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ORIGINAL ARTICLE

Tensile and flexural properties of polymer composites reinforced by flax, jute and sisal fibres

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Abstract

Natural fibre composites are a class of materials that are currently replacing the synthetic fibre composites for practical applications. This paper deals with the fabrication and investigation of hybrid natural/natural fibre composites obtained by a new technique based on statistical analysis of variance (ANOVA) by using the properties of the individual fibre composites. The influences of type of fibres, such as flax, jute and sisal, the type of chemical treatment and the volume fraction of fibre on the mechanical properties such as tensile strength, tensile modulus, flexural strength and flexural modulus of the composites, were evaluated. Mathematical models for mechanical properties were developed using the response surface methodology (RSM). Statistical analysis of the results showed that the mechanical properties are influenced principally by the volume fraction of fibre, then the type of fibres. On the opposite side, the type of chemical treatment has a very weak significance effect. Then, the best mechanical proprieties of composites were achieved at the highest volume fraction of fibre and when used the sodium bicarbonate $NaHCO_3$ for treated fibres. Finally, the developed hybrid composite exhibited superior properties compared to the previous composites based on individual fibre composites when the fibre content is at 80 wt% of jute and 20 wt% of flax.

Keywords Natural fibres · Hybrid composites · RSM · Statistical analyses · Mechanical testing

Nomenclature

ANOVA	Analysis of variance
ASTM	American Standards of Technical Material
B	Width of the beam, mm
BBD	Box-Behnken design
DF	Degrees of freedom
E_f	Mean flexural modulus, GPa
E_t	Mean tensile modulus, MPa
L	Support span, mm
M	Slope, N/mm
MS	Mean squares
P_f	Maximum load, N
R^2	Coefficient of determination
RSM	Response surface methodology
SC	Sum of squares
VF	Volume fraction of fibre, cm^3
W_m	Weight of matrix, g
W_f	Weight of fibre, g
X_1	Type of fibres
X_2	Type of chemical treatment
X_3	Volume fraction of fibre, wt.%
σ_f	Mean flexural strength, MPa
σ_t	Mean tensile strength, GPa
ρ_f	Density of fibre, g/cm^3
ρ_m	Density of matrix, g/cm^3

1 Introduction

Natural fibre composites are proposed to replace synthetic materials in many engineering applications due to several advantages such as renewability, less abrasiveness to equipment and biodegradability, and also they are low density, cheaper and acceptable specific properties [1–3]. The natural fibres can be classified as substances produced by plants or vegetable, animals and minerals. Plant fibres exist in many varieties, such as kenaf, jute, alfa, bamboo, hemp, banana and flax

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extracted from the stem of the plant. Sisal and abaca were extracted from leaves. Coir and cotton were extracted from the fruit of the plant. Agopyan [4] listed 18 types of vegetable fibres potentially useful for civil construction. Table 1 lists the mechanical properties of some natural fibres used as reinforcement in composites materials [5–12].

The properties of natural fibre-reinforced composites depend on a number of parameters such as volume fraction of the fibres, fibre aspect ratio, orientation, fibre type and controlled by the properties of the matrix and interfacial adhesion between the fibre and matrix [13].

Research studies have been conducted on the mechanical properties of natural fibre-based composite materials. For example, Rao et al. [6] studied the mechanical properties such as tensile strength and flexural strength of vakka, sisal, bamboo and banana fibre-reinforced polyester resin matrix. Similarly, Prasad and Rao [7] studied the mechanical behaviours such as tensile and flexural tests of jowar, sisal and bamboo fibre-reinforced polyester composites. Another, El-Shekeil et al. [14] studied the mechanical properties (i.e. tensile, flexural, impact, hardness and abrasion resistance) and thermal of Kenaf bast fibre-reinforced thermoplastic polyurethane (TPU) composites. The influences of moisture absorption of bamboo/vinylester composites [15], the effect of oil palm fibre volume fraction of oil palm/polyester composites [16]and the influence of the fibre orientation and the volume fraction of alfa fibre/polyester composites with a volume fraction in fibres of 45% have been studied by Ben Brahim and Cheikh [12]. Recently, García del Pino et al. [17] investigated the effect of fibre weight fraction, treatment concentration, treatment time and nanoparticle content on tensile strength and flexural strength using three-level factorial designs (Taguchi matrix L9) during tensile and three-point bending tests. Results show that the mechanical proprieties are mainly influenced by fibre weight fraction. Another, Belaadi et al. [18] have determined the quasi static tensile mechanical properties of more than 150 of natural sisal yarns. The authors used five different gauge lengths GL = 50, 100, 150, 200 and 300 mm to determine the influence of this parameter (GL) on the mechanical performance of the yarns. During the tests, the researchers noticed that the result obtained contain a large dispersion. To remedy this behaviour, the results were studied and analysed by probabilistic and statistical tools such as the Weibull distribution with two and three parameters using different probability and estimation index and also one-way analysis of variance (ANOVA).

The quality of the interface between the fibre and the matrix plays a vital role in the mechanical properties of the composite. Surface modification of fibres is often required to achieve maximum compatibility fibre-matrix interfaces [19]. In this case, several researchers employed different percentage of alkali solution (5%, 7%, 10% and 15% in weight) and immersion times (1, 3 and 24 h) to treated natural fibres [20–22]. In particular, Vilay et al. [22] compared the effect of NaOH and acetic acid on the flexural properties of the bagasse–polyester composite. NaOH-treated fibre composites showed better flexural strength and modulus (increase by about 11% and 20%, respectively) compared to untreated fibre composites. In another recent study presented by Bedjaoui et al. [23], the effects of concentration of sodium bicarbonate ($NaHCO_3$) (4–8 vol.%) for a period of 80 h at room temperature on the tensile properties of flax/polyester composites have been studied. The results obtained were analysed by applying probabilistic and statistical approaches such as the Weibull distribution. Asumani et al. [24] studied the alkali and silane-treated kenaf fibre-reinforced polypropylene

Table 1 Mechanical properties of some natural fibres and epoxy resin used in this study

Fibres	Mechanical properties						
	Density (g/cm^3)	Flexural strength (MPa)	Flexural modulus (GPa)	Tensile strength (MPa)	Young's modulus (GPa)	Diameter (μm)	Refs.
Banana	1.35	–	2–5	550–560	20	120–126	[5–12]
Cotton	1.51	–	–	287–800	5.5–12.6	–	[5–12]
Ramie	1.5	–	–	938–220	128–44	20–80	[10, 12]
Hemp	1.48	–	3–5	550–900	70	–	[10–12]
Jute*	1.30	–	–	52.97	0.720	880 ± 80	This work
Flax*	1.50	–	–	216.93	14.880	17 ± 10	This work
Sisal*	1.48	–	–	248.02	3.006	240 ± 40	This work
Epoxy resin	–	19.07	0.35	3.63	0.098	–	This work

*Gage length is the 10 mm

composites. It has been noted that the tensile strength and modulus increased significantly by 25% and 11%, respectively, after treatment with 5% alkali. Another study, Cao et al. [25], investigated the effect of alkali (NaOH) on the mechanical properties of bagasse fibre-reinforced polyester composites. NaOH solutions of 1, 3 and 5% concentrations were used. Superior properties were obtained for the composites made from 1% NaOH-treated bagasse fibres. The authors observed a 13% improvement in tensile strength, 14% in flexural strength and 30% in impact resistance, respectively, due to chemical modification.

The usage of hybrid polymer composites is increasing day to day because of their outstanding properties. There is a great potentiality of hybrid composite materials in the field of engineering due to low cost, strength-to-weight ratio and ease of manufacturing. Combining two or more fibre in a single matrix could offer a possibility of achieving combined properties such as increasing fatigue life, stiffness, ductility and strength compared to single fibre-reinforced composites. The composites obtained by using two or more different kinds of fibres in a single matrix are termed as hybrid composites [26–28].

Hybrid composites have long taken the attention of many researchers as a way to enhance the mechanical properties of composites. However, hybrid composites using natural fibres have been studied rarely and the most published reports limited to the hybrid composite consist of one kind of natural fibre and one kind of non-natural fibre. For example, using of natural–natural fibres has been reported (references in Table 2).

Boopalan et al. [33] 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. Recently, Venkateswaran et al. [34] reported that sisal/banana hybrid natural fibre composite specimens were prepared with different rations 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. [35] studied the mechanical properties such as tensile strength, flexural strength, impact strength and water absorption rate of sisal and banana fibre-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.

In this study, three different natural fibres (flax, jute and sisal) were used to reinforce epoxy resin, using in each case a fibre content (or, volume fraction of fibre) ranging from 10 to 20 wt%. The effects of input variables such as type of fibres (X_1), types of chemical treatment (X_2) and volume fraction of fibre (X_3) on the mechanical properties of composite materials were investigated. The properties such as tensile and flexural tests are presented in detail in uni-factorial and multi-factorial form. The main goals of this work are to prepare natural hybrid composite plates using response surface methodology (RSM) and the desirability function approach by using the properties of the individual fibre composites. Then, the different tests realized to characterize the mechanical properties of the optimal natural hybrid composite reinforced epoxy resins, which were experimentally analysed.

2 Materials and experimental method

2.1 Materials

In this present investigation flax (*Linum usitatissimum*), jute (*Corchorus capsularis* of Tiliaceae) and sisal (*Agave sisalana*) fibres are used for fabricating the composite specimens. All of the materials employed in this work were obtained from commercial sources and used as received. The definitions of fibres are discussed in detail as follows:

2.1.1 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 is 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

Table 2 Some natural fibre-based hybrid composites studied by researchers

S no.	Natural fibre 1	Natural fibre 2	Natural fibre 3	Matrix	Refs.
1	Wood flour	Kenaf		Polypropylene	[25]
2	Banana	Sisal		Polyester	[29]
3	Oil palm fibre	Jute		Epoxy	[30]
4	Banana	Sisal		Polyester	[31]
5	Kenaf	Bamboo	Coir	Polylactic acid	[32]

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. [36, 37]. Morphology, physical and mechanical properties of flax fibres were presented in detail by Baley et al. [38].

2.1.2 Jute fibre

Jute is a best 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. Its abundance in availability with cheaper cost has acquired importance of its use in polymer composites [39]. 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 and aqueous extracts [40].

2.1.3 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*, is of a Mexican origin and is grown in Brazil, East Africa particularly in Tanzania, Haiti, India, Indonesia and Thailand [41]. This plant consists of sword-shaped leaves of normally 1.5 m length and a typical plant produces around 150 leaves during its life span of 6 years, and each leaf contains 1200 ± 1000 fibre bundles, which are composed of 4 wt% fibre, 0.75 wt% cuticle, 8 wt% dry matter and 87.25 wt% water [42]. 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 [43].

2.2 Fibre preparation methods

In order to improve the interfacial properties between the fibres (flax, jute and sisal 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 $NaHCO_3$ for 12 h.

2.2.1 Treatment with NaOH

In this study, the untreated flax, jute and sisal fibres were respectively soaked in 7, 9 and 1 wt% NaOH solution for 12 h at room temperature. Then the fibres were washed several times by deionized water until the residual NaOH was removed (examined by test paper) and then the fibres were dried at room temperature until a constant weight was reached.

