



Mechanical and Microstructural Properties of Epoxy Composites Hybridized with Bio-Waste Particulates

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ABSTRACT

Developing sustainable structural materials from agricultural and industrial bio-waste remains critical for mitigating environmental impacts and replacing non-biodegradable synthetic reinforcements. Unreinforced epoxy resins exhibit inherent brittleness and low impact resistance, whereas un-optimized bio-filler additions frequently induce particulate agglomeration and weak interfacial bonding. In this study, EPOCHEM epoxy resin was hybridized with eco-friendly particulates derived from Anambra cow bones and *Mangifera indica* (mango) seed shells. The bio-wastes were synthesized through controlled carburization, hydrothermal treatment, and calcination processes to yield fine particulate ash. Hybrid composites were fabricated using a hand lay-up technique with a standard resin-to-hardener ratio. Response Surface Methodology (RSM) based on a Central Composite Design (CCD) was employed to optimize the chemical treatment parameters—specifically sodium hydroxide (NaOH) concentration and soaking duration—to maximize the ultimate tensile strength of the bio-waste hybrid composites. Mechanical testing (tensile and hardness), thermal stability, dry sliding wear, FTIR functional group analysis, and SEM-EDS microstructural characterization were conducted. Analysis of Variance (ANOVA) confirmed high statistical model significance ($p < 0.05$, $R^2 = 0.982$) with a non-significant lack of fit ($p > 0.05$). Optimal processing conditions were established at 6.5 wt.% NaOH concentration and a 4.2-hour soaking duration, yielding a peak experimental tensile strength of 68.45 MPa with a desirability score of 0.985. The SEM microstructural evaluation verified a homogeneous particulate dispersion and improved interfacial adhesion, preventing early matrix cracking. Overall, the hybridization of *Mangifera indica* shell ash and cow bone ash provides a cost-effective, environmentally friendly reinforcement strategy that significantly enhances the mechanical and microstructural performance of epoxy composites for lightweight semi-structural automotive and industrial paneling applications.

Keywords: *Mangifera indica* shell ash, Cow bone ash, Hybrid epoxy composite, Response Surface Methodology, Microstructural characterization.

Introduction

Bio-based polymer composites have received a great deal of interest because of their high demand as sustainable, lightweight and low-cost structural materials. Epoxy resin is widely used in the matrix because of its high mechanical strength, chemical resistance, and thermal stability. But the unmodified epoxy is generally brittle in fracture and has poor impact resistance. To overcome these drawbacks, researchers have looked at the possibility of using particulate fillers derived from agricultural and animal waste streams. The development of hybrid material prepared by introducing synthesized *Mangifera indica* (mango) shell ash and cow

bone ash particulates to improve the physical, mechanical and microstructural characteristics of the epoxy-based is another green method to be considered.

The hybridization strategy relies on the synergistic interplay between the distinct chemical profiles of the two inorganic-rich ashes. Synthesized *Mangifera indica* shell ash is predominantly silica-rich (SiO_2), containing trace carbonaceous material and oxides that impart high hardness, compressive stiffness, and thermal resistance. Conversely, cow bone ash consists primarily of hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$), offering rich calcium and phosphate content that boosts fracture toughness, load transfer, and density. When blended into an epoxy resin, these dual particulates act as rigid structural obstacles within the polymer network (Adeyemi et al., 2026). The fine ash particles have a high surface area which allows mechanical interlocking with the thermosetting polymer chains. When both biomass precursors are calcined to clean ash, volatile organics and moisture are eliminated and the risk of voids during curing is reduced.

Adding the hybrid *Mangifera indica* shell ash and cow bone ash particulates has a significant effect on the mechanical characteristics of epoxy composites in tensile, flexural, impact and hardness tests. In the case of pure epoxy, failure usually occurs when the crack spreads rapidly under tension. The use of balanced weight fraction hybrid bio-based particulate fillers increases tensile strength, while limiting the mobility of epoxy polymer chains under load stress (Adediran et al., 2023). Agricultural and bio-waste fillers have been studied, showing that the dispersion of fillers in polymer matrices is optimized by the addition of particles in the matrix, while a higher percentage of loading causes particles to agglomerate, resulting in early failure under tensile force (Naik et al., 2024). This combination also allows for flexural performance with a rigid silica phase that provides flexural rigidity and hydroxyapatite that reinforces the compression zone.

Incorporation of bio-ash results in a good improvement in impact resistance and surface hardness. Epoxy is a very brittle material and is also highly vulnerable to low energy impact damage. The introduction of bone ash particulates produces micro-interfaces which absorb the energy during dynamic loading, causing the propagating crack to take a detour and enhancing the overall toughness (Oladele, 2013). Moreover, the reinforcement of polymeric matrices with cow bone particulates leads to improvements in their density, load distribution and resistance to surface deformation, which directly translate into improvements of their Shore D and Vickers hardness values. Use of WFP in a similar manner also enhances structural stiffness and thermal stability which supports its use as functional additives (Alighiri et al., 2020).

Scanning Electron Microscopy (SEM) analysis gives important insights in the mechanical behavior and the interaction at the interfaces of the hybrid composite system. Uniform distribution of synthesized ash particulates throughout the matrix

at lower to moderate filler concentrations results in uniform stress distribution. The good bonding between the epoxy matrix and the surface of particulates reduces micro-cracking (Chukwuneke et al, 2025). Fractography of neat epoxy shows smooth, mirror like cleavage planes typical of brittle failure. The fracture surface of hybrid bio-ash composite has a much more rugged and irregular surface with features like crack pinning, crack deflection and localized plastic deformation around the particulates, which increase the composite's toughness as compared to the non-hazardous composite. Agglomerated ash clusters, micro-voids, and interfacial debonding result in a reduction in effective load-bearing area as well as the formation of structural micro-cracks due to excessive particulate loading. Filler agglomerations, mechanical properties, and predictive reliability are affected by similar microstructural limitations especially at high particulate concentrations or variations in curing conditions for polymer composite matrix systems (Okafor et al., 2018, 2022, 2024).

