Fibers and Polymers 2020, Vol.21, No.10, 2321-2337
DOI 10.1007/s12221-020-1299-1

ISSN 1229-9197 (print version)
ISSN 1875-0052 (electronic version)

Flexural Mechanical Properties of Natural Fibre Reinforced Polymer Composites - A Statistical Investigation

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(Received November 30, 2019; Revised December 28, 2019; Accepted January 5, 2020)

Abstract: The objectives and novelty of this paper are to create a hybrid-natural fibre composite by the Response Surface Methodology RMS technique, and then compared this hybrid composite with the individual fibre reinforced composites in the bending test. The first aim of this study is devoted to analyse, modelize and optimise the various independent variables such as the type of fibres (X_1), the types of chemical treatment (X_2), the volume fraction of fibre (X_3) and the treatment duration (X_4) used on the output parameters which are the mechanical characteristics namely, ultimate flexural stress and flexural modulus in the bending test using a Box-Behnken experimental design. Mathematical models for ultimate flexural strength and flexural modulus were developed using the response surface methodology (RSM). These models would be helpful in selecting independent variables in order to maximize the flexural mechanical properties. Statistical analysis of the results showed that selected variables had a significant effect on the flexural properties, except the treatment time that has a very weak significance effect on the flexural properties. In the second section, the impact behaviors of the natural hybrid composites found by the RMS method were confirmed experimentally. Finally, the experimental results indicate that the flexural properties of the natural hybrid composites increase with an increase in the composition of jute fibres.

Keywords: Flexural properties, Natural fibres, Hybrid composites, RSM, Optimisation

Introduction

Companies and researchers are driven by current environmental requirements to develop new materials that can replace synthetic fibres (e.g. carbon and glass fibres) with enhanced life cycle performance. In the recent days natural fibres such as sisal, flax, abaca, and jute fibres are replacing the glass and carbon fibres owing to their easy availability and cost [1,2].

The natural fibres can be classified as substances produced by plants or vegetable, animals, and minerals. But the plant fibres are the most common natural fibres used as reinforcement in fibre reinforced composites [1]. Plant fibres exists in many varieties, such as kenaf, jute, alfa, bamboo, hemp, banana, and flax extracted from the stem of the plant. Sisal and abaca extracted from leaves. Coir and cotton extracted from the fruit of the plant. Agopyan [3] listed 18 types of vegetable fibres potentially useful for civil construction. The use of natural fibres has many advantages such as, being derived from a renewable resource, they require low energy inputs in their manufacture. The natural fibre composites are not only biodegradable and renewable but also possess several other advantages such as lightweight, low cost, high specific strength, high modulus, reduced tool wear, and safe manufacturing process [4]. Table 1 lists the mechanical properties of some natural fibres used as reinforcement in composites materials [5-13].

The mechanical characteristics of a polymer composite reinforced by natural fibres are mainly result of the quantity and fibres type, besides the interfacial strength between reinforcement and matrix. Research studies have been conducted on the mechanical properties of natural fibre-based composite materials. Salman *et al*. [14] investigated the influence of fibre content on the mechanical properties of woven kenaf fibre-reinforced polyvinyl butyral composites. The composites were prepared with various fibre contents: 0 %, 10 %, 20 %, 30 %, 40 %, 50 %, and 60 % (by weight). It was noticed that the composites with 40 wt.% fibre content attested the highest mechanical properties. Similarly, Lee *et al*. [15] investigated the kenaf/polypropylene composites fabricated with different fiber content, varying from 10 % to 70 % weight fraction with 10 % increment. The results indicated that the tensile strength and modulus of kenaf/PP composites increased with increasing kenaf fiber contents, reaching a maximum value at 40 %, and then decreased. Aother, Ku *et al*. [16] described a remarkable increment of mechanical behaviors in the polymeric composites by increasing the natural fiber content.

In other words, the adhesion between the reinforcing fibres and the matrix plays an important role in the final mechanical properties of the materials, several authors [17-24] have reported that the mechanical efficiency of the fibre reinforced polymer composites depends on fibre-matrix interface and the ability to transfer stress from the matrix to

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fibre. For example, Ray *et al*. [17] and Mishra *et al*. [18] treated jute and sisal fibres with 5 % aqueous NaOH solution for 2 h up to 72 h at room temperature. Another similar work [19] presents the effect of different concentration of NaOH (0.5, 1, 1.5, 2.5 and 5 %) at a temperature of 100 °C for 1 h of time on date fibres. In this work, the average mechanical properties obtained are 840 MPa and 165 GPa for the strength and Young's modulus, respectively. In another recent study presented by Bedjaoui *et al*. [20], the effect of concentration of sodium bicarbonate ($NaHCO_3$) (4-8 vol.%) for a period of 80 hours at room temperature on the tensile properties of flax/polyester composites have been studied. Asumani *et al*. [21] studied the alkali and silane treated kenaf fibre reinforced polypropylene composites. It has been noted that the tensile strength and modulus increased significantly by 25 % and 11 % respectively after treatment with 5 % alkali. In [22], Vilay *et al*. investigated the effect of fibre surface treatment (NaOH) and fibre loading (0-20 vol.%) on the flexural properties of bagasse fibre reinforced unsaturated polyester composites. NaOH treated fibre composites showed better flexural strength and modulus (increase by about 11 % and 20 % respectively) compared to untreated fibre composites.

The main objective of this work are to create hybrid-natural (sisal, jute and flax) fibres composite by the RMS technique. The hybrid bio-composites can be designed by combining two or more dissimilar kinds of fibre in a single matrix. Hybrid composites can be made from artificial fibres, natural fibres and with a combination of both artificial and natural fibres. The advantages of using a hybrid composite containing two or more types of different fibres are that the advantages of one type of fibre could complement what are lacking in the other. The mechanical performance of the hybrid composites varies according to the type of reinforcement, stacking sequence of fibre layers, fibre orientation, fibre-matrix compatibility, fibre weight fraction and manufacturing process [25,26]. For this reason, we have done this work to answer these questions by applying a statistical study (RMS).

In the literature, there are few studies on hybrid composites under flexural loading are presented below. Boopalan *et al*. [27] investigated the mechanical properties of hybrid raw jute/banana fibre reinforced epoxy composites with varying fibre weight ratio of 100/0, 75/25, 50/50, 25/75 and 0/100, respectively. This study shows that the addition of banana fibre in jute/epoxy composites of up to 50 % by weight results in increasing the mechanical and thermal properties and decreasing the moisture absorption property. Addition of the jute in the composites results in the 17 % increase in the tensile strength, 4.3 % increase in flexural strength and 35.5 % increase in impact strength. A similar trend, Dixit and Verma [9] have investigated that the effect of hybridization on the mechanical properties on coir and sisal-reinforced polyester composite, coir and jute-reinforced polyester composite, jute and sisal-reinforced polyester composite was evaluated experimentally. The results showed that the tensile and flexural properties of hybrid composites are improved as compared to unhybrid composites. Alavudeen [28] studied the mechanical properties such as tensile, bending and impact of woven banana fibre, kenaf fibre and banana/kenaf hybrid fibre composites. They found that the tensile, flexural and impact strength of the hybrid composite is greater than that of the separately used fibres. Jawaid and Abdul Khalil [11] evaluated mechanical performance such as bending and impact characteristics of a hybrid composite (oil palm empty fruit bunches and jute) using an epoxy matrix. They tested specimens by sandwich theory (jute/EFB/jute and EFB/jute/EFB). They found that the bending characteristics of the hybrid composite are better than the pure EFB in contrast to the impact that is important in the composite purely in EFB than witch of hybridization.

In this present study, the flexural properties of individual jute, sisal and flax fibre were studied, and a detailed study of the mechanical performance of hybrid fibre reinforced polymer composites have been made with reference to the relative volume fraction of the two fibres. Then, the morphological study of impact fracture surfaces was investigated on the effect of fibre-matrix bonding and breakage of the composites with the help of scanning electron microscopy (SEM).

Experimental

Materials

In this present investigation sisal (Agave sisalana), jute (Corchorus Capsularis of Tiliaceae) and flax (Linum usitatissimum) fibres are used for fabricating the composite specimens. The definitions of fibres are discussed in detail as follows:

Jute Fibre

Jute is a bast fibre whose scientific name is Corchorus Capsularis of Tiliaceae family originated from Tiliaceae family and takes nearly 3 months to grow to a height of 12-15 feet. Jute plant is cut and kept immersed in the water for retting process during rainy season. Jute is a natural biodegradable fibre with advantages such as high tensile strength, excellent thermal conductivity, and coolness etc. Its abundance in availability with cheaper cost has acquired importance of its use in polymer composites [29,30]. Jute fibre extracted from the bark of jute plant has three major categories of chemical compounds namely cellulose (58-63 wt.%), hemicellulose (20-24 wt.%), and lignin (12-15 wt.%) and some other small quantities of components like fats, pectins, aqueous extracts, etc [1].

Sisal Fibre

Natural sisal fibre is a hard fibre extracted from the leaves

of the sisal plant in the form of long fibre bundle. This plant, scientifically named Agave sisalana Perrine, is of Mexican origin and is grown in Brazil, East Africa particularly in Tanzania, Haiti, India, Indonesia and Thailand [31]. A sisal plant produces about 200-250 leaves and each leaf contains 1000±1200 fibre bundles which are composed of 4 % fibre, 0.75 % cuticle, 8 % dry matter and 87.25 % water [32]. So normally a leaf weighing about 600 g will yield about 3 % by weight of fibre with each leaf containing about 1000 fibres. Sisal fibres with excellent mechanical property are mainly used as textiles, strings, mats, yarns, art ware and reinforced material.

