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Statistical Modelling and Mechanical Behaviour of Biochar-Reinforced Hybrid Composites: A Response Surface Approach

Velmurugan Ganesan[1*], *Jasgurpreet Singh* Chohan[2], *Gowthami* Duttaluru[3], *Vasa* Anil Kumar [4], *Singarapu* Lokesh Sai[5], *Bommepalli* Harsha Vardhan Reddy[6]

[1,3,4,5 & 6]Department of Mechanical Engineering, School of Engineering, Mohan Babu University, Tirupati-517 102, Andhra Pradesh, India.
[2]Department of Mechanical Engineering, University Centre for Research and Development, Chandigarh University, Gharuan, Mohali-140 413, Punjab, India.

Abstract. The present research investigates the mechanical properties (tensile, flexural & impact) of kenaf-flax-based hybrid composites with the incorporation of various weight percentages of coconut shell biochar. The hybrid composites were fabricated through compression molding with the following parameters: varying flax to kenaf ratios (0-100%), biochar content (2-6 wt.%), and fiber loading (20-40 wt.%) according to a Response Surface Methodology (RSM) and Box-Behnken Design (BBD). The ANOVA analysis of the composites confirmed that the quadratic models were statistically significant for high coefficients of determination, i.e., R^2>98. Among the three variables, the flax-to-kenaf weight ratio and fiber loading were found to be the maximum leading variables, while the biochar also provides the best results up to 4 wt.%. The RSM method demonstrates the maximum mechanical properties (tensile 110 MPa, flexural 150 MPa, and impact 13 kJ/m²) were achieved at 60% of flax, 40% of kenaf, 35 wt.% of fiber loading, and 4 wt.% of biochar. The fracture analysis of the hybrid composites was evaluated through scanning electron microscopy (SEM). The obtained results of hybrid composites reveal that the inclusion of biochar content enhanced the performance of natural fiber composites, providing sustainable alternatives for structural applications.

1 Introduction

Throughout history, fiber crops have been an integral part of human civilization. Humans have historically gathered natural resources to make textiles and ropes. Societies subsequently learned how to grow these kinds of crops.

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* Corresponding author: velresearch032@gmail.com

Over the years, people kept on trying to cultivate natural fiber crops, which are among the oldest known domesticated plants [1]. Numerous fiber crop varieties were created via selective breeding in response to societal demands and ideals. Natural fibers are increasingly being used in composite goods. These polymer composites have several advantages over artificial fibers, such as lower tool wear, lower density, reduced cost, better affordability, and improved environmental performance [2]. he most commonly used plant-based materials are bast fibers, which include cotton, hemp, flax, kenaf, and jute. One reason for this growing interest is that natural materials exhibit higher specific resistivity and comparable specific elasticity to glass fibers. Theoretically, due to their inherent characteristics and lower-cost resources, these bio-fibers provide suitable specific properties and stiffness at a reduced cost [3].

Kenaf is one of the numerous organic (plant) fibers used as strengthening fibers in Polymeric Matrix Composites (PMCs). It was recently discovered that kenaf (Hibiscus cannabinus, L., family Malvaceae) is an essential source of fiber for plastics and other uses in manufacturing. Overall, the kind of fiber, its arrangement (unidirectional or random), its quantity and shape (fabric or fiber), and the kind of mixing or plasticizer employed all affect the tension and flexural characteristics of kenaf-impregnated polymers. For instance, Ochi [4] found that up to 50% fiber percentage increases the flexural and tensile strength of PLA materials linearly supplemented with kenaf. Kenaf strengthening materials had tensile and flexural properties of around 231 MPa and 274 MPa, respectively, in samples that had a 60% fiber content. Nishino et al. [5] conducted a more recent investigation on the impact of fiber content on the mechanical characteristics of kenaf fiber-strengthened composite. In general, the amount of reinforcing fiber employed in the composites determines their ideal tensile characteristics and Young's modulus. Despite the notable benefits of kenaf fiber as a reinforced material for sustainability because of its specific strength, biodegradability, and low cost, it does have some challenges. Kenaf fiber has high moisture absorption, resulting in weaker mechanical properties and moisture-related degradation [6]. Moreover, its high variability in fiber morphology and low interface adhesion with the polymer matrix limit its use in structural applications, as it cannot withstand much load. Flax fibers are often added to kenaf fibers to address these issues. Flax fibers offer greater tensile strength, stiffer material, and low moisture absorbance, which, in turn, increases the durability and mechanical performance of the hybrid composites [7]. Therefore, the addition of flax fibers to kenaf fibers not only balances the mechanical properties and mitigates moisture absorption but also makes the system more favorable for structural and engineering applications.

