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Tensile mechanical properties of natural fibre composites – a statistical approach

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Abstract:

The main objective and the originality of this work are to create a hybrid-natural fibre composite by the RMS method. Hybrid composites are manufactured by combining two or more dissimilar kinds of fibre in a single matrix. In the first section, Response Surface Methodology (RSM) using a Box-Behnken experimental design and the Analysis of Variance (ANOVA) are applied to investigate the effects of the type of fibres, chemical treatment, volume fraction and treatment time on the mechanical properties (ultimate tensile strength and Young's modulus) in the tensile quasi-static loading when used two resins namely, epoxy and polyester. In the studied range, statistical analysis of the results showed that selected variables had a significant effect on the mechanical properties, except the treatment time that has a very weak significance effect on the mechanical properties. Then, to maximize the mechanical properties, the optimal conditions coded by RSM were found: the type of fibres (X_1) of $[-0.28$ and $-0.33]$, the chemical treatment (X_2) of -1 , the volume fraction of fibre (X_3) of 1 and the treatment duration (X_4) of $[-0.97$ and $-1]$ for epoxy resin matrix. Similarly, when used the polyester resin matrix; the type of fibres (X_1) of -0.26 , the chemical treatment (X_2) of -1 , the volume fraction (X_3) of 0.99 and the sinking time (X_4) of $[-0.94$ and $-0.93]$. The obtained optimum parameters were confirmed experimentally in the second section.

Keywords: Natural fibres, hybrid composites, RSM, modelling, optimization

Contributions

LAOUICI Hamdi, results analysis and article writing; Asma Benkhelladi, elaboration of test specimens and mechanical properties tests; Ali Bouchoucha, equipment preparation and supervision.

Ethics approval

Not applicable.

Consent to participate

Not applicable

Consent for publication

Not applicable.

Competing interests

The authors declare no competing interests

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1. Introduction

Natural fibres have been used to reinforce materials for over 3000 years. More recently, the natural fibre composites are receiving more attention during the last thirty years due to their ecologically friendly behavior [1]. Researchers focus their investigations on composite materials based on renewable resources with comparable properties to synthetic fibres for reducing petroleum consumption and pollution [2–6]. The natural fibres are lighter than synthetic fibres which revert as fuel reduction when this material is used by the automotive industry. Moreover, the natural fibres have low density, low cost, its availability, renewability, easy recyclability, low process energy, good thermal and acoustical insulation properties, acceptable specific strength and modulus, and reduced tool wear (non-abrasive) in machining operations [7–10]. Table 1 shows physical properties of some natural fibres used as reinforcement in composites materials [11–16].

The properties of the composite depend not only on the properties of the fibre but are also controlled by the properties of the matrix, interfacial adhesion between the fibre and matrix and the design of the hybrid system. In order to improve their properties researchers turned their focus on the study of effect on mechanical properties due to hybridization of natural–natural fibres i.e. Hybrid composites consist of an amalgamation of two or more fibres to form a single matrix. The possible combinations of hybrid composites include artificial–artificial, natural–artificial and natural–natural fibre types [14, 17]. In this paper, sisal, jute and flax fibres were used as reinforcements because they are abundantly found in nature.

There are few studies in the literature investigated and addressed the competitiveness, capabilities and suitability of hybrid natural fibres as reinforced fillers in polymeric matrices. Since, Schneider and Karmaker [18] developed composites using jute and kenaf fibre and polypropylene resins and they reported that jute fibre provides better mechanical properties than kenaf fibre. Recently, Venkateswaran et al. [19] reported that sisal/banana hybrid natural fibre composite specimens were prepared with different ratios by taking 0.4 volume fraction and tensile properties of these hybrid natural fibre composites are also examined using rule of mixtures (RoHM). In the next study, Venkateswaran et al. [20] studied the mechanical properties such as tensile strength, flexural strength, impact strength and water absorption rate of sisal and banana fibres reinforced epoxy composite materials. They have observed when the hybridization of the sisal fibre with banana/epoxy composites up to 50% by weight increases the mechanical properties and also decreases the water absorption properties. Boopalan et al. [21] studied the mechanical and thermal properties of jute and banana fibre

reinforced epoxy hybrid composites. Jute fibre was hybridized with banana fibre, in order to enhance the better mechanical properties of composites.

In other words, the adhesion between the reinforcing fibres and the matrix plays an important role in the final mechanical properties of the materials; when natural fibre used as reinforcement in composite materials, many problems occur at the interface due to incompatibility. Fibre–matrix interaction can be improved by surface or structural modification of the fibres using various processes such as alkali treatment, bleaching, acetylation and steaming. It is worth to mention that the chemical treatment of the fibres can either increase or decrease the strength of the fibres, and hence good understanding of what occurs structurally is required [22].

In this paper, we used two types of chemical treatment namely, alkaline (sodium hydroxide) NaOH and sodium bicarbonate NaHCO_3 . In alkaline treatment, fibres are immersed in NaOH solution for a given period of time. Ray et al. [23] and Mishra et al. [24] treated jute and sisal fibres with 5% aqueous NaOH solution for 2 h up to 72 h at room temperature. Similar treatments were attempted by Morrison et al. [25] to treat flax fibre. Asumani et al. [22] 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. Another, Rajesh and Prasad [26] studied short jute fibre/PLA composites with different concentrations of NaOH and H_2O_2 treatments on jute fibres. The effect of fibre loading and alkali concentrations used for fibre treatment on the mechanical properties of the composites were investigated. It was reported that the tensile properties of composites with treated fibre at higher fibre loadings were better than those of untreated fibre.

