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2019

Implementation and Evaluation of Green Materials in Technology Development:

Emerging Research and Opportunities

M.T. Mastura
Universiti Teknikal Malaysia Melaka, Malaysia

S. M. Sapuan
Universiti Putra Malaysia, Malaysia

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Chapter 6
Optimizing the Mercerisation Effect on the Mode I Fracture Toughness of Bambusa Vulgaris Bamboo Using Surface Response Method

Mohamad Zaki Hassan
https://orcid.org/0000-0002-2321-0604
Universiti Teknologi Malaysia, Malaysia

Siti Amni Roslan
Universiti Teknologi Malaysia, Malaysia

Zainudin A. Rasid
Universiti Teknologi Malaysia, Malaysia

Mohd Zuhri Mohamed Yusoff
Universiti Putra Malaysia, Malaysia

S. M. Sapuan
Universiti Putra Malaysia, Malaysia

Firdaus Muhammad-Sukki
https://orcid.org/0000-0002-5415-2259
Robert Gordon University, UK

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ABSTRACT

Alkaline treatment is widely being promoted to treat natural fibres and improves the fibre bundle surface for better interlocking with the polymer matrix. The aim of this study is to optimize the merceration parameter including natrium hydroxide (NaOH) concentration, soaking and drying time for Bambusa Vulgaris bamboo using response surface methodology (RSM). Here, the treatment conditions were employed by the Box-Behnken design (BBD). The comparative study of the treated and untreated fibre on crack propagation behaviour, Mode I interlaminar fracture toughness (GIC) of the bamboo along the longitudinal direction test was carried out. Through the statistical analysis approach (ANOVA), it is suggested that bamboo treated with 1.5 wt.% concentration of NaOH is capable to reach the fracture toughness value up to 367.25 J/m2. It is also shown that all proposed variables for treatment in this study (i.e., the concentration of the NaOH is highly significant with the 2.85 hours of soaking and drying for 72.5 hours).

INTRODUCTION

Normally, fiber reinforced polymer materials are laminate composites consisting of high strength of synthetic fibers (i.e. glass, carbon and kevlar) reinforcement in polymeric matrices. The reinforcement by those fibers provides the polymer substantially enhanced mechanical properties and makes the fiber reinforced polymer composites suitable for a large number of diverse applications. Although this synthetic fiber reinforced polymer possess exclusive mechanical strength, they have also got some serious drawbacks such as high cost, poor recycling and non-biodegradable properties.

For these reasons, natural plant fibers reinforced polymer composites are increasingly gaining attention as viable alternative to replace the synthetic fiber reinforced polymer counterpart. These renewable fiber closely high specific strength to weight ratio and offering less abrasion as compared to glass fibers. Many researcher has studied the mechanical properties of these natural fibers such as bamboo, kenaf and jute (Chen et al., 2017; Bakhori et al., 2017; Bakhori, Zuikafly, Ahmad, & Hassan, 2017). Interestingly, fiber derived from bamboos have attracted more interests due to their offering higher performance and plenty sources for production (Yu, Jiang, Fei, Wang, & Wang, 2011).

In addition, an extensively study have been conducted on chemical treatment for natural fiber. This treatment is being suggested in order to decrease the incompatibility between natural fibre and polymer matrix due to hydrophilic and hydrophobic interaction. The common alkaline treatment (NaOH) on natural fibre has been considered as one of most popular and cost affective methods. The effect of chemical

treatments of the bamboo fiber is reported by Yang et al. (2017). They immersed the bamboo fibers in a NaOH solution of 5, 10 and 25% concentration for further removing hemicellulose and heated at 75 °C for 2 h in a water bath. It is found that a higher concentration of NaOH degrades the long chain cellulose molecules at the fiber interface and consequently, weakens fiber load transfer. Reddy and Dhoria (2018) examined the effect of NaOH concentrations of 5% (w/t) on kenaf fibre reinforced composites. Mechanical testings including tensile, flexural and impact are obtained to determine tensile strength, bending and impact strength of the composites. It is found that the chemically treated fiber composites offer good mechanical properties compared to untreated fibers. Similarly, Mishra et al. (2003) reported that sisal fibre treated with 5% concentration of NaOH exhibited good tensile properties than those treated with 10% of alkaline concentration. They suggested that the excessive alkali concentration would cause delignification and weaken the sisal fibre. On the other hand, a comprehensive characterization of wettability and interfacial properties of kenaf fibers polyester composites fabricated by resin transfer molding is reported by Ariawan et al. (2017). Here, kenaf fibers are chemically modified by immersing in 6% NaOH concentration for 1 to 5 h to enhance the interaction between fiber and matrices. In order to investigate the effect of soaking time, surface energy and the interlaminar shear strength value of the composites are evaluated. They reported that 3 h treated of kenaf fiber composite enhanced the interface bonding characteristic of the composite laminate. Furthermore, the effect of alkali treatment under various conditions on physical properties of kenaf fiber is investigated by Khan et al. (Khan, Yousif, & Islam, 2017). In this study, kenaf fiber were treated at alkali concentrations 2, 6 and 10 (w/v%), immersion durations at 30, 240 and 480 minute and temperatures at 27, 60 and 100^{o} C. Kenaf fiber weight loss and density value are decreased after alkali treatment compared to untreated kenaf fiber. Furthermore, Rao et al. (2010) reported that 1% concentration of NaOH is the optimum condition to treat bamboo polymeric composite. On the other hand, few studies claimed that a higher NaOH concentration is the best in treating natural fibre (Yan, Chouw, Huang, & Kasal, 2016) and capable of removing excess moisture thoroughly. In spite of that, the NaOH concentration is the most dominant factor that gives effect on the natural fibre and still remain inconsistency.