According to the previous literature, the addition of flax fiber to other natural fibers like kenaf-based composites improves the mechanical properties. Flax has proven to provide higher tensile and flexural strength over other bast fibers due to superior cellulose content and lower microfibril angle, which aid in stiffness and load-bearing capacity [8]. It is well documented that flax and other natural fibers, such as kenaf or jute, when hybridized, result in improved tensile, flexural, and impact resistance. In addition, biochar has recently emerged as a filler with reinforcing properties that are beneficial when incorporated in polymer and natural-fiber composites. It has been demonstrated that the specific surface area, porosity, and thermal stability of biochar can strengthen the bonding, respectively, due to an increase in ductility and modulus. Likewise, Das and co-workers [9] published results where 24 wt% biochar was added to a wood-polypropylene composite, increasing tensile modulus from 3.1 GPa to 3.5 GPa and flexural modulus from 2.3 GPa to 3.4 GPa, demonstrating the reinforcing capacity biochar has increased. Consequently, the incorporation of biochar into flax–kenaf hybrid composites increases not only mechanical strength and stiffness but also dimensional stability and sustainability as well, reinforcing the value of the flax–kenaf–biochar hybrid.

In this work, biochar from coconut shells was used to reinforce flax-kenaf hybrid composites, whose mechanical properties—tensile, flexural, and impact strength—were evaluated. The flax-kenaf hybridization was done with the intention to address the limitations associated with single-fiber composites, while the addition of biochar was expected to improve bonding and stress transfer because of its porous and high surface area structure. To optimize and model the composite response, Response Surface Methodology (RSM) was used with a Box-Behnken Design (BBD). The flax-to-kenaf ratio, biochar content, and fiber loading to matrix were set as three independent process variables (0–100%, 2–6 wt%, and 20–40 wt%, respectively). The BBD produced 15 experimental runs, replicating center points for error estimation and capturing non-linear effects. The second-order quadratic polynomial was fitted to the data with the model adequacy assessed by ANOVA, R^2, and lack-of-fit tests. Subsequent multi-response optimization increased the simultaneous maximal values of tensile, flexural, and impact strengths, with experimental predictions validating the optimal values. This approach founds a systematic background for modifying the mechanical outcome of hybrid composites with nominal investigational trials. To my knowledge, no study has explored the optimization of the mechanical response by integrating flax–kenaf hybridization with biochar reinforcement together with a Response Surface Methodology (RSM)-based Box–Behnken Design (BBD) framework (RSM-BBD) for the simultaneous optimization of tensile, flexural, and impact strength. The novelty of the present work lies in combining this specific hybrid material system with a systematic statistical optimization approach, which has not been reported in earlier studies. Other than the selected parameters, the study does not emphasize any additional mechanical or functional properties.

2 Experimental Works

2.1 Material

Flax and kenaf fibers, with a density of 1.29 g/cm³ and 300 gsm, were provided by Deekshi Natural Fiber Industry, Salam, Tamil Nadu, India. Both fibers have a smooth surface texture and are suitable to be used as surface or backing plies. Coconut biochar with a size of 50 µm was procured from Green Solution, Madurai, Tamil Nadu, India. In the current research, the max clear grade epoxy resin is used as a matrix material. Before fabricating the composite materials, both natural fibers were pretreated with NaOH solution for 4 hrs at normal atmospheric temperature to improve the interfacial adhesion between the matrix and reinforcement. Photographic pictures of matrix components, filler, and reinforced materials are shown in Figure 1.

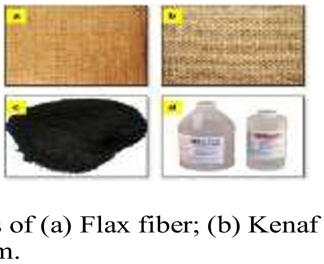


Fig.1. Photographic images of (a) Flax fiber; (b) Kenaf fiber; (c) Coconut shell biochar & (d) Matrix system.