2.2.2 Treatment with $NaHCO_3$

Similarly, the second treatment used in this study consisted of soaking the raw of the flax, jute and sisal fibres in 25, 25 and 10 wt% $NaHCO_3$ solution for 12 h at room temperature, respectively. After this process the fibres were thoroughly washed several times with distilled water and dried at room temperature until a constant weight was reached.

2.3 Fabrication of composite

Many techniques such as compression moulding, vacuum moulding, hand lay-up and others are available for manufacturing of composites. In this work the composites are fabricated by hand-lay-up technique followed by static compression. The working surfaces were treated with releasing waxes to facilitate easy removal of specimens from the mould surface after curing. The matrix was prepared by mixing accelerator and catalyst by 1.5% by weight in unsaturated polyester resin and stirred well to insure homogeneity of the system. Composites were made using a wood mould measuring 300 mm × 250 mm × 5 mm length, width and depth, respectively. Four beadings of a glass plate were used to maintain a 5-mm thickness all around the mould plates. 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. Then, each composite were cured by applying compression pressure using dead weights on the top of the mould and cured at room temperature for 24 h. The plates were kept in open air for 5 days to obtain a complete polymerization of the resin. Finally, dimension of specimens was cut as per ASTM standard by using a diamond saw blade.

The volume fraction of fibre (*VF*) is calculated by using the following relation [44, 45].

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

VF is fibre volume fraction, W_f are the weight (g) of fibres (sisal, jute and flax) and W_m is the weight (g) of matrix and 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 maker microscope with × 4.5 magnifications and 1-µm 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.

2.4 Mechanical property characterization

The test specimens were prepared and undergone in different mechanical tastings namely tensile test and flexural test as per the ASTM standards.

2.4.1 Tensile strength testing

The measurement of material capability to withstand forces and to what extent the material stretches before breaking is vital in material characterization. In this case, the tensile stress was applied in the direction of fibre axis (longitudinal axis). The tensile strength and modulus of the all the composites were measured using a Universal Testing Machine EZ20, equipped with a load cell of 20 kN. Test specimens of dimensions 250 mm × 25 mm × 5 mm were cut as per ASTM D 3822-0 specifications. The test speed was maintained at 2 mm/min. The tests are carried out at a condition of 23 °C and an average relative humidity of 30%. In each case, three specimens were tested and the average values were reported in Table 3 for every material.

2.4.2 Flexural strength testing

Flexural strength is carried out to find the ability of the material to resist the deformation under the load. The flexural specimens are prepared as per the ASTM D790 standards. The flexural tests were investigated using the three-point bending fixture with rectangular shape three-point bending specimens, 150 mm × 15 mm × 5 mm for each composite materials. Specimen deflection is measured by the crosshead position. Test results include flexural strength and displacement. The testing process involves placing the test specimen in the universal testing machine and applying force to it until it fractures and breaks. The tests are carried out at a condition of 23 °C and an average relative humidity of 30%. Three specimens of each formulation were tested, and average values were reported in Table 3 for every material. In addition, the flexural modulus of elasticity, which is the ratio of stress to strain at any point on the stress vs strain curve, was calculated using the following expressions:

$$\text{Flexural strength}: \sigma_f = \frac{3P_f L}{2bh^2} \tag{2}$$

$$\text{Flexural modulus}: E_f = \frac{ML^3}{4bh^3} \tag{3}$$

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) and M is the slope of the tangent to the initial straight-line portion of the load-deflection curve (N/mm) of deflection.

3 Results and discussion

3.1 Tensile properties

Tensile properties of a composite material are mainly depending on fibre strength, modulus, fillers, fibre length and orientation, fibre/matrix interfacial bonding and fibre content. Also, is influenced by the strength and modulus of fibres.

The effect of natural fibre-reinforced epoxy composites on tensile properties is shown in Fig. 1. As it can be seen from this figure the jute reinforced composite showed a higher tensile mechanical properties (σ_t and E_t) than other composites. This is because the jute fibres are characterized by high diameter and stronger stiffer than the flax and sisal fibres. The mechanical properties of the fibres used in this work are presented in Table 1. In practice, the mean tensile strength and the mean tensile modulus of the flax, jute and sisal reinforced composites are in the range of [(35.85 MPa and 1.54 GPa), (41.08 MPa and 1.66 GPa) and (29.96 MPa and 1.29 GPa)], respectively. In addition, the mean value ratios for $\sigma_{\text{Jute}}/\sigma_{\text{Flax}}$, $\sigma_{\text{Jute}}/\sigma_{\text{Sisal}}$, $E_{\text{Jute}}/E_{\text{Flax}}$ and $E_{\text{Jute}}/E_{\text{Sisal}}$ are 1.14, 1.37, 1.07 and 1.28, respectively.

Then, the effects of the type of treatment on the tensile properties of the natural-reinforced composites were investigated. The tensile strength and tensile modulus were determined and shown in Fig. 2 a and b, respectively. From these figures, it can be apparently seen that the tensile strength and

Table 3 Levels of various independent variables at coded values of RSM experimental design

No.	Independent variables	Symbol code, X_i	Units	Levels		
				− 1	0	+ 1
1	Type of fibres	X_1	–	Flax	Jute	Sisal
2	Type of chemical treatment	X_2	–	$NaHCO_3$	Raw	NaOH
3	Volume fraction of fibre	X_3	wt%	10	15	20

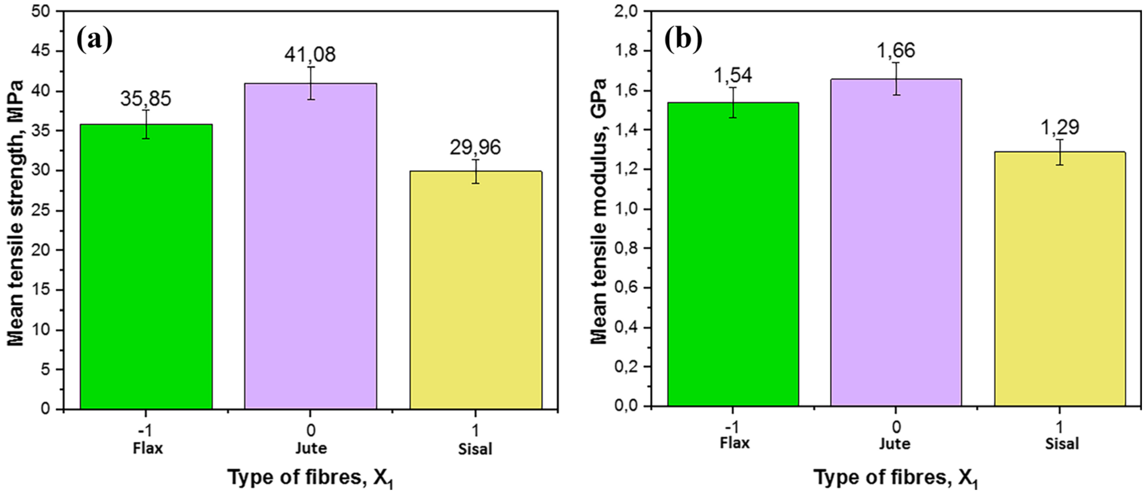


Fig. 1 Effect of type natural fibre composites on tensile properties: **a** mean tensile strength and **b** mean tensile modulus

tensile modulus of all fibre-reinforced composites improved for treated fibres compared to those of the untreated tensile composites are due to removal of outer surface. Hence, the surface modifications could remove surface impurities and produce a rough surface topography. This topography offers better fibre matrix interface adhesion and an increase in mechanical properties of the composites (see Figs. 7, 8 and 9). The effects of fibre surface treatment were discussed by Zhou [46] and Alawar [47]. In another recent study presented by Bedjaoui et al. [23], the effects of concentration of sodium bicarbonate ($NaHCO_3$) on the tensile properties of flax/polyester composites have been discussed.

In comparison to the neat resin, the untreated fibre-reinforced composites showed 1377% and 1617% increases in tensile strength and tensile modulus, respectively. On the other hand, the sodium hydroxide NaOH and sodium bicarbonate $NaHCO_3$-treated fibre-reinforced composites showed around 10% improvement in tensile strength for both treatments, while the tensile modulus increased by 7.70% and 12.40% for sodium hydroxide and sodium bicarbonate-treated composites, respectively, compared to the untreated composites.

The results presented on Fig. 3 a and b show the evolution of the tensile properties according to the volume fraction of fibre and the type of chemical treatment (X_2) is kept at the middle level ($NaHCO_3$). From these figures, it is observed that the tensile strength and tensile modulus of all composites increased with an increase in the volume fraction of fibre, which were evident from Fig. 3. Similar results were reported by Ben Brahim [12], when they study the effect of volume fraction of fibres on the tensile properties (longitudinal modulus and the longitudinal stress) of unidirectional alfa-polyester composites.

The analysis of the effects of the volume fraction of fibre (X_3) on the tensile properties is as follows: the increase in the volume fraction of fibre from 10 to 20 wt% increases the average tensile properties (σ_t and E_t) successively of [($\sigma_{\text{Flax}} = 135.63\%$, $\sigma_{\text{Jute}} = 103.90\%$ and $\sigma_{\text{Sisal}} = 56.83\%$) and

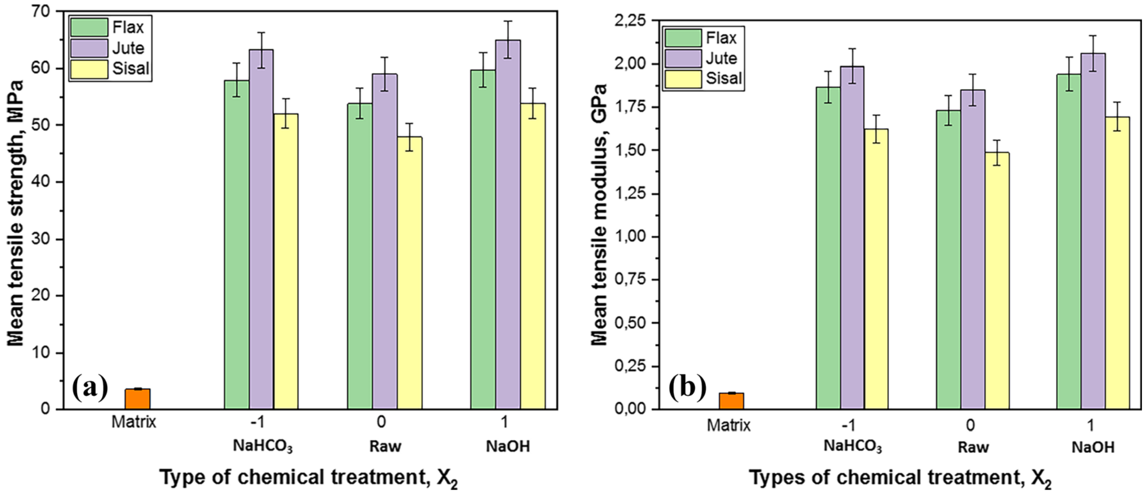


Fig. 2 Effect of type of chemical treatment on tensile properties of jute natural fibre composites: **a** mean tensile strength and **b** mean tensile modulus

