Tensile mechanical properties of natural fibre composites – a statistical approach

Hamdi Laouici • Asma Benkhelladi • Ali Bouchoucha

H. Laouici
Innovative Technologies Laboratory (LTI), Higher National School of Technology, ex biomedical, Dergana 16087, Algiers, Algeria

Corresponding author:
H. Laouici
e-mail: hamdi.aouici@enst.dz
A. Benkhelladi • A. Bouchoucha
Department of Mechanical Engineering, Faculty of Technology Sciences, University of Mentouri Brothers Constantine P.O Box 325 Ain-El-Bey Way, Constantine, Algeria 25017

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 (X1) of [-0.28 and -0.33], the chemical treatment (X2) of -1, the volume fraction of fibre (X3) of 1 and the treatment duration (X4) of [-0.97 and -1] for epoxy resin matrix. Similarly, when used the polyester resin matrix; the type of fibres (X1) of -0.26, the chemical treatment (X2) of -1, the volume fraction (X3) of 0.99 and the sinking time (X4) 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

Acknowledgements
This work was carried out at the Higher National School of Technology, Alger, Algeria as part of a PRFU research project, Code: A11N01ES161220180003. The authors thank the Directorate General of Scientific Research and Technological Development (DGRSDT) of (MESRS) for the financial support.
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 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. [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 NaHCO₃. 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 H₂O₂ 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 (X₁), chemical treatment (X₂), volume fraction of fibre (X₃) and treatment duration (X₄) 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 ±2mm long pieces, washed with distilled water and oven dried at 45 ºC until obtaining a
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].

\[
VF = \frac{W_f / \rho_f}{(W_f / \rho_f) + (W_m / \rho_m)}
\]

(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, \( \rho_f \) and \( \rho_m \) 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 ×4.5 magnifications and 1μm 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±40 μm, 880±80 μm and 17±10 μm, respectively.

![Optical microscopy image of a longitudinal sisal, jute and flax fibres](image)

**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**

| Material       | Physical Properties | Mechanical Properties |
|----------------|---------------------|-----------------------|
| Sisal          |                     |                       |
| Jute           |                     |                       |
| Flax           |                     |                       |
| Epoxy          |                     |                       |
| Polyester      |                     |                       |

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                         |              |
| 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 NaHCO$_3$

Similarly, the second treatment method consisted of soaking the raw of the sisal, jute and flax fibres in 25, 25 and 10 wt% NaHCO$_3$ solution for various times 4, 12 and 24 hrs at room temperature, respectively. The fibres were then taken out of the solution, drained, and washed several times with tap water to remove any residual NaHCO$_3$ traces sticking on the fibre surface. 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 (X₁, type of fibre; X₂, chemical treatment; X₃, volume fraction of fibre; and X₄, treatment duration) at three levels. In addition, there are two qualitative variables (X₁, type of fibre and X₂, chemical treatment) and the other two are quantified (volume fraction of fibre and X₄, 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.

\[
N = 2^n + 2n + Nc = 2^4 + 2\times4 + 5 = 29 \text{ runs} \tag{2}
\]

where \(N\) is the total experimental runs, \(n\) is the number of variables and \(Nc\) 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):

\[
Y = a_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i,j}^{k} b_{ij} X_i X_j + \sum_{i=1}^{k} b_{ii} X_i^2
\]

(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-\( R^2 \) (\( R^2_{\text{adj}} \)), and predicted-\( R^2 \)
(\( R^2_{\text{pred}} \)). 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 (StatEase). 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.

| No | Independent variables                  | Symbol code, \( X_i \) | Units | Levels | Type          |
|----|----------------------------------------|------------------------|-------|--------|---------------|
| 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 |
Independent variables
✓ Type of fibres
✓ Chemical treatment
✓ Volume fraction
✓ Treatment duration

Resins used
✓ Epoxy resin
✓ Polyester resin

Fig. 2. Experimental setup.
### Table 3
Box-Behnken Design (BBD) with independent variables and response values.