Flax Fibre

Flax, Linum usitatissimum, belongs to the best fibres. It is grown in temperate regions and is one of the oldest fibre crops in the world. It's an 80 to 120 cm high plant which possesses strong fibres all along its stem and contains 70 % of cellulose. These cellulose based fibres have low density, good tensile strength, stiffness and non-abrasive qualities. Disadvantages of these materials are the low thermal resistance and, as much of the natural materials, variability of fibre quality according to the local climate, nature of the ground, etc [7]. Morphology, physical and mechanical properties of flax fibres were presented in detail by Baley *et al*. [33].

Fibre Preparation Methods

The composite specimens were produced in rectangular size as per ASTM D790 standard for flexural testing. The plats of various composites are shown in Figure 1. The volume fraction of fibre (*VF*) is calculated by using the following relation [34].

$$VF = \frac{(W_f/\rho_f)}{(W_f/\rho_f)+(W_m/\rho_m)} \quad (1)$$

VF is fibre volume fraction, W_f(i.e. flax, jute and sisal) and W_m are the weight (g) of fibres and matrix respectively, ρ_f and ρ_m are the density (g/cm^3) of fibres and matrix, respectively. Also, the diameters of sisal, jute and flax fibres were evaluated by a Visual machine 250 tool makers microscope with ×4.5 magnifications and 1μm resolution at three different random locations along the single fibre and the average value is taken. The average diameters detected of sisal, jute and flax fibres were 240±40 μm, 880±80 μm and 17±10 μm, respectively. Table 1 show diameter and mechanical properties of natural fibres (sisal, jute and flax) used as reinforcement in composites materials were assembled from several sources [5-13].

Surface Chemical Treatments

In this study, fibres were treated with sodium hydroxide NaOH and sodium bicarbonate $NaHCO_3$, with various times 4, 12 and 24 hrs.

Treatment with NaOH

In this process untreated sisal, jute and flax fibres, they were respectively immersed in 7, 9 and 1 wt.% NaOH solution for various times 4, 12 and 24 hrs at room temperature. The fibres were then washed several times with fresh water to remove any NaOH sticking to the fibre surface, neutralized with dilute acetic acid and finally washed again with distilled water. Finally, pH was maintained at 7. The fibres were then dried at room temperature until a constant weight was reached.

Treatment with $NaHCO_3$

Similarly, the second treatment method consisted of soaking the raw of the sisal, jute and flax fibres in 25, 25 and 10 wt.% $NaHCO_3$ solution for various times 4, 12 and 24 hrs at room temperature, respectively. The fibres were then taken out of the solution, drained, and washed several times with tap water to remove any residual $NaHCO_3$ traces sticking on the fibre surface. Fibres were then neutralised with dilute acetic acid, and finally rinsed again with distilled water. Finally, the fibres were then dried at room temperature until a constant weight was reached.

Fabrication of Composites

In this present work, the individual and hybrid fibre

Table 1. Diameter and mechanical properties for some plant fibres [5-13]

Type of fibres	Properties					
	Density (g/cm^3)	Flexural modulus (GPa)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (wt.%)	Diameter (μm)
Banana	1.35	5-2	550-560	20	-56	120-126
Cotton	1.51	-	287-800	5.50-12.60	7-8	-
Flax	1.40-1.50	3.4	343-2000	45	1-4	12-600
Jute	1.30-1.48	11.9-14.7	320-800	13-26.50	1.5-1.16	25-200
Hemp	1.48	3-5	550-900	70	1.6	-
Ramie	1.5	-	938-220	128-44	3.8-2.0	20-80
Sisal	1.33-1.41	12.50-17.50	350-370	12.80	3-7	8-230

composites were fabricated by a hand lay-up technique using a wooden mould (200 mm×200 mm×5 mm) under uniform pressure of 0.75 MPa for 1h. Four beadings a glass plate were used to maintain a 5 mm thickness all around the mould plates. A fine layer of mould releasing agent is applied over this mould cavity to avoid the bonding of resin other than composite material and to easily recover the mould without bonding with resins and wax is applied to the secondary plate for covering and to equally support the weight. Then, each composite was cured under a load of 15 kg for 24 hours before removing from mould at room temperature. The plates were kept in open air for 5 days to obtain a complete polymerization of the resin. The specimens were then cut from the plates using a diamond saw, following the recommendations of the ASTM D790-03 standards.

Mechanical Properties

In order to evaluate the effect of the type of fibre, chemical treatment, volume fraction and treatment time on the flexural properties (ultimate flexural stress and flexural moduli) of the modified composites were measured using a Universal Testing Machine EZ20, equipped with a load cell of 20 kN. A flexural test imposes tensile stress on the convex side and compressive stress on the concave side of the specimen which causes a shear stress along the centre line. Three-point bending testing was used to measure the flexural properties as per the ASTM D790-03 standards (American Society for Testing and Materials) [35,36]. In bending test, the load cell of 20 kN was used with the cross head speed of 2 mm/min. Five samples with dimensions 150 mm×15 mm×5 mm were measured and the average values of the properties are reported in this article. In addition, flexural modulus and flexural strength were then obtained using the following expressions;

$$E_f = \frac{ML^3}{4bh^3} \tag{2}$$

$$\sigma_f = \frac{3P_f L}{2bh^2} \tag{3}$$

where E_f is the flexural modulus of elasticity (MPa), σ_f is the ultimate flexural stress (MPa), P_f is the maximum load (N), L is the support span (mm), b is the width of the beam (mm), h is the thickness of beam (mm), M is the slope of the force-vs-deflection curve (N/mm).

RSM Approach

Response surface methodology (RSM) is an efficient and flexible experimental design technique for the modelling and analysis of problem in which a response of interest is influenced by several variables [37]. Compared to conventional optimization method, RSM is an economic and time-saving technology for it can provide more information from less number of experiments. RSM has been widely used to describe the interactive and synergistic effects among experimental variables as well as operation conditions optimization. The procedure of optimization has been represented in the form of a flowchart as shown in Figure 1 [38,39].

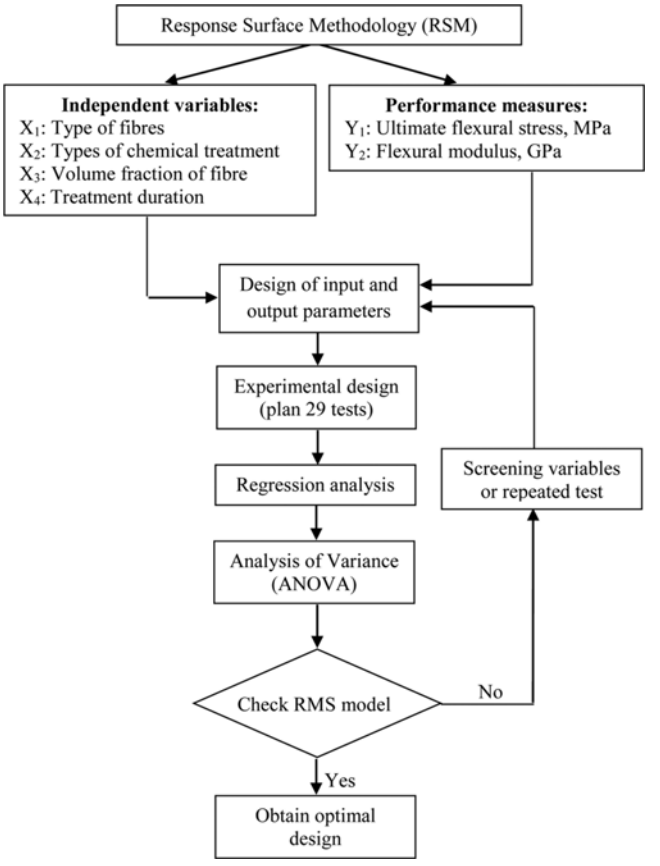

Figure 1. Procedure of response surface methodology.

In this study, the 4-factors and 3-levels BBD (low, medium and high coded as -1, 0, and +1) of each factor was employed to investigate the mechanical properties the bending tests of natural fibre composites. The RSM was used for evaluation of combined effects of type of fibres (X_1), types of chemical treatment (X_2), volume fraction of fibre (X_3) and treatment duration (X_4) on the bending tests. The ranges of independent variables and experimental conditions derived from BBD are summarized in Table 1. Total number of experiments carried out was 29, including eight axial, sixteen factorial and five centre points (calculated based on equation (4)). Table 2 provides the detail of the 29 experimental conditions and the experimental values of output variables (Ultimate flexural stress and flexural modulus).

$$N = 2^n + 2n + Nc = 2^4 + 2 \times 4 + 5 = 29 \text{ runs} \tag{4}$$

where N is the total experimental runs, n is the number of variables and Nc replicate runs at the centre.