Various technique have been explored by researchers to accurately determine the optimum condition for alkaline treatment of the natural fiber. Singh, Mukhopadhyay, & Das (2017)used a three-factor and three-level Box-Behnken design method to optimize the mass fraction of fibres, percentage of crosslinking and plasticizing agent. Initialy, they treated the sugarcane baggase fiber with 6% of sodium hydroxide. Liu et al. (2018) suggested optimized solution using the Box-Benhken design for pretreatment of corn straw by the alkaline solution. Under the optimized conditions, the cellulose and hemicellulose of the corn straw increased and the lignin content

was also reduced. Furthermore, a three-level Box–Behnken design, which is subset of the response surface methodology (RSM), has been investigated by Yaghoobi and Fereidoon (2018). The effect of three independent variables incuding kenaf fiber load, fiber length and polypropylene-grafted maleic anhydride (PP-g-MA) compatibilizer content have been investigated on the tensile strength and modulus of the biocomposite. The optimal tensile strength and tensile modulus are to be 32.70 MPa and 2,182.33 MPa, respectively; and achieved at 28.95 wt% of the kenaf fiber, fiber length of 6.22 mm and PP-g-MA content of 5 wt%.

In this study, the Box-Behnken design (BBD) of the response surface method is employed to obtain an experimental design of alkaline treatment conditions for the *bambusa vulgaris* bamboo. Three experimental conditions including the concentration of NaOH, soaking and drying time are considered. Mode I interlaminar fracture toughness (G_{IC}) test is conducted to investigate the effect of alkaline treatment conditions on crack propagation behaviour along the longitudinal direction of the bamboo.

EXPERIMENTAL METHOD

Box-Benhken Design Iteration

Table 1 shows the design points for low, middle and high level of each variable conditions. This input data then was randomized and modelled by BBD using the Design-expert (6.0.8) software. It offers a total number of 17 experimental runs that include of 12 runs and 5 replication runs of the centre point.

Material and Testing

The bamboo material used in this study belongs to *bambusa vulgaris* family. The raw bamboo is cut approximately 5 metres above the ground. The age of bamboo is approximately four years old and taken from Jeli, Kelantan at the north-eastern

Table 1. The initial setting of Box-Behnken design (BBD)

Factors / Independent Variables	Symbols	Coded and Actual Levels		
		Low (-1)	Middle (0)	High (+1)
Concentration of NaOH (%)	X_1	1	2	3
Soaking duration (hours)	X_2	3	6	9
Drying duration (hours)	X_3	2	48	72

state of Malaysia. The Mode I testing specimens are prepared as a double cantilever beam (DCB) according to the ASTM D5528 standard (ASTM D5528, 2001). The smaller parts of dried bamboo culm are trimmed into narrow strips and those strips were edge squared. The dimension of the DCB specimen is, longitudinal direction, $w = 200$ mm; tangential direction, $h = 20$ mm; and radial thickness, $b = 9$ mm with initial crack length, $\alpha_0 = 40$ mm. Initially, two holes of 5 mm in diameter are made at the point crack initiation as shown in Figure 1.

Following this, the specimens are chemically treated with an alkali solution. The concentration of this solution, sodium hydroxide (NaOH), is prepared by weight per volume (*w/v*) percentage. Here, 1 gram of NaOH pellets is diluted in 100 ml of distilled water to obtain 1 wt.% concentration of NaOH. After undergoing alkali treatment and drying process, an initial crack is cleaved along the middle-line of the bamboo DCB specimen parallel to grain by a stiff razor. The length of the initial crack (α_0) controlled at 40 mm from the centre of the loading holes. The purpose of this cut is to stimulate a naturally sharp crack during the test. Then, the crack tip is marked to obtain correct pre-crack length. Later, the bamboo DCB specimen is fitted to the U-shaped hook steel which connected to a 10 kN load cell on a Shidmazu universal testing machine as illustrated in Figure 2.