| N° | X₁  | X₂  | X₃  | X₄  | Type of fibres | Types of chemical treatment | Volume fraction of fibre (%) | Treatment duration (hrs) | Epoxy resin | Polyester resin |
|----|-----|-----|-----|-----|----------------|-----------------------------|-----------------------------|--------------------------|-------------|----------------|
| 1  | 0   | 0   | 1   | 1   | Jute          | Raw                         | 20                          | 24                       | 59.495      | 54.679         | 1.9618       |
| 2  | 0   | 0   | 0   | 0   | Jute          | Raw                         | 15                          | 12                       | 36.923      | 30.901         | 1.4993       |
| 3  | 0   | 1   | 0   | 1   | Jute          | NaOH                        | 15                          | 24                       | 41.082      | 34.747         | 1.5021       |
| 4  | 0   | 0   | 0   | 0   | Jute          | Raw                         | 15                          | 12                       | 36.923      | 30.901         | 1.4993       |
| 5  | 0   | 0   | -1  | 1   | Jute          | Raw                         | 10                          | 24                       | 25.199      | 22.618         | 1.4621       |
| 6  | -1  | 0   | -1  | 0   | Flax          | Raw                         | 10                          | 12                       | 17.076      | 13.778         | 0.7593       |
| 7  | 0   | -1  | -1  | 0   | Jute          | NaHCO₃                      | 10                          | 12                       | 28.366      | 25.947         | 1.6540       |
| 8  | 0   | 1   | 1   | 0   | Jute          | NaOH                        | 20                          | 12                       | 67.413      | 57.601         | 1.5742       |
| 9  | 0   | 0   | 0   | 0   | Jute          | Raw                         | 15                          | 12                       | 36.923      | 30.901         | 1.4993       |
| 10 | -1  | 0   | 0   | -1  | Flax          | Raw                         | 15                          | 4                        | 32.942      | 24.234         | 1.1124       |
| 11 | 0   | 0   | 0   | 0   | Jute          | Raw                         | 15                          | 12                       | 36.923      | 30.901         | 1.4993       |
| 12 | -1  | 0   | 0   | 1   | Flax          | Raw                         | 15                          | 24                       | 32.942      | 24.234         | 1.3088       |
| 13 | 1   | 0   | 1   | 0   | Sisal         | Raw                         | 20                          | 12                       | 43.840      | 38.720         | 1.7574       |
| 14 | 1   | -1  | 0   | 0   | Sisal         | NaHCO₃                      | 15                          | 12                       | 37.024      | 30.838         | 1.6765       |
| 15 | 0   | 0   | 0   | 0   | Jute          | Raw                         | 15                          | 12                       | 36.923      | 30.901         | 1.4993       |
| 16 | -1  | -1  | 0   | 0   | Flax          | NaHCO₃                      | 15                          | 12                       | 39.717      | 31.649         | 1.4565       |
| 17 | -1  | 0   | 1   | 0   | Flax          | Raw                         | 20                          | 12                       | 59.844      | 56.638         | 1.8817       |
| 18 | 1   | 0   | 0   | -1  | Sisal         | Raw                         | 15                          | 4                        | 34.765      | 23.527         | 1.5393       |
| 19 | 1   | 0   | 0   | 1   | Sisal         | Raw                         | 15                          | 24                       | 34.765      | 23.527         | 1.5393       |
| 20 | 0   | -1  | 0   | -1  | Jute          | NaHCO₃                      | 15                          | 4                        | 46.715      | 37.903         | 1.7309       |
| 21 | 0   | 0   | -1  | -1  | Jute          | Raw                         | 10                          | 4                        | 25.199      | 22.618         | 1.4621       |
| 22 | 1   | 1   | 0   | 0   | Sisal         | NaOH                        | 15                          | 12                       | 22.031      | 12.224         | 1.3624       |
| 23 | 0   | -1  | 1   | 0   | Jute          | NaHCO₃                      | 20                          | 12                       | 59.072      | 62.454         | 1.9810       |
| 24 | -1  | 1   | 0   | 0   | Flax          | NaOH                        | 15                          | 12                       | 36.421      | 28.502         | 1.3450       |
| 25 | 0   | 1   | -1  | 0   | Jute          | NaOH                        | 10                          | 12                       | 45.424      | 16.992         | 1.6726       |
| 26 | 0   | -1  | 0   | 1   | Jute          | NaHCO₃                      | 15                          | 24                       | 53.340      | 42.794         | 1.7191       |
| 27 | 0   | 1   | 0   | -1  | Jute          | NaOH                        | 15                          | 4                        | 55.576      | 40.837         | 1.6420       |
| 28 | 1   | 0   | -1  | 0   | Sisal         | Raw                         | 10                          | 12                       | 26.633      | 16.338         | 1.3659       |
| 29 | 0   | 0   | 1   | -1  | Jute          | Raw                         | 20                          | 4                        | 59.495      | 54.679         | 1.9618       |
3.2. Regression equations