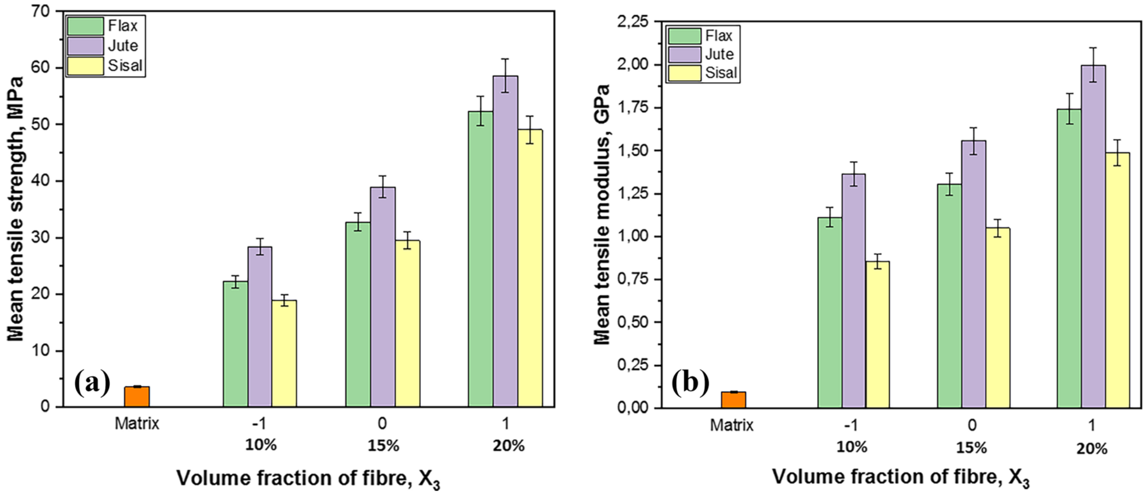


Fig. 3 Effect of volume fraction of fibre on tensile properties of various natural fibre composites treated with $NaHCO_3$: **a** mean tensile strength and **b** mean tensile modulus

($E_{Flax} = 135.63\%$, $E_{Jute} = 46.30\%$ and $E_{Sisal} = 73.79\%$)]. It is noted that the mean tensile strength is very affected by the volume fraction of fibre. The maximum values of the tensile properties (σ and E) are about up 58 MPa and 2 GPa. These last are recorded at the volume fraction of 20 wt% and when used composites reinforced by jute fibre.

3.2 Flexural properties

Flexural properties represent the flexibility of the materials, and good flexural strength indicates that the materials have brittle properties and high hardness [14]. Figure 4 show the variation of flexural strength and modulus for various types of composites, while the volume fraction of fibre (X_3) in the composite and the type of chemical treatment (X_2) are kept at the middle level. Flexural property behaviour, a similar trend to tensile property behaviour, exhibits higher values compared to the tensile properties. Also, the results indicated that the jute reinforced composites performed the other types of composites tested. On the experimental plan, the jute reinforced composites generate higher values of the flexural properties (flexural strength and flexural modulus) than flax and sisal reinforced composites. For example, $\sigma_{Flax} = 1.43\ \sigma_{Jute}$, $E_{Flax} = 1.89\ E_{Jute}$, $\sigma_{Sisal} = 1.61\ \sigma_{Jute}$ and $E_{Flax} = 2\ E_{Sisal}$, respectively.

Figure 5 expresses the evolution of flexural properties of pure epoxy resin and untreated/treated flax, jute and sisal reinforced composites. Figure 5 a shows the variation of mean flexural strength of the specimens of untreated/treated natural fibres used in the composites, at constant volume fraction of fibre (20 wt%). Similarly, Fig. 5b shows the variation of mean flexural modulus of untreated/treated reinforced composites at constant volume fraction of fibre (20 wt%).

For untreated specimens, the mean flexural strength of flax, jute and sisal reinforced composites increased 224.86%,

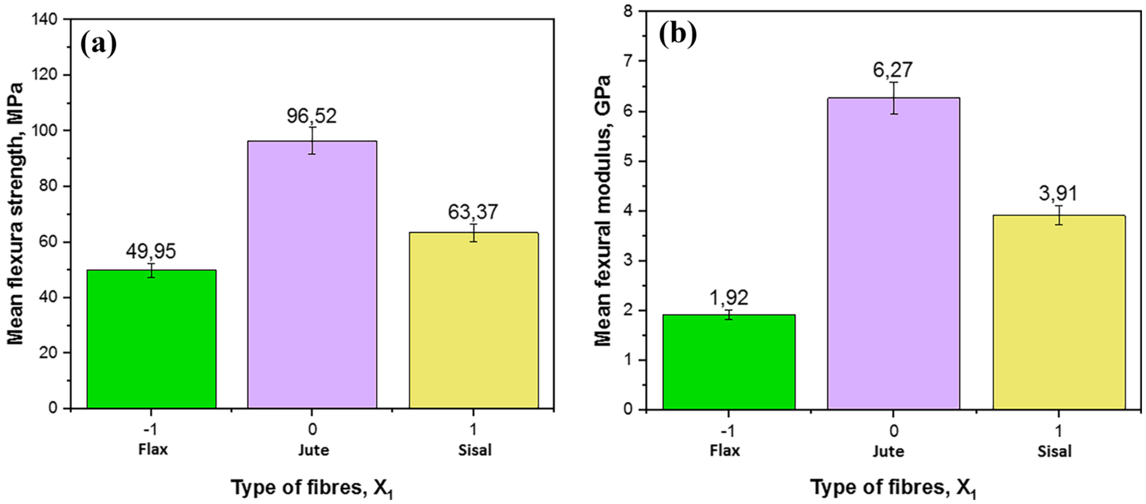


Fig. 4 Effect of type natural fibre composites on flexural properties: **a** mean flexural strength and **b** mean flexural modulus

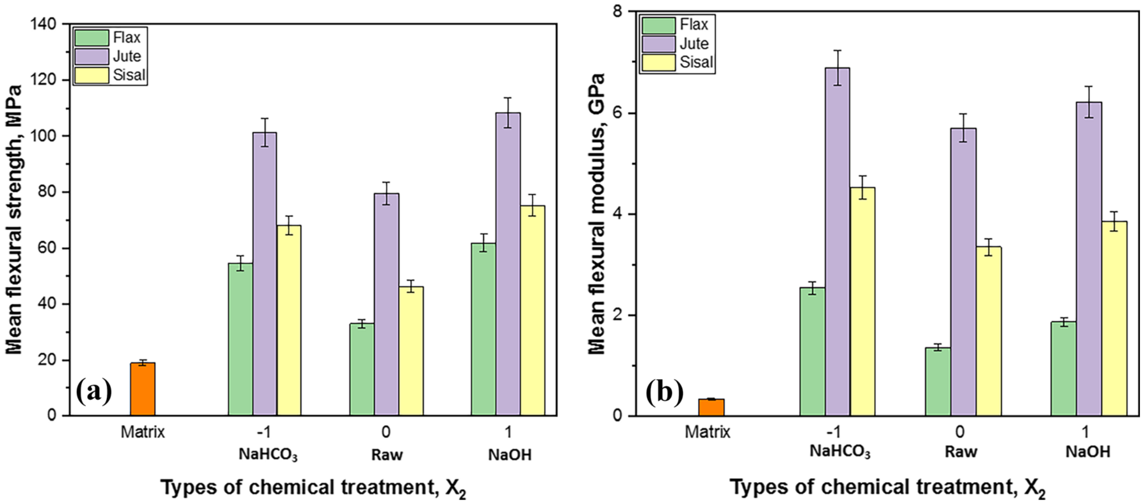


Fig. 5 Effect of type of chemical treatment on flexural properties of jute natural fibre composites: **a** mean flexural strength and **b** mean flexural modulus

469.06% and 295.23%, respectively, compared to pure epoxy (19.07 MPa), respectively (Fig. 5a). The mean flexural modulus of flax, jute and sisal reinforced composites are 628.29%, 1869.49% and 1196.40% higher than that of pure epoxy (0.35 GPa), respectively (Fig. 5b). Similarly, for treated specimens, the mean flexural strength of flax, jute and sisal reinforced composites increased 76.37%, 31.73% and 54.34%, respectively, compared to untreated specimens (33.10, 79.67 and 46.52) MPa, respectively (Fig. 5a). The mean flexural modulus of flax, jute and sisal reinforced composites are 61.87%; 14.80% and 25.19% higher than that of untreated specimens (1.36, 5.71 and 3.35) GPa, respectively (Fig. 5b). This is because of a good bonding between fibres and epoxy matrix. Hence, the fibre–matrix interface plays an important role in determining the mechanical properties of composite materials.

Figure 6 shows the effect of different volume fraction of fibre on the mean flexural strength and mean flexural modulus for various fibre types of composites. Flexural properties behaved with a similar trend to tensile property behaviour and exhibit higher values compared to the tensile properties. For all three composites, the addition of fibres increased the flexural properties of the composites with results depending on the fibre type and loading. The mean flexural strength increased from a value of 19.07 MPa for an unfilled matrix (pure matrix) to a maximum value of 93.20 MPa for a 20 wt% untreated-jute fibre composite. Values of 61.78 MPa and 55.23 MPa have also been found for untreated-flax and untreated-sisal composites, respectively. This corresponds to a 388.72%, 123.96% and 189.61% increase in mean flexural strength, respectively. Similar result was obtained from mean flexural modulus measurement; we successively record an increase in the mean flexural modulus of the fibre composite (flax, jute and sisal) from 737.14%, 1977.14% and 1302.85%, when compared to unfilled matrix

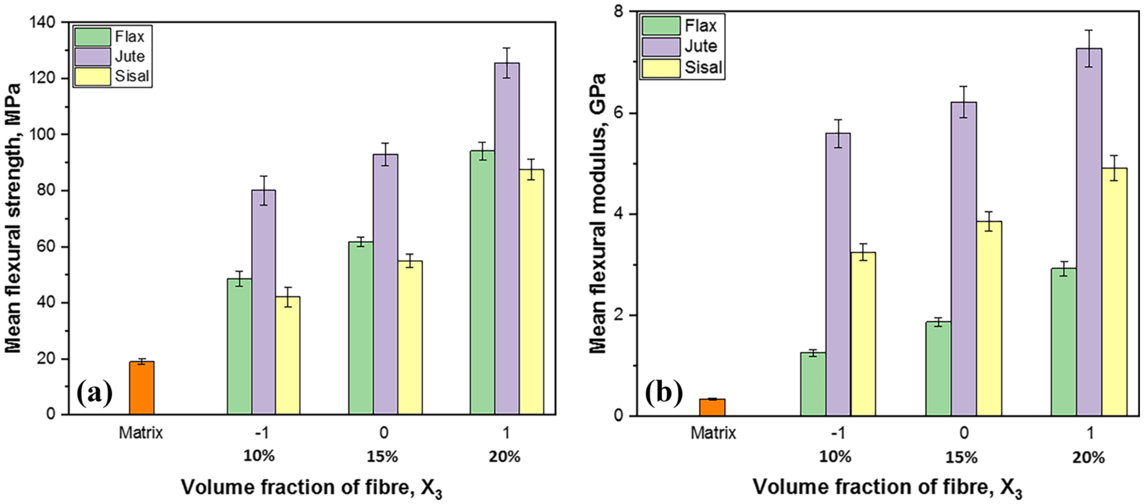


Fig. 6 Effect of volume fraction of fibre on flexural properties of natural fibre epoxy composites: **a** mean flexural strength and **b** mean flexural modulus

(0.35 GPa). Similarly, de Albuquerque et al. [48] have reported the same trend on the jute reinforced polyester composites.