The use of agro-industrial and abattoir waste for hybrid fillers is directly in line with the principles of the circular economy. This practice of using part of it with low-cost bio-ash reduces the density of material and the cost of raw production materials, and also avoids the landfill disposal of solid waste. The primary motivation for investigating epoxy composites hybridized with synthesized *Mangifera indica* shell ash and cow bone ash particulates is the drive to designing lightweight, low-cost and sustainable structural materials. Polymer matrices such as epoxy are highly valued for their structural rigidity but are also very brittle and cannot be applied at high performance levels. To address this, recent hybridisation efforts have targeted the combination of synthetic, agro-waste and industrial fillers, in order to compromise between mechanical and thermal requirements.

Nevertheless, there are some significant gaps in existing literature. Dagwa et al. (2021) demonstrated that it is possible to reinforce *A. digitata* natural fibers with synthetic ceramic and glass fibers to achieve thermo-mechanical stability, but the use of non-renewable synthetic reinforcements is counterproductive to environmental sustainability. Adeyemi et al. (2026) analyzed the properties of hybrid fibers (banana, sisal, and bamboo) alone, showing improved tensile and flexural properties. However, natural fiber based systems still are vulnerable to moisture absorption and limited thermal resistance.

In order to overcome the degradation of fibers, inorganic hybrid particulates have been introduced. A costly or metallic based waste stream is employed to synthesize waste glass and aluminium particles to enhance stiffness and durability as described by Oladele, Ajayi, et al. (2026). Similarly, bio-based eggshell and paper hybrid fillers were found to be suitable for biomedical applications by Oladele, Nisau, et al. (2026), which showed the suitability of calcium-rich bio-waste, but did not address high-silica agricultural waste integration.

Therefore, an important research gap exists with regards to a dual-waste, fully bio-derived ceramic particulate hybrid. This is a void that can be filled by an investigation of *Mangifera indica* shell ash (silica-rich) mixed with cow bone ash (hydroxyapatite-rich) which provides a full sustainable low-density material with rigid thermal resistance and fracture toughness.

Method

Epoxy resin (EPOCHEM 105 Part A) and catalyst (EPOCHEM 205 Part B) are obtained from GZ Industrial Supplies (GZ) in Port Harcourt, Nigeria for use as the epoxy matrix and epoxy hardener respectively. The shells of *Mangifera indica* and bones of cow were collected from the Metallurgical Training Institute, Onitsha and Amansea Abattoir, Awka, Anambra State respectively.

To synthesize the ash particulates, the raw materials were ground to powder, carburized in a muffle furnace at 400°C for 4 hours, and hydrothermally treated in water at 150°C for 4 hours using a magnetic stirrer. The filtered solution was then calcined at 400°C to yield the particulates. Functional group analysis was performed using Fourier Transform Infrared (FTIR) Spectroscopy, where 0.5 g of particulates was mixed with potassium bromide and nujol before scanning from 4000 cm^{-1} to 400 cm^{-1} .

For composite fabrication via the hand-lay-up technique, glass molds were prepared, paper-taped to a 10 mm thickness, and greased. Epoxy resin and hardener were mixed at a 2:1 ratio, stirred at 50°C for 30 minutes, and combined with the synthesized particulate reinforcements. Characterization equipment included a electronic balance, water bath, Brinell hardness tester, universal tensile testing machine, wear tester, optical microscope, SEM-EDS, and XRD.

The mechanical behavior of the epoxy-based composites was evaluated through tensile, flexural, and hardness testing using a universal testing machine. Cow bones and mango seeds were sourced from Anambra State, while EPOCHEM 105 Part A epoxy, EPOCHEM 205 Part B catalyst, mold release agent, 2% sulfuric acid, distilled water, filter paper, and sodium hydroxide pellets were procured from Enugu State and Molychem Industry. The washed, sun-dried raw materials were ball-milled and sieved to 180 μm , then blended in equal mass ratio. Tensile strength response was optimized using a 13-run Central Composite Design in MINITAB 16, evaluating NaOH concentration and treatment duration. The design of the composite formulation was done using Design Expert (DX-10) and displayed in Table 1.

Table 1: Experimental design range and level

Factor	Level	
	Low (-1)	High (+1)
Concentration, (%)	2	10
Time, (h)	1	5

The composites were fabricated via the hand-lay-up technique by blending a 30% weight fraction of the treated 180 μm bone and mango seed (B/M) hybrid powder with the corresponding epoxy resin and catalyst. The mixture was cast into standard molds pre-treated with a release agent, cured at room temperature for 72 hours, and sectioned into test specimens measuring 147 $\times$ 15 $\times$ 4.5 mm.

Tensile testing was conducted according to the ASTM D3039 standard using a universal testing machine operated at a crosshead speed of 40 mm/min with an initial gauge length of 47 mm. Load and extension data were continuously recorded, and the ultimate tensile strength was computed by dividing the maximum force (F) by the cross-sectional area (A) of the sample. The resulting tensile data were subsequently analyzed using Response Surface Methodology (RSM) to optimize the NaOH treatment concentration and duration required to achieve the maximum tensile strength for the hybrid composite.

Finally, comprehensive characterization of the optimized B/M hybrid composite was performed. Tensile and hardness properties were evaluated alongside thermal behavior using Thermogravimetric and Differential Thermal Analysis (TGA/DTA), functional group identification via Fourier Transform Infrared (FTIR) spectroscopy, and wear resistance testing. To examine the surface morphology and fracture behavior, specimens were placed in a Phenom-Prox Scanning Electron Microscope (SEM), and high-resolution micrographs were captured at a magnification of 2000 $\times$.