In the empirical approach, prediction of ultimate tensile strength ($\sigma_u$) 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 fibre ($X_1$), 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**

$$\sigma_u = 36.92 - 1.66X_1 + 0.3094X_2 + 15.11X_3 - 0.6557X_4 - 7.23X_1 \times X_1$$

$$+ 7.05X_2 \times X_2 + 5.15X_3 \times X_3 + 3.21X_4 \times X_4$$

$$E = 1.53 - 0.128X_1 - 0.0398X_2 + 0.3162X_3 + 0.00965X_4 - 0.3725X_1 \times X_1$$

$$+ 0.164X_2 \times X_2 + 0.133X_3 \times X_3 + 0.0478X_4 \times X_4$$

**Polyester resin**

$$\sigma_u = 30.90 - 2.88X_1 - 3.33X_2 + 17.21X_3 - 0.0999X_4 - 8.24X_1 \times X_1$$

$$+ 4.04X_2 \times X_2 + 6.71X_3 \times X_3 + 2.13X_4 \times X_4$$

$$E = 1.5 + 0.1148X_1 - 0.0933X_2 + 0.2285X_3 + 0.0037X_4 - 0.1711X_1 \times X_1$$

$$+ 0.1054X_2 \times X_2 + 0.1276X_3 \times X_3 + 0.0586X_4 \times X_4$$

Fig. 3 presents a comparison between predicted and measured values of the ultimate tensile strength ($\sigma_u$) 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 ($\sigma_u$ 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 ($\sigma_u$) 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 ($\leq 0.05$) 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 $\sigma_{\text{epoxy}}$ 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 $E_{\text{epoxy}}$ and 7.37 for $E_{\text{polyester}}$) and low P-values (both < 0.0001 for $E_{\text{epoxy}}$ 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 (adj-$R^2_{\text{epoxy}} = 0.7233$ and adj-$R^2_{\text{epoxy}} = 0.6454$) 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 ($X_3$) 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 ($\sigma_u$).

| 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  | 338.8 | 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 5
Analysis 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 | –         |
| $X_1 \times X_3$ | 0.1146 | 1 | 0.1146 | 3.2    | 0.0886 | 4.37 | Insignificant |
| $X_1 \times X_4$ | 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 |
| $X_1 \times X_3$ | 0.1056 | 1 | 0.1056 | 4.59   | 0.0446 | 8.26   | Significant |
| $X_1 \times X_4$ | 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 ($\sigma_u$ 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 $\sigma_u$ 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 $\sigma_u$ and $E$. Therefore, the volume fraction ($X_3$) seemed to have a major role for improving the mechanical properties ($\sigma_u$ 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 $\sigma_u$. 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 $E$. 

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3.5. Effect of operating parameters on grafting

In order to present the relationship between independent variables (type of fibre \(X_1\), types of chemical treatment \(X_2\) and volume fraction of fibre \(X_3\)) on response parameters (ultimate tensile strength \(\sigma_u\) 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 \((\sigma_u\) 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 NaHCO₃ 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](image-url)
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.
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 $\sim1$ 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   |
| (X₁) : Type of fibres              | In range           | -1                | +1                |
| (X₂) : Types of chemical treatment | In range           | -1                | +1                |
| (X₃) : Volume fraction of fibre (%)| In range           | -1                | +1                |
| (X₄) : Treatment duration (hrs)    | In range           | -1                | +1                |