The test performed at a crosshead displacement rate of 1 mm/min with five specimens tested at each treatment conditions. During loading, the crack propagated in the bamboo DCB specimen is measured. The corresponding applied load versus the opening displacement (F-δ) is recorded by a computer. Once the load (F) suddenly dropped, the loading machine stopped. Here, the crack tip on the inner (α_{1inner}) and outer (α_{1outer}) parts of the specimen are marked and the average value is taken as the

Figure 1. Schematic diagram of DCB specimen

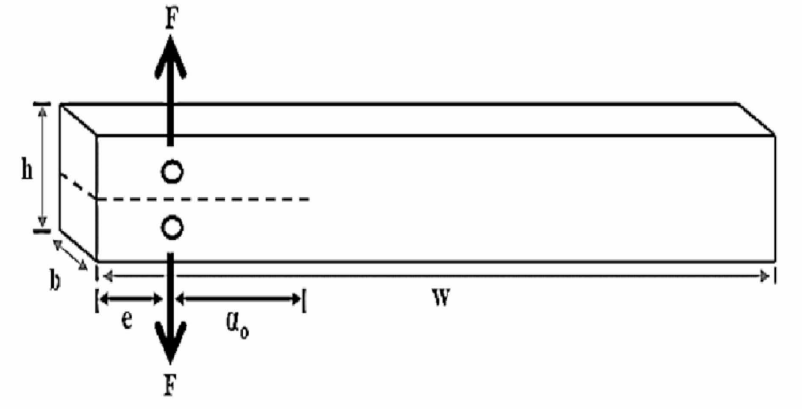
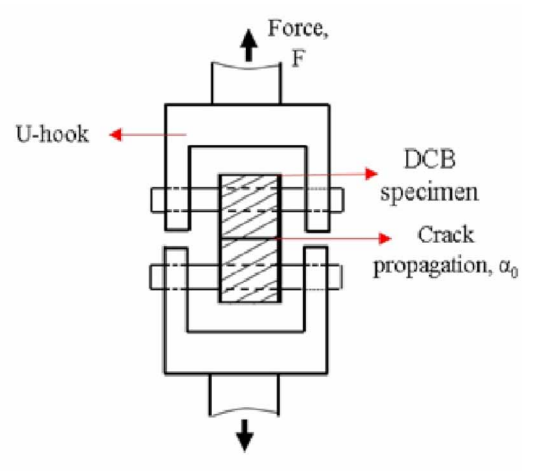


Figure 2. Schematic diagram of DCB specimen connected to U-hook



corresponding crack length (α_1). Afterwards, the specimen is unloaded and reloaded repeatedly until the specimen completely fractured. The equivalent crack length α_2, α_3, α_4… α_n can be obtained and recorded.

G_{IC} Analysis

The Mode I interlaminar fracture toughness (G_{IC}) is measured by symmetric bending tests using the DCB specimen. Hence, the energy release rate G of interlaminar fracture mode can be calculated according to Equation 1.

$$G = \frac{1}{b}.\frac{\mathrm{d}U}{\mathrm{d}a} \tag{1}$$

where U is the total strain energy in the test specimen, b is the specimen width, and a is the delamination length. Then, U can also be written as

$$U = \frac{1}{2}F\delta = \frac{1}{2}F^2C \tag{2}$$

where F is the load, δ is opening displacement of the DCB specimen and C is the DCB specimen compliance (δ/F) namely the reciprocal of the slope (k). It can be calculated based on two points from the F-δ traces obtained. The relationship between them can be explained in the following Equation 3.

$$C = \frac{1}{k} = \frac{\delta_2 - \delta_1}{F_2 - F_1} \tag{3}$$

So, the Mode I interlaminar fracture toughness (G_{IC}) is calculated using Equation 4.

$$G_{IC} = -\frac{1}{b} \cdot \frac{d(-\frac{1}{2} Fcr^2 C)}{da} = \frac{Fcr^2}{2b} \cdot \frac{dc}{da} \tag{4}$$

where Fcr is the critical load. It is only necessary to measure the differential increase in compliance (dC) depending on the differential increase in the crack length (da).