2.2 Response Surface Methodology (RSM) Design

Response surface methodology (RSM) employing a Box-Behnken Design (BBD) was used to optimize the mechanical attributes of flax-kenaf-biochar hybrid materials. It was decided that three independent factors relative to the material and process be (A) flax-to-kenaf fiber ratio, (B) biochar content (wt%), and (C) total fiber loading in the matrix (wt%). Each of these parameters was set to three levels, which can be coded as -1, 0, and +1 as shown in Table 1. Based on the BBD, a total of 15 experimental runs were obtained. 3 replicate center points were included to capture experimental noise and to test the goodness of fit of the quadratic model [10,11]. The optimization responses focused on the tensile and flexural strength as well as the impact strength of the composites. The experimental data were fitted to a second-order polynomial regression, and the adequacy of the model was tested using ANOVA, and the residuals were examined using diagnostic plots. Table 1 reveals the processing parameters of hybrid composites.

Table 1. RSM Design Process Parameters

Sl.No	**Parameters**	**Symbols**	**Levels**		
			-1	**0**	**+1**
1	Flax-to- Kenaf ratio (%)	A	0:100	50:50	100:0
2	Biochar content (%)	B	2	4	6
3	Fiber loading (%)	C	20	30	40

2.3 Composite Fabrication

The flax-kenaf-biochar hybrid composites were produced using the compression molding method. The flax and kenaf fibers as well as the coconut shell biochar and epoxy resin were measured based on the experimental combinations obtained from the Box–Behnken Design (BBD) of RSM. The fibers were cut to specific lengths and placed in the mold in a way that ensured alignment and uniform distribution.

A mechanical stirrer was used at 1000 rpm for 30 minutes to evenly mix the epoxy resin with the dispersive biochar of coconut shells, which is 50 micrometers in size. The biochar–epoxy mixture was meticulously absorbed in the prepreg fiber mats and underwent hand lay-up to eliminate air pockets and achieve thorough wetting. This was followed by a compression stage in a mold that had been preheated to 80°C and subjected to a constant pressure of 40 bar for 45 minutes [12]. The composites were then kept at room temperature for 24 hours to perform post-curing. The laminate composites were then demolded, polished to the required specimen sizes, and preconditioned before testing. The RSM experimental design matrix parameters were adhered to during fabrication to achieve statistically reliable results for the optimization study.

2.4 Mechanical characterization

The tensile properties of both original and hybridized composites were measured in accordance with the ASTM D3039-76 standard. The dimensions of the object being studied were 250 x 25 x 3 mm. ASTM D3039-76 states that the crosshead velocities were 2 mm/min and the measurement instrument length was 150 mm during the course of the investigation. The Instron-3389 Universal Testing Apparatus was used to perform the evaluation. The ASTM D7264 standard was used for this bending evaluation. The dimensions of the object being tested were 70 x 12.7 x 3 mm. The investigation's length of span was mostly 60 mm, and the depth proportion was 20:1. The impact strength of polymeric hybrid composites has been determined using the ASTM D256 criteria. A sample with dimensions of 65 x 12.7 x 3 mm was used for the assessment. Every specimen underwent three trials, and the mean outcome was recorded. A scanning electron microscope (SEM) was used to evaluate the samples' ruptured regions.

3 Results and Discussion

3.1 Tensile strength

It was also found that for the case of ANOVA, the tensile strength of the flax–kenaf–biochar hybrid composites were highly dependent on certain input variables. Furthermore, the quadratic model was very important on its own ($F = 61.10$, $p < 0.0001$) with an equally high coefficient of determination ($R^2 = 0.9910$) and adjusted R^2 of 0.9748. From this, we can determine the great precision of modeled versus empirical data. The data's lack of fit did not seem significant ($p = 0.1642$), which casts doubt on model validity. Of the linear factors, the ratio of flax to kenaf (A) was the most significant ($p < 0.0001$), followed by the fiber (C) ($p = 0.0004$) and the presence of biochar (B) ($p = 0.0016$). It's easy to see that flax will always have relatively much greater tensile stiffness than kenaf, while kenaf, on the other hand, will improve tensile ductility; hence, the ratio will always have a great role to play in tensile performance [13]. Figure 2 shows the surface plots of the tensile strength of a hybrid composite.