Although we mentioned many studies about composites, there are few studies in literature studied the mechanical properties of natural–natural hybrid fibre reinforced polymer composites, i.e. The main goals of the first part of this work are to prepare natural hybrid composite plates using response surface methodology (RSM) and the desirability function approach. Then, the ANOVA study involves the effects of input parameters, namely and coded; type of fibre (X_1), chemical treatment (X_2), volume fraction of fibre (X_3) and treatment duration (X_4) on mechanical properties of composites. In the second part of this work, the different tests realized to characterize the mechanical properties of the optimal natural hybrid composite reinforced with polyester or with epoxy resins which were experimentally analyzed.

2. Materials and methods

2.1. 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:

2.1.1. Jute fiber

Jute is a bast fibre whose scientific name is *Corchorus Capsularis* of Tiliaceae family. 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 [27]. 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 [28].

2.1.2 Sisal fiber

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 [29]. A sisal plant produces about 200-250 leaves and each leaf contains 1000 ± 1200 fibre bundles which are composed of 4 wt% fibre, 0.75 wt% cuticle, 8 wt% dry matter and 87.25 wt% water [30]. 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 [31].

2.1.3. Flax fiber

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 fibers all along its stem and contains 70% of cellulose. These cellulose based fibres have low density, good tensile strength, stiffness and high aspect ratio [32–33].

2.2. Fibre preparation methods

In order to improve the interfacial properties between the fibres (the sisal, jute and flax fibres) and the matrix, we were subjected to several surface treatments. The fibres were cut into 300 ± 2 mm long pieces, washed with distilled water and oven dried at 45 °C until obtaining a

constant weight. In this study, fibres were treated with sodium hydroxide NaOH and sodium bicarbonate NaHCO₃, with various times 4, 12 and 24 hrs.

The volume fraction of fibre (VF) is calculated by using the following relation [34–35].

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

VF is fiber volume fraction, W_f are the weight (g) of fibres (sisal, jute and flax) and W_m is the weight (g) of matrix, ρ_f and ρ_m are the density (g/cm³) of fibers and matrix, respectively. Also, the diameters of sisal, jute and flax fibres were evaluated by a Visual machine 250 tool makers microscope with $\times 4.5$ magnifications and 1 μ m resolution at three different random locations along the single fibre and the average value is taken, as shown in Fig. 1. The average diameters detected of sisal, jute and flax fibres were $240 \pm 40 \mu\text{m}$, $880 \pm 80 \mu\text{m}$ and $17 \pm 10 \mu\text{m}$, respectively.

Fig. 1. Optical microscopy image of a longitudinal sisal, jute and flax fibres

Table 1 shows the average values of physical and mechanical properties real of natural fibres (sisal, jute and flax) and resins (epoxy and polyester) used in this study. Ten identical specimens from each fibre and resin were tested and the average value is tabulated.

Table 1

Physical and mechanical properties of sisal, jute, flax fibres and resins used in this study.

Gage length (mm)	Density (g/cm ³)	Average ultimate tensile strength (MPa)	Average Young's modulus (GPa)	Average elongation at break (%)	Diameter (μm)
a) Jute fibre					
10	1.30	52.97	0.7200	13.86	800-960
30		31.95	0.2038	20.91	
50		29.02	0.3750	14.28	
b) Sisal fibre					
10	1.48	248.02	3.0060	13.80	220-260
30		212.36	8.8500	4.85	
50		242.28	9.0160	3.68	
c) Flax fibre					
10	1.50	216.93	14.8800	2.09	16-18
d) Resins					
Epoxy resin	—	3.6310	0.0985	9.1273	—
Polyester resin	—	17.3655	0.6983	3.6685	—

2.2.1. 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. Then, The fibres were washed several times with fresh water to remove any NaOH sticking on the fibre surface, neutralized with dilute acetic acid and after that, 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.

2.2.2. Treatment with NaHCO₃

Similarly, the second treatment method consisted of soaking the raw of the sisal, jute and flax fibres in 25, 25 and 10 wt% NaHCO₃ 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 NaHCO₃ traces sticking on the fibre surface. Then, fibres were neutralised with dilute acetic acid. After that, rinsed again with distilled water. Finally, the fibres were dried at room temperature until a constant weight was reached.

2.3. Mechanical properties

In order to evaluate the effect of the fibres' type, chemical treatment, volume fraction and treatment time on the mechanical properties (ultimate tensile strength and Young's modulus) the modified composites were measured using a Universal Testing Machine EZ20, equipped with a load cell of 20 KN. The clamps used during the tests have self-concentric alignment and are manually adjusted by mechanical springs. The tensile static tests were performed at a constant speed of 2 mm/min and the longitudinal strain was measured using an extensometer with 30 mm gauge length. All tests were conducted at a room temperature of 26°C and a relative humidity of approximately 30%. Tensile tests were conducted according to the ASTM

D 3822-01 specifications. In each case, five specimens for each composite were tested and the average value is tabulated (Table 3).