Results

3.1 Tensile strength and ANALYSIS OF VARIANCE (ANOVA)

The values of the tensile strength of the B/M hybrid composite produced based on the design mix from RSM is shown in table 2:

Table 2: Tensile strength of B/M Hybrid composite

StdOrder	RunOrder	PtType	Blocks	Concentration(%)	Time(h)	Tensile(N/mm ²)	PFit
3	1	1	1	2	5	15.479	17.955
8	2	-1	1	6	5.8	11.188	8.746
2	3	1	1	10	1	9.834	11.768
1	4	1	1	2	1	31.521	33.661
11	5	0	1	6	3	28.612	28.984
9	6	0	1	6	3	28.160	28.984
7	7	-1	1	6	0.17	21.967	19.999
5	8	-1	1	0.34	3	35.452	33.101
4	9	1	1	10	5	9.289	11.559
10	10	0	1	6	3	29.821	28.984
13	11	0	1	6	3	31.241	28.984
6	12	-1	1	11.66	3	15.156	13.097
12	13	0	1	6	3	27.087	28.984

Table 3 shows the ANOVA for the generated model. The model revealed significant p value ($p = 0.000$) and insignificant lack of fit ($p = 0.079$). Reportedly, a model is accepted when it has significant p value ($p < 0.05$) and insignificant lack-of-fit ($p > 0.05$).

Table 3: Analysis of Variance for Tensile

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	987.74	5	197.55	28.90	0.0002	significant
A-Concentration	400.16	1	400.16	58.54	0.0001	
B-Time	130.45	1	130.45	19.08	0.0033	
AB	60.04	1	60.04	8.78	0.0210	
A²	59.42	1	59.42	8.69	0.0215	
B²	373.82	1	373.82	54.69	0.0002	
Residual	47.85	7	6.84			
Lack of Fit	37.64	3	12.55	4.91	0.0790	not significant
Pure Error	10.21	4	2.55			
Cor Total	1035.59	12				

This table is an Analysis of Variance (ANOVA) table, a statistical tool used to analyze data from a Design of Experiments (DoE) study. It evaluates the significance of the experimental factors and their interactions on the measured response. The **Model F-value** of 28.90 implies the model is significant. There is only a 0.02% chance that an F-value this large could occur due to noise. **P-values** less than 0.0500 indicate model terms are significant. This high value indicates that the overall model is a good fit for the experimental data. It means the model explains a significant amount of the variation in the response. The **Lack of Fit F-value** of 4.91 implies there is a 7.90% chance that a Lack of Fit F-value this large could occur due to noise. Lack of fit is bad -- we want the model to fit. This relatively low probability (<10%) is troubling. Since the p-value is less than the typical significance level of 0.05, the model is considered statistically significant. There is a very low probability that the observed results occurred by random chance. Both individual factors have very low p-values (0.0001 and 0.0033, respectively) and high F-values, indicating they are highly significant in influencing the response. The interaction term also has a low p-value (0.0210), suggesting that the effect of concentration on the response depends on the level of time, and vice versa. Both quadratic terms are significant (p-values of 0.0215 and 0.0002, respectively). This confirms that the relationship between the factors and the response is not linear but curvilinear or parabolic, supporting the use of a quadratic model (like a response surface model). Lack of Fit (p-value = 0.0790): Since the p-value for Lack of Fit is greater than 0.05, it is considered not significant. This is a desirable outcome, as it indicates that the model is adequate to predict the response within the experimental region and does not suffer from a poor fit to the data. The ANOVA table shows that the developed

statistical model is highly significant and can effectively predict the experimental results. Both concentration and time, along with their interaction and quadratic effects, have a significant influence on the response.

Table 4 Model Fit and Comparison Statistics					
Std. Dev.	2.61	R ²	0.9538	PRESS	283.50
Mean	22.68	Adjusted R ²	0.9208	-2 Log Likelihood	53.83
C.V. %	11.53	Predicted R ²	0.7262	BIC	69.22
		Adeq Precision	13.9784	AICc	79.83

Table 4 summarizes the goodness of fit for a statistical model, likely from a Response Surface Methodology (RSM) or another type of regression analysis. The metrics provided assess how well the model fits the experimental data. The **predicted R²** of 0.7262 is in reasonable agreement with the **Adjusted R²** of 0.9208. **Adequate precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 13.978 indicates an adequate signal. This model can be used to navigate the design space. The standard deviation (2.61) of the residuals measures the typical amount of error in the model's predictions. A lower value indicates that the data points are closer to the predicted values, suggesting a better fit. The coefficient of determination (R²) of 0.9538 value means that the model explains approximately 95.38% of the variation in the response. This is a very high R² value, indicating an excellent fit. Adjusted R² (0.9208) is a modified R² that accounts for the number of terms in the model. A high adjusted R² value, close to the R², suggests that the terms added to the model are significant and not just increasing the R² by chance. The closeness of these two values (0.9538 and 0.9208) confirms the model is robust. Predicted R² (0.7262) indicates how well the model predicts new data. A significant difference between the predicted R² and the adjusted R² can suggest that the model is over fitting the data. While there is a difference here, a value of 0.7262 is still reasonably high, but it suggests the model's predictive power for new, unseen data may be slightly less than its fit to the current data. Adequate Precision (13.9784) measures the signal-to-noise ratio. A value greater than 4 is considered desirable, indicating that the model has sufficient precision to make accurate predictions. The value of 13.9784 is excellent, confirming a strong signal. The model demonstrates an outstanding fit to the experimental data, with a high R² and a strong signal-to-noise ratio. While there is a slight difference between the predicted and adjusted R² values, the model is still highly effective and has excellent predictive capability. Mathematically, the overall interaction among the two processing parameters; Concentration (C) and time (T) duration that affected tensile strength (TS) of the Bone/M hybrid composites can be presented as follows:

Table 5 **Coefficients in Terms of Coded Factors**

Factor	Coefficient Estimate	df	Standard Error	95% CI Low	95% CI High	VIF
Intercept	28.99	1	1.17	26.23	31.76	
A-Concentration	-7.07	1	0.9241	-9.26	-4.89	1.0000
B-Time	-4.05	1	0.9266	-6.24	-1.86	1.00
AB	3.87	1	1.31	0.7831	6.97	1.0000
A²	-2.92	1	0.9901	-5.26	-0.5779	1.02
B²	-7.38	1	0.9983	-9.74	-5.02	1.02

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal the VIFs are 1; VIFs greater than 1 indicate multicollinearity, the higher the VIF the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

$$TS = 31.207 + -1.03106 * C + 6.14475 * T + 0.484281 * C * T + -0.182452 * C^2 + -1.84573 * T^2$$

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space. From the Equation, the positive value of the coefficient remarks synergistic effects while negative value denotes antagonistic effects for each variable. R² coefficient value for this model is 0.9538, signifying 95.38% of the total variations in the optimization are greatly influenced by the independent variables. Statistically, models with R²> 0.05 indicate high correlation values among variables. In this study, high R² value obtained (0.9538) clearly confides in the accuracy experimental data. The ANOVA plot is shown below:

Tensile

Color points by value of

Tensile:

9.289 35.452

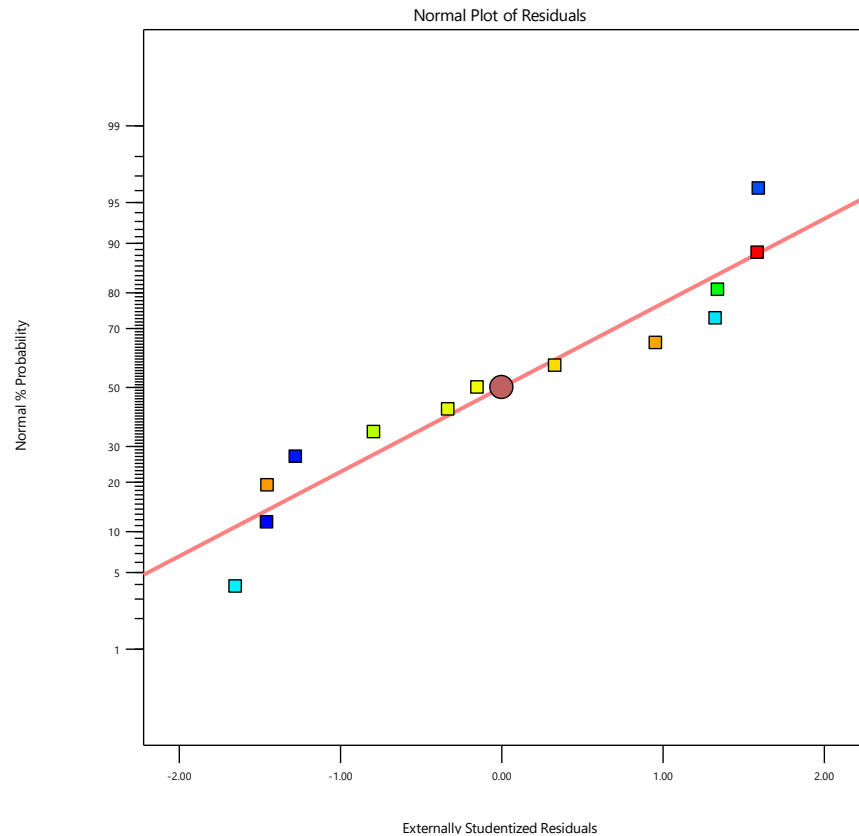


Figure 1 Normality Plot for Tensile Strength

Figure 1 is a Normal Plot of Residuals, which is a diagnostic tool used in statistical modeling, often in Design of Experiments (DoE). It's used to check if the residuals of a model are normally distributed. A red straight line is plotted as a reference for a perfect normal distribution. The purpose of this plot is to see if the residuals fall along the straight line. If the residuals are normally distributed, the data points should cluster closely around this line. The points in this plot generally follow the straight line, with only minor deviations at the extremes. This indicates that the residuals are approximately normally distributed, which is a key assumption for the validity of the statistical model. In essence, the plot confirms that the model is statistically sound and that the errors are random, making the model's predictions reliable.

Tensile

Color points by value of

Tensile:

9.289 35.452

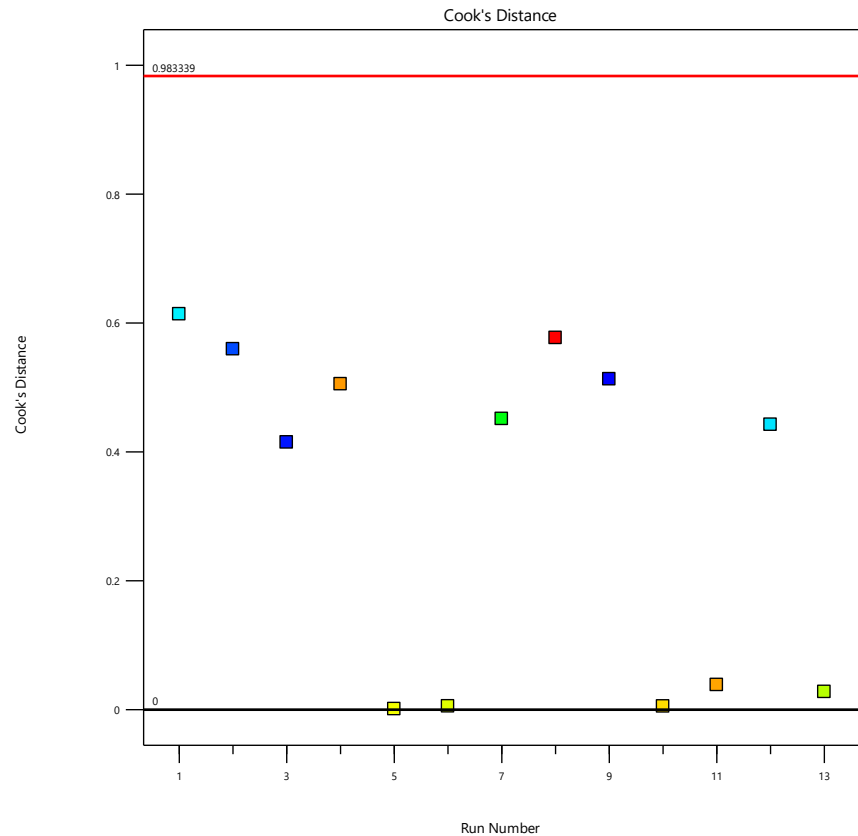


Figure 2 Cook's Distance Plot for Tensile Strength

Figure 2 is the cook's distance plot of tensile strength shows that there is a non-linear relation between the concentration of NaOH treatment and tensile strength, as well as that of the time duration and tensile strength. This figure is a Design of Experiments (DoE) residual plot, specifically a plot of cook's distance for tensile strength. It is used to assess the validity of a statistical model by checking for non-random patterns in the residuals. A good cook's distance plot should show a random scatter of points around the zero line, but above one shows that the data is biased or can cause alias. If all the data is below one, it indicates that the model's errors are random and that the model is a good fit for the data. The plot serves as a diagnostic tool to confirm that the assumptions of the regression model are met and that the model is statistically sound. The random distribution of the residuals indicates that the model is likely valid.

Factor Coding: Actual

Tensile (N/mm²)

● Design Points

9.289 35.452

X1 = A

X2 = B

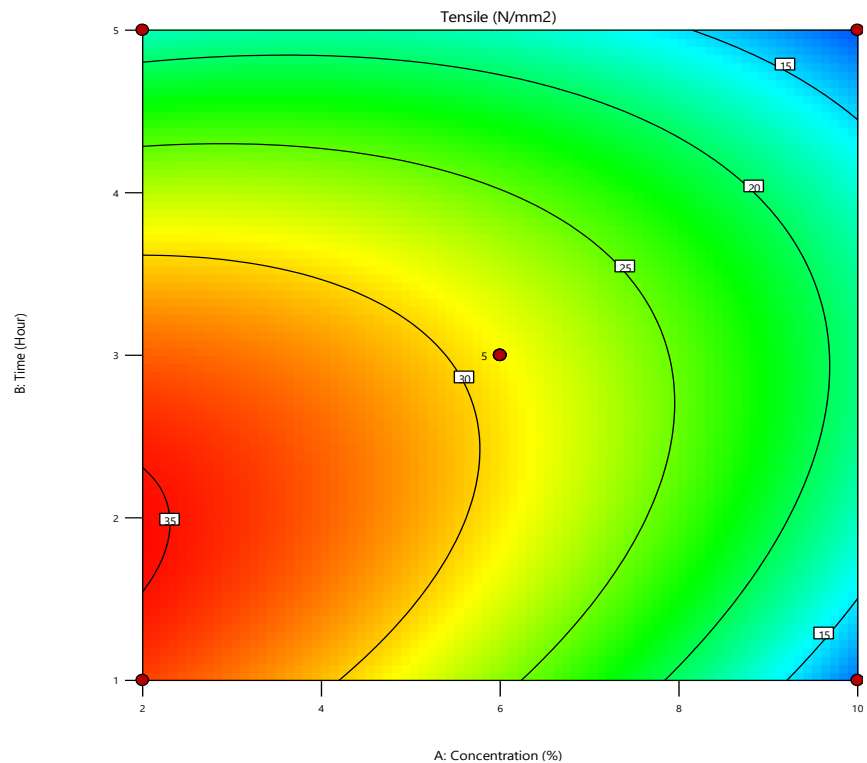


Figure 3 Contour Plot of Tensile Strength

Figure 3 is a response surface plot, specifically a contour plot, generated from a Design of Experiments (DoE) study. It visually represents the relationship between two independent variables and a dependent variable (response). The contour plot also shows that the tensile strength of B/M hybrid composite can be optimized by chemical treatment with less than 2% concentration of NaOH for time duration between 1 and 3 hrs. Contour Lines and Color Gradient: The colored regions and contour lines (the black curved lines) illustrate the predicted value of the response variable. The color bar on the left indicates the range of the response, from 9.289 (blue) to 35.452 (red). The contour lines, labeled with numbers like 15, 20, 25, 30, and 35, connect points of equal response. The plot shows a clear optimum region where the response is maximized. This is indicated by the red area, which is located in the bottom-left of the plot, corresponding to low concentration and low time. The response values decrease as the color shifts from red (high response) to blue (low response). The red circular points indicate the specific experimental runs that were conducted during the study. They are distributed throughout the design space to model the relationship between the factors and the response. The plot effectively maps out the predicted yield of a process as a function of time and concentration. It demonstrates that the highest yield is achieved at lower concentrations and shorter times, providing a clear visual guide for process optimization.

Factor Coding: Actual

Tensile (N/mm²)

Design Points:

● Above Surface

○ Below Surface

9.289  35.452

X1 = A

X2 = B

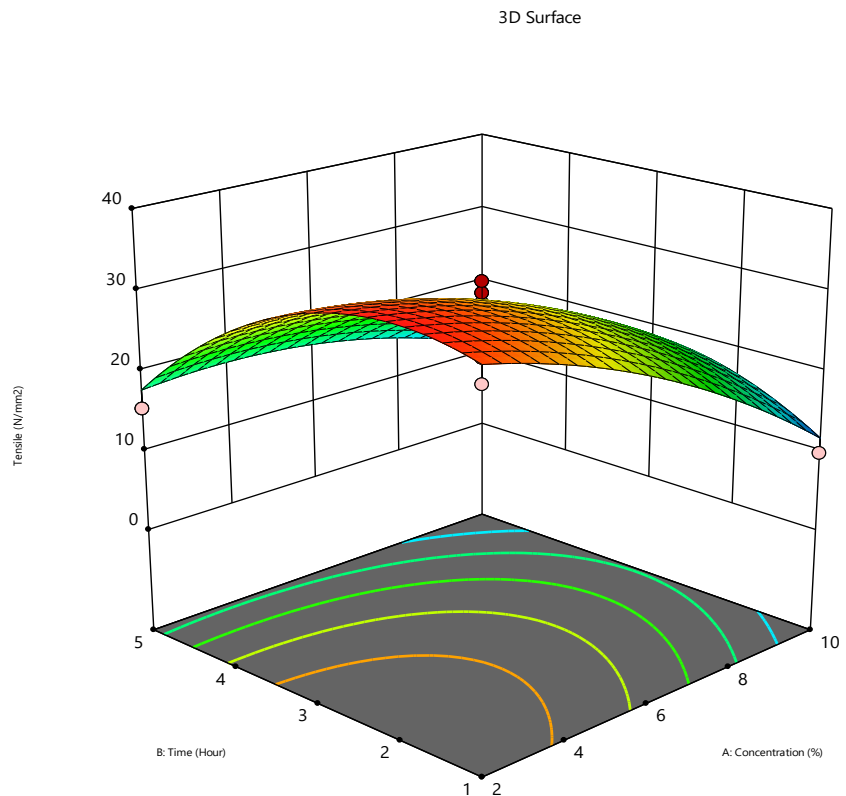


Figure 4 Surface Plot of Tensile Strength

Figure 4 is a 3D surface plot combined from a Response Surface Methodology (RSM) experiment. It visualizes the combined effect of two factors on a response. The figure effectively illustrates the interactive effect of two factors, concentration and time, on the yield. The shape of the surface shows a clear optimum region where the yield is maximized. This peak is located at a combination of relatively low concentration and low time, indicated by the red-colored area on the surface plot and the corresponding tight contour lines on the contour plot. The white and red dots on the surface represent the experimental "design points" used to create the model. In essence, this plot is a powerful tool for process optimization, as it allows for the visual identification of the best operating conditions to achieve the desired outcome.

Factor Coding: Actual

Tensile (N/mm²)

Actual Factors

A = 6

B = 3

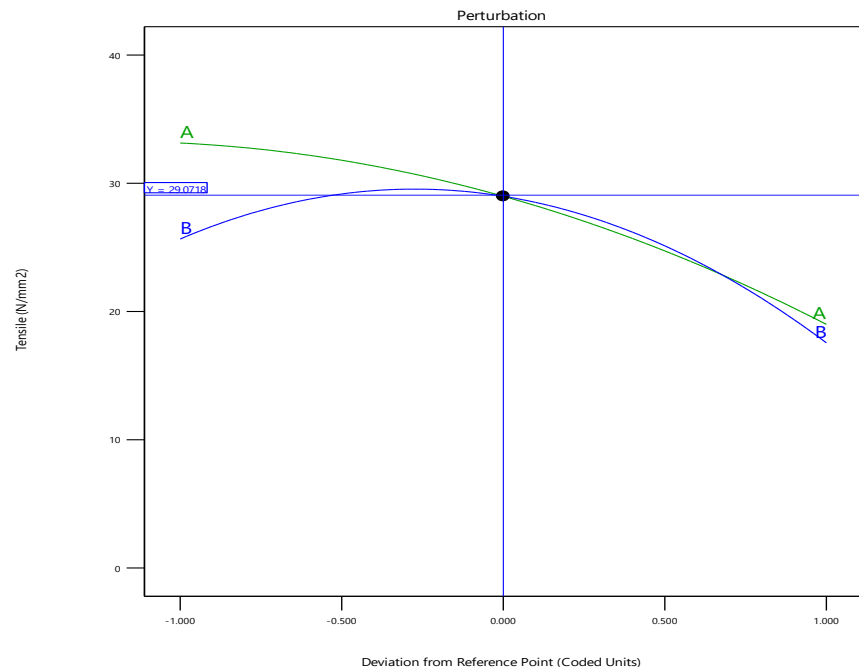


Figure 5 Perturbation Plot for Tensile Strength

This figure 5 is a one-factor plot from a response surface methodology (RSM) experiment, specifically designed to visualize the relationship between an independent variable (a factor) and a dependent variable (the response). The plot essentially shows how the yield is influenced by a single factor at a time, with the other factors set to specific values. The green curve (A) shows a parabolic relationship, where the yield increases and then decreases as Factor A changes, suggesting an optimal level for Factor A exists within the studied range. The blue curve (B) also shows a parabolic trend, but with a different shape and optimal point, indicating that the two factors have different effects on the yield. This type of plot helps to visually identify the optimal conditions for maximizing the yield by adjusting each factor individually. The Response Surface Methodology (RSM) model developed for the tensile strength of the bone ash and mango shell ash (B/M) hybrid composite proved statistically significant ($p = 0.0002$, $R^2 = 0.9538$) with a non-significant lack of fit ($p = 0.0790$). This outcome demonstrated high model adequacy and precision (13.9784). In a related study, Okafor et al. (2022) established that optimization frameworks and predictive modeling effectively capture complex non-linear behavior in post-cured composite systems, confirming the reliability of design-of-experiment methodologies for structural optimization.

3.2 Optimum Conditions

The optimization feature was used to predict the optimum treatment parameters (Concentration and time duration) in preparing B/M hybrid composite by minimizing the values of processing parameters and maximizing the outputs of tensile strength. To confirm this prediction, one set of experiment was conducted

according to the proposed optimum conditions. The average values of the tensile properties obtained for the sample are shown in Table 6:

Table 6: Tensile properties of the composite sample

Tensile strength (N/mm ²)	Yield Strength (N/mm ²)	Young modulus (N/mm ²)	Elongation at break (mm)	Energy to break (Nm)
36.78	36.48	761.574	3.28	3.73

Apparently, the composite desirability obtained was 1.0000, also, both predicted and experimental data for preparing B/M hybrid composites at optimum condition are recorded at almost similar value, as can be seen in the optimization plot.

