

Optimization of Formulation Parameters for Tensile Strength of Powdered *Pentaclethra Macrophylla* Pod–Gum Arabic Bio-composites

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ABSTRACT

The tensile behaviour of powdered *Pentaclethra macrophylla* pod–gum Arabic bio-composites was optimized using response surface methodology based on a Central Composite Design (CCD). *Pentaclethra macrophylla* pods, an underutilized agro-waste, were cleaned, dried, pulverized and fractionated into 150µm and 210µm size classes, with part of the powder subsequently carbonized at 950°C. Composite plates were produced via pre-impregnation and hot pressing, varying pod content between 10 and 50 wt. % at fixed processing conditions, while maintaining constant levels of other additives. Tensile tests were performed in accordance with ASTM D638. Maximum tensile strength occurred at 10 wt. % reinforcement and 150µm particle size for both carbonized and un-carbonized systems, with peak values of 14.2MPa and 16.4MPa, respectively. Analysis of variance showed that reinforcement content, particle size and surface condition (carbonized vs. un-carbonized) significantly affected tensile strength ($p \leq 0.05$). The developed quadratic CCD model exhibited excellent agreement between predicted and experimental values ($R^2 = 0.9957$), comparable to optimized natural-fibre systems reported for kenaf, bamboo and banana fibre composites using response surface methodologies. The results define an optimal formulation window for *Pentaclethra* pod–gum Arabic composites and corroborate broader evidence that moderate filler loadings and fine particle sizes are required to avoid agglomeration, void formation and interfacial debonding in agro-waste reinforced bio-composites. These findings support the potential of *Pentaclethra macrophylla* pod and gum Arabic as sustainable constituents for low-to-moderate load engineering applications.

Keywords: Bio-composites; *Pentaclethra macrophylla* pod; Gum Arabic; Tensile strength; Central Composite Design; Agro-waste reinforcement.

1. INTRODUCTION

Growing environmental concerns and the need to replace synthetic reinforcements have intensified interest in bio-composites based on natural fibres and agro-waste particulates, which offer low density, renewability, biodegradability and competitive mechanical properties when compared to conventional synthetic reinforcements (Daramola et al., 2023; Taghipoor & Mirzaei, 2023; Hassan et al., 2019; Sultana et al., 2020; Okechukwu et al., 2025).

Pentaclethra macrophylla pods, an abundant agro-waste from the African oil bean tree, have been reported to possess favorable chemical composition, including silica- and alumina-rich oxides and high fixed carbon after heat treatment, which supports their application in composite and refractory materials (Iloabachie et al., 2022). *Pentaclethra macrophylla*, generates large quantities of pod waste whose fibres and particulates have recently been explored as reinforcement in polymer composites. Mechanical performance of *Pentaclethra*-based composites is strongly influenced by particle size, filler loading, and surface treatment (Iloabachie et al., 2023; Okechukwu et al., 2025).

Gum Arabic (GA) is a natural polysaccharide binder with good film-forming and adhesion properties, widely used in bio-based formulations (Khan et al., 2023; Brzyski, 2021; Mohamed et al., 2025). In lime–meta-kaolin binders and thermoplastic starch matrices, low-to-moderate GA additions significantly enhanced flexural, compressive and tensile properties, whereas excessive GA or filler contents promoted porosity, cracking and strength reduction (Brzyski, 2021; Terán et al., 2024). Similar non-monotonic trends have been reported where

small GA contents (≈ 0.5 – 1 wt. %), improved stiffness and tensile-related properties of glass ionomer cements, but higher GA fractions or loadings in incompatible matrices led to degradation (Khan et al., 2023; Alsunbul et al., 2023; Alsadon et al., 2022).

At low loadings (≈ 0.5 – 1 wt. %), oxidized gum Arabic improved nano hardness, elastic modulus, compressive and diametral tensile strength of glass ionomer cement (Khan et al., 2023). In lime–meta kaolin pastes for hemp concrete, 3–5 wt.% powdered gum Arabic substantially increased flexural ($\approx 300\%$) and compressive strengths (up to 60%) while slightly reducing water absorption (Brzyski, 2021). In banana fibre–gum Arabic boards, intermediate GA contents (15–25 wt. %) maximized tensile and flexural strength by enhancing fibre bonding and stiffness (Sangarote et al., 2025). However, in Poly methyl Methacrylate (PMMA) denture resins