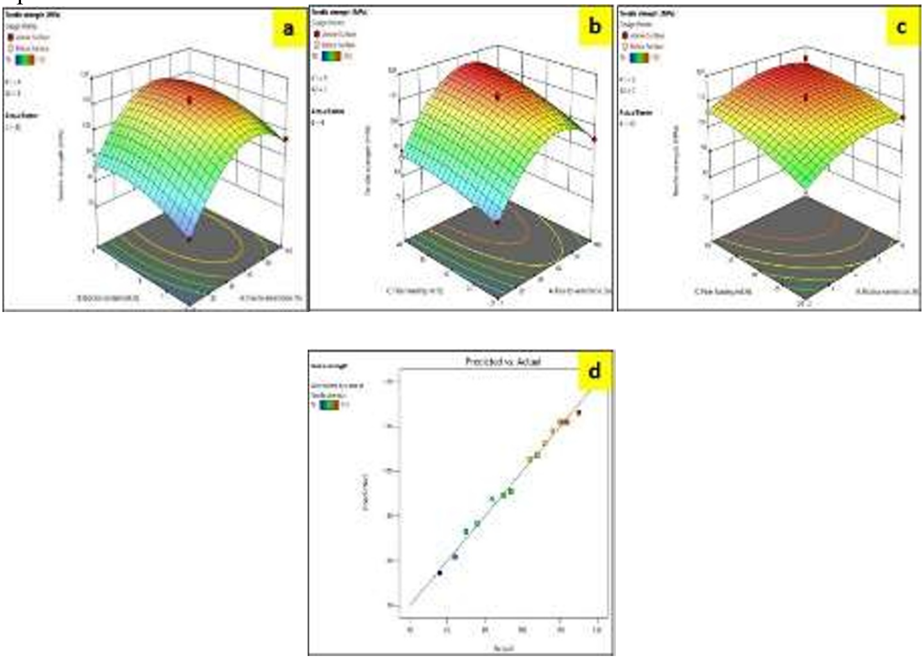


Fig. 2. Surface and predicted vs actual plots of tensile strength based on various process parameters.

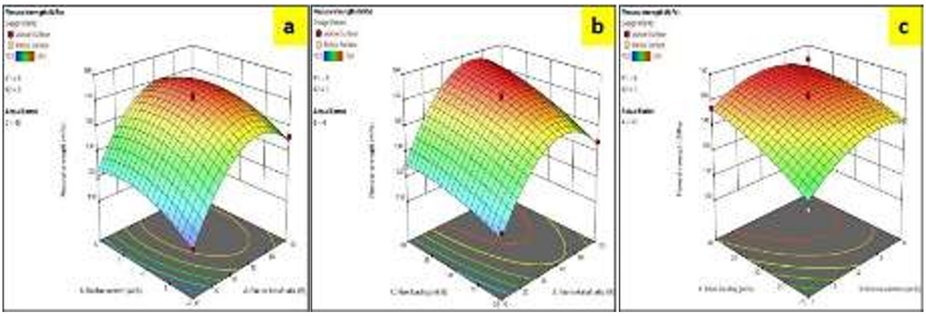
The quadratic A^2 and B^2 ($p < 0.0001$ and $p = 0.0047$, respectively) were also significant, which indicates the range of levels is not linear and is more complex than that. The flexible response surface graphs showed that with the constant increase in the flax ratio, tensile strength also increased; however, it did reach a maximum of around 60% before dropping due to poor fiber-fiber bonding and stress transfer. Concerning the results, the maximum tensile strength of 115 MPa and flexural strength of 350 MPa are obtained with an average weight of 60% flax, 40% kenaf, and 35% of total fiber loading with 4 wt.% of biochar; no infusion pores were observed with the naked eye. The concordance with earlier cited works of the composite fillers, particularly with respect to the filler percent and their ratio, is remarkable [14]. It undeniably affirms the augmented tension results of the synergism, which were optimized through the RSM technique of composite flax and kenaf fibers and added biochar from coconut shells. Table 2 indicates the ANOVA results for tensile strength based on process parameters.

Table 2. ANOVA results for tensile strength based on process parameters

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1952.25	9	216.92	61.1	0.0001	significant
A-Flax-to-kenaf ratio	595.12	1	595.12	167.64	< 0.0001	
B-Biochar content	136.12	1	136.12	38.35	0.0016	
C-Fiber loading	242	1	242	68.17	0.0004	
AB	1	1	1	0.2817	0.6183	
AC	12.25	1	12.25	3.45	0.1223	
BC	2.25	1	2.25	0.6338	0.4621	
A^2	915.92	1	915.92	258.01	< 0.0001	
B^2	83.31	1	83.31	23.47	0.0047	
C^2	14.77	1	14.77	4.16	0.0969	
Residual	17.75	5	3.55			
Lack of Fit	15.75	3	5.25	5.25	0.1642	not significant
Pure Error	2	2	1			
Cor Total	1970	14				