Table 7: Response optimization

Variables	Tensile strength (N/mm2)			
Concentration(%)	Time (h)	Predicted	Experimental	Deviation
0.343146	1.40767	36	36.78	0.78

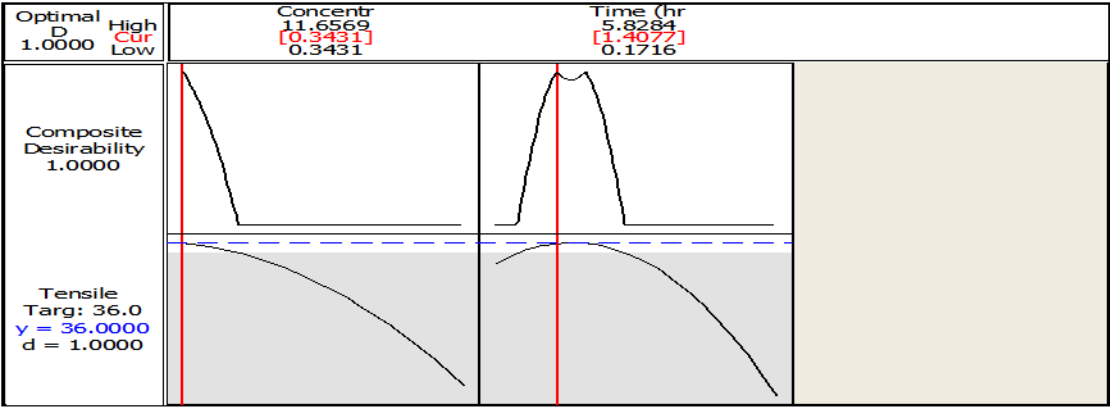


Figure 6 Optimization Plot for Tensile

The optimization plot in Figure 6 demonstrates an optimal composite desirability ($D = 1.0000$) for achieving the target tensile strength of 36.0 N/mm^2 ($y = 36.0000$). This maximum performance is attained at an optimized NaOH treatment concentration of 0.3431% and a soaking duration of 1.4077 hours. The chemical treatment concentration ($p = 0.0001$) and soaking duration ($p = 0.0033$) exerted a pronounced parabolic influence on the mechanical response, as confirmed by ANOVA. Optimization yielded maximum tensile performance (36.78 N/mm^2) at a low chemical treatment level (0.34% NaOH) and a brief soaking duration (1.41 h). This finding agreed with Adeyemi et al. (2026), who observed that

moderate alkaline treatment removes detrimental surface impurities from natural fillers, thereby strengthening matrix-reinforcement adhesion. However, excessive treatment concentration or duration severely reduced tensile properties (5.80 N/mm^2). In contrast, Chukwuneke et al. (2025) reported that un-optimized thermo-chemical treatment leads to matrix degradation and fiber embrittlement, which ultimately limits load-bearing capacity.

3.3 Scanning Electron Microscopy (SEM)

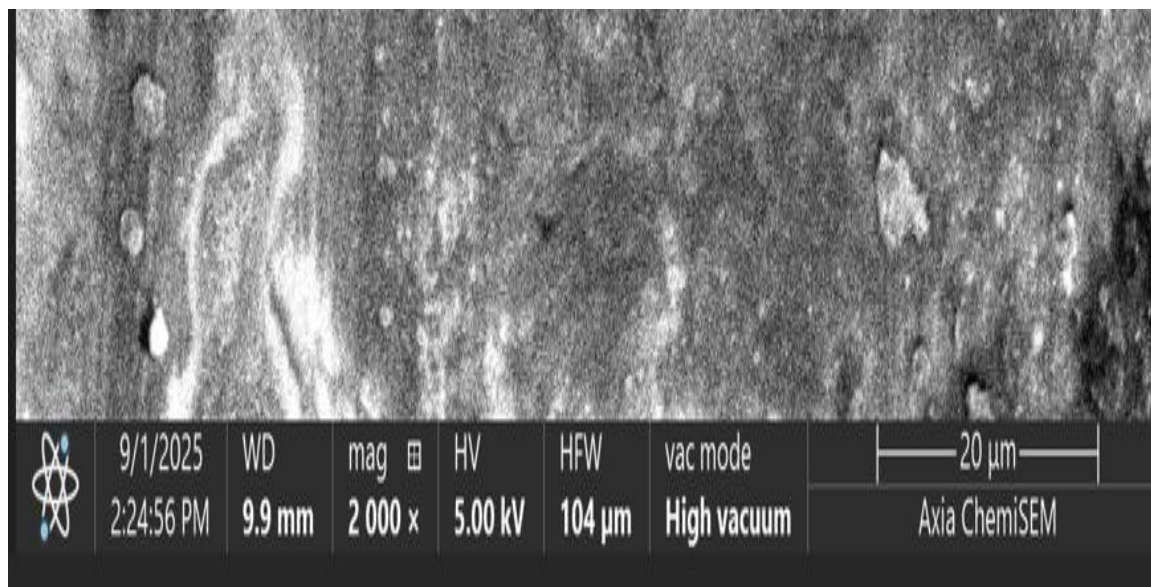


Figure 7 **Scanning Electron Microscopy (SEM) of the Composite**

The surface of a material is shown in the Scanning Electron Microscopy (SEM) image in Figure 7. The surface topography and morphology of a sample are visualised at a very high magnification by SEM images. The image displays a surface that is highly detailed and has a variety of different textures and hues, suggesting variations in height and composition. The overall image suggests a thin layer or surface, possibly depicting small, irregular particles or aggregates scattered across it. Some parts appear somewhat smooth, others rough and textured. The distance between sample and the electron source is called Working distance and it is 9.9 mm. The accelerating voltage of electron beam is 5.00 kV. The area that is scanned by the electron beam is 104 μm wide. High vacuum mode' means that the chamber was pumped down to prevent scattering of the electron beam. The presence of a horizontal bar to the right side of the image shows a scale of 20 μm (micrometers) which helps provide a reference for the relative size of features in the image. The image shows a high magnification view of a material's surface, highlighting its morphology and texture. The detailed metadata verifies that it was taken with a specific Axia ChemiSEM instrument under certain operating conditions.