Generally, increasing the volume fraction of fibre on the composites will result in a significant increase of composite stiffness, as tensile and impact strengths are increased through the addition of natural fibres. A similar trend was observed by other authors [25, 46, 47]. For example, Salman et al. [49] 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.

3.3 Influence of NaOH and $NaHCO_3$ treatments on the surface morphology of the fibres

To clarify the effect of NaOH and $NaHCO_3$ treatments on flax, jute and sisal fibres, surface examinations were carried out on the untreated and treated fibres. The scanning electron microscope (SEM) photographs of morphology in diameter direction and in the cross section of both untreated and treated surfaces are exhibited in Figs. 7, 8 and 9. As shown in Figs. 7a, 8a and 9a the surface of untreated flax, jute and sisal fibres were found to be considerably covered with waxy substances and impurities. Relatively, micrograph of the treated fibre (Figs. 7b, c; 8b, c; and 9b, c) shows an improvement in the surface morphology after NaOH and $NaHCO_3$ treatments; i.e. using NaOH and $NaHCO_3$ treatments removed the waxy layer and impurities from surface, and the treated surface of fibre becomes rather rougher and fibrillation as compared to that of untreated fibre. Moreover, it can be seen that the fibres have been spitted into finer fibres. This could lead to high interlock and adhesion between the fibres and the matrix.

4 Statistical analysis

4.1 RSM experimental design

The response surface methodology (RSM), firstly induced by Box and Wilson [50, 51], is a method for the accurate prediction of engineering system input–output relationships by taking a full consideration for parameter interactions. It has been widely applied in numerous manufacturing fields for the design, development and formulation of

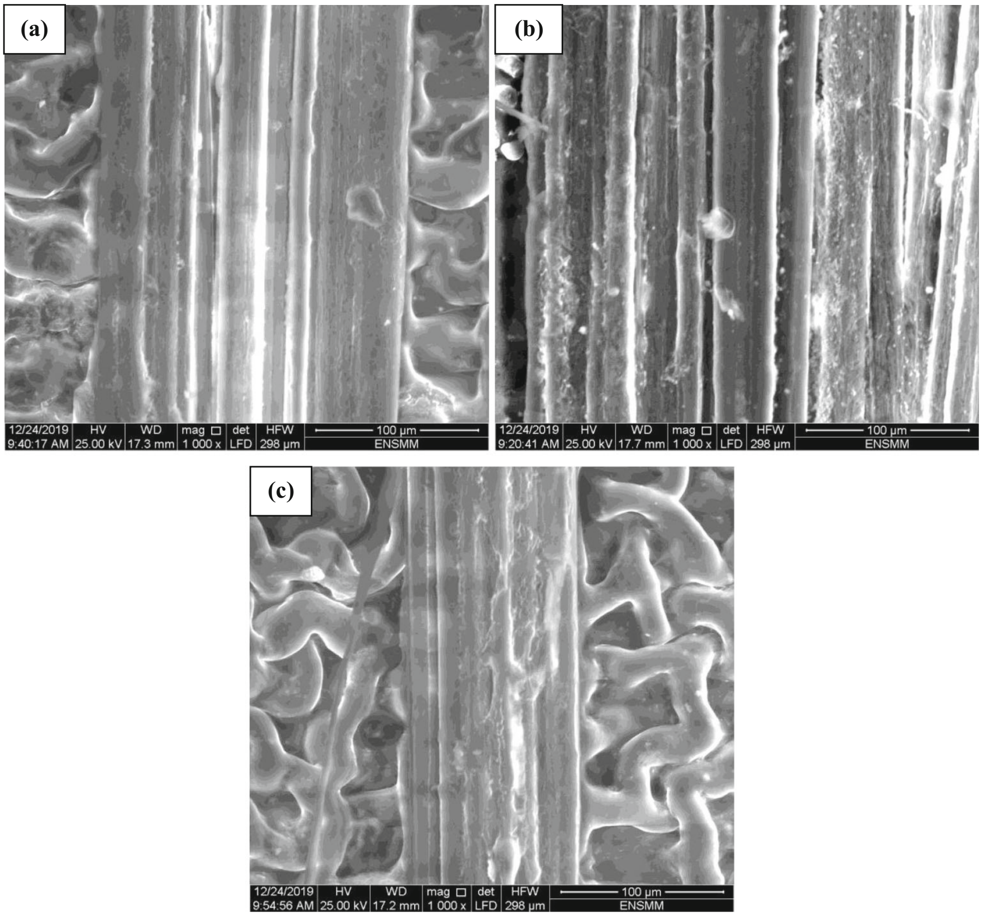


Fig. 7 SEM micrographs of flax fibre surface of **a** untreated flax fibre, **b** $NaHCO_3$-treated flax fibre and **c** NaOH-treated flax fibre

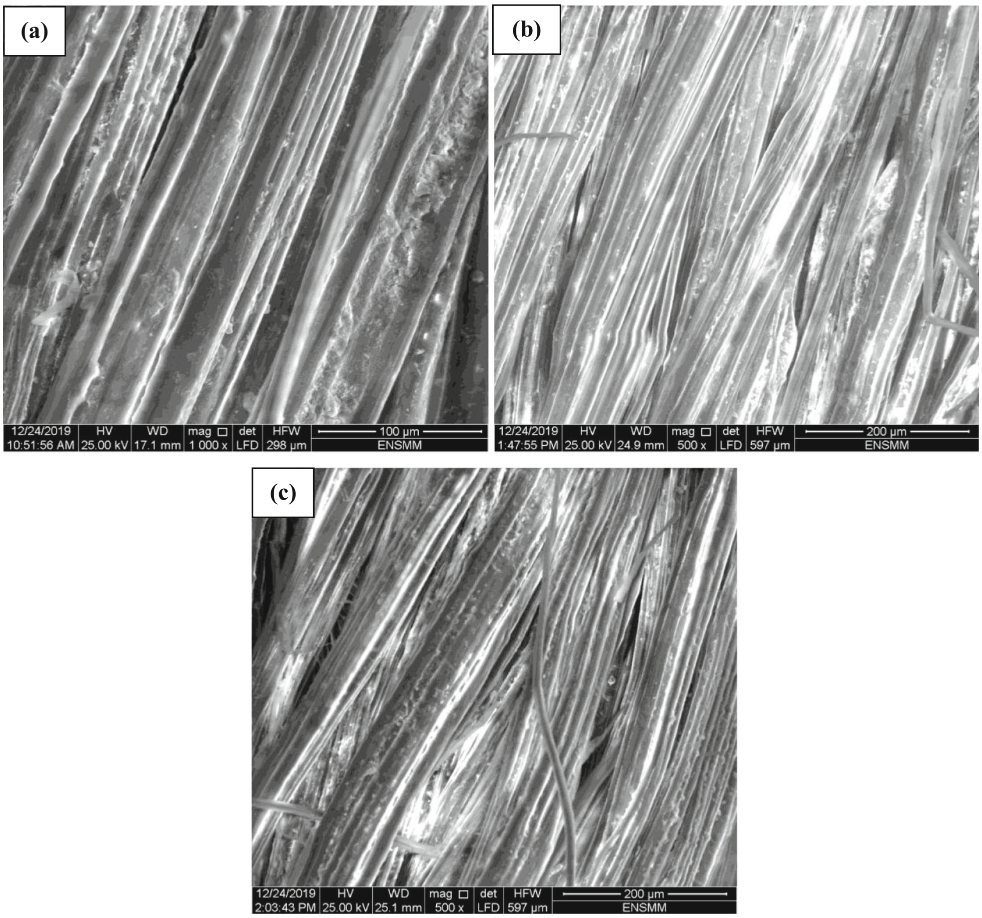


Fig. 8 SEM micrographs of jute fibre surface of **a** untreated jute fibre, **b** $NaHCO_3$-treated jute fibre and **c** NaOH-treated jute fibre

new products, as well as in the improvement of existing product designs.

The RSM is a collection of mathematical and statistical techniques useful for the modelling and analysis of problems in which response of interest is affected by several variables and the purpose is to optimize this response [50]. An important advantage of RSM is to reduce the 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) and Central Composite Design (CCD). The BBD is selected to generate the design matrix since it needs fewer experiments when the number of factors is about 3–4. A Box–Behnken experimental design with three independent variables: X_1, type of fibre; X_2, chemical treatment and X_3, volume fraction of fibre, at three levels were chosen. 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. The complete design consisted of seventeen combinations including five replicates of a central point (Table 3). Each combination was repeated three times.

The relationship of the independent variables and response was calculated by the quadratic polynomial equation. The quadratic polynomial equation was used to study the effects of the linear and square terms of the input variables, as shown is Eq. (4):

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 \tag{4}$$

where X_1, X_2 and X_3 are independent variables, which influence the response variable Y; β_0 is the intercept; β_1, β_2 and β_3 are the linear coefficient, on the response Y; and β_{11}, β_{22} and β_{33} are the quadratic coefficient on the response Y. k is the number of independent variables ($k = 3$ in this study). The quality of the model was expressed by the 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.