3. Statistical analysis

3.1. RSM experimental design

The response surface methodology (RSM), firstly induced by Box and Wilson [36–38], is a method for the accurate prediction of engineering system input–output relationships by taking a full consideration for parameter interaction. It has been widely applied in numerous manufacturing fields for the design, development and formulation of new products, as well as in the improvement of existing product designs. RSM is a collection of mathematical and statistical techniques that are useful for the modeling and analysis of problems in which a response of interest is influenced by several variables and the purpose is to optimize this response [37]. An important advantage of RSM is the reduced number of experimental trials required to evaluate multiple parameters and their interactions. The RSM offers several experimental designs depending on the number of design factors, such as Box–Behnken Design (BBD), Central Composite Design (CCD), etc. The BBD is selected to generate the design matrix since it needs fewer experiments when the number of factors is about 3–4.

A BBD was performed with four independent variables (X_1 , type of fibre; X_2 , chemical treatment; X_3 , volume fraction of fibre; and X_4 , treatment duration) at three levels. In addition, there are two qualitative variables (X_1 , type of fibre and X_2 , chemical treatment) and the other two are quantified (volume fraction of fibre and X_4 , treatment duration). The range of the independent variables, the levels of the independent variables and the results of the whole design consisting of 29 (calculated based on Eq. (2)) experimental points performed in random order were presented in Table 3; these conditions were based on the results of the preliminary experiments.

$$N = 2^n + 2n + N_c = 2^4 + 2 \times 4 + 5 = 29 \text{ runs} \quad (2)$$

where N is the total experimental runs, n is the number of variables and N_c replicate runs at the centre. For statistical calculation, each variable was coded at three levels: “–1”, “0” and “+1”, where “–1” is the lowest level, “0” is the central level and “+1” is the highest level. And each combination was repeated five times.

A complete description of the process behavior requires a quadratic or higher order polynomial model. Hence, the full quadratic models were established by using the method of least squares, which included all interaction which is termed to calculate the predicted

response. The quadratic model is usually sufficient for industrial applications. For n factors the full quadratic model is shown in Eq. (3):

$$Y = a_0 + \sum_{i=1}^k b_i X_i + \sum_{i,j}^k b_{ij} X_i X_j + \sum_{i=1}^k b_{ii} X_i^2 \quad (3)$$

where Y is the response; X_i and X_j are the variables (i and j range from 1 to k); a_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 adjusted- R^2 (R^2 adj), and predicted- R^2 (R^2 pred). The other important coefficient, R^2 , which is called coefficient of determination in the resulting ANOVA tables, is defined as the ratio of the explained variation to the total variation and is a measure of the fit degree. When R^2 approaches to unity, it indicates a good correlation between the experimental and the predicted values. The regression analyses, graphical analyses, analyses of variance (ANOVA) and analyses of response surfaces were carried out using Design Expert software V8 (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). The variables, units, symbol code and levels were shown in Table 2.

Table 2

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

N°	Independent variables	Symbol code, X_i	Units	Levels			Type
				-1	0	+1	
1	Fibres type	X_1	–	Flax	Jute	Sisal	Qualitative
2	Types of chemical treatment	X_2	–	NaHCO ₃	Raw	NaOH	Qualitative
3	Volume fraction of fibre	X_3	(%)	10	15	20	Quantitative
4	Treatment duration	X_4	hrs	4	12	24	Quantitative

Fig. 2. Experimental setup.

Table 3

Box-Behnken Design (BBD) with independent variables and response values.

N°	Coded values of independent variables				Actual values of independent variables				Response parameters			
									Epoxy resin		Polyester resin	
	X ₁	X ₂	X ₃	X ₄	Type of fibres	Types of chemical treatment	Volume fraction of fibre (%)	Treatment duration (hrs)	Ultimate tensile strength σ_u (MPa)	Young's modulus E (GPa)	Ultimate tensile strength σ_u (MPa)	Young's modulus E (GPa)
1	0	0	1	1	Jute	Raw	20	24	59.495	2.1294	54.679	1.9618
2	0	0	0	0	Jute	Raw	15	12	36.923	1.5264	30.901	1.4993
3	0	1	0	1	Jute	NaOH	15	24	41.082	1.5296	34.747	1.5021
4	0	0	0	0	Jute	Raw	15	12	36.923	1.5264	30.901	1.4993
5	0	0	-1	1	Jute	Raw	10	24	25.199	1.5610	22.618	1.4621
6	-1	0	-1	0	Flax	Raw	10	12	17.076	1.0054	13.778	0.7593
7	0	-1	-1	0	Jute	NaHCO ₃	10	12	28.366	1.1244	25.947	1.6540
8	0	1	1	0	Jute	NaOH	20	12	67.413	2.0876	57.601	1.5742
9	0	0	0	0	Jute	Raw	15	12	36.923	1.5264	30.901	1.4993
10	-1	0	0	-1	Flax	Raw	15	4	32.942	1.3088	24.234	1.1124
11	0	0	0	0	Jute	Raw	15	12	36.923	1.5264	30.901	1.4993
12	-1	0	0	1	Flax	Raw	15	24	32.942	1.1124	24.234	1.3088
13	1	0	1	0	Sisal	Raw	20	12	43.840	1.2686	38.720	1.7574
14	1	-1	0	0	Sisal	NaHCO ₃	15	12	37.024	1.4630	30.083	1.6765
15	0	0	0	0	Jute	Raw	15	12	36.923	1.5264	30.901	1.4993
16	-1	-1	0	0	Flax	NaHCO ₃	15	12	39.717	1.5556	31.649	1.4565
17	-1	0	1	0	Flax	Raw	20	12	59.844	1.9096	56.638	1.8817
18	1	0	0	-1	Sisal	Raw	15	4	34.765	0.9320	23.527	1.5393
19	1	0	0	1	Sisal	Raw	15	24	34.765	0.9320	23.527	1.5393
20	0	-1	0	-1	Jute	NaHCO ₃	15	4	46.715	1.7592	37.903	1.7309
21	0	0	-1	-1	Jute	Raw	10	4	25.199	1.5610	22.618	1.4621
22	1	1	0	0	Sisal	NaOH	15	12	22.031	1.3172	12.224	1.3624
23	0	-1	1	0	Jute	NaHCO ₃	20	12	59.072	2.0088	62.454	1.9810
24	-1	1	0	0	Flax	NaOH	15	12	36.421	1.4885	28.502	1.3450
25	0	1	-1	0	Jute	NaOH	10	12	45.424	1.5518	16.992	1.6726
26	0	-1	0	1	Jute	NaHCO ₃	15	24	53.340	2.0876	42.794	1.7191
27	0	1	0	-1	Jute	NaOH	15	4	55.576	1.5458	40.837	1.6420
28	1	0	-1	0	Sisal	Raw	10	12	26.633	0.9320	16.338	1.3659
29	0	0	1	-1	Jute	Raw	20	4	59.495	2.1294	54.679	1.9618