According to the compliance method, the reciprocal of the slope is the corresponding compliance (Ci) of the DCB specimen with a certain crack length (α_i). The relationship between C and α can be described as stated in following Equation 5.

$$C = q\alpha m \tag{5}$$

where q and m are the fitting coefficients of the compliance curve of the DCB specimen. So, after taking a logarithm of Equation 5, the equation meets the linear model as stated in Equation 6.

$$\lg C = \lg q + m \lg \alpha \tag{6}$$

Following the Mode I test, the results are analysed and evaluated statistically using the Analysis of Variance (ANOVA).

ESULTS AND DISCUSSION

GI_c Characterization of Bamboo DCB Specimen

A typical force-displacement trace of an untreated bamboo specimen for a 42 mm of crack length (α_1) is shown in Figure 3. Initially, the trace increase linearly from the loading start to the maximum force. It begins to waver after a while due to the overbearing load capacity, which caused unstable crack propagation along the longitudinal direction of bamboo DCB specimen. Subsequently, the trace drops and the test suddenly stopped. At this point, maximum force is recorded as the critical force, F_{cr}, to indicate the decisive point of rapid cracking in the bamboo specimen.

Figure 4 shows an untreated bamboo DCB specimen subjected to the multi-point method of the Mode I fracture testing. The opening crack lengths for the outer layer

of the bamboo that was recorded approximately 1 to 2 mm longer than those of the inner layer as can be seen in Figure 4. A similar observation also recorded on the treated bamboo DCB specimens. Shao, Fang, and Tian (2009) reported the resistance against crack propagation of bamboo is controlled by interlaminar strength between the bamboo fibres. As the amount of bamboo fibres on the outer layer is less than that of the inner layer, resistance against crack propagation exhibited by the outer layer is weaker. It indicates that the outer layer has lower interlaminar strength than the inner layer as described in (Shao, Fang, & Tian, 2009).

Table 2 summarises the average G_{IC} values for bamboo specimens with the corresponding treatment conditions subjected to Mode I testing. From the table, it can be seen that all of the treated specimens exhibited lower G_{IC} values compared to untreated specimens. The average of G_{IC} value for untreated bamboo specimens is recorded at 365.20 J/m^2. In contrast, the highest average G_{IC} value for the treated bamboo is shown by the treatment condition No.1, which is 362.85 J/m^2. The lowest G_{IC} value is found at the treatment condition No.13, exhibited more than half G_{IC} value of the treatment condition No.1. The lower G_{IC} value of treated bamboo specimens may be attributed to the softening effect caused by alkaline treatment which influences the stress transfer process along the specimen during the test is conducted (Shao, Fang, & Tian, 2009). It should be noted that even though the presence of alkaline lowers the value of the mean, G_{IC}, in the natural fibre composite application, the alkaline will improve the bonding between the fibre and the matrix and improve the mechanical properties of the composite.

Figure 3. The typical force-displacement trace of an untreated bamboo specimen

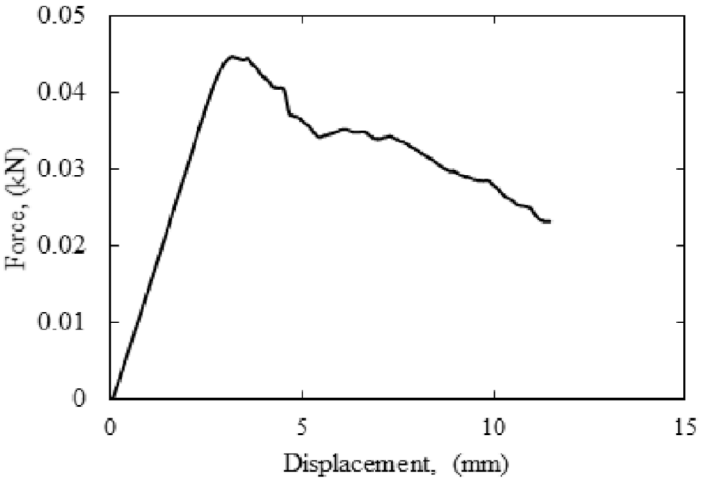
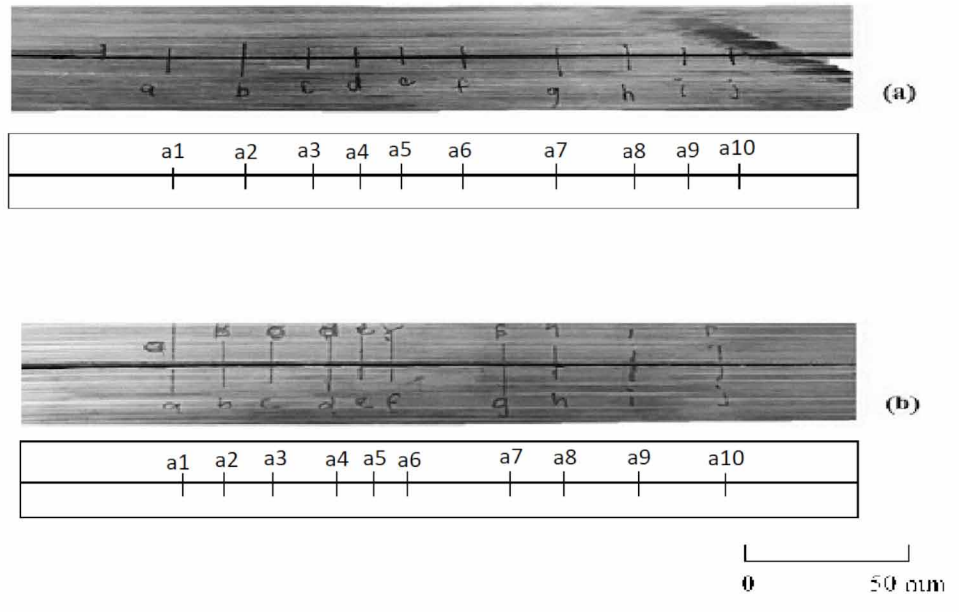