A quadratic regression was used to develop the relationship

of output variables with the independent variables in the form of the second order polynomial equation (5):

$$Y = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 \quad (5)$$

where Y is the response; X_i and X_j are the variables (i and j range from 1 to k); b_0 is the model intercept coefficient; b_i, b_{ii} and b_{ij} are the interaction coefficients of linear, quadratic and the second order terms, respectively; k is the number of independent variables ($k = 4$ in this study). The quality of the model was expressed by the coefficient of determination (R^2), adjusted-R^2 (R^2 adj), and predicted-R^2 (R^2 pred). When R^2 approaches to unity, it indicates a good correlation between the experimental and the predicted values. These values can be determined using the following equations [38]:

$$R^2 = 1 - \frac{SS_{residual}}{SS_{model} + SS_{residual}} \quad (6)$$

$$R^2_{adj} = 1 - \frac{SS_{residual} / DF_{residual}}{(SS_{model} + SS_{residual}) / (DF_{model} + DF_{residual})} \quad (7)$$

In this equation, SS is the sum of squares and DF is degrees of freedom. Equations (6) and (7) and an F-test in the program were used to check the model's adequate precision ratio (AP) to determine the statistical importance of the model:

$$AP = \frac{\max(Y) - \min(Y)}{\sqrt{\overline{V}(Y)}} \quad (8)$$

$$\overline{V}(Y) = \frac{1}{n}\sum_{i=1}^{n}(Y) = \frac{p\sigma^2}{n} \quad (9)$$

where Y is the predicted response, p represents the number of model parameters, residual mean square is described as σ^2, and n is the number of experiments. After the F-test had been performed, the insignificant terms were found and eliminated from the model. Thereafter, the finalized model was introduced based on the significant variables. Eventually, optimum values were determined.

Also, the variables, units, symbol code and levels were shown in Table 2. The regression analyses, graphical analyses, analyses of variance (ANOVA) and analyses of response surfaces were carried out using Design-Expert Statistical Software (Version 8.0, Stat-Ease). The significance of the independent parameters and their interactions and the adequacy of the developed model were estimated by analysis of variance (ANOVA). In ANOVA table, P-value is the probability (ranging from 0 to 1) that the results observed in a study (or results more extreme) could have occurred by chance.

· If P value ≤ 0.05, the parameter is significant;
· If P value > 0.05, the parameter is insignificant.

Results and Discussion

Development of Regression Model Equation

An empirical relationship between the performance measures (ultimate flexural stress σ_f and flexural modulus E_f) and the independent variables such as: type of fibres (X_1), types of chemical treatment (X_2), volume fraction of fibre (X_3) and treatment duration (X_4) were modelled by quadratic regression. Based on the RMS method using the quadratic model of equaion (5), the approximated quadratic equation is obtained in terms of coded values for both responses are presented in equaions (10) to (13).

- Epoxy resin

$$\sigma_f = 75.97 + 5.451X_1 + 3.572X_2 + 14.311X_3 + 0.8235X_4 - 39.037X_1 \times X_1 + 26.130X_2 \times X_2 + 6.448X_3 \times X_3 + 6.154X_4 \times X_4 \quad (10)$$

$$E_f = 6.17 + 0.943X_1 - 0.282X_2 + 0.951X_3 + 0.052X_4 - 3.537X_1 \times X_1 + 0.649X_2 \times X_2 + 0.160X_3 \times X_3 - 0.517X_4 \times X_4 \quad (11)$$

- Polyester resin

$$\sigma_f = 93.20 - 2.501X_1 - 1.667X_2 + 24.082X_3 + 2.37X_4 - 31.22X_1 \times X_1 + 6.894X_2 \times X_2 + 7.864X_3 \times X_3 - 2.063X_4 \times X_4 \quad (12)$$

$$E_f = 7.54 - 0.254X_1 + 0.019X_2 + 2.496X_3 + 0.067X_4 - 3.934X_1 \times X_1 + 0.358X_2 \times X_2 + 0.156X_3 \times X_3 + 0.095X_4 \times X_4 \quad (13)$$

Table 2. Levels of various independent variables at coded values of RSM experimental design

Independent variables	Units	Levels		
		-1	0	+1
X_1 : Type of fibres	-	Flax	Jute	Sisal
X_2 : Types of chemical treatment	-	$NaHCO_3$	Raw	NaOH
X_3 : Volume fraction of fibre	(%)	10	15	20
X_4 : Treatment duration	hrs	4	12	24

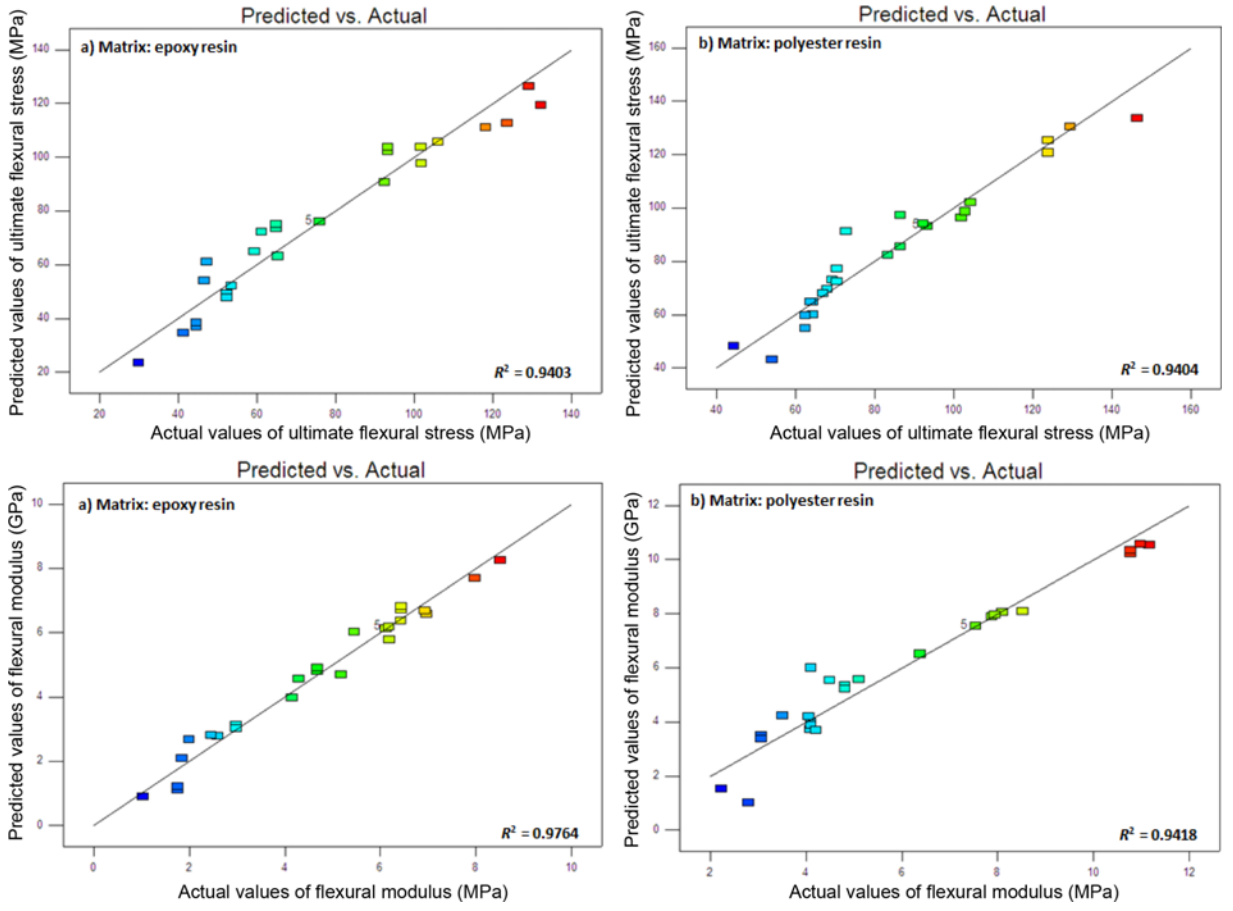

Figure 2. Comparison plot between predicted and actual values for ultimate flexural stress and flexural moduli.

The goodness of fit of the model was verified by the correlation coefficient (R^2) between the actual and model predicted values of the response variable. Since, the high value of R^2 advocates a correlation between experimental and predicted values of response. In the present study, R^2 for ultimate flexural stress σ_f from epoxy and polyester resins were found to be 0.9403, 0.9404, and 0.9764, 0.9418 for the flexural modulus E_f, respectively. In addition, the predicted values of the responses (σ_f and E_f) using the equations were compared with the other experimental results as shown in Figure 2. Obviously, the actual values are distributed fairly close to the straight line, illustrating that the predicted values of the ultimate flexural stress σ_f and flexural modulus E_f by the models are in good agreement with the actual experimental data for both resin matrices (epoxy and polyester).

Perturbation Plots

Perturbation plots in RSM design revealed significant independent variables and which one have a positive or a negative effect on the performance measures by displaying changes in response of each factor as each factor moves from the reference point, which is the zero coded level of each factor, with all other factors held constant at the reference value. In Figure 3, it is clear that types of fibres (A or X_1) had both increases and decreases effect for all responses while other two factors (B or X_2 and C or X_3) had a significant positive effect on the mechanical properties (ultimate flexural stress and flexural modulus) of composites. It is notable that the treatment duration (D or X_4) was not significant for responses.