Ultimate tensile strength $\sigma$ (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 | Remarks |
|---------|---------------|----------------------|--------------|---------|
|         | $X_1$ | $X_2$ | $X_3$ | $X_4$ | Ultimate tensile strength $\sigma$ (MPa) | Young’s modulus $E$ (GPa) | D | |
| Epoxy resin |           |           |           |           | 67.4484 | 2.2211 | 1 | Selected |
| 1       | -0.28  | -1.00  | 1.00  | -0.97 | 67.4236 | 2.2165 | 1 | |
| 2       | -0.33  | -0.99  | 1.00  | +1.00 | 66.9613 | 2.1954 | 0.9966 | |
| 3       | -0.47  | -1.00  | 1.00  | -1.00 | 59.8618 | 1.9188 | 0.9488 | |
| Polyester resin |           |           |           |           | 63.8168 | 2.0487 | 1 | Selected |
| 1       | -0.26  | -0.97  | 0.99  | -0.94 | 63.5924 | 2.0431 | 1 | |
| 2       | -0.26  | -0.95  | 0.99  | -0.93 | 60.6524 | 1.9988 | 0.9488 | |
| 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 |           |           |           |           | 72% of jute and 28% of flax | NaHCO₃ | 20 | 4h14 |
| 1       | -0.28  | -1       | +1       | -0.97  | 67% of jute and 33% of flax | NaHCO₃ | 20 | 4h14 |
| 2       | -0.33  | -1       | +1.00    | -1.00  | 67% of jute and 33% of flax | NaHCO₃ | 20 | 4h14 |
| Polyester resin |           |           |           |           | 74% of jute and 26% of flax | NaHCO₃ | 19.80 | 4h28 |
| 1       | -0.26  | -1       | +0.99    | -0.94  | 74% of jute and 26% of flax | NaHCO₃ | 19.80 | 4h28 |
| 2       | -0.26  | -1       | +0.99    | -0.93  | 74% of jute and 26% of flax | NaHCO₃ | 19.80 | 4h28 |
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 NaHCO₃, 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

| 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 $\sigma_u$ (MPa) | Young’s modulus $E$ (GPa) | $\sigma_u$ (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 \([-0.28 \text{ and } -0.33]\), the chemical treatment \((X_2)\) of \(-1\), the volume fraction of fibre \((X_3)\) of 1 and the treatment duration \((X_4)\) of \([-0.97 \text{ and } -1]\) 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 \([-0.94 \text{ and } -0.93]\).

(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 \((8.96\%, 3.86\%, 9.90\% \text{ and } 2.92\%, 10.04\%, 21.46\%)\) respectively for epoxy resin and by \((0.27\%, 5.68\%, 14.9\% \text{ and } 2.02\%, 6.03\%, 5.36\%)\) 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.

References
1. Ramzy A, Beermann D, Steuernagel L, Meiners D, Ziegmann G. Developing a new generation of sisal composite fibres for use in industrial applications. Compos Part B: Engineering 2014;66:287–98.
2. Orue A, Jauregi A, Peña-Rodriguez C, Labidi J, Eceiza A, Arbelaitz A. The effect of surface modifications on sisal fibre properties and sisal/poly (lactic acid) interface adhesion. Compos Part B: Engineering 2015; 73:132–8.
3. Arbelaitz A, Fernández B, Ramos JA, Retegi A, Llano-Ponte R, Mondragon I. Mechanical properties of short flax fibre bundle/polypropylene composites: influence of matrix/fibre modification, fibre content, water uptake and recycling. Composites Science and Technology 2005;65:1582–92.
4. Lu T, Liu S, Jiang M, Xu X, Wang Y, Wang Z, Gou J, Hui D, Zhou Z. Effects of modifications of bamboo cellulose fibres on the improved mechanical properties of cellulose reinforced poly (lactic acid) composites. Compos Part B: Eng 2014;62:191–7.
5. Awal A, Rana M, Sain M. Thermorheological and mechanical properties of cellulose reinforced PLA bio-composites. Mech Mater 2015;80:87–95.
6. Yu Y, Huang X, Yu W. A novel process to improve yield and mechanical performance of bamboo fibre reinforced composite via mechanical treatments. Compos Part B: Eng 2014;56:48–53.
7. Li X, Tabil LG, Panigrahi S. Chemical treatments of natural fiber for use in natural fiber-reinforced composites: a review. Journal of Polymers and the Environment 2007; 15:25–33.

8. Belaadi A, Bourchak M, Aouici H. Mechanical properties of vegetal yarn: Statistical approach. Composites Part B 2016;106:139–153.

9. Kiruthika AV. A review on physico-mechanical properties of bast fibre reinforced polymer composites. Journal of Building Engineering 2017;9:91–99.

10. George M, Bressler DC. Comparative evaluation of the environmental impact of chemical methods used to enhance natural fibres for composite applications and glass fibre based composites. Journal of Cleaner Production 2017;149:491–501.