Figure 4. The delamination of crack lengths with geometric views for (a) the outer and (b) the inner parts of an untreated bamboo DCB specimen



Quadratic Model Fitting and Analysis of Variance (ANOVA)

The G_{IC} results of treated bamboo are further investigated using analysis of variance (ANOVA) to determine which variables that significantly affect the G_{IC} value of treated bamboo. By applying a multiple regression analysis of the responses, the outcome proposed the highest order polynomial in which the additional terms are significant and the model is not aliased. Following that, backwards elimination method was applied to exclude insignificant terms automatically. The ANOVA for the reduced quadratic models that summarised in the G_{IC} value, is shown in Equation 7.

$$G_{IC} = 573.235 - 160.801\ X_1 - 14.743\ X_2 - 1.229\ X_3 + 27.696\ X_1^2 - 0.679\ X_2^2 + 0.033\ X_3^2 + 3.936\ X_1X_2 - 1.095\ X_1X_3 \quad (7)$$

Equation 7 represents the relationship between NaOH concentration (X_1), soaking time (X_2) and drying time (X_3) towards G_{IC} value of treated bamboo. This equation was also used to generate predictions of the response for a given level of each variable. Table 3 depicts the predicted G_{IC} values using Equation **7** and the experimental finding value. It is in good agreement between the yield predicted and the experimental responses, with a small difference in G_{IC} value are measured

Table 2. The effect of treatment conditions on G_{IC} values of bamboo

No.	NaOH concentration, X_1 (wt.%)	Soaking time, X_2 (hours)	Drying time, X_3 (hours)	Measurement numbers, k	Sample numbers, n	Mean G_{IC} (J/m^2)
0	0	0	0	30	3	365.20
1	1	3	48	35	3	362.85
2	1	6	24	33	3	314.88
3	1	6	72	34	3	356.94
4	1	9	48	33	3	251.32
5	2	3	24	33	3	270.83
6	2	3	72	33	3	264.13
7	2	6	48	53	5	205.48
8	2	9	24	30	3	186.11
9	2	9	72	30	3	163.97
10	3	3	48	30	3	185.06
11	3	6	24	32	3	200.77
12	3	6	72	30	3	142.71
13	3	9	48	30	3	120.76

as can see in Table 3. The percentages of error are also calculated to determine the precision of calculations. Here, the error percentage of each run is less than 4%, which is considered effective (Montgomery, 2005). The bamboo treated with 1 wt.% concentration of NaOH offers the fracture toughness value up to 365.86 J/m^2, which 0.82% of error from the testing result. The G_{IC} value of treated bamboo specimens is declined with the increasing NaOH concentration and soaking time. Again, a higher NaOH concentration and soaking time could reduce the mechanical properties of the fibre due to the softening effect (Bledzki, Fink, & Specht, 2004). Longer drying time, however, has a positive correlation with the response. The model afterwards was inspected statistically using the F-test and regression coefficient, R^2 for validity purposes.