Analysis of Variance ANOVA

An analysis of variance (ANOVA) is necessary to check the model significance, and the results are presented in Tables 1 and 2. ANOVA is a statistical analysis that is part of the analysis in Response Surface Methodology [38]. It has been applied to investigate the difference between two or more variables that vary in an experiment and is usually used to indicate that there is a significant result from the experiment. The statistical significance of each coefficient of the model equations (5)-(8), were checked by P-values and F-values. According to the results, for the ultimate flexural stress, the high F-values (39.38 for $\sigma_{f\text{-epoxy}}$ and 39.48 for

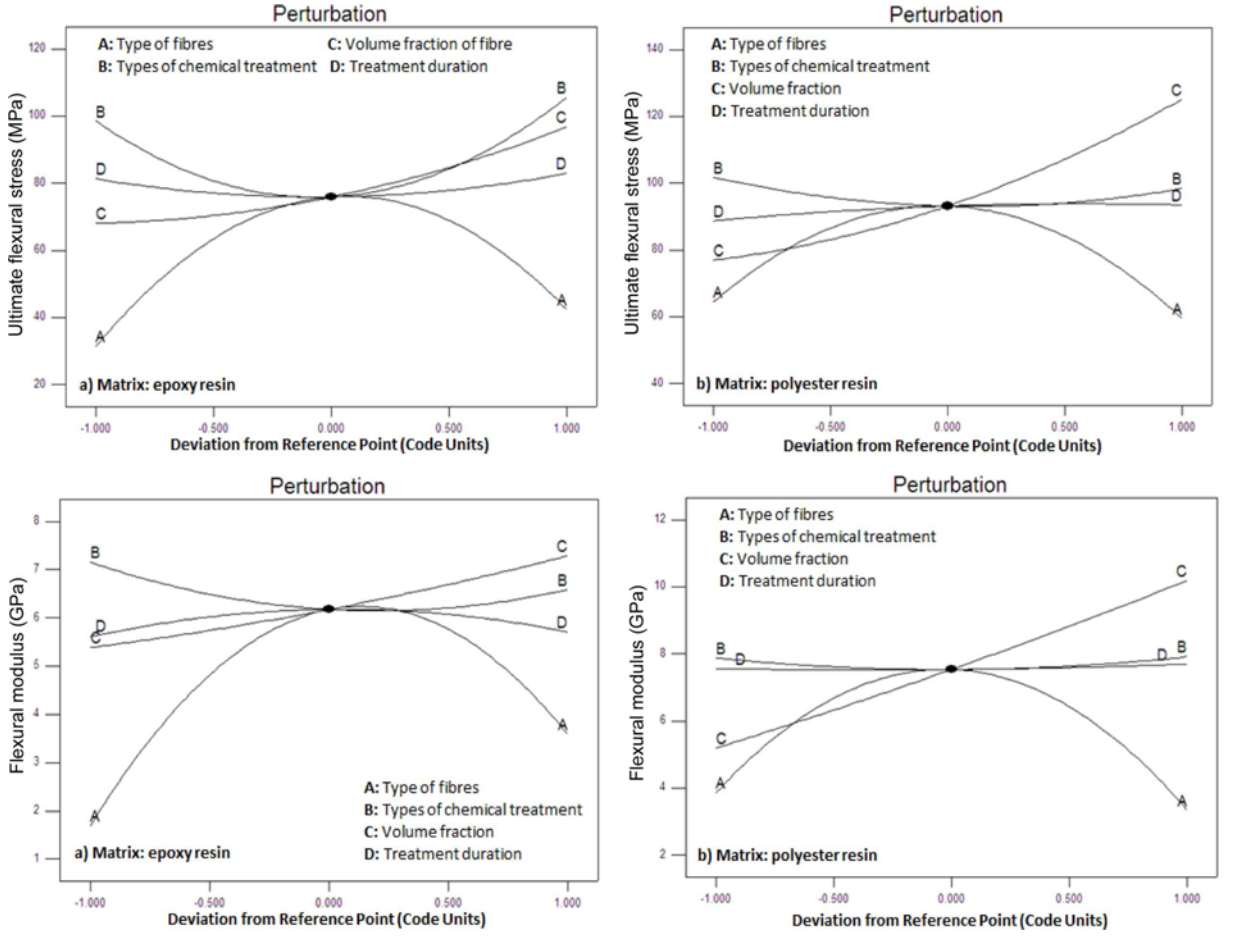

Figure 3. Effect of independent variables on flexural mechanical (σ_f and E_f).

$\sigma_{f\text{-polyester}}$) and small P-values (< 0.0001 for $\sigma_{f\text{-epoxy}}$ and <0.0001 for $\sigma_{f\text{-polyester}}$ value, both p-values < 0.05) suggested that the regression models are more significant. In the same way, for the flexural modulus, the models F-value of (103.53 for $E_{f\text{-epoxy}}$ and 40.52 for $E_{f\text{-polyester}}$) with very low probability (P < 0.0001; P < 0.0001 and both p-values < 0.05), implies that these models are significant. The importance of each independent variable on the dependent variables can be estimated by the sum of square (SS) of independent variables. The larger sum of square of independent variables indicates a relatively larger effect on dependent variables. Firstly, Table 4 presents ANOVA results for ultimate flexural stress σ_f. It can be seen that the volume fraction of fibre (X_3) is the most important factor affecting σ_f in a considerable way, with (13.80 and 49.02) % contribution when used epoxy and polyester resins matrices respectively. A similar trend was observed by other authors [14,40-42]. For example, Salman *et al.* [14] investigated the influence of fibre content on the mechanical properties of woven kenaf fibre-reinforced polyvinyl butyral composites. It was noticed that the composites with 40 wt.% fibre content attested the highest mechanical properties. Then, the second factor influencing σ_f is the type of fibres (X_1) with (2 and 0.53) % contribution values. For this factor, lots of work has been done to study the effect of fibre loading on the mechanical properties [40-42]. Rao *et al*. [42] it has been found that the mechanical properties of natural fibre composite are directly related to type fibre and fibre content. Then, the types of chemical treatment (X_2) and the treatment duration (X_4) have a very weak significance effect on σ_f when compared the effect of X_3. This dispersion of the results is mainly due to the differences in diameter and age of the fibres. Similar results were reported by Alvarez [43].

Finally, according to ANOVA results of flexural modulus E_f presented in Table 5, the term X_3 (volume fraction of fibre) represents the higher statistical significance on flexural modulus with the contribution of (9.99 and 42.21) %. A study conducted by Ng [44] obtained a similar trend where the Kenaf fibre-reinforced thermoplastic polymers PP composites with various fibre contents 10 %, 20 % and 30 % (by weight) displayed the highest flexural proprieties. The term X_1 is also significant with smaller contribution of (9.82

Table 3. The levels of the factors values and the results of the experiments for σ_f and E_f

N°	Coded factors				Actual factors				Polyester resin		Epoxy resin	
									Performance measures			
	X_1	X_2	X_3	X_4	Type of fibres	Types of chemical treatment	Volume fraction	Treatment duration (hrs)	Ultimate flexural stress σ_f (MPa)	Flexural modulus E_f (GPa)	Ultimate flexural stress σ_f (MPa)	Flexural modulus E_f (GPa)
1	0	0	1	1	Jute	Raw	20	24	123.77	10.78	87.67	5.20
2	0	0	0	0	Jute	Raw	15	12	93.20	7.54	75.97	6.18
3	0	1	0	1	Jute	NaOH	15	24	102.91	8.52	123.61	5.45
4	0	0	0	0	Jute	Raw	15	12	93.20	7.54	75.97	6.18
5	0	0	-1	1	Jute	Raw	10	24	70.45	4.81	64.87	4.68
6	-1	0	-1	0	Flax	Raw	10	12	44.33	2.23	29.90	1.03
7	0	-1	-1	0	Jute	$NaHCO_3$	10	12	86.47	4.49	92.41	6.43
8	0	1	1	0	Jute	NaOH	20	12	129.50	10.98	129.16	7.98
9	0	0	0	0	Jute	Raw	15	12	93.20	7.54	75.97	6.18
10	-1	0	0	-1	Flax	Raw	15	4	64.34	4.10	44.55	1.76
11	0	0	0	0	Jute	Raw	15	12	93.20	7.54	75.97	6.18
12	-1	0	0	1	Flax	Raw	15	24	59.33	3.87	37.13	1.28
13	1	0	1	0	Sisal	Raw	20	12	72.72	4.10	65.27	5.18
14	1	-1	0	0	Sisal	$NaHCO_3$	15	12	66.87	4.21	59.38	4.29
15	0	0	0	0	Jute	Raw	15	12	93.20	7.54	75.97	6.18
16	-1	-1	0	0	Flax	$NaHCO_3$	15	12	69.29	4.06	46.55	1.98
17	-1	0	1	0	Flax	Raw	20	12	101.93	6.38	53.52	2.46
18	1	0	0	-1	Sisal	Raw	15	4	69.05	3.10	59.96	3.11
19	1	0	0	1	Sisal	Raw	15	24	62.42	3.06	52.34	2.98
20	0	-1	0	-1	Jute	$NaHCO_3$	15	4	86.47	7.88	101.65	6.97
21	0	0	-1	-1	Jute	Raw	10	4	70.45	4.81	64.87	4.68
22	1	1	0	0	Sisal	NaOH	15	12	63.73	4.09	61.17	4.15
23	0	-1	1	0	Jute	$NaHCO_3$	20	12	146.40	11.17	132.24	8.51
24	-1	1	0	0	Flax	NaOH	15	12	68.03	3.51	47.28	1.84
25	0	1	-1	0	Jute	NaOH	10	12	83.33	5.10	101.81	6.19
26	0	-1	0	1	Jute	$NaHCO_3$	15	24	104.22	8.10	106.06	6.93
27	0	1	0	-1	Jute	NaOH	15	4	92.22	7.94	118.14	5.45
28	1	0	-1	0	Sisal	Raw	10	12	54.08	2.80	41.27	2.59
29	0	0	1	-1	Jute	Raw	20	4	153.12	11.61	93.34	6.29

and 0.44) %. On the other hand, the chemical treatment (X_2) and the treatment duration (X_4) have a very weak significance effect on E_f. Effects of the chemical treatment and treatment duration on mechanical properties were studied previously [19,20]. On the opposite side, it can be noted that the product ($X_1 \times X_1$) affects E_f in a considerable way. Its contribution is (74.67 and 56.71) % for both matrices; epoxy and polyester resins, respectively. Hence, the use of more rigid fibres leads to an improvement of the composite flexural properties in a considerable way. A similar trend was observed by other authors [40,42].