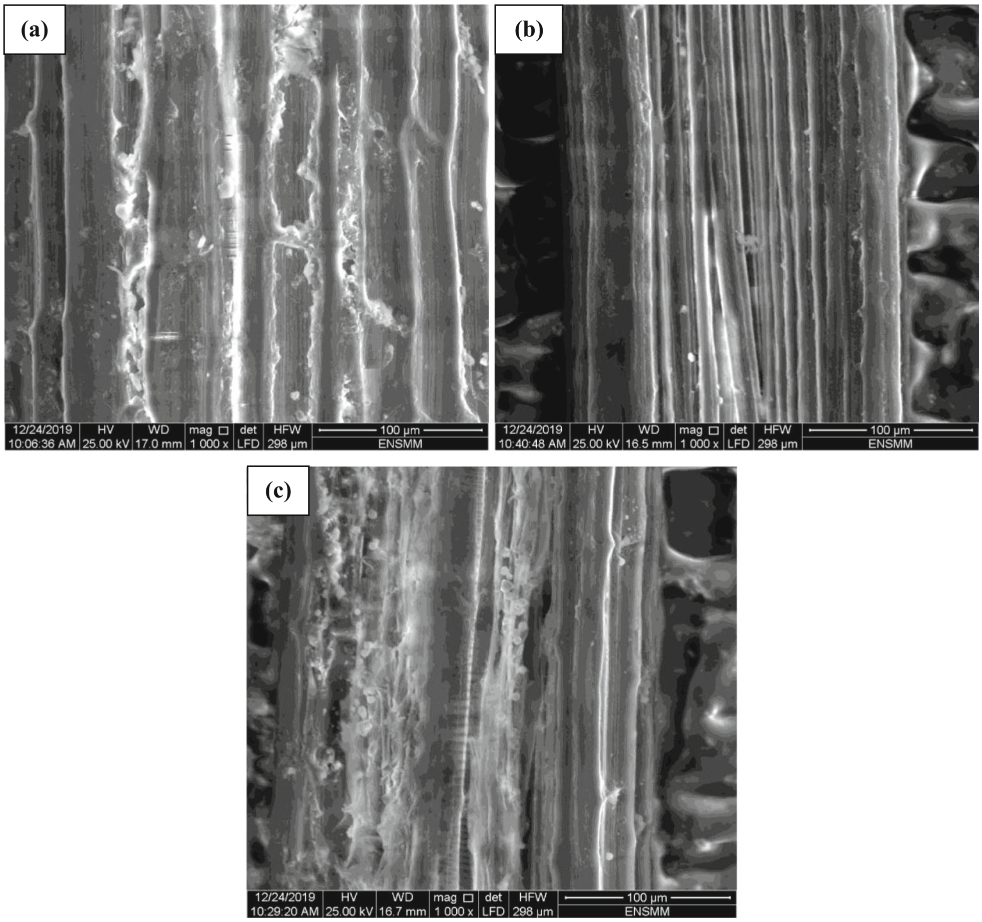


Fig. 9 SEM micrographs of sisal fibre surface of **a** untreated sisal fibre, **b** $NaHCO_3$-treated sisal fibre and **c** NaOH-treated sisal fibre

When R^2 approaches to unity, it indicates a good correlation between the experimental and the predicted values. The value of coefficient of determination R^2 was calculated by the following equation [52]:

$$R^2 = 1 - \frac{SS_{\text{Error}}}{SS_{\text{Total}}} \tag{5}$$

where SS_{Error} is the residual and SS_{Total} is the total sum of squares.

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 3.

4.2 Regression equations

In the empirical approach, predictions of mechanical properties were done based on the regression analysis of the experimental data (Table 4). The correlations between the response parameters (mean tensile strength σ_t, mean tensile modulus E_t, mean flexural strength σ_f and mean flexural modulus E_f) and third independent variables, which are type of fibres (X_1), type of chemical treatment (X_2) and volume fraction of fibre (X_3), were modelled by quadratic regressions. The final quadratic models of response equation in terms of coded terms were observed from Eq. (4) as

The tensile properties are given the following equation:

$$E_t = 36.923 - 2.941X_1 + 0.888X_2 + 14.08X_3 - 8.17X_1^2 + 5.04X_2^2 + 8.09X_3^2 \tag{6}$$

($R^2 = 82.85\%$; adj-$R^2 = 72.56\%$)

$$\sigma_t = 1.526 - 0.122X_1 + 0.036X_2 + 0.332X_3 - 0.242X_1^2 + 0.172X_2^2 - 0.005X_3^2 \tag{7}$$

($R^2 = 86.90\%$; adj-$R^2 = 79.04\%$)

The flexural properties are given in the following equation:

$$E_f = 93.2 - 3.27X_1 - 3.055X_2 + 22.79X_3 - 34.69X_1^2 + 8.47X_2^2 + 9.755X_3^2 \tag{8}$$

Table 4 The 3-factor BBD matrix and response parameters

No.	Independent variables						Response parameters			
	Coded values			Actual values			Tensile properties		Flexural properties	
	X_1	X_2	X_3	Type of fibres	Type of chemical treatment	Volume fraction of fibre (wt%)	Mean tensile strength σ_t (MPa)	Mean tensile modulus E_t (GPa)	Mean flexural strength σ_f (MPa)	Mean flexural modulus E_f (GPa)
1	0	0	0	Jute	Raw	15	36.92	1.52	93.20	7.54
2	0	0	0	Jute	Raw	15	36.92	1.52	93.20	7.54
3	−1	0	−1	Flax	Raw	10	17.07	1.01	44.33	2.23
4	0	−1	−1	Jute	$NaHCO_3$	10	28.36	1.12	86.47	4.49
5	0	1	1	Jute	NaOH	20	67.41	2.08	129.50	10.98
6	0	0	0	Jute	Raw	15	36.92	1.52	93.20	7.54
7	0	0	0	Jute	Raw	15	36.92	1.52	93.20	7.54
8	1	0	1	Sisal	Raw	20	43.84	1.26	72.72	4.10
9	1	−1	0	Sisal	$NaHCO_3$	15	37.02	1.46	66.87	4.21
10	0	0	0	Jute	Raw	15	36.92	1.52	93.20	7.54
11	−1	−1	0	Flax	$NaHCO_3$	15	39.72	1.55	69.29	4.06
12	−1	0	1	Flax	Raw	20	59.84	1.91	101.93	6.38
13	1	1	0	Sisal	NaOH	15	22.03	1.32	63.73	4.09
14	0	−1	1	Jute	$NaHCO_3$	20	59.07	2.01	146.40	11.17
15	−1	1	0	Flax	NaOH	15	36.42	1.49	68.03	3.51
16	0	1	−1	Jute	NaOH	10	45.42	1.55	83.33	5.10
17	1	0	−1	Sisal	Raw	10	26.63	0.93	54.08	2.80

$(R^2 = 94.35\%;\ \text{adj-}R^2 = 90.96\%)$

$$\begin{aligned} E_f &= 7.54 - 0.122X_1 - 0.031X_2 + 2.25X_3 - 3.815X_1^2 \\ &\quad + 0.24X_2^2 + 1.152X_3^2 \end{aligned} \tag{9}$$

$(R^2 = 91.52\%;\ \text{adj-}R^2 = 86.43\%)$

4.3 Analysis of variance

In order to understand the interaction between the independent variables and the responses, a variance analysis (ANOVA) was done in accordance with the suggested model. In this case, the ANOVA of the data with the mean strength (σ) and mean elastic modulus (E), with the objective of analysing the influence of type of fibres (X_1), types of chemical treatment (X_2) and volume fraction of fibre (X_3) on the total variance of the results, was 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 [53].

ANOVA results for the mean strength (σ) are summed up in Table 5. As can be seen from Table 5, the regression models for σ were found to be highly significant from the Fisher's test, which have a high F values (8.0536 and 27.8565) with very low probability ($P = 0.0023$ and $P < 0.0001$) according to tensile and flexural tests, respectively. Also, analysis of variance (ANOVA) enables the classification of the three independent variables representing the X_1, X_2 and X_3 along with their quadratic terms of their influence on the mean strength: it can be seen that the volume fraction of fibre (X_3) is the most important factor affecting σ. Their contributions are (68.19% for $\sigma_{\text{-tensile}}$ and 41.21% for $\sigma_{\text{-flexural}}$). The next largest factor influencing σ is the type of fibre (X_1) (2.97% for mean tensile strength and 0.85% for mean flexural strength) contributions. The types of chemical treatment (X_2) with (0.27% for mean

Table 5 Analysis of variance for mean strength

Source	SS	DF	MS	*F* value	*P* value	Cont. %	Remarks
(a) Tensile test							
Model	2299.56	6	383.259	8.0536	0.0023		Significant
X_1	69.21	1	69.21	1.4543	0.2556	2.97	Insignificant
X_2	6.32	1	6.32	0.1328	0.7232	0.27	–
X_3	1586.82	1	1586.82	33.3443	0.0002	68.19	Significant
$X_1 \times X_1$	281.23	1	281.23	5.9096	0.0354	12.09	–
$X_2 \times X_2$	107.29	1	107.29	2.2545	0.1641	4.61	Insignificant
$X_3 \times X_3$	276.11	1	276.11	5.8019	0.0368	11.87	Significant
Residual	475.89	10	475.89				
Total	2775.44	16				100	
(b) Flexural test							
Model	9861.298	6	1643.550	27.8565	< 0.0001		Significant
X_1	85.674	1	85.674	1.4521	0.2559	0.85	Insignificant
X_2	74.664	1	74.664	1.2655	0.2869	0.74	–
X_3	4155.984	1	4155.984	70.4398	< 0.0001	41.21	Significant
$X_1 \times X_1$	5066.931	1	5066.931	85.8794	< 0.0001	50.24	–
$X_2 \times X_2$	302.067	1	302.067	5.1197	0.0472	2.99	–
$X_3 \times X_3$	400.674	1	400.674	6.7910	0.0262	3.97	–
Residual	590.006	10	590.006				
Total	10,451.30	16				100	

Table 6 Analysis of variance for mean elastic modulus

Source	SS	DF	MS	*F* value	*P* value	Cont. %	Remarks
(a) Tensile test							
Model	1.3701	6	0.2283	11.0576	0.0006		Significant
X_1	0.1196	1	0.1196	5.7934	0.0369	8.62	–
X_2	0.0108	1	0.0108	0.5207	0.4871	0.78	Insignificant
X_3	0.8851	1	0.8851	42.8625	< 0.0001	63.80	Significant
$X_1 \times X_1$	0.2472	1	0.2472	11.9695	0.0061	17.82	–
$X_2 \times X_2$	0.1245	1	0.1245	6.0295	0.0339	8.97	–
$X_3 \times X_3$	0.0001	1	0.0001	0.0055	0.9421	0.01	Insignificant
Residual	0.2065	10	0.0207				
Total	1.5766	16				100	
(b) Flexural test							
Model	101.9707	6	16.9951	17.9849	< 0.0001		Significant
X_1	0.1201	1	0.1201	0.1270	0.7289	0.12	Insignificant
X_2	0.0079	1	0.0079	0.0083	0.9291	0.01	–
X_3	40.5495	1	40.5495	42.9110	< 0.0001	39.63	Significant
$X_1 \times X_1$	61.2769	1	61.2769	64.8455	< 0.0001	59.90	–
$X_2 \times X_2$	0.2474	1	02474	0.2618	0.6200	0.24	Insignificant
$X_3 \times X_3$	0.0978	1	0.0978	0.1035	0.7543	0.10	–
Residual	9.4497	10	9.4497				
Total	111.4204	16				100	

tensile strength and 0.74% for mean flexural strength) contributions have a very weak significance effect on mean strength σ. Similarly, the quadratic coefficients ($X_1 \times X_1$ and $X_3 \times X_3$) have significant effect on mean strength σ.

Similarly, results from ANOVA (Table 6) for the quadratic model of mean elastic modulus (*E*) showed that the polynomial models were highly statistically significant, as suggested by the high model *F* values (11.0576 for $E_{\text{-tensile}}$ and 7.37 for $E_{\text{-flexural}}$) and low *P* values ($P = 0.0001$ for $E_{\text{-tensile}}$ and $P < 0.0001$ for $E_{\text{-flexural}}$). 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. According to Table 6, it can be apparently the volume fraction of fibre (X_3), which is the most important factor affecting on the mean elastic modulus (*E*). Its contribution is (Cont. = 63.80% and Cont. = 39.64%) for tensile and flexural tests, respectively. The next factor influencing *E* is the type of fibres (X_1) with (Cont. = 8.62% and Cont. = 0.12%); there are lots of work that have been done to study the effect of fibre loading on the mechanical properties [6, 7]. Whereas, the chemical treatment (X_2) with (Cont. = 0.78% and Cont. = 0.01%) was found to be less significant on mean elastic modulus of tensile and flexural tests, respectively.