3.2. Regression equations

In the empirical approach, prediction of ultimate tensile strength (σ_u) and Young's modulus (E) were done based on the regression analysis of the experimental data. A statistical model gives relationship between response variables (ultimate tensile strength and Young's modulus) and four independent parameters which are; type of fibres (X_1), type of chemical treatment (X_2), volume fraction of fibre (X_3) and treatment duration (X_4) for both resins matrix used (epoxy and polyester). Based on the RSM method using the quadratic model of Eq. (2), the following equations are the final empirical models in terms of coded factors for:

❖ Epoxy resin

$$\begin{aligned}\sigma_u = & 36.92 - 1.66X_1 + 0.3094X_2 + 15.11X_3 - 0.6557X_4 - 7.23X_1 \times X_1 \\ & + 7.05X_2 \times X_2 + 5.15X_3 \times X_3 + 3.21X_4 \times X_4\end{aligned}\quad (4)$$

$$\begin{aligned}E = & 1.53 - 0.128X_1 - 0.0398X_2 + 0.3162X_3 + 0.00965X_4 - 0.3725X_1 \times X_1 \\ & + 0.164X_2 \times X_2 + 0.133X_3 \times X_3 + 0.0478X_4 \times X_4\end{aligned}\quad (5)$$

❖ Polyester resin

$$\begin{aligned}\sigma_u = & 30.90 - 2.88X_1 - 3.33X_2 + 17.21X_3 - 0.0999X_4 - 8.24X_1 \times X_1 \\ & + 4.04X_2 \times X_2 + 6.71X_3 \times X_3 + 2.13X_4 \times X_4\end{aligned}\quad (6)$$

$$\begin{aligned}E = & 1.5 + 0.1148X_1 - 0.0933X_2 + 0.2285X_3 + 0.0037X_4 - 0.1711X_1 \times X_1 \\ & + 0.1054X_2 \times X_2 + 0.1276X_3 \times X_3 + 0.0586X_4 \times X_4\end{aligned}\quad (7)$$

Fig. 3 presents a comparison between predicted and measured values of the ultimate tensile strength (σ_u) and Young's modulus (E) of different composites corresponding to different combinations of independent variables were presented in Table 3. The results of the comparison prove that predicted values of the ultimate tensile strength and Young's modulus are very close to those experimentally recorded. Similarly, the Figure 4a–4b shows the comparison between the values of the response parameters (σ_u and E) of all fibre reinforced composites corresponding to different combinations of independent variables. The fibre reinforced epoxy composite provides higher values than the fibre reinforced polyester composites in term ultimate tensile strength.

Fig. 3. Comparison between measured and predicted values for ultimate tensile strength and Young's modulus of different composites (natural fibres/epoxy and natural fibres/polyester).

Fig. 4. Comparison between different composites (natural fibres/epoxy and natural fibres/polyester) for; a) ultimate tensile strength; b) Young's modulus.

3.3. Analysis of variance ANOVA

The ANOVA of the data with the ultimate tensile strength (σ_u) and Young's modulus (E), with the objective of analysing the influence 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 total variance of the results were carried out. The table of ANOVA shows the degrees of freedom (DF), sum of squares (SC), mean squares (MS), F-values (F) and probability (P). The last column of table shows the factor contribution percentage (Cont. %) on the total variation for each factor and different products. The statistical significance of each coefficient was checked by P-values and F-values. A low P-value (≤ 0.05) indicates statistical significance for the source on the corresponding response (i.e., $\alpha = 0.05$, or 95% confidence level), this indicates that the obtained models are considered to be statistically significant, which is desirable; as it demonstrates that the terms in the model have a significant effect on the response, and the higher F-values for each coefficient suggest more significance of that term in the model [39].

