH concentrations. Figure presents the response surface and corresponding contour plot s for the interaction effect of NaOH concentration (X1) and soaking time (X2) on the tensile strength of bamboo reinforced epoxy composites at 48 h drying time (X3). As can be seen in Figure 5, the highest tensile strength of 335.84 MPa was recorded at the highest NaOH concentration (3 wt.%) and soaking time (9 h). In contrast, the tensile strength of composites was the lowest at 245.67 MPa when treated with 1 wt.% concentration of

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NaOH for 3 h. Figuratively, the tensile strength performance increase with NaOH concentration and soaking time.



Figure 4: Typical stress-strain curves bamboo/epoxy composites under 1 %wt of NaOH concentrations.



Figure 5: Response surface plots of X1X2 in 3D model

4. Conclusion

The experimental results showed that the alkali treatment enhanced the bonding between bamboo fibre and epoxy matrix. It was proven by the high tensile strength value of alkali-treated bamboo/epoxy composite compared to untreated. According to ANOVA analysis, all the variables and their interactions were significant. This indicated that the tensile strength of bamboo/epoxy composite greatly influenced by NaOH concentration, soaking and drying time. Based on analysis conducted, the suitable treatment condition for treating bamboo fibre reinforced with epoxy matrix is 3 wt.% of NaOH concentration, 9 h of soaking and 55 h of drying time (3 wt.%-9hr-55hr). The tensile strength under this treatment condition was recorded to be 335.84 MPa. The increment of drying time after 55 h were found have reduced the tensile strength of composite due to the reduction of the alkalizing effect.

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