3.2 Flexural strength

It was found that the developed quadratic model for flexural strength had statistical significance (F = 36.83, p = 0.0005), while the coefficient of determination remained high ($R^2 = 0.9851$) with adjusted R^2 at 0.9584, which confirmed that the model explained more than 95% of the data variability. The lack of fit was not significant (p = 0.0736), which suggested the model was appropriate for the captured data. Among the main effects, the most significant factor was the flax-to-kenaf ratio (A) (p = 0.0001), followed by fiber loading (C) (p = 0.0012) and then biochar (B) (p = 0.0073). This implies that the addition of biochar to composites was effective in controlling the flexural behavior of the composites, since with sufficient fiber volume and a balanced fiber architecture, biochar was able to effectively regulate the overall volume contribution [15]. Figure 3 shows the surface plots of the flexural strength of a hybrid composite.



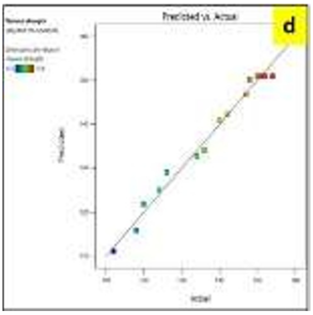


Fig. 3. Surface and predicted vs actual plots of flexural strength based on various process parameters

The terms quadratic A^2 ($p < 0.0001$) and B^2 ($p = 0.0076$) were also significant, thus indicating that the optimal levels for both fiber ratio and biochar addition are present and non-linear in nature. Having a certain amount of fiber ratio and biochar in the composites, as shown in the response surface plots, flexural strength increased as the flax fraction increased and peaked at about 60%, after which the fibers began to collapse, causing a slight reduction due to interfacial mismatch. With a certain amount of biochar in the composites, as shown in the response surface plots, flexural strength increased as the biochar fraction increased. However, clustering reduced the transfer efficiency, and beyond about 3–4 wt% biochar the improvement was limited. Fiber loading had a strong positive effect, with flexural strength peaking at about 35–38 wt% and remaining sustained at about 40 wt%. At these levels, stress transfer between fiber and matrix was maximized, while below 30–35 wt% the load-bearing efficiency dropped due to voids and insufficient wetting [16].

The optimum flexural strength of around 150–155 MPa was obtained for the ratio of 60% flax, 40% kenaf, 3–4 wt% biochar, and 35 wt% total fiber loading. These results prove that both the ratio and content of the fibers, together with the reinforcing action of biochar, strongly influence flexural performance. The hybrid flax–kenaf architecture combined with biochar provided a moderate but effective reinforcement, improving stiffness and confirming the sensitivity of flexural properties to fiber–matrix interactions [17]. Therefore, in addition to enhancing stiffness, the addition of coconut shell biochar reduced the moisture sensitivity of the natural fibers, resulting in a composite that is more dimensionally stable and mechanically stronger. Table 3 indicates the ANOVA results for flexural strength based on process parameters.

Table 3. ANOVA results for flexural strength based on process parameters

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	2668.68	9	296.52	36.83	0.0005	significant
A-Flax-to-kenaf ratio	882	1	882	109.57	0.0001	
B-Biochar content	153.12	1	153.12	19.02	0.0073	
C-Fiber loading	351.12	1	351.12	43.62	0.0012	
AB	4	1	4	0.4969	0.5123	
AC	16	1	16	1.99	0.2177	
BC	20.25	1	20.25	2.52	0.1736	
A^2	1146.98	1	1146.98	142.48	< 0.0001	
B^2	150.06	1	150.06	18.64	0.0076	
C^2	20.83	1	20.83	2.59	0.1686	
Residual	40.25	5	8.05			
Lack of Fit	38.25	3	12.75	12.75	0.0736	not significant
Pure Error	2	2	1			

Cor Total	2708.93	14				

3.3 Impact strength

The impact strength of the model has always been confirmed with prediction, and experimental values showed great agreement ($R^2 = 0.9879$, Adj-$R^2 = 0.9662$). It is worth noting that the lack of fit, while significant ($p=0.0486$), is more attributable to the response's sensitivity to impact strength because of the defects like voids and micro-cracks. The ANOVA impact strength of fiber loading ($p = 0.0004$) was the most important, then biochar ($p = 0.0043$) and flax-to-kenaf ratio ($p = 0.0074$), with the A × C interaction also significant ($p = 0.0355$). The A^2, B^2, and C^2 showed positive quadratic effects, confirming the presence of optimum values for each factor. Surface profiles of the blended material's impact resistance are shown in Figure 4. The impact strength ANOVA findings dependent on process factors are shown in Table 4.