As can be seen from Table 4, the regression model for ultimate tensile strength was found to be highly significant from the Fisher's test which had a high F-values (12.27 and 35.73) with very low probability (both $P < 0.0001$ and both p-values < 0.05) according to, epoxy and polyester resins, respectively. Judging by the F-values of the items in the regression models, the order in which the independent variables influenced the ultimate tensile strength degradation efficiency was: the volume fraction of fibre (X_3) which is the most important factor affecting the tensile strength. Its contribution is (74.33% for σ_{epoxy} and 82.46% for $\sigma_{\text{polyester}}$). Similar results were reported by Ben Brahim [40], when they study the effect of volume fraction of fibers on the tensile properties (longitudinal modulus and the longitudinal stress) of unidirectional alfa-polyester composites. The type of fibres (X_1), types of chemical treatment (X_2) and treatment duration (X_4) have a very weak significance effect on the ultimate tensile strength when compared volume fraction of fibre. The quadratic coefficients ($X_1 \times X_1$, $X_2 \times X_2$ and $X_3 \times X_3$) were found to be significant ($P < 0.05$). The increasing of volume fraction of fibre ($X_3 \times X_3$) had a negative effect on the ultimate tensile strength. Our finding is against the report by Idicula et al. [41]; i.e. they have found that the quality of the composites has been increased with the boost of volume fraction of the sisal and banana fibers. The permissible fibre volume fraction was mentioned as 40% beyond that the proper stress transfer from fibres to matrix was not occurred due to fibre agglomeration.

Moreover, the determination coefficients of the models (R^2) have very high values (0.8308 and 0.9346), which mean that 83.08 and 93.46% of the total variation is explained by this quadratic regression model. Besides, the values of the adjusted determination coefficient (R^2_{Adjusted}) are 0.7631 and 0.9084, for epoxy and polyester matrix, respectively; indicating that the predicted and experimental ultimate tensile strength efficiencies have a high degree of accordance.

Similarly, results from ANOVA (Table 5) for the quadratic model showed that the polynomial models were highly statistically significant, as suggested by the high model F-values (10.15 for E_{epoxy} and 7.37 for $E_{\text{polyester}}$) and low P-values (both < 0.0001 for E_{epoxy} and $E_{\text{polyester}}$, both p-values < 0.05). The F and P values are used to check the significance of each coefficient. The lower P-value and higher F-value indicated the more significance of corresponding coefficient. The high values of determination coefficients ($R^2_{\text{epoxy}} = 0.8024$ and $R^2_{\text{polyester}} = 0.7467$), and adjusted determination coefficients ($\text{adj-}R^2_{\text{epoxy}} = 0.7233$ and $\text{adj-}R^2_{\text{polyester}} = 0.6454$) showed a high degree of correlation between the experimental and the predicted values of Young's modulus. In addition, according to Table 5, it can be apparently the volume fraction of fibre (X_3) which is the most important factor affecting on Young's modulus (E). Its

contribution is (Cont. = 45.78 % and Cont. = 48.99 %) for epoxy and polyester resins, respectively. The next factor influencing E is the type of fibres (X_1) with (Cont. = 7.50 % and Cont. = 12.35 %), there are lots of work that has been done to study the effect of fiber loading on the mechanical properties [42–44]. Whereas, the chemical treatment (X_2) with (Cont. = 0.72 % and Cont. = 8.17 %) was found to be less significant on Young's modulus of epoxy and polyester resins, respectively. Similarly, the treatment duration (X_4) has a very weak effect on the Young's modulus due to higher P-value ($P > 0.05$). Its contribution is (Cont. = 0.04 % and Cont. = 0.02 %) for epoxy and polyester resins, respectively.

Table 4

Analysis of variance for ultimate tensile strength (σ_u).

Source	SS	DF	MS	F-value	P-value	Cont. %	Remarks
(a) Epoxy resin							
Model	3822.65	8	477.83	12.27	< 0.0001		Significant
X_1	32.95	1	32.95	0.8462	0.3686	0.90	Insignificant
X_2	1.15	1	1.15	0.0295	0.8653	0.03	–
X_3	2737.99	1	2737.99	70.32	< 0.0001	74.46	Significant
X_4	5.16	1	5.16	0.1325	0.7197	0.14	Insignificant
$X_1 \times X_1$	338.8	1	338.80	8.70	0.0079	9.21	Significant
$X_2 \times X_2$	321.98	1	321.98	8.27	0.0093	8.76	–
$X_3 \times X_3$	172.36	1	172.36	4.43	0.0482	4.69	–
$X_4 \times X_4$	66.93	1	66.93	1.72	0.2047	1.82	Insignificant
Error	778.75	20	38.94				
Total	4601.4	28				100	
(b) Polyester resin							
Model	4835.41	8	604.43	35.73	< 0.0001		Significant
X_1	99.86	1	99.86	5.90	0.0247	2.15	–
X_2	132.85	1	132.85	7.85	0.0110	2.85	–
X_3	3552.83	1	3552.83	210	< 0.0001	76.33	–
X_4	0.1198	1	0.1198	0.0071	0.9338	0.01	Insignificant
$X_1 \times X_1$	440.47	1	440.47	26.04	< 0.0001	9.47	Significant
$X_2 \times X_2$	106.10	1	106.10	6.27	0.0210	2.28	–
$X_3 \times X_3$	292.09	1	292.09	17.26	0.0005	6.28	–
$X_4 \times X_4$	29.35	1	29.35	1.74	0.2027	0.63	Insignificant
Error	338.36	20	16.92				
Total	5173.77	28				100	

Table 5Analysis of variance for Young's modulus (E).