4.4 Perturbation plots

The main effects of the single factor (X_1, X_2 and X_3) and the perturbation plots for both response parameters (σ and *E*) are illustrated in Fig. 10. They confirm the ANOVA results demonstrated in Tables 5 and 6. The *x*-axis in the graphs is the low and high levels of the design factor, and the *y*-axis is the mean value of the response parameter at a specific design factor

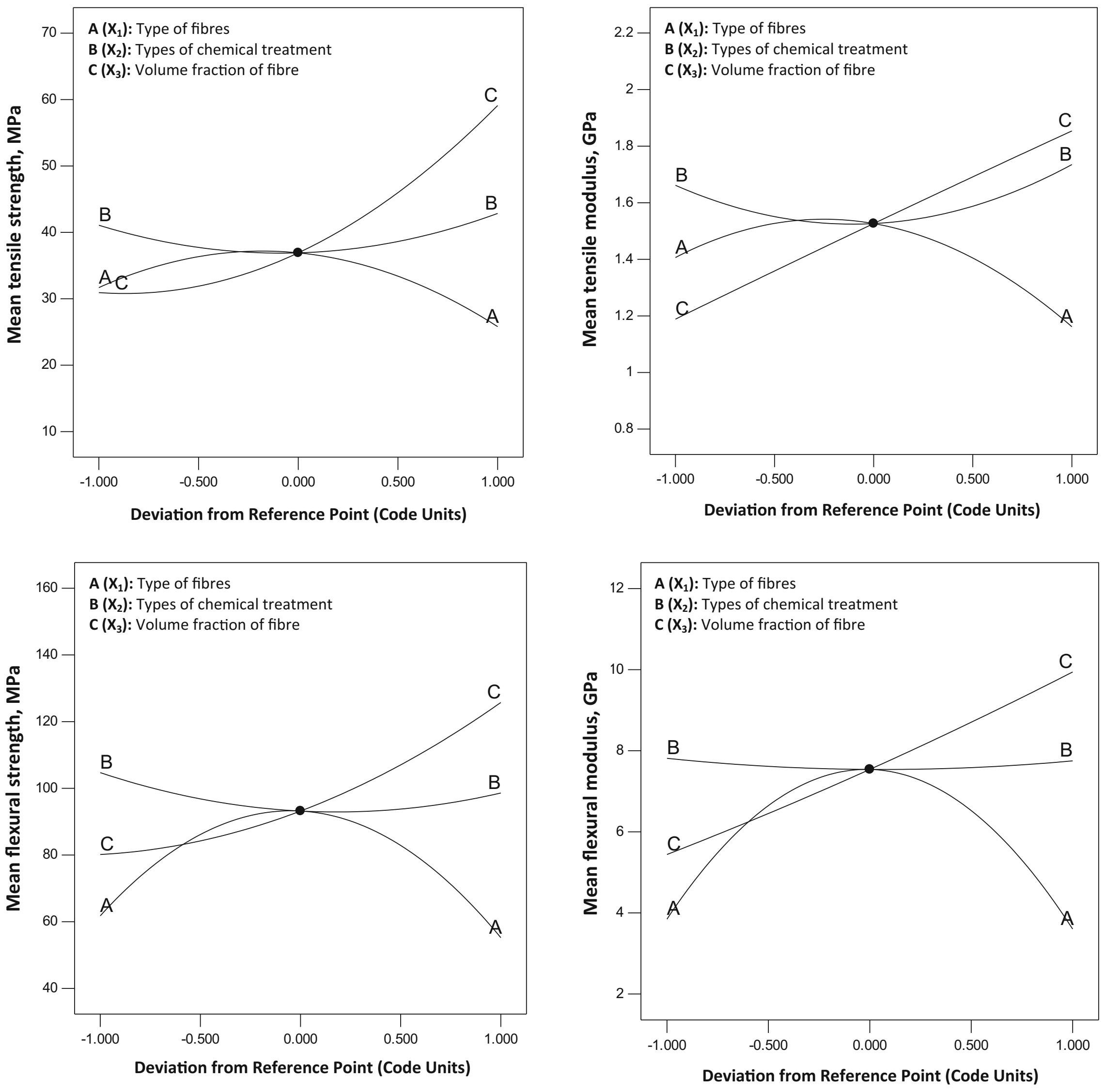


Fig. 10 Main effect graphs for tensile and flexural proprieties versus X_1, X_2 and X_3

level. According to this plot, we understand that the third factors X_1, X_2 and X_3 have a positive quadratic influence on mechanical properties (σ and E). The values of mechanical properties (σ and E) will increase greatly as the value of X_3 increases, while X_2 has less effect on mechanical properties (σ and E).

4.5 Response surface analysis

The response surfaces were generated by Matlab after model fitting of BBD, as shown in Figs. 11 and 12. The influences of each factor (X_1: type of fibres, X_2: types of chemical treatment and X_3: volume fraction of fibre) and their interactions on the response parameters for both tests, namely tensile test and flexural test, were investigated.

Figure 11 a shows the estimated mean strength for the corresponding volume fraction of fibre (X_3) and the types of chemical treatment (X_2) when the type of fibres (X_1) is fixed at the middle level (jute fibre). Volume fraction of fibre has the most effect on mean strength, and its variation is very high when compared to other independent variables. This figure also displays that the mean strength is the less sensitive as the types of chemical treatment are changed. In addition, the highest mean strength were achieved at the highest volume fraction of fibre and when used treated specimens.

Next, the effect of types of chemical treatment (X_2) and type of fibres (X_1) on the mean strength when the volume fraction of fibre (X_3) is fixed at the middle level (10 wt%) is shown in Fig. 11b. It can be seen that an intermediate X_2, around the middle level (raw), favours low mean strength

Fig. 11 Response surface 3D plots of mean strength as a function of type of fibres (X_1), types of chemical treatment (X_2) and volume fraction of fibre (X_3) under tensile and flexural tests

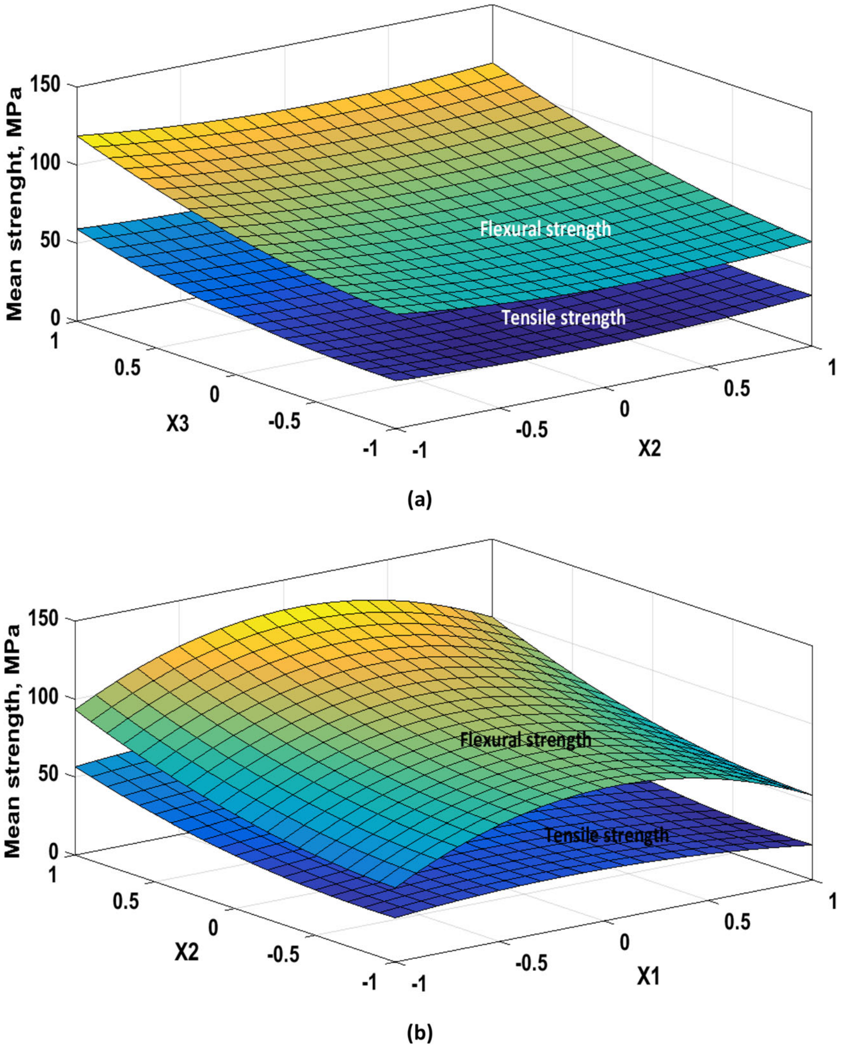
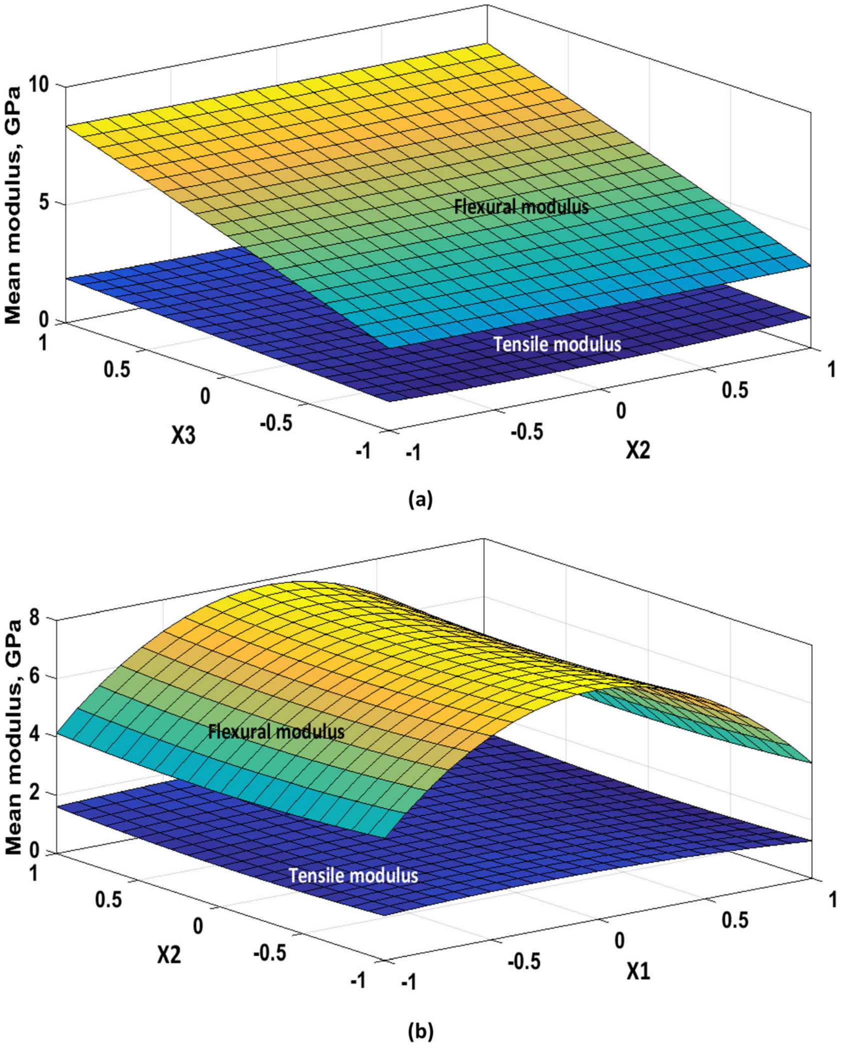


Fig. 12 Response surface 3D plots of mean modulus as a function of type of fibres (X_1), types of chemical treatment (X_2) and volume fraction of fibre (X_3) under tensile and flexural tests



for both tests. Contrary, it also seems that an intermediate X_1, around the middle level (jute fibre), also favours high strength for both tests.