Model Accuracy Analysis Check

Table 4 lists the statistical data for analysis of variance of G_{IC} value. The coefficient of regression (R^2) value of 0.998 implies that the model competently represents the relationship between the significant terms. In fact, the closer R^2 value to 1, the higher reliability of the empirical model of the obtained data. A similar observation is noticed in the Adjusted R-squared of 0.996 where its value is almost 1, indicates

Table 3. The BBD analysis predicted and experimental G_{IC} values

Run	Independent Variables						G_{IC} (J/m²)			
	Coded values			Actual values			Experimental	Predicted	Residual	Error (%)
	X_1	X_2	X_3	X_1	X_2	X_3				
1	0	+1	-1	2	9	24	186.11	182.40	3.71	2.03
2	0	0	0	2	6	48	205.64	208.42	-2.78	1.33
3	0	-1	-1	2	3	24	270.83	272.58	-1.75	0.64
4	0	0	0	2	6	48	207.19	208.42	-1.23	0.59
5	0	+1	+1	2	9	72	163.97	169.94	-5.97	3.51
6	+1	-1	0	3	3	48	185.06	184.32	0.74	0.40
7	0	0	0	2	6	48	207.63	208.42	-0.79	0.38
8	0	-1	+1	2	3	72	264.13	260.12	4.01	1.54
9	-1	+1	0	1	9	48	251.32	252.06	-0.74	0.29
10	+1	0	+1	3	6	72	142.71	143.60	-0.89	0.62
11	0	0	0	2	6	48	208.73	208.42	0.31	0.15
12	-1	-1	0	1	3	48	362.85	365.86	-3.01	0.82
13	-1	0	+1	1	6	72	356.94	354.09	2.85	0.80
14	-1	0	-1	1	6	24	314.88	313.99	0.89	0.28
15	0	0	0	2	6	48	212.91	208.42	4.49	2.15
16	+1	+1	0	3	9	48	120.76	117.76	3.00	2.55
17	+1	0	-1	3	6	24	205.77	208.62	-2.85	1.37

that the suggested empirical model is significantly reliable. Adjusted *R*-squared of 0.996 is also in good agreement with the Predicted *R*-squared.

The coefficient of variation (C.V.) value measures the degree of precision with which treatments were executed and must be lower than 10% (Montgomery, 2005). At the same time, a lower C.V. value implied a higher reliability of the experiment. Therefore, the C.V. value of 1.81% indicates the experiment conducted in this study is reliable. Meanwhile, Adequate Precision value measures the signal to noise ratio were a ratio greater than 4 are desirable (Montgomery, 2005). The ratio for the studied model of 82.783 indicates an adequate signal, thus suggests the proposed model can be utilized to navigate the design space.

Response Analysis

The adequacy of the model is inspected to verify whether the suggested model provides sufficient approximation to the actual system (Shao, Fang, & Tian, 2009).

Table 4. The statistical data of ANOVA for G_{IC}

Source	Response value
R-Squared (R^2)	0.998
Adjusted R-Squared	0.996
Predicted R-Squared	0.987
Standard Deviation	4.120
Coefficient of Variation (C.V.) (%)	1.810
Adequate Precision	82.783
Mean	227.500

To evaluate the model satisfaction, the internal studentised residuals can be verified with the assumption of ANOVA. Here, the internally studentised residual is used to obtain the standard deviation between the experiment and predicted values. Figure 5 presents a relationship of normal probability distribution and the internally studentised residuals. All the residuals point fall closer to the line and fits with the model of the data as can be observed in Figure 5. It can be suggested that no response transformation is required. In addition, the examine residual is also having no apparent problem with the normality.