Source	SS	DF	MS	F-value	P-value	Cont. %	Remarks
(a) Epoxy resin							
Model	2.90	8	0.3629	10.15	< 0.0001		Significant
X_1	0.1965	1	0.1965	5.49	0.0295	7.50	–
X_2	0.019	1	0.019	0.5327	0.4739	0.72	Insignificant
X_3	1.20	1	1.2	33.61	< 0.0001	45.78	Significant
X_4	0.0011	1	0.0011	0.0313	0.8615	0.04	Insignificant
$X_1 \times X_1$	0.9005	1	0.9005	25.18	< 0.0001	34.35	Significant
$X_2 \times X_2$	0.1748	1	0.1748	4.89	0.0388	6.67	–
$X_3 \times X_3$	0.1146	1	0.1146	3.2	0.0886	4.37	Insignificant
$X_4 \times X_4$	0.0148	1	0.0148	0.4144	0.5271	0.56	–
Error	0.7151	20	0.0358				
Total	3.62	28				100	
(b) Polyester resin							
Model	1.36	8	0.1695	7.37	0.0001		Significant
X_1	0.158	1	0.158	6.87	0.0163	12.35	–
X_2	0.1045	1	0.1045	4.54	0.0456	8.17	–
X_3	0.6265	1	0.6265	27.25	< 0.0001	48.99	–
X_4	0.0002	1	0.0002	0.0072	0.933	0.02	Insignificant
$X_1 \times X_1$	0.1899	1	0.1899	8.26	0.0094	14.85	Significant
$X_2 \times X_2$	0.072	1	0.072	3.13	0.092	5.63	Insignificant
$X_3 \times X_3$	0.1056	1	0.1056	4.59	0.0446	8.26	Significant
$X_4 \times X_4$	0.0222	1	0.0222	0.9672	0.3371	1.74	Insignificant
Error	0.4599	20	0.023				
Total	1.82	28				100	

3.4. Perturbation plots

The main effects of the single factor (X_1 , X_2 , X_3 and X_4) and the perturbation plots for both response parameters (σ_u and E) are illustrated in Fig. 4. They confirm the ANOVA results demonstrated in Tables 4 and 5. The x -axis in the graphs is the low and high level of the design factor and y -axis is the mean value of the response parameter at a specific design factor level. Fig. 4a illustrates the perturbation plot of σ_u for both resins. According to this plot, we understand that the forth factors X_1 , X_2 , X_3 and X_4 have a positive quadratic influence on σ_u and E . Therefore, the volume fraction (X_3) seemed to have a major role for improving the mechanical properties (σ_u and E) of natural composites while the treatment duration (X_4) played the minor. In addition, the factors (X_1 , X_2 and X_3) at different levels have different influence on the σ_u . The Same way, Fig. 4b represents the perturbation plot for E . From this graph, we can figure out that X_1 , X_2 and X_3 have significant impacts on the E , while X_4 has less effect. At the same time, all the four processing parameters have positive effects on the E .

Fig. 5. Main affects graphs for σ_u and E versus X_1 , X_2 , X_3 and X_4 of the both resin matrices (epoxy and polyester)

3.5. Effect of operating parameters on grafting

In order to present the relationship between independent variables (type of fibres (X_1), types of chemical treatment (X_2) and volume fraction of fibre (X_3)) on response parameters (ultimate tensile strength (σ_u) and Young's modulus E), the response surface plots of the models were generated and illustrated on the three-dimensional space by varying two variables within the experimental range for both resins matrix tested (epoxy and polyester), while the treatment duration (X_4) is kept at the middle level (12 hrs) and when using three different natural fibres, namely and coded; flax fibre (-1), jute fibre (0) and sisal fibre (+1). From Figures 4 and 5, we could observe the remarkable increases of both response parameters (σ_u and E) with increases in volume fraction of fibre and chemical treatment in the composites arranged in specific order which is jute, sisal and flax. In addition, these two variables exhibited significant positive quadratic effects in response of response parameters (Eqs. 4 and 5). Moreover, the surface modifications could remove surface impurities and increase surface roughness. These modifications increase to bonding of the fibre with the resins matrix there

by improving the fibre-matrix interaction, subsequently, significantly increased the ultimate tensile strength and Young's modulus of the composites. Several authors [45–48], have focussed the studies on the treatment of fibres to improve the bonding with resin matrix. For example, Zou et al. [49] studied the effects of alkali and silane surface treatments on sisal fibre properties and they observed that the surface treatments facilitated good adhesion between fibers and thermoplastic matrix resulting in composites with improved mechanical properties.