11. Akil HM, Omar MF, Mazuki AAM, Safiee S, Ishak ZAM, Abu Bakar A. Kenaf fiber reinforced composites: A review. Materials & Design 2011;32:4107–4121.

12. Jawaid M, Abdul Khalil HPS, Abu Bakar A. Woven hybrid composites: Tensile and flexural properties of oil palm-woven jute fibres based epoxy composites. Materials Science and Engineering: A 2011;528:5190–5.

13. Kistaiah N, Udaya Kiran C, Ramachandra Reddy G, Sreenivasa Rao M. Mechanical characterization of hybrid composites: A review. Journal of Reinforced Plastics and Composites 2014;33(14):1364–1372.

14. Nunna S, Ravi Chandra P, Shrivastava S, Jalan AK. A review on mechanical behavior of natural fiber based hybrid composites. Journal of Reinforced Plastics and Composites. 31(11) 759–769.

15. Dittenber DB, GangaRao HVS. Critical review of recent publications on use of natural composites in infrastructure. Composites: Part A 2012; 43:1419–1429.

16. Sathishkumar TP, Navaneethakrishnan P, Shankar S, Rajasekar R, Rajini N. Characterization of natural fiber and composites – A review. Journal of Reinforced Plastics and Composites 32(19):1457–1476.

17. Saba N, Paridah MT, Jawaid M. A Review on Potentiality of Nano Filler/Natural Fiber Filled Polymer Hybrid Composites. Polymers 2014, 6, 2247-2273; doi:10.3390/polym6082247.

18. Schneider JP, Karmaker AC. Mechanical performance of short jute fibre reinforced polypropylene. Journal of Materials Science Letters 1996;15(3): 201–202.

19. Venkateshwaran N, Elayaperumal A, Sathiya GK. Preidction of tenstile propreties of hybrid-natural fibre composites. Composites Part B: Engineering 2012;43(2):793–796.
20. Venkateshwaran N, ElayaPerumal A, Alavudeen A, Thiruchitrambalam M. Mechanical and water absorption behaviour of banana/sisal reinforced hybrid composites. Materials & Design 2011;32(7):4017–4021.

21. Boopalan M, Niranjanaa M, Umapathy MJ. Study on the mechanical properties and thermal properties of jute and banana fibre reinforced epoxy hybrid composites, Compos Part B: Engineering 2013;51:54–57.

22. Asumani OML, Reid RG, Paskaramoorthy R. The effects of alkali–silane treatment on the tensile and flexural properties of short fibre non-woven kenaf reinforced polypropylene composites. Composites Part A: Applied Science and Manufacturing 2012;43:1431–1440.

23. Ray D, Sarkar BK, Rana AK, Bose NR. Effect of alkali treated sisal fibres on composite properties. Bulletin of materials science 2001; 24: 129–135.

24. Mishra S, Misra M, Tripathy SS, Nayak SK, Mohanty AK. Graft Copolymerization of Acrylonitrile on Chemically Modified Sisal Fibers. Macromolecular Materials Engineering 2001;286:107.

25. Morrison Iii W.H, Archibald DD, Sharma HSS, Akin DE. Chemical and physical characterization of water- and dew-retted flax fibers. Industrial Crops and Products 2000;12(1):39–46.

26. Rajesh G, Prasad AVR. Tenstile propreties of successive alkali treated short jute fibre reinforced PLA composite, Procedia Mat. Sci. 5, 2188-2196. Doi: 10.1016/j.mspro.201.07.425.

27. Roy A, Chakraborty S, Prasad Kundu S, Kumar Basak R, Basu Majumder S, Adhikari B. Improvement in mechanical properties of jute fibres through mild alkali treatment as demonstrated by utilisation of the Weibull distribution model. Bioresource Technology 2012;107:222–228.

28. Vijaya Ramnath B, Manickavasagam VM, Elanchezhian C, Vinodh Krishna C, Karthik S, Saravanan K. Determination of mechanical properties of intra-layer abaca–jute–glass fiber reinforced composite. Materials and Design 2014;60:643–652.

29. Palani Kumar K, Shadrach Jeya Sekaran A. Some natural fibers used in polymer composites and their extraction processes: A review. Journal of Reinforced Plastics and Composites 2014;33(20):1879–1892.