Figure 5. The normal probability plot of residuals

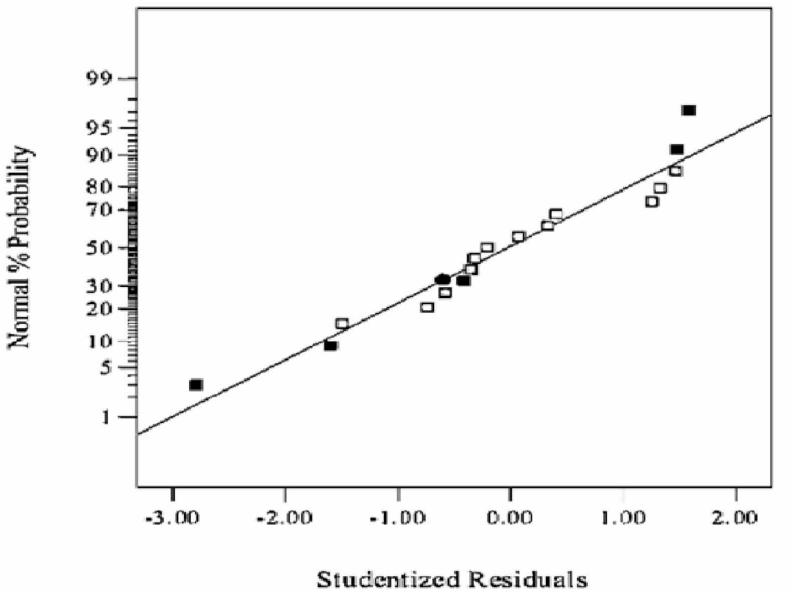


Figure 6 shows the relationship between studentized residuals and predicted response. In this figure, the studentized residuals was scattered randomly and spread in a constant range across the plot around the "0" line. It is indicated that no clear patterns in this plots and validating the initial assumption of constant variance competently. As can be seen from the Figure 6, the residuals and thus confirmed the adequacy of the model. Figure 7 presents a correlation between the experimental and predicted values of G_{IC}. Here, all scatter points are properly distributed close to the line, which suggests a high degree of correlation between the experiment and predicted values. It is therefore suggested that the closer data points to the reference line, the greater accuracy of the model (Montgomery, 2005). Thereby, all the fundamental analyses described are closely fitted, and the selected empirical model is acceptable in predicting the G_{IC} value.

In order to attain a better understanding of the results, the perturbation plot that provides a silhouette view of the response surface is presented in Figure 8. The perturbation plot demonstrates the response of a particular variable as it moves from the chosen reference point while the other variables are fixed to this point. In the Design-Expert software, the reference point is set at the centre of the design space that is the zero-coded level of each variable. So, the zero-coded level of each variable in this study is NaOH concentration $(X_1) = 2.00$, soaking time $(X_2) = 6.00$ and drying time $(X_3) = 48.00$. From Figure 8, the increasing variables X_1 and X_2 clearly

Figure 6. The plot of studentized residuals versus predicted response

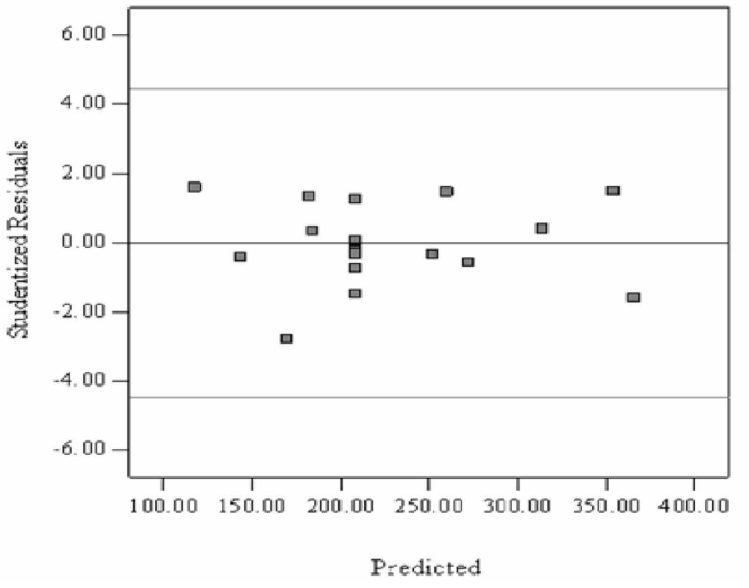
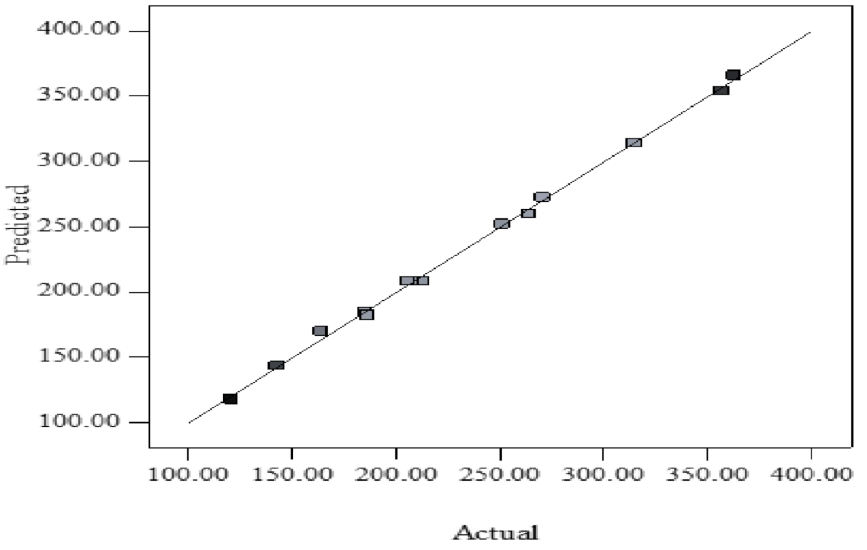


Figure 7. The plot of the predicted versus actual response



offer negative effect on G_{IC} values. Conversely, variable X_3 shows a slight positive effect on G_{IC} values of bamboo where the G_{IC} values increase with increasing X_3.