Generally, the plots indicate that the highest ultimate tensile strength and Young's modulus can be achieved at the volume fraction of fibre is higher (level +1) with NaHCO_3 chemical treatment (level -1), when using jute fibre (level 0) and when reinforcing composites with epoxy matrix. Also, for all types of fibres a quadratic increase in the response parameters with increasing the volume fraction of fibre were observed. This is due to the high fibres tensile modulus compared to resins matrix.

a) Epoxy composites

b) Polyester composites

Fig. 6. Comparison of response surface for ultimate tensile strength versus X_1 , X_2 , and X_3 at the middle level of X_4 ; a) epoxy matrix and b) polyester matrix.

a) Epoxy composites

b) Polyester composites

Fig. 7. Comparison of response surface for Young's modulus versus X_1 , X_2 , and X_3 at the middle level of X_4 ; a) epoxy matrix and b) polyester matrix.

3.6. Multiple response optimizations

In this section, the statistical method of RSM was used to calculate desirability for the optimization analysis. 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. The Derringer function or desirability (D) function is the most important and most currently used multi-criteria methodology in the optimization of analytical procedures [39]. The global D value was analyzed basing on individual desirability's. 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 independent variables and responses are reported in Table 7 in order of decreasing desirability level, the optimal process factors were found to be as follows: the type of fibres (X_1) of [-0.28 and -0.33], the type of chemical treatment (X_2) of -1, the volume fraction of fibre (X_3) of 1 and the treatment duration (X_4) of [-0.97 and -1] for epoxy composites. Similarly, when using the polyester matrix; the type of fibres (X_1) of -0.26, the type of chemical treatment (X_2) of -1, the volume fraction of fibre (X_3) of ~1 and the treatment duration (X_4) of [-0.94 and -0.93].

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	
Ultimate tensile strength σ (MPa)	Maximize	17.076	12.224	67.413	62.454
Young's modulus E (GPa)	Maximize	0.9320	0.7593	2.1294	1.9810

Table 7

Response optimization for ultimate tensile strength and Young's modulus.

Test N°	Coded factors				Performance measures		Desirability D	Remarks
	X ₁	X ₂	X ₃	X ₄	Ultimate tensile strength σ (MPa)	Young's modulus E (GPa)		
Epoxy resin								
1	-0.28	-1.00	1.00	-0.97	67.4484	2.2211	1	Selected
2	-0.33	-0.99	1.00	-1.00	67.4236	2.2165	1	
3	-0.47	-1.00	1.00	-1.00	66.9613	2.1954	0.9966	
Polyester resin								
1	-0.26	-0.97	0.99	-0.94	63.8168	2.0487	1	Selected
2	-0.26	-0.95	0.99	-0.93	63.5924	2.0431	1	
3	-0.40	-0.54	1.00	-1.00	59.8618	1.9188	0.9488	

4. Validation of experimental results

4.1. Creation of the hybrid composites

Once the optimal level of the process parameters is selected, the final step is to predict and verify the improvement of the performance characteristics (ultimate tensile strength and Young's modulus) using the optimal levels of the process parameters presented in terms of coded factors in section 3.4. To make the confirmation tests, we converted the coded values presented in Table 8 to the actual values. The rule of mixture fibre has been presented in Fig. 5.

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 (%)	Type of chemical treatment	Volume fraction of fibre (%)	Treatment duration (hrs)
Epoxy resin								
1	-0.28	-1	+1	-0.97	72% of jute and 28% of flax	NaHCO ₃	20	4h14
2	-0.33			-1	67% of jute and 33% of flax			4h00
Polyester resin								
1	-0.26	-1	+0.99	-0.94	74% of jute and 26% of flax	NaHCO ₃	19.80	4h28
2				-0.93				4h33

Fig. 8. Rule of hybrid mixture.

4.2. Hybrid composites manufacturing methods

In this study the hybrid composites are fabricated by hand-lay up method. Composites were made using a wood mould measuring: 300 × 150 × 5 mm length, width and depth, respectively. Four beadings a glass plate were used to maintain a 5 mm thickness all around the mould plates. Initially, the moulds are cleaned, dried and silicon spray was used as releasing agent. Secondly, untreated jute, and flax fibres were, weighed, bagged and formulated according to the various fibre contents indicated in Table 8. In this section, fibres were treated with sodium bicarbonate NaHCO_3 , with various times 4 to 4.33 hrs (see Table 9). Then, the fibres were mixed with resin matrices (epoxy or polyester) for the fabrication of composite. Then, uniform pressure of 5 Pa was applied over the mould plates (purpose of this is to maintain uniform thickness and to avoid void formation during curing) for 1 h at room temperature curing. Finally, the composites were cut and shaped in rectangular form (250 × 25 × 5 mm) according to ASTM standard by using a diamond saw blade. Four identical samples were prepared for each volume fraction of fibres and all the specimens were tested at a strain rate of 2 mm/min using an electronic tensometer.

Table 9
Designations and composition of hybrid composites.

n°	Code	Composites	Volume fraction		Resin content (wt %)	Treatment duration (hrs)
			Jute fibre content (wt %)	Flax fibre content (wt %)		
Epoxy resin						
1	HCE1	Hybrid composite	14.40	05.60	80	4h14
2	HCE2	Hybrid composite	13.40	06.60	80	4h00
Polyester resin						
1	HCP1	Hybrid composite	14.65	05.15	80.20	4h28
2	HCP2	Hybrid composite	14.65	05.15	80.20	4h33

4.3. Confirmation test results

The four different hybrid composite specimens: HCE1, HCE2, HCP1 and HCP2 (see Table 8) are tested in the universal testing machine to find the tensile properties. Each test was repeated four times and the results are displayed as strength against strain (Figure 3a–d). Also, Figure 3a–c. shows the tensile testing data of various hybrid composites with different fibres content reinforced with epoxy or polyester resins are linear and follows Hooke's law. Then, the Young's modulus is calculated by taking the corresponding values of stress and strain from the linear portion of the graph before the first damage zone. The averages of tensile properties (ultimate tensile strength and Young's modulus) are summarized in the Table 1.