Effect of Independent Parameters on Surface Response Performance Measures

In order to better understand the interaction effect of independent on response factors, three-dimensional response surface plots of the model were prepared for quantifying the optimal values to obtain the optimum ultimate flexural stress and flexural modulus. The response surface plots (3-D) of the interaction effects of the different variables while the treatment duration is kept at the middle level (12 hrs) are shown in Figures 4 and 5, which displayed a visual interpretation of various effects from a steep degree of 3-D

Table 4. Analysis of variance for ultimate flexural stress σ_f

Source	SS	DF	MS	F-value	P-value	Cont. %	Remarks
(a) Epoxy resin							
Model	21265.18	8	2658.14	39.38	< 0.0001		Significant
X_1: Type of fibres	356.57	1	356.57	5.28	0.0324	2.00	-
X_2: Chemical treatment	153.16	1	153.16	2.27	0.1476	0.86	Insignificant
X_3: Volume fraction	2457.85	1	2457.85	36.42	< 0.0001	13.80	Significant
X_4: Treatment duration	8.13	1	8.13	0.12	0.7320	0.05	Insignificant
$X_1 \times X_1$	9885.07	1	9885.07	146.47	< 0.0001	55.52	Significant
$X_2 \times X_2$	4428.85	1	4428.85	65.62	< 0.0001	24.87	-
$X_3 \times X_3$	269.74	1	269.74	3.99	0.0594	1.51	Insignificant
$X_4 \times X_4$	245.68	1	245.68	3.64	0.0709	1.38	-
Error	1349.76	20	67.48				
Total	22614.95	28				100	
$R^2 = 0.9403$			R^2 Adjusted = 0.9164			R^2 Predicted = 0.8657	
(b) Polyester resin							
Model	15511.80	8	1938.97	39.48	< 0.0001		Significant
X_1: Type of fibres	75.10	1	75.10	1.52	0.2306	0.53	Insignificant
X_2: Chemical treatment	33.33	1	33.33	0.67	0.4198	0.23	-
X_3: Volume fraction	6959.16	1	6959.16	141.69	< 0.0001	49.02	Significant
X_4: Treatment duration	67.40	1	67.40	1.37	0.2552	0.47	Insignificant
$X_1 \times X_1$	6323.68	1	6323.68	128.75	< 0.0001	44.55	Significant
$X_2 \times X_2$	308.30	1	308.30	6.27	0.0210	2.17	-
$X_3 \times X_3$	401.15	1	401.15	8.16	0.0097	2.83	-
$X_4 \times X_4$	27.61	1	27.61	0.56	0.4621	0.19	Insignificant
Error	982.26	20	49.11				
Total	16494.07	28				100	
$R^2 = 0.9404$			R^2 Adjusted = 0.9166			R^2 Predicted = 0.8660	

plots, The steepness of the response surface indicated the degree of interaction influences of two experimental variables.

The ultimate flexural stress of different composites affected by different types of chemical treatment (X_2) and the volume fraction of fibre (X_3) were shown in Figures 4a and 4b, with treatment duration (X_4) fixed at a middle level (12 hrs) for both resin matrices (epoxy and polyester). It showed that the ultimate flexural stresses of different composites were affected significantly by the volume fraction of fibre and types of chemical treatment. Also, when the types of chemical treatment was kept at a middle level (raw) provides lower values of ultimate flexural stresses of different composites than the other levels (-1: $NaHCO_3$ and +1: NaOH). Chemical modifications of natural fibres could remove surface impurities and increased the surface roughness. These modifications increase to the bonding of the fibre with the resins matrix there by improving the fibre-matrix interaction, subsequently, significantly increased the ultimate tensile strength and flexural modulus of the composites [19,45]. The effect of interfacial adhesion was discussed by Wong [46] and Yang [47]. In addition, as shown in Figures 5a and 5b, types of chemical treatment (X_2) and the volume fraction of fibre (X_3) indicated quadratic and linear effects on the flexural modulus of different reinforced composites, when treatment duration (X_4) was fixed at a middle level (12 hrs), respectively. As a result of good interfacial bonding between fibre and matrix, the fibres are effectively participating in the stress transfer. The flexural modulus increased rapidly with the increase of the volume fraction of fibre, but increased slowly as the types of chemical treatment changed.

In general, the flexural properties of all individual composites considered in the present study increases with volume fraction of fibre in the composite (concentration of the fibre in the composite) increases. It is also observed that the flexural properties (ultimate flexural stress σ_f and flexural modulus E_f) of jute fibre composite generate higher values than the other fibres. For example, the average values of flexural properties under the following conditions (X_2 is the low level: $NaHCO_3$, X_3 is the high level: 20 % and X_4 is

Table 5. Analysis of variance for flexural modulus E_f

Source	SS	DF	MS	F-value	P-value	Cont. %	Remarks
(a) Epoxy resin							
Model	118.91	8	14.86	103.53	< 0.0001		Significant
X_1: Type of fibres	10.67	1	10.67	74.38	< 0.0001	9.82	-
X_2: Chemical treatment	0.95	1	0.95	6.67	0.0177	0.88	-
X_3: Volume fraction	10.85	1	10.85	75.61	< 0.0001	9.99	-
X_4: Treatment duration	0.03	1	0.03	0.22	0.6405	0.03	Insignificant
$X_1 \times X_1$	81.16	1	81.16	565.35	< 0.0001	74.67	Significant
$X_2 \times X_2$	3.11	1	3.11	21.65	0.0002	2.86	-
$X_3 \times X_3$	0.16	1	0.16	1.16	0.2934	0.15	Insignificant
$X_4 \times X_4$	1.73	1	1.73	12.08	0.0024	1.60	Significant
Error	2.87	20	0.14				
Total	121.78	28				100	
$R^2 = 0.9764$				R^2 Adjusted = 0.9669		R^2 Predicted = 0.9469	
(b) Polyester resin							
Model	190.05	8	23.75	40.52	< 0.0001		Significant
X_1: Type of fibres	0.77	1	0.77	1.32	0.2631	0.44	Insignificant
X_2: Chemical treatment	0.01	1	0.01	0.01	0.9321	0.01	-
X_3: Volume fraction	74.75	1	74.75	127.51	< 0.0001	42.21	Significant
X_4: Treatment duration	0.05	1	0.05	0.09	0.7661	0.03	Insignificant
$X_1 \times X_1$	100.41	1	100.41	171.27	< 0.0001	56.71	Significant
$X_2 \times X_2$	0.83	1	0.83	1.42	0.2471	0.47	Insignificant
$X_3 \times X_3$	0.15	1	0.15	0.27	0.6097	0.09	-
$X_4 \times X_4$	0.05	1	0.05	0.10	0.7539	0.03	-
Error	11.72	20	0.58				
Total	201.77	28				100	
$R^2 = 0.9418$				R^2 Adjusted = 0.9186		R^2 Predicted = 0.8692	

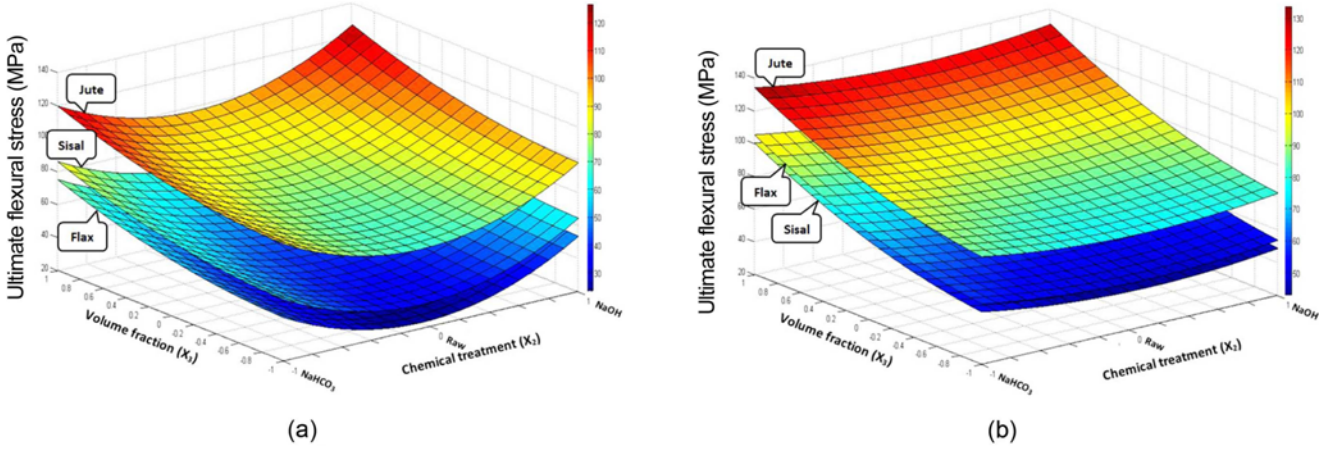

Figure 4. Comparison of response surface for ultimate flexural stress versus X_1, X_2, and X_3 at the middle level of X_4; (a) epoxy resin and (b) polyester resin.

the medium level: 12 hrs) are: $\sigma_{f\text{-sisal}}$~1,36$\sigma_{f\text{-jute}}$, $\sigma_{f\text{-flax}}$~1,40$\sigma_{f\text{-jute}}$, $E_{f\text{-sisal}}$~1,57$E_{f\text{-jute}}$, $E_{f\text{-flax}}$~2,35$E_{f\text{-jute}}$. This is due to lower percentage strain of jute fibre composite compared to sisal and flax composites.