Table 7 Constraints for optimization of independent variables

Conditions	Goal	Lower limit	Upper limit
(X_1): Type of fibres	In range	− 1	+ 1
(X_2): Type of chemical treatment	In range	− 1	+ 1
(X_3): Volume fraction of fibre (%)	In range	− 1	+ 1
Mean tensile strength σ_t, MPa	Maximize	17.07	67.41
Mean tensile modulus E_t, GPa	Maximize	0.93	2.09
Mean flexural strength σ_f, MPa	Maximize	44.33	146.40
Mean flexural modulus E_f, GPa	Maximize	2.23	11.17

Concerning now mean modulus, a similar phenomenon can be observed in Fig. 12. The influences of volume fraction of fibre (X_3) and types of chemical treatment (X_2) on the mean modulus are shown in Fig. 12a, with the type of fibres (X_1) fixed at level 0 (jute fibre) for both tests. According to the figure, the volume fraction of fibre has a very significant influence on mean modulus. Also, this figure also displays that the mean modulus is the less sensitive as the types of chemical treatment are changed compared to that of the volume fraction of fibre. When the volume fraction of fibre (X_3) is fixed at level 0 (10 wt%), the effects of types of chemical treatment (X_2) and type of fibres (X_1) on the mean modulus (E) are shown in Fig. 12b. It can be seen that an intermediate X_1, around the middle level (jute fibre), favours high mean modulus for both tests.

Table 8 Response optimisation in coded terms

No.	Coded factors			Tensile properties		Flexural properties		Desirability D
	X_1	X_2	X_3	Mean tensile strength σ_t (MPa)	Mean tensile modulus E_t (GPa)	Mean flexural strength σ_f (MPa)	Mean flexural modulus E_f (GPa)	
1	− 0.09	+ 1	+ 1	65.23	2.07	131.18	10.14	0.9179
2	− 0.10	+ 1	+ 1	65.25	2.07	131.15	10.13	0.9178
3	− 0.20	+ 1	+ 1	65.30	2.07	130.38	10.02	0.9139

In summary, the mechanical properties were significantly affected by volume fraction of fibre (X_3) and the type of fibres (X_1). However, the influence of types of chemical treatment (X_2) was relatively smaller.

5 Multiple response optimizations

According to Elbah et al. [53] desirability function approach has been used for multiple response parameters (mean tensile strength σ_t, mean tensile modulus E_t, mean flexural strength σ_f and mean flexural modulus E_f) optimization. The optimization module searches for a combination of factor levels that simultaneously satisfy the requirements placed on each of the responses and factors in an attempt to establish the appropriate model. During the optimization process, the aim is to find the optimal values of independent variables in order to produce the highest mechanical properties of composites (σ_t, E_t, σ_f and E_f). To resolve this type of parameter design problem, an objective function, $F(x)$, is defined as follows [54]:

$$Df = \left(\prod_{i=1}^{n} d_i^{w}\right)^{\frac{1}{\sum_{j-1}^{n} w_i}} \tag{10}$$

$$F(x) = -Df \tag{11}$$

where d_i is the desirability defined for the ith targeted output and w_i is the weighting of d_i. For various goals of each targeted output, the desirability, d_i, is defined in different forms. If a goal is to reach a specific value of T_i, the desirability d_i is:

$$di = 0 \text{ if } Y_i \leq \mathrm{Low}_i$$

$$d_i = \left[\frac{Y_i - Low_i}{T_i - Low_i}\right] \quad \text{if} \quad Low_i \leq Y_i \leq T_i \tag{12}$$

$$d_i = \left[\frac{Y_i - High_i}{T_i - High_i}\right] \quad \text{if} \quad T_i \leq Y_i \leq High_i \tag{13}$$

$$di = 0 \text{ if } Y_i \geq High_i \tag{14}$$

For a goal to find a maximum, the desirability is shown as follows:

$$di = 0 \text{ if } Y_i \geq Low_i \tag{15}$$

$$d_i = \left[\frac{Y_i - Low_i}{High_i - Low_i}\right] \quad \text{if} \quad Low_i \leq Y_i \leq High_i \tag{16}$$

$$di = 0 \text{ if } Y_i \geq High_i \tag{17}$$

For a goal to search for a minimum, the desirability can be defined by the following formulas:

$$di = 0 \text{ if } Y_i \geq Low_i \tag{18}$$

$$d_i = \left[\frac{High_i - Y_i}{High_i - Low_i}\right] \quad \text{if} \quad Low_i \leq Y_i \leq High_i \tag{19}$$

$$di = 0 \text{ if } Y_i \geq High_i \tag{20}$$

where the Yi is the value found of the ith output during optimization processes and the Low_i and the $High_i$ are, respectively, the minimum and the maximum values of the experimental data for the ith output. In Eq. (11), w_i is set to one since the d_i is equally important in this study. The Df is a combined

Table 9 Optimal levels of factors in actual terms

No.	Coded factors			Actual factors		
	X_1	X_2	X_3	Type of fibre content (%)	Types of chemical treatment	Volume fraction of fibre (%)
1	− 0.09	+ 1	+ 1	91% of jute and 9% of flax	NaOH	20
2	− 0.10			90% of jute and 10% of flax		
3	− 0.20			80% of jute and 20% of flax		

Table 10 Designations and composition of hybrid composites

No	Composites	Code	Volume fraction			Resin content (%)
			Flax fibre content (%)	Jute fibre content (%)	Sisal fibre content (%)	
1	Single composite: Jute/epoxy	SCJ	0	20	0	80
2	Single composite: Flax/epoxy	SCF	20	0	–	–
3	Hybrid composite: Intimate mix	HC01	1.80	18.20	–	–
4	Hybrid laminate composite : (jute/flax/jute)	HC02	–	–	–	–
5	Hybrid laminate composite : (flax/jute/flax)	HC03	–	–	–	–
6	Hybrid composite: Intimate mix	HC04	2	18	–	–
7	Hybrid laminate composite : (jute/flax/jute)	HC05	–	–	–	–
8	Hybrid laminate composite : (flax/jute/flax)	HC06	–	–	–	–
9	Hybrid composite: Intimate mix	HC07	4	16	–	–
10	Hybrid laminate composite : (jute/flax/jute)	HC08	–	–	–	–
11	Hybrid laminate composite : (flax/jute/flax)	HC09	–	–	–	–

desirability function, and the objective is to choose an optimal setting that maximizes a combined desirability function *Df*, i.e. minimizes *F*(*x*).

The constraints used during the optimization process are summarized in Table 7. The best optimal values of independent variables and response parameters are reported in Table 8 in order of decreasing desirability level; the optimal process factors were found to be as follows: in coded terms are (Table 8) the type of fibres (X_1) of [− 0.09 and − 0.20], the type of chemical treatment (X_2) of + 1 and the volume fraction of fibre (X_3) of + 1. Then, the optimal values in actual terms are presented in Table 9.

6 Confirmation test

6.1 Manufacturing process 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 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 (Table 10). The rule of mixture fibre is presented in Fig. 6.

The hybrid composites for this section were fabricated by hand-lay up method technique using a wooden mould (300 mm × 25 mm × 5 mm) (Fig. 13). After 7 days of curing, the plates were cut according to ASTM standards. Help designations and composition of hybrid composites are presented in Table 10, nine different kinds of composites were prepared with stacking sequences and the different configurations of composites are represented also in Table 10.

6.2 Comparative studies of the mechanical properties of the hybrid composites

Figure 14 shows that the stress–strain diagrams of three similar samples of hybrid laminate composite with different configurations of composites at the highest volume fraction of fibre (see Table 10) are linear and follow Hooke's law. Also, Fig. 15 shows the effect of different configurations of composites at the highest volume fraction of fibre on the flexural