The contour plots and response surface for the interaction effect of NaOH concentration (X_1) and soaking time (X_2) on G_{IC} values of bamboo at 72 hours of drying time (X_3) are presented in Figure 9. The highest G_{IC} value of 365.86 J/m^2 was recorded at the lowest NaOH (1% wt) concentration and 3 hours of soaking time.

Validation of Optimization Process

Validation experiments are conducted to determine the performance of chemical treatment by evaluating the level of concentration, drying time and soaking duration studies at the optimum favorable conditions through Design Expert Software. The results recorded at (1.5% wt) NaOH concentration, 2.85 hours of soaking time and 72.5 hours of drying time (X_3) offers the highest G_{IC} value of 367.25 J/m^2. Three additional experiments are repeated and the average value for Mode 1 fracture is approximately 85%.

CONCLUSION

Based on the statistical analysis conducted using ANOVA, the proposed variables are found to significantly influence the G_{IC} values of treated bamboo. The analysis

Figure 8. The perturbation plot of the effect of all variables on G_{IC} value

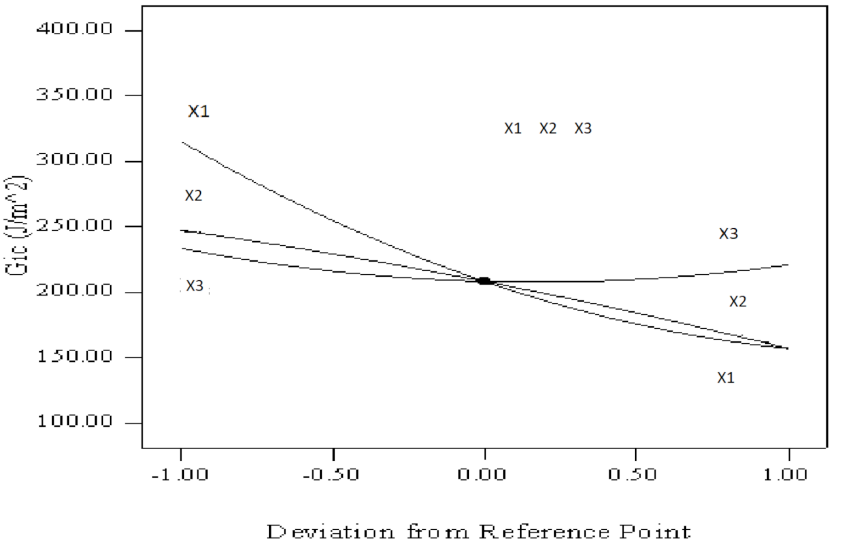
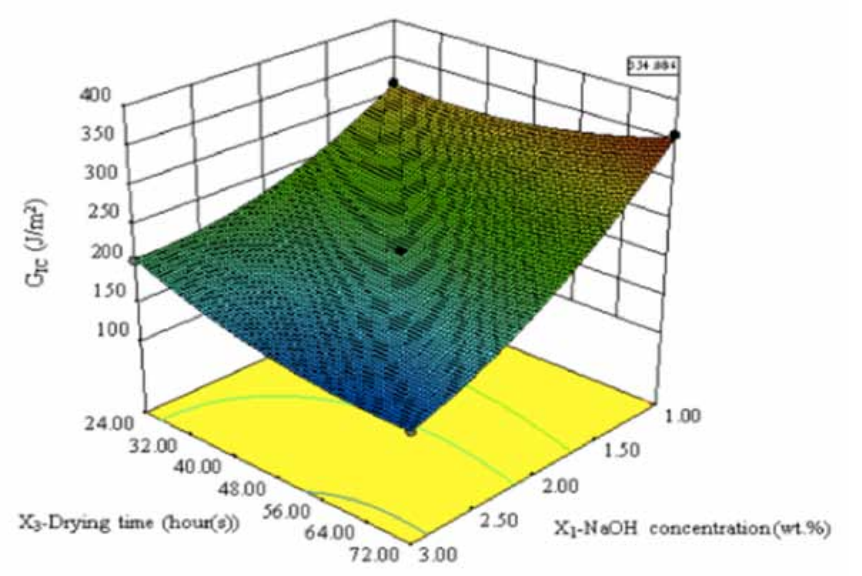


Figure 9. The effect of NaOH and drying time on G_{IC} value



suggested that the bamboo needs to be treated with low NaOH concentration for a short soaking period and dried at a longer drying time, to obtain the optimum G_{IC} value of bamboo at room temperature. The highest value of G_{IC} for treated

bamboo is evaluated at 367.25 J/m^2 when the bamboo treated with 1.5 wt.% of NaOH concentration for 2.85 hours and dried up to 72.5 hours at room temperature (1.5wt.%-2.85hours-72.5hours).

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