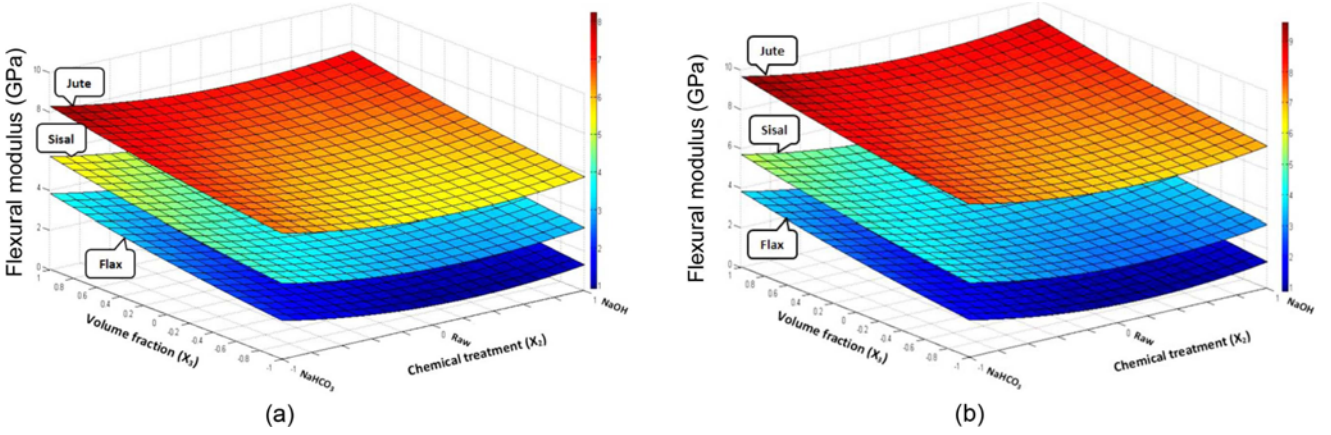

Figure 5. Comparison of response surface for flexural modulus versus X_1, X_2, and X_3 at the middle level of X_4; (a) epoxy resin and (b) polyester resin.

RSM Optimisation

The optimal levels of the process factors were determined with the aim of achieving the maximizing the values of ultimate flexural stress and flexural modulus using the RMS approach. This approach is a multi-criteria methodology often applied when various responses have to be considered at the same time and it is necessary to find optimal comprises between the total numbers of responses taken into account [37]. Statistical analyses were performed operating the Design-Expert software V8 (Stat-Ease). The constraints used during the optimization process are summarized in Table 6. The best optimal values of the process factors are reported in Table 7 in order of decreasing desirability level. Values of optimal independent variables: are found to be as follows: The epoxy composite at (0.13 and 0.19) of the type of fibres, -1 of the type of chemical treatment, +1 of the volume fraction of fibre and (-0.76 and -0.52) the treatment duration ensure the maximize response parameters (ultimate flexural stress and flexural modulus). Then, it is found that (0.13 and 0.19) of the type of fibres, -1 of the type of chemical treatment, +1 of the volume fraction of fibre and (-0.76 and -0.52) the treatment duration are responsible for

Table 6. Constraints for optimization of independent variables

Conditions	Goal	Lower limit		Upper limit	
		Epoxy resin	Polyester resin	Epoxy resin	Polyester resin
X_1 : Type of fibres	In range	-1		+1	
X_2 : Types of chemical treatment	In range	-1		+1	
X_3: Volume fraction of fibre, %	In range	-1		+1	
X_4 : Treatment duration, hrs	In range	-1		+1	
Y_1: Ultimate flexural stress, MPa	Maximize	29.90	44.33	132.24	146.40
Y_2: Flexural modulus, GPa	Maximize	1.03	2.23	8.51	11.17

Table 7. Response optimization for ultimate flexural stress and flexural modulus

Test N°	Coded factors				Performance measures		Desirability D	Remarks
	X_1	X_2	X_3	X_4	Ultimate flexural strength σ_f (MPa)	Flexural modulus E_f (GPa)		
Epoxy resin								
1	0.13	-1	+1	-0.76	125.55	8.23	0.9488	Selected
2	0.10	-1	+1	-0.52	123.67	8.34	0.9465	
Polyester resin								
1	-0.13	-1.00	+1	-0.04	137.97	10.80	0.9381	Selected
2	-0.18	-1.00	+1	-0.37	136.44	10.82	0.9311	

Table 8. Optimal levels of factors in actual terms

N°	Coded factors				Actual factors			
	X_1	X_2	X_3	X_4	Type of fibres content (%)	Types of chemical treatment	Volume fraction of fibre (%)	Treatment duration (hrs)
Epoxy resin								
01	0.13	-1	+1	-0.76	87 % of jute and 13 % of sisal	$NaHCO_3$	20	5h55
02	0.10			-0.52	90 % of jute and 10 % of sisal			7h50
Polyester resin								
01	-0.13	-1	+1	-0.04	83 % of jute and 17 % of flax	$NaHCO_3$	20	11h41
02	-0.18			-0.37	82 % of jute and 18 % of flax			9h02

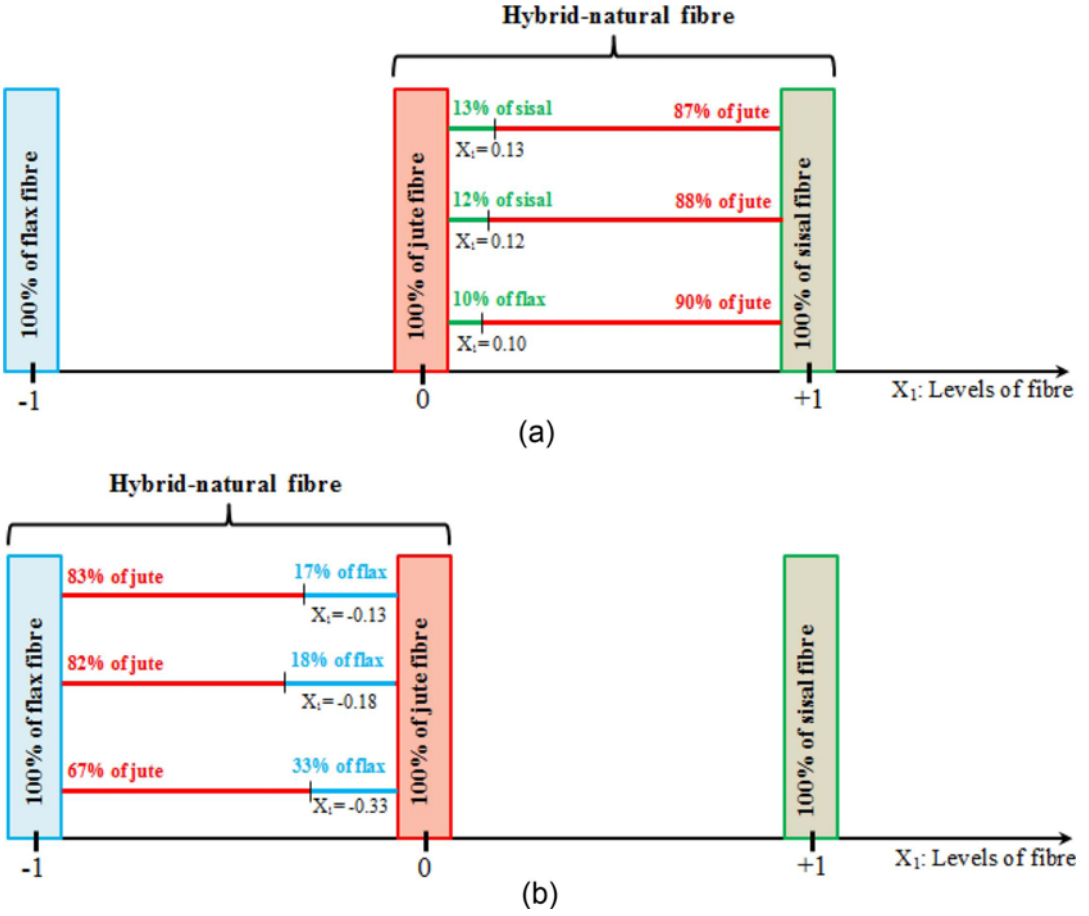

Figure 6. Rule of hybrid mixture; (a) epoxy resin and (b) polyester resin.

the maximize response parameters (σ_f and E_f) when used the polyester composite.

Once the optimal level of the process parameters is selected, the final step is to predict and verify the improvement of the performance characteristics using the optimal levels of the process parameters presented in terms of coded factors in above section. To make the confirmation tests, we converted the coded values presented in Table 8 to the actual values, then were fabricated by a hand lay-up technique. The rule of mixture fibre has been presented in Figure 6.