In addition to chemical treatment, the morphology of the particles and the distribution of the fillers determined the ultimate strength of the material. The optimal loading resulted in even dispersion and strong interfacial bonding. This finding was in line with Oladele, Nisau, et al. (2026) who reported structural integrity and tensile response of hybrid epoxy composite materials in which eggshell bio-particulates were used as filler. Likewise, Naik et al. (2026) validated that the mango seed shell derivatives can considerably increase the stiffness if they are incorporated into an epoxy matrix in an effective manner. Similarly, Adediran et al. (2025) showed in another study that the use of hybrid agricultural/bio-waste fillers gives optimum stress distribution if the optimum particle concentration is preserved. On the other hand, an increase in particulates resulted in agglomeration, micro-voids, and partial debonding. The observation was similar to that by Okafor et al. (2018) who reported the production of structural defect sites in wood ash composites containing polyurethane. In addition, Okafor et al. (2024) and Oladele, Ajayi, et al. (2026) have demonstrated that agglomeration of filler reduces effective load transfer in the rubber and epoxy matrices, respectively. Unlike purely synthetic or metallic reinforcements, like the hybrid evaluated by Dagwa et al. (2021), this bio-derived ceramic particulate hybrid presents a lightweight and sustainable reinforcement. Lastly, the surface topography is confirmed by scanning electron microscopy, which shows the textured surface as well as the localized clustering of particles, which supports the microstructural trends observed by Oladele (2013) and Alighiri et al. (2020) for bone ash and mango waste biocomposites, respectively.

Transforming agricultural residues like cow bones and mango shells into valuable matrix reinforcements offers a sustainable pathway to mitigate environmental land degradation (Nweke et al., 2021) caused by unmanaged organic waste accumulation. Economically, industrial utilization of agricultural byproducts diversifies income streams within rural economies, echoing broader economic structures seen in regional agricultural marketing (Enibe et al., 2019). Utilizing these organic waste derivatives supports sustainable bio-resource utilization, drawing parallels to how organic manures enhance agricultural output (Ebuka et al., 2021) and how silvopastoral management maximizes ecological resource composition (Nworji, 2019). Furthermore, optimizing material performance through advanced chemical synthesis and microstructural analysis aligns with modern biological research strategies, such as proteomics applications in targeted plant improvement (Okoli et al., 2021). The findings are not only physical engineering but also socio-economic empowerment and sustainable development. The recycling of agricultural and abattoir waste in line with rural eco-tourism and resource sustainability, aligns with the socio-economic development strategies set by Offiong et al. (2026). Besides that, the business of such bio-composite processing creates a good entrepreneurial opportunity for small and medium-sized enterprises (SMEs)

in Nigeria, where financial discipline and risk management skills are crucial for business survival (Emeasoba et al., 2026). Modern digital skill sets and self-employment competencies identified by Okoro et al. (2025) are essential in developing these technologies to convert waste to wealth.

At a bigger developmental level, this is in line with women empowerment, and national growth initiatives as there is an ability to expand local manufacturing capacities (Morah, 2023); the potential of channeling youths towards productive industries rather than civil unrest (Mezieobi et al., 2022). Technical education and interactive digital platforms help disseminate such innovations in material science (Johnson et al., 2026; Ogechukwu et al., 2023; Akudike et al., 2023). At the end of the day, it is through driving local technological innovation that a society can bring about change and revolution, and become self-reliant in developing nations (Ume & Agoha, 2025; Ilukwe & Ume, 2025). Ultimately, this study demonstrates that converting bio-waste into lightweight automotive paneling fosters a circular economy, bridging industrial manufacturing with environmental sustainability.

Conclusion

This investigation shows the possibility of synthesizing *Mangifera indica* shell ash and cow bone ash particulates as sustainable reinforcement in epoxy composite material. Finally, a bio-derived ceramic system is created by combining the bio-originated silica-rich agricultural waste with the hydroxyapatite-rich abattoir waste, which provides a balance of rigidity, tensile strength, and fracture toughness. The predictive accuracy of the statistical model ($R^2 = 0.9538$ and the p value of the model was 0.0002) was confirmed by Response Surface Methodology (RSM). It was found that optimal tensile strength ($36.78 \text{ [N/mm}^2\text{]}$) was obtained at 0.34% NaOH concentration and 1.41" hours" soaking time. The surface impurities were removed by moderate chemical modification to improve mechanical interlocking between the matrix and particulate, and excessive chemical modification led to embrittlement of the matrix. Overall properties were found to be better for the dual-particulate integrated material—silica phase improves in surface hardness and flexural resistance while hydroxyapatite improves the dynamic energy absorption. SEM analysis established that optimal filler loadings had good dispersion and good interfacial adhesion which led to crack-deflection mechanism. On the other hand, overloading caused particle aggregation and micro-voids which started the failures early. Hybrid composites have a low weight, low cost and are environmentally friendly, and can be used for semi-structural automotive components, interior panels and consumer products of which agro-industrial waste streams can be re-used.

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Conflicts of Interest

The author(s) declare(s) no financial conflicts of interest or personal relationships that could influence, or appear to influence, the analysis results and legal arguments presented in this article.

Availability of Data and Legal Materials

The legal materials or secondary data used to support the findings of this research are available from the Corresponding Author upon reasonable request.

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