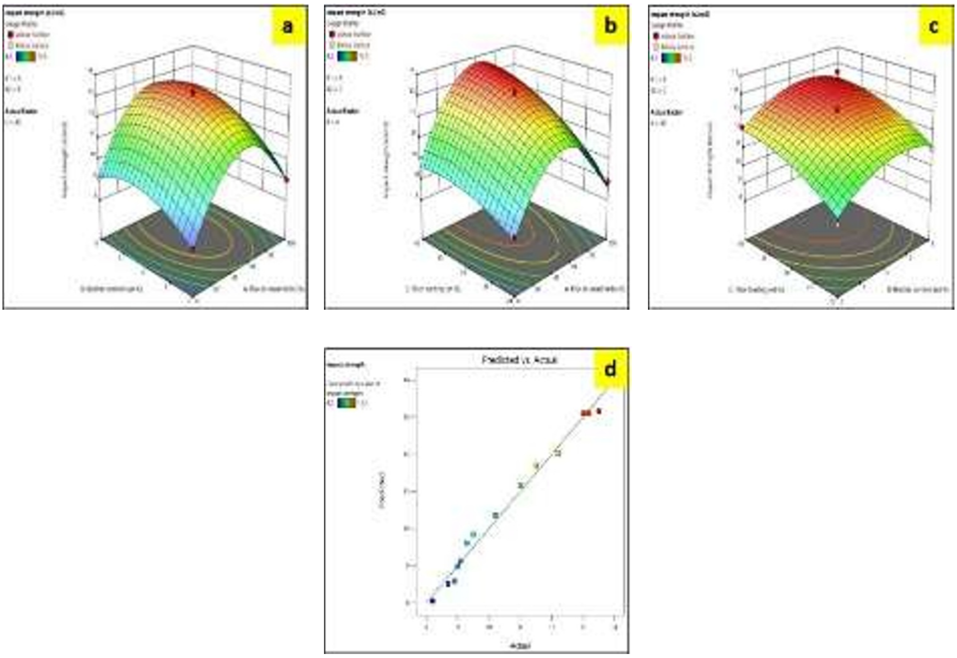


Fig. 4. Surface and predicted vs actual plots of impact strength based on various process parameters

Impact strength response showed the most effect and increased with flax content to above ~60% before decreasing due to brittleness. Incorporated biochar enhanced overall fiber loading, peaking at 4 wt%, and improved toughness by crack deflection, then higher content clustering. Better crack bridging with energy absorption was achieved while fiber loading was around 35 wt%. Toughness was reduced at higher loadings [18]. The blended flax–kenaf–biochar system, at a ratio of approximately 60% flax and 40% kenaf with 4 wt% biochar and 35 wt% fiber loading, demonstrated the highest impact strength of 13–14 kJ/m², proving the enhancement of stiffness and energy absorption for semi-structural applications.

Table 4. ANOVA results for impact strength based on process parameters

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	50.1	9	5.57	45.44	0.0003	significant
A-Flax-to-kenaf ratio	2.31	1	2.31	18.87	0.0074	
B-Biochar content	3	1	3	24.5	0.0043	
C-Fiber loading	8.82	1	8.82	72	0.0004	
AB	0.0225	1	0.0225	0.1837	0.6861	
AC	1	1	1	8.16	0.0355	
BC	0.01	1	0.01	0.0816	0.7866	
A^2	32.41	1	32.41	264.53	< 0.0001	
B^2	3.79	1	3.79	30.9	0.0026	
C^2	1.07	1	1.07	8.71	0.0319	
Residual	0.6125	5	0.1225			
Lack of Fit	0.5925	3	0.1975	19.75	0.0486	significant
Pure Error	0.02	2	0.01			
Cor Total	50.71	14				

From the perspective of the current study, the optimized flax–kenaf hybrid composite was compared with other recently reported biochar-based hybrids in terms of their potential mechanical performance. The studies listed in Table 5 were reviewed and summarized for their tensile, flexural, and impact strength. Five works were selected due to their close relevance to natural-fiber and hybrid composite research. The benchmark used in the present work remains appropriate despite differences in related studies. The advanced work presented here includes an optimized composite and flexural strength, with superior tensile properties compared to other hybrid composites. This highlights the strong potential of composites developed with biochar and hybrid fibrous architectures. The consistency and thoroughness of the reporting are noteworthy.