30. Belaadi A, Bourchak M, Aouici H. Mechanical properties of vegetal yarn: Statistical approach. Composites Part B 2016;106:139–153.
31. Arthanarieswaran VP, Kumaravel A, Kathirselvam M. Evaluation of mechanical properties of banana and sisal fiber reinforced epoxy composites: Influence of glass fiber hybridization. Materials and Design 2014;64:194–202.
32. Faruka O, Bledzki AK, Fink HP, Sain M. Biocomposites reinforced with natural fibers: 2000–2010. Progress in Polymer Science 2012;37:1552–1596.
33. John MJ, Anandjiwala RD. Chemical modification of flax reinforced polypropylene composites. Composites Part A: Applied Science and Manufacturing 2009;40:442–8.
34. Mirbagheri J, Tajvidi M, Ghasemi I, Hermanson JC. Prediction of the Elastic Modulus of Wood Flour/Kenaf Fibre/Polypropylene Hybrid Composites. Iranian Polymer Journal 2007;16(4): 271–278.
35. Suresh kumar SM, Duraibabu D, Subramanian K. Studies on mechanical, thermal and dynamic mechanical properties of untreated (raw) and treated coconut sheath fiber reinforced epoxy composites. Mater Des 2014; 59: 63c69.
36. Box GEP, Wilson KB. On the experimental attainment of optimum conditions, Journal of the Royal Statistical Society: Series B (Methodological) 1951;13(1):1–45.
37. Aouici H, Yallese MA, Chaoui K, Mabrouki T, Rigal J-F. Analysis of surface roughness and cutting force components in hard turning with CBN tool: prediction model and cutting conditions optimization. Measurement 2012;45:344–353.
38. Ferreira SLC, Bruns RE, Ferreira HS, Matos GD, David JM, Brandao GC, da Silva EGP, Portugal LA, dos Reis PS, Souza AS, dos Santos WNL. Box-Behnken design: An alternative for the optimization of analytical methods. Analytica Chimica Acta 2007;597: 179–186.
39. Aouici H, Bouchelaghem H, Yallese MA, Elbah M, Fnides B. Machinability investigation in hard turning of AISI D3 cold work steel with ceramic tool using response surface methodology, International Journal of Advanced Manufacturing Technology 2014;73:1775–1788.
40. Ben Brahim S, Ben Cheikh R. Influence of fibre orientation and volume fraction on the tensile properties of unidirectional Alfa-polyester composite. Composites Science and Technology 2007;67:140–147.
41. Idiculla M, Joseph K, Thomas S. Mechanical performance of short banana/sisal hybrid fiber reinforced polyester composites. J Reinf Plast Compos 2010; 29(1): 12–29.
42. Ratna Prasad AV, Mohana Rao K. Mechanical properties of natural fibre reinforced polyester composites: jowar, sisal and bamboo. Mater Des 2011;32:4658–63.
43. Vilay V, Mariatti M, Mat Taib R, Todo M. Effect of fiber surface treatment and fiber loading on the properties of bagasse fiber-reinforced unsaturated polyester composites. Compos Sci Technol 2008;68:631–8.

44. K. Murali Mohan Rao, K. Mohana Rao, A.V. Ratna Prasad. Fabrication and testing of natural fibre composites: Vakka, sisal, bamboo and banana. Materials and Design 2010; 31:508–513.

45. Rout J, Misra M, Tripathy SS, Nayak SK, Mohanty AK. The influence of fibre treatment on the performance of coirpolyester composites. Compos Sci Technol 2001; 61:1303–10.

46. Herrera-Franco PJ, Valadez-González A. A study of the mechanical properties of short natural-fiber reinforced composites. Compos Part B 2005; 36:597–608.

47. Carmisciano S, De Rosa IM, Sarasini F, Tamburrano A, Valente M. Basalt woven fiber reinforced vinylester composites: Flexural and electrical properties. Materials & Design 2011; 32:337–42.

48. Paiva MC, Ammar I, Campos AR, Ben Cheikh R, Cunha AM. Alfa fibres: Mechanical, morphological and interfacial characterization. Composites Science and Technology 2007; 67:1132–38.

49. Zou H, Wang L, Gan H, Yi C. Effect of fiber surface treatments on the properties of short sisal fiber/poly (lactic acid) biocomposites. Polymer Composites 2012;33:1659–66.