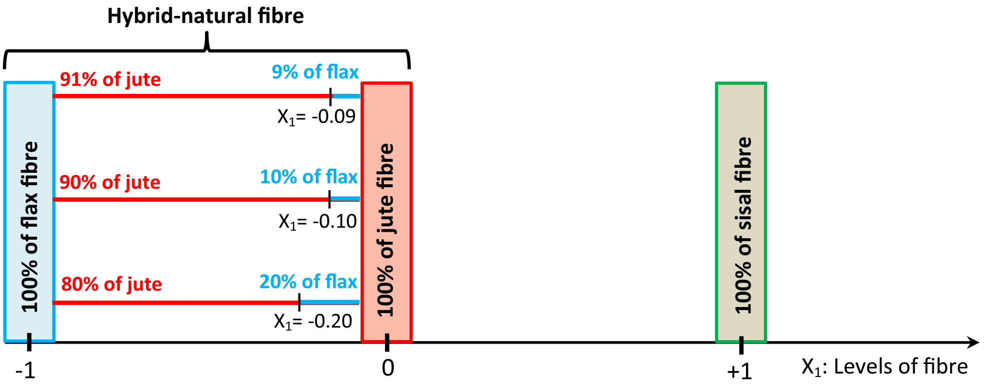


Fig. 13 Rule of hybrid mixture

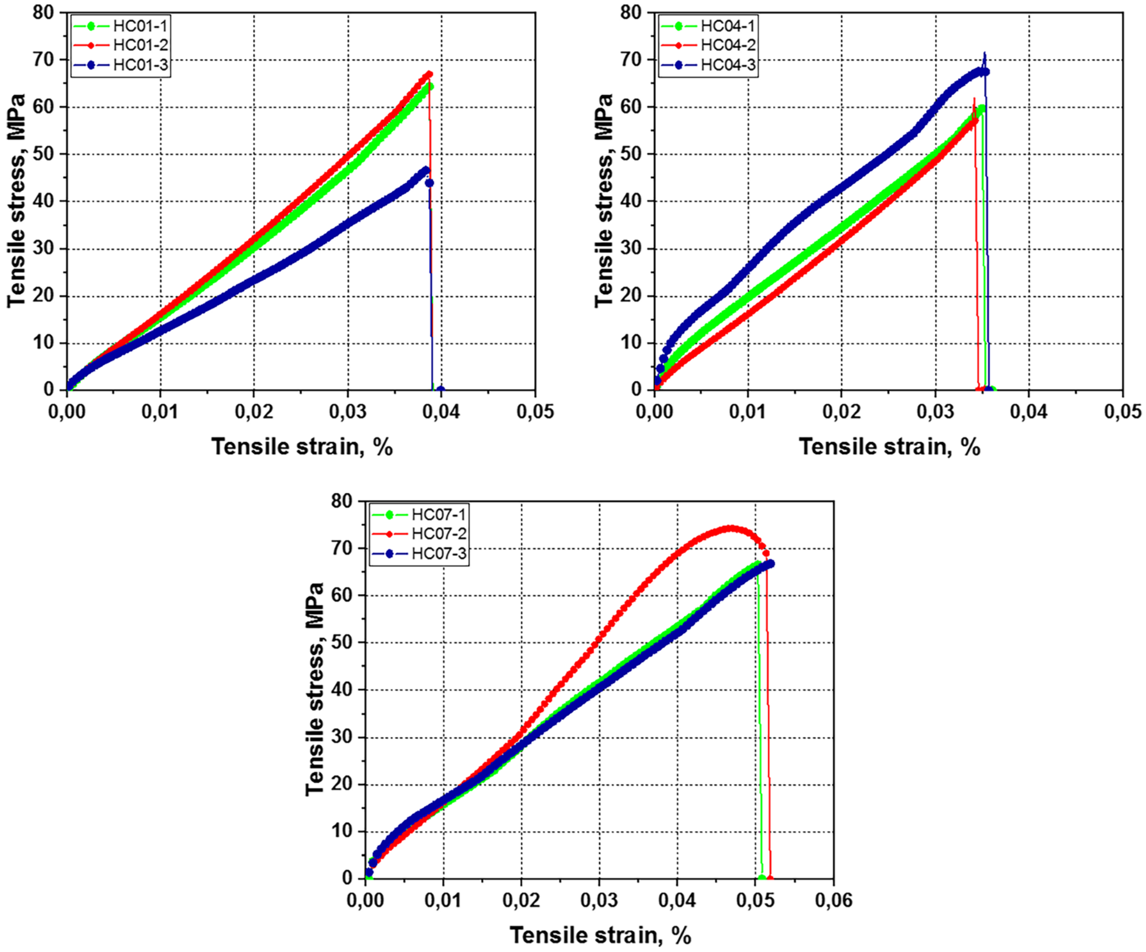


Fig. 14 Stress–strain curves for hybrid laminate composite in tensile tests (fibre content 20 wt%)

properties of hybrid laminate composites. Flexural strength behaved with a similar trend to tensile strength behaviour.

The experimental mechanical properties of the hybrid composites (HC01, HC04 and HC07) having a fibre content of 20 wt% were evaluated, and the values are given in Tables 11 and 12. It was found that the mean tensile strength σ_t, mean tensile modulus E_t, mean flexural strength σ_f and mean flexural modulus E_f of the HC01 (91% of jute and 9% of flax) were found to be 59.37 MPa, 1.84 GPa, 125.92 MPa and 9.97 GPa, respectively. Flax-based (HC07: 80% of jute and 20% of flax) hybrid composites made of 20% fibre significantly improved the mechanical properties (σ_t, E_t, σ_f and E_f). σ_t, E_t, σ_f and E_f for the HC07 hybrid composite were found 69.30 MPa, 2.13 GPa, 99.58 MPa and 10.47 GPa, respectively. From this investigation, it was clear that jute composites gained huge mechanical properties over the matrix material and thus indicated good fibre matrix adhesion.

Finally, Tables 11 and 12 show the comparison between the predicted and experimentally values of mechanical properties of hybrid composites (σ_t, E_t, σ_f and E_f). It is concluded that the results of the comparison prove that predicted values of the mechanical properties of hybrid composites are very close to those experimentally recorded.

7 Conclusion

The present investigation deals about the mechanical properties such as tensile and flexural tests were studied for flax, jute and sisal fibre-reinforced epoxy composites. The important findings are mentioned in the following specific conclusions:

1. In tensile test, the sodium hydroxide NaOH and sodium bicarbonate $NaHCO_3$-treated fibre composites had an increase of 10% in tensile strength, while the tensile modulus increased by 7.7% and 12.4%, respectively.
2. In the flexural test, the flexural properties of the fibre-reinforced epoxy composites are highly influenced by the surface characteristics of fibres. NaOH and $NaHCO_3$ treatments highly enhanced the interfacial adhesion of the fibre with the matrix leading to better flexural properties compared to the untreated fibres.

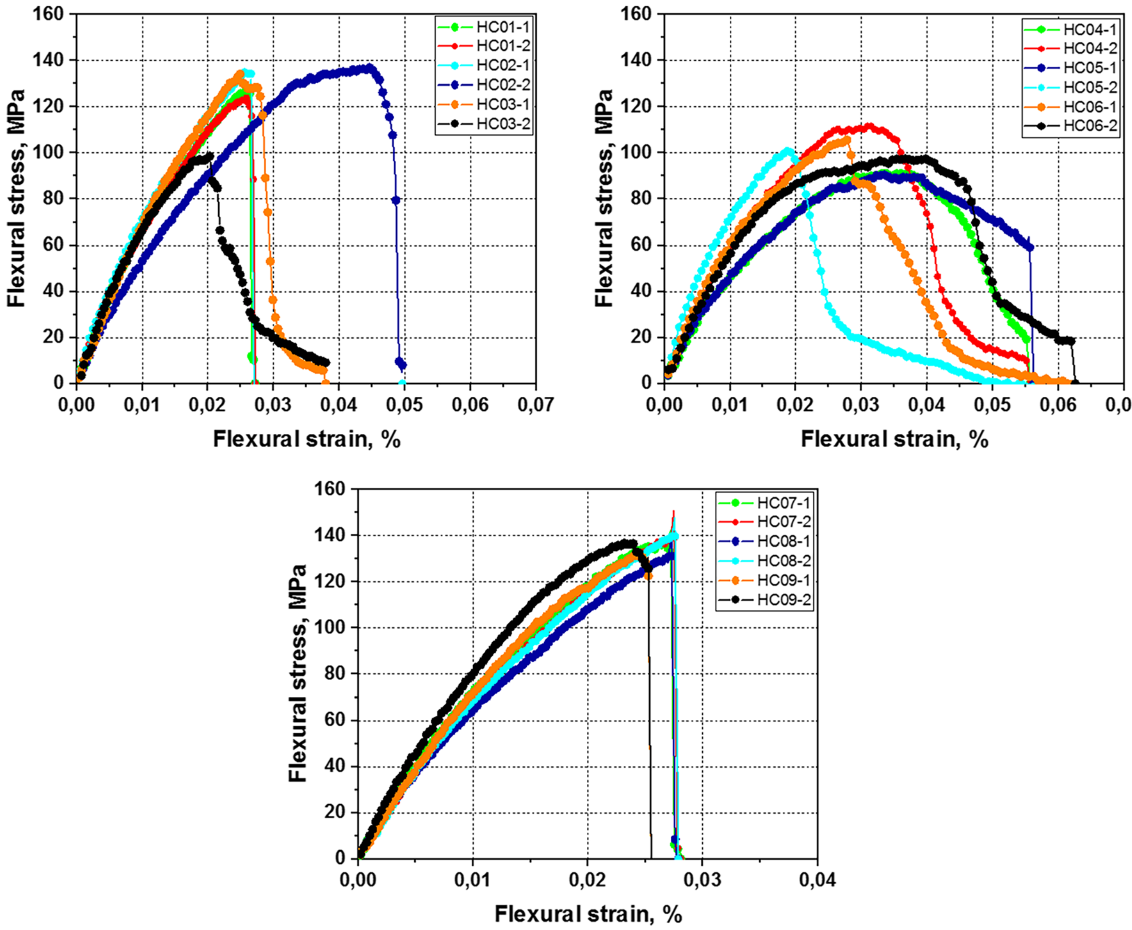


Fig. 15 Stress–strain curves for hybrid laminate composite in flexural tests (fibre content 20 wt%)

3. In summary, the sodium bicarbonate $NaHCO_3$-treated fibres showed significant increase in mechanical properties when compared to NaOH-treated fibres. Also, in flexural test, the specimens tend to behave the same way as in the tensile test and exhibits higher values compared to the tensile properties.
4. The ANOVA shows the following:

(a) The analysis of independent variables using RSM technique allows investigating the influence of each one on the output parameters such as mean tensile strength, mean tensile modulus, mean flexural strength and mean flexural modulus.

(b) According to presented results, the mechanical proprieties are highly affected by volume fraction of fibre, whereas the volume fraction of fibre has a positive effect, but the types of chemical treatment have a very small

Table 11 Tensile properties of hybrid composites, HC01, HC04 and HC07

Sample code	Tensile properties of hybrid composites				Error, %	
	Experimental		Predicted (RMS)			
	Mean tensile strength, σ_t (MPa)	Mean tensile modulus, E_t (GPa)	Mean tensile strength, σ_t (MPa)	Mean tensile modulus, E_t (GPa)	σ_t	E_t
HC01	59.37	1.84	65.23	2.07	9	11.38
HC04	61.56	2.02	65.25	2.07	5.65	2.31
HC07	69.30	2.13	65.30	2.08	5.78	2.35

Table 12 Flexural properties of hybrid composites, HC01, HC04 and HC07

Sample code	Flexural properties of hybrid composites				Error, %	
	Experimental		Predicted (RMS)			
	Mean flexural strength, σ_f (MPa)	Mean flexural modulus, E_f (GPa)	Mean flexural strength, σ_f (MPa)	Mean flexural modulus, E_f (GPa)	σ_f	E_f
HC01	125.92	9.97	131.18	10.14	4.01	1.63
HC04	99.58	10.47	131.15	10.13	24.07	3.37
HC07	136.06	10.07	130.38	10.02	4.36	0.50

influence on mechanical proprieties of fibre-reinforced epoxy composites.

(c) The mathematical models elaborated for σ_t, E_t, σ_f and E_f are very reliable, and they represent an important industrial interest, since they help to make predictions within the range of the actual experimentation.

5. From multi-objective optimization:

(a) Based on the response surface optimization and the desirability method of RSM, the optimization process is done by maximizing both mechanical proprieties (tensile and flexural properties) and is found to be as the following: the type of fibres (X_1) of [− 0.09 and − 0.29], the chemical treatment (X_2) of + 1 and the volume fraction of fibre (X_3) of + 1. The best mechanical proprieties of composites were achieved at the highest volume fraction of fibre and when used the sodium bicarbonate $NaHCO_3$ for treated fibres.

(b) It is concluded that the results of the comparison prove that predicted values of the mechanical properties of hybrid composites are very close to those experimentally recorded.

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