Fig. 9. Stress vs strain curve for tensile test of various hybrid composites at highest volume fraction of fibre (20%).

Table 10

Result of tensile test of different composites.

n°	Sample code	Experimental		Error (%)	
		Ultimate tensile strength σ_u (MPa)	Young's modulus E (GPa)	σ_u (MPa)	E (GPa)
a) Epoxy resin					
1	HCE1	64.7100	2.1900	4.06	4.10
2	HCE2	60.2600	2.1800	10.62	1.65
b) Polyester resin					
1	HCP1	62.6200	1.8122	1.88	11.54
2	HCP2	62.6800	1.9400	1.43	5.05

The average values of the ultimate tensile strength and Young's modulus of different composite formulations are plotted in Figure 10 for better comparison. It is observed that the hybrid composite HCE1 had the highest tensile properties (ultimate tensile strength and Young's modulus) especially when reinforced with epoxy. The lowest tensile properties were produced by jute composite when reinforced with epoxy. The modulus value for the hybrid composite HCP2 fell within these two values.

Fig. 10. Tensile properties of various composites for both resin matrices: a) ultimate tensile strength and b) Young's modulus.

5. Conclusion

In this paper, the application of RSM for the tensile quasi-static loading of biocomposites was presented. Mathematical models of ultimate tensile strength and Young's modulus evolutions according to the influence of independent variables are tested through ANOVA and found to be adequate at 95% confidence interval. The following conclusions are drawn from the present investigation:

- 1) The ANOVA shows that:
 - (a) The volume fraction of fibre (concentration of the fibre in the composite) plays a significant role on the mechanical properties (ultimate tensile strength and Young's modulus) of composites with contributions between (74.46 and 45.78)% for epoxy matrix and (76.33 and 48.99)% for polyester matrix, respectively. It is followed by the type of fibres, then the chemical treatment. After that, the treatment duration comes in forth position.
 - (b) The mean ultimate tensile strength and the mean Young's modulus of jute fibre composite at in range of volume fraction of fibre in the present study is much higher than those of flax and sisal composites is this for both resins tested. It is also concluded that the mechanical properties of composite reinforced with an epoxy matrix provides higher properties than the composite reinforced with polyester matrix.
 - (c) Comparison of experimental and predicted values of ultimate tensile strength and Young's modulus shows that good agreement has been achieved between them. Therefore, the developed model can be recommended to be used for predicting ultimate tensile strength and Young's modulus.

- (d) The optimization process is done by maximizing both ultimate tensile strength and Young's modulus. It depends on the following factor combinations: the type of fibres (X_1) of [-0.28 and -0.33], the chemical treatment (X_2) of -1, the volume fraction of fibre (X_3) of 1 and the treatment duration (X_4) of [-0.97 and -1] for epoxy matrix. Similarly, when used the polyester matrix: the type of fibres (X_1) of -0.26, the chemical treatment (X_2) of -1, the volume fraction of fibre (X_3) of ~1 and the treatment duration (X_4) of [-0.94 and -0.93].
- (e) The new hybrid material which is obtained by the optimisation of input parameters confirms the predicted results.
- 2) The mechanical characteristics (tensile strength and the Young's modulus) of hybrid materials are better than the jute, flax and pure sisal characteristics by (8.96%, 3.86%, 9.90% and 2.92%, 10.04%, 21.46%) respectively for epoxy resin and by (0.27%, 5.68%, 14.9% and 2.02%, 6.03%, 5.36%) respectively for polyester resin.
- 3) In addition, the hybrid composite specimens HCE1 when reinforced with an epoxy matrix possess good tensile strength and can withstand the strength up to 64.71 MPa; hence provide higher Young's modulus of 2.19 GPa.

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Figures

Figure 1

Optical microscopy image of a longitudinal sisal, jute and flax fibres

Figure 2

Experimental setup.

Figure 3

Comparison between measured and predicted values for ultimate tensile strength and Young's modulus of different composites (natural fibres/epoxy and natural fibres/polyester).

Figure 4

Comparison between different composites (natural fibres/epoxy and natural fibres/polyester) for; a) ultimate tensile strength; b) Young's modulus.

(a)

(b)

Figure 5

Main affects graphs for σ_u and E versus X_1 , X_2 , X_3 and X_4 of the both resin matrices (epoxy and polyester)

Figure 6

Comparison of response surface for ultimate tensile strength versus X_1 , X_2 , and X_3 at the middle level of X_4 ; a) epoxy matrix and b) polyester matrix.

Figure 7

Comparison of response surface for Young's modulus versus X_1 , X_2 , and X_3 at the middle level of X_4 ; a) epoxy matrix and b) polyester matrix.

Figure 8

Rule of hybrid mixture.

Figure 9

Stress vs strain curve for tensile test of various hybrid composites at highest volume fraction of fibre (20%).

Figure 10

Tensile properties of various composites for both resin matrices: a) ultimate tensile strength and b) Young's modulus.