Table 5. Comparative analysis of the current research with previous studies

Authors	**Matrix / Reinforcement**	**Tensile strength**	**Flexural strength**	**Impact strength**	**Reference**
Proposed research	Epoxy (flax+ kenaf+ Coconut shell biochar)	115 MPa	154 MPa	13.5 kJ/m^2	-
Dal Pont et al.,	Epoxy (biobased resin blends) + flax fabric (30 vol.%)	45 MPa	108.5 MPa	11.9–12.2 kJ/m^2	[19]
Ojha et al.,	Unsaturated polyester + coconut shell ash (CSA) particulates (10–40 wt.%)	(predicted) ~87.9 MPa; validated ~90.5 MPa	(predicted) ~85.6 MPa; validated ~88.3 MPa.	(predicted) ~3.88 kJ/m^2; validated ~4.03 kJ/m^2.	[20]

Han et al.,	ABS + kenaf fibres (various vol.% for FDM filament / printed parts)	11.48 to 23.20 MPa	26–113 MPa	-	[21]

3.4 Microstructural analysis

SEM was used to analyze the fractured composite surfaces post-tensile testing to gain insights on the failure mechanisms and interfacial behavior. The absence of biochar reinforcement at lower fiber loadings was characterized by weak fiber adhesion and poor interfacial stress transfer. The microstructural images taken after the hybridization of flax and kenaf showed that the stiffness and interfacial bonding were greatly improved due to the contribution of flax, which permitted efficient fiber pull-out and enhanced fiber alignment (Figure 5 (a) & (b)) [22]. The tensile strength improved drastically because of the addition of ~4 wt% coconut shell biochar uniformly dispersed around the fibers and micro-voids and promoting cavity deflection (Figure 5 (c)). With excessive addition of biochar (>6 wt%) or fibers (>40 wt%), the SEM images were dominated by particle agglomerates, resin-rich zones, and micro-cracks, which behaved as stress concentrators and led to premature fracture (Figure 5 (d)). These results confirm the RSM-optimized parameters, which showed that the strongest fiber–matrix adhesion and highest tensile performance were achieved from balanced flax–kenaf hybridization and a modest biochar addition [23].

Fig. 5 Microstructural analysis of hybrid composites after tensile testing

4 Conclusion

The refined results showed the development and optimization of the composites of biochar with flax and kenaf fibers using response surface methodology (RSM), and these were successful. The calculated values of the tensile, flexural, and impact strengths were statistically significant, showing that the modeling approach using a quadratic model fitted the data well. The most significant parameters affecting the mechanical properties were the ratios of flax and kenaf, the fiber volume, and the biochar volume. The hybrid combination of kenaf and flax provided the most reinforcement, with 60% and 40%, respectively, and the biochar at 4%, and the fiber approximately 35%, all at moderate levels. The study shows that the biochar used serves as a micro-filler reinforcing the bonds in the fiber–matrix, and during energy absorption, the hybridization of flax and kenaf overcomes the problems in composites of a single fiber. As a whole, the results demonstrate that the composites reinforced with coconut shell biochar, flax, and kenaf represent novel materials that are low-cost, strong, and lightweight, making them suitable for structural and semi-structural applications.

5 Limitations and Future Scope

This investigation has focused only on analysing the mechanical behaviour of flax/kenaf/biochar hybrid composite materials, and determines their performance in terms of tensile, flexural, and impact responses within a controlled parameter range. Other key functional properties such as thermal stability, dynamic mechanical behaviour, moisture absorption, and long-term durability were not considered, and the results are limited to coconut-shell biochar of a specific particle size and the compression-moulding conditions used. In spite of these constraints, there are clear opportunities for further investigation. Subsequent studies could explore different biochar sources, particle morphologies, thermal and viscoelastic properties, and durability under environmental ageing for similar flax/kenaf/biochar composite systems. Extending the statistical model to include multi-objective optimisation, examining alternative composite formulations, and assessing suitability for automotive or structural applications would significantly enhance the practical relevance of this work. Additionally, integrating artificial intelligence or machine learning with response surface methodology could further improve the accuracy of performance prediction.

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