Confirmation Experiments

The hybrid composites for this section were fabricated by hand-lay up method technique using a wooden mould (200 mm×25 mm×5 mm). After 7 days of curing, the plates were cut according to ASTM standards. Help designations and composition of hybrid composites presented in Table 9,

Table 9. Designations and composition of hybrid composites

n°	Composites	Code	Volume fraction			Resin content (%)	Treatment duration (hrs)
			Jute fibre content (%)	Flax fibre content (%)	Sisal fibre content (%)		
Epoxy resin							
1	Single composite: Jute/epoxy	CEJ	20	-	0	80	12h
2	Single composite: Sisal/epoxy	CES	0	-	20		
3	Hybrid composite: Intimate mix	HCE01	17.40	-	2.60		5h55
4	Hybrid composite: jute/sisal/jute	HCE02					
5	Hybrid composite: sisal/jute /sisal	HCE03					
6	Hybrid composite: Intimate mix	HCE04	18	-	2		7h50
7	Hybrid composite: jute/sisal/jute	HCE05					
Polyester resin							
1	Single composite: Jute/polyester	CPJ	20	0	-	80	12h
2	Single composite: Flax/polyester	CPF	0	20	-		
3	Hybrid composite : Intimate mix	HCP01	17.60	3.40	-		11h41
4	Hybrid composite: jute/flax/jute	HCP02					
5	Hybrid composite: Intimate mix	HCP03	16.40	3.60	-		9h02
6	Hybrid composite: jute/flax/jute	HCP04					
7	Hybrid composite: flax/jute/flax	HCP05					

twelve different kinds of composites were prepared with stacking sequences and the different configurations of composites are represented in Figures 6a-6e. Intimate mix is presented in Figure 7a (HCE01 and HCP01), in Figure 7b, jute is the skin material and sisal is the core material and it is in the reverse order in Figure 7c. Figure 7d, jute is the skin material and flax is the core material and it is in the reverse order in Figure 7e.

Flexural Test Confirmation

The flexural test for the hybrid composite samples is performed in a universal testing machine according to ASTM D 790. The graphs comparing stress-strain of all configurations of composites are presented in Figure 8. All configurations show a similar mechanical behaviour and the initial linear portion of the hybrid composite curves show the elastic behaviour of the composite. Modulus, which was determined from the initial slope of the stress-strain curve, was similar for all reputation. It has also been noticed that the weave-fabricated fibres are uniform in term of distribution of fibres and spaces between them. Then, the average flexural properties of different hybrid composites are regrouped in Figure 9.

From Figure 9a, it is understood that, the results of hybrid composites (HCE05 and HCP05) are higher than the other composites tested which can withstand the ultimate flexural stress of 136.04 and 156.517 MPa, respectively. This indicates that the flexural properties of the composite increase with an increase in the composition of jute fibres.

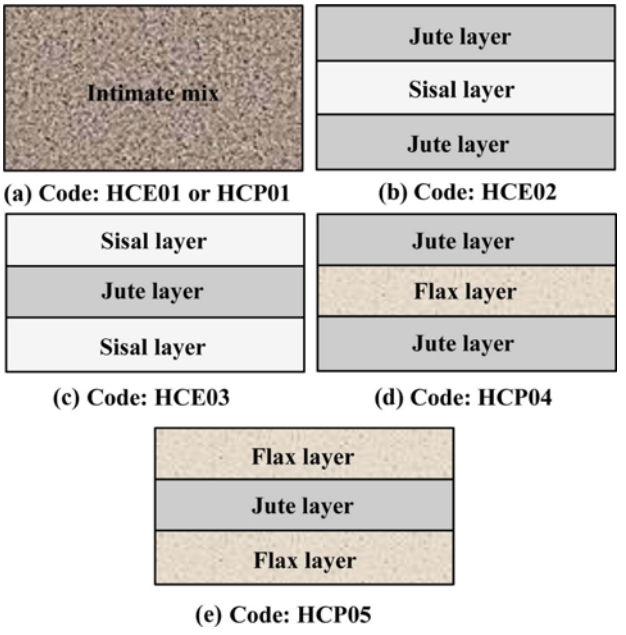

Figure 7. Schematic representation of different layering patterns of hybrid composites.

By comparing the flexural modulus (Figure 9b), it is noted that the hybrid composites (HCE04 and HCP05) have higher modulus than the other hybrid composites. This is due to the presence of 90 % of jute in HCE04 and 82 % of jute in HCP05. It is also observed that the change in order of fibres

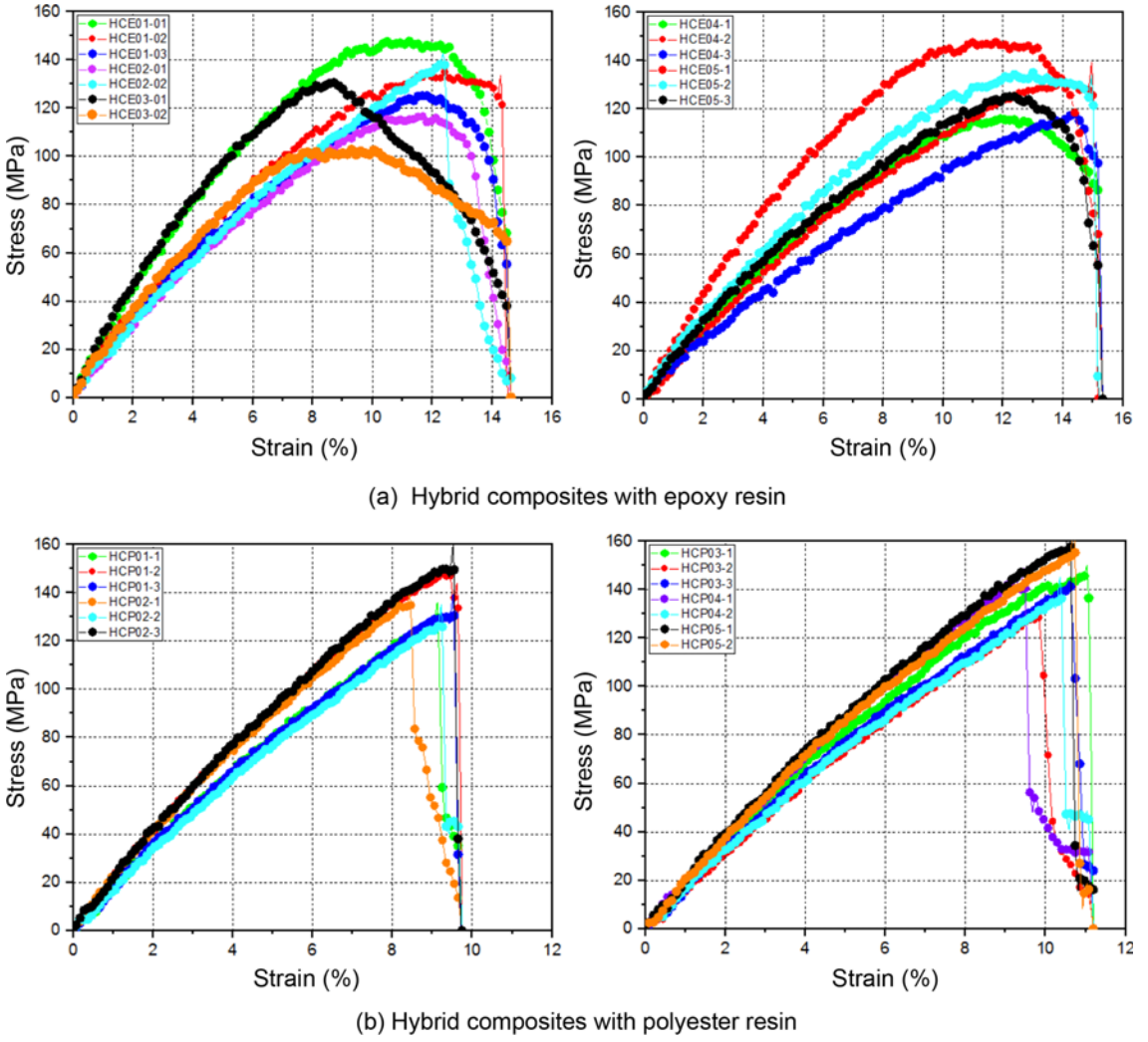

Figure 8. Flexural stress versus strain for hybrid composites.

has its effect on the flexural properties of hybrid composites. It is seen that the composite exhibits higher flexural properties when the order fibres are jute is the skin material and sisal is the core material (HCE02 or HCE05) for epoxy matrix and when the flax is the skin material and jute is the core material (HCP05) for polyester matrix.

Scanning Electron Microscopy Studies

A microstructural analysis of the fractured surfaces of hybrid jute/sisal epoxy composites (HCE02) of flexural tested specimens were examined with the help of Scanning Electron Microscopy SEM (Model: JSM 6360LV). The all samples have been covered with a thin layer of gold to make them conductive. The magnification and the voltage are displayed on the microphotographs of the samples. The SEM micrograph for hybrid composite fracture is shown in Figures 10 and 11, respectively. Firstly, it can be seen from the cross section of Figure 10, a uniform distribution of fibres of different layers is observed. The jute is the skin material and sisal is the core material (HCE02).

Secondly, is clearly observed strong interfacial bonding between the fibres and matrix. These give strong evidence for the higher mechanical properties of these composites. It is also clear from SEM image (Figure 10b) that only little evidence of fibre pull out is visible, which indicates that chemical treatments led to good interfacial adhesion between the fibre and matrix leading to better stress transfer efficiency with increased mechanical properties.

Conclusion

In this paper, the flexural properties of jute, sisal, flax fibre reinforced individual and hybrid polymer composites were studied. Based on the results, the following conclusions are drawn:

1. The analysis of independent variables using RSM technique

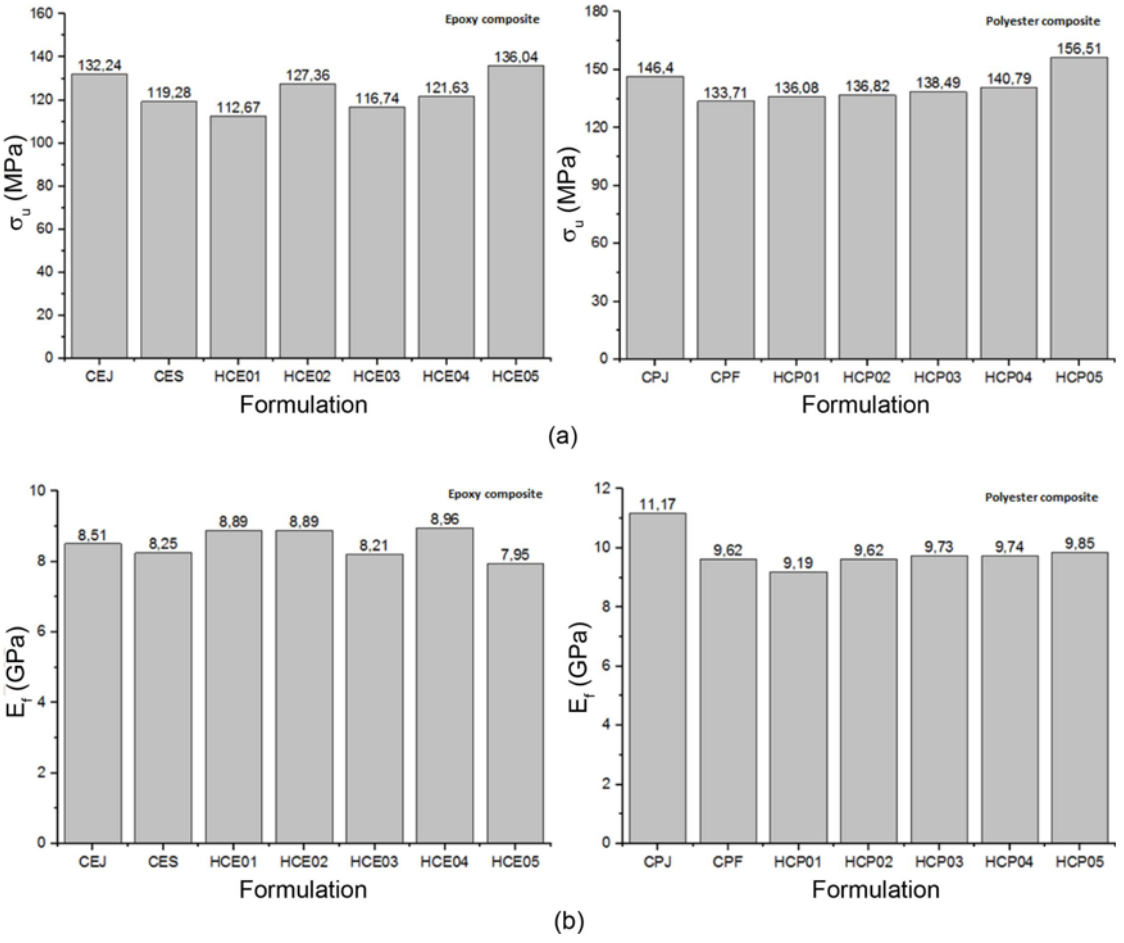

Figure 9. Comparison of the flexural properties of various composites for both resin matrices; (a) ultimate flexural and (b) flexural modulus.

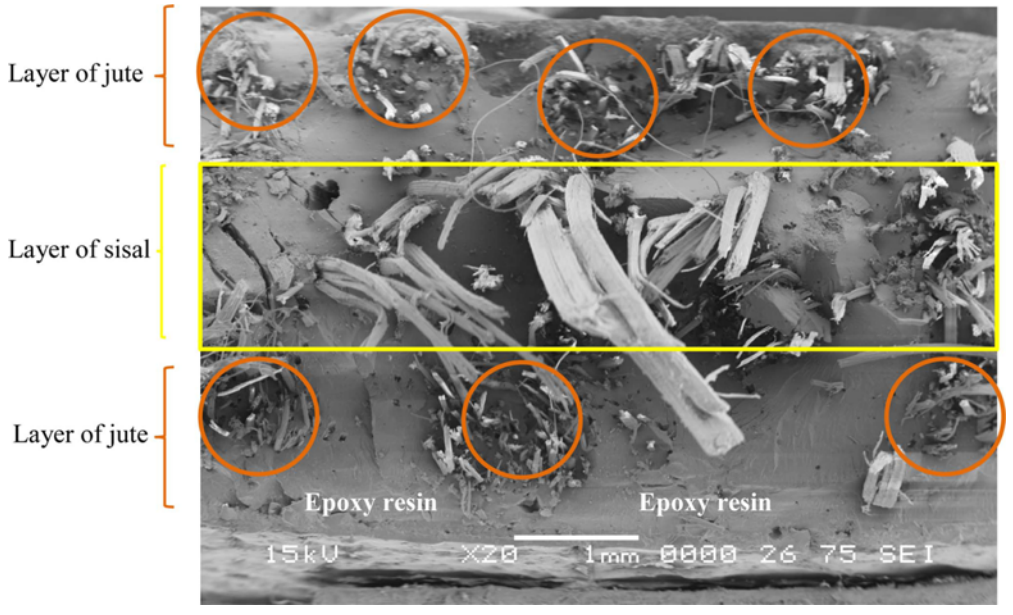

Figure 10. Transversal cross section of hybrid composites (HCE02).

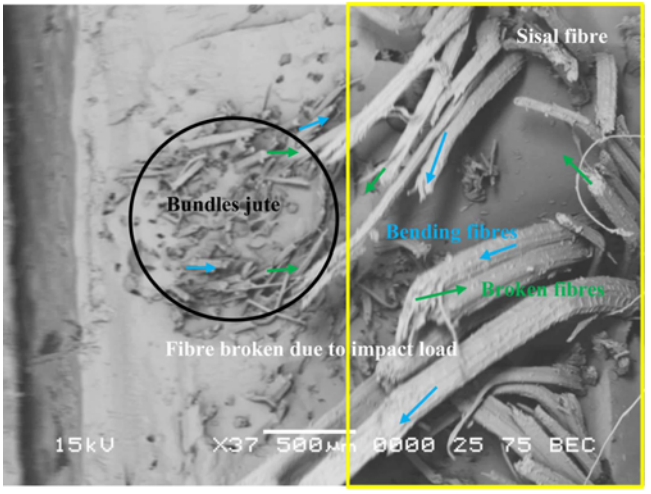

Figure 11. Fracture surface of the flexural test in hybrid composites (HCE02).

allows investigating the influence of each one on the output parameters which are the mechanical characteristics namely, ultimate flexural strength σ_f and flexural modulus E_f.

2. The ANOVA shows that:
   (a) The ultimate flexural strength σ_f is strongly influenced by the volume fraction of fibre with contributions between ($Cont._{epoxy\text{-}resin} \approx 13.80$ and $Cont._{polyester\text{-}resin} \approx 49.02$) %. The next largest factor influencing on σ_f is the type of fibres with contributions between ($Cont._{epoxy\text{-}resin} \approx 2.00$ and $Cont._{polyester\text{-}resin} \approx 0.53$) %. The types of chemical treatment and the treatment duration comes in the last position with contributions of [($Cont._{epoxy\text{-}resin} \approx 0.86$ and $Cont._{polyester\text{-}resin} \approx 0.23$) % and ($Cont._{epoxy\text{-}resin} \approx 0.05$ and $Cont._{polyester\text{-}resin} \approx 0.47$)] %, respectively.
   (b) In the same way, the volume fraction of fibre has the highest physical as well statistical influence on the flexural modulus followed by type of fibres. But the types of chemical treatment and the treatment duration have a very small influence.
   (c) The results indicated a clear correlation between fibre volume fractions and the flexural properties (ultimate flexural strength and flexural modulus) of the composite.
3. From multi-objective optimization:
   (a) Comparison of experimental and predicted values of the, ultimate flexural strength and flexural modulus show that a good agreement has been achieved between them. However, the validity of the procedure is limited to the range of factors considered for the experimentation.
   (b) The epoxy composite at [0.13 and 0.10] of the type of fibres, -1 (actual factor is $NaHCO_3$) of the type of chemical treatment, +1 (actual factor is 20 %) of the volume fraction of fibre and [-0.76 and -0.52] (actual factor are 5h55 and 7h50) the treatment duration ensure the maximize response parameters (σ_f and E_f).
   (c) Then, it is found that [-0.13 and -0.18] of the type of fibres, -1 (actual factor is $NaHCO_3$) of the type of chemical treatment, +1 (actual factor is 20 %) of the volume fraction of fibre and [-0.04 and -0.37] (actual factor are 11h41 and 9h02) the treatment duration are responsible for the maximize response parameters (σ_f and E_f) when used the polyester composite.
4. The hybridization improved the flexural properties for all the composites studied when compared to individual fibres. For jute+sisal (HCE05) and flax+jute (HCP05) composites, the improvement in ultimate flexural stress was by approximately 8 % and 12 %, respectively compared to individual type of natural fibers reinforced.
5. The SEM micrographs of flexural fractured specimens reveal that jute+sisal+jute fiber-epoxy hybrid composites (HCE02) show better interfacial bonding between the fibres and matrix. These give strong evidence for the higher mechanical properties of these composites.
6. Finally, it can be concluded that the reinforcement of jute, sisal, flax fibre in epoxy/or polyester matrices results in a positive hybrid effect for flexural properties. Therefore, value added and cost-effective composites having high flexural properties could be well developed by the judicious selection of jute, sisal and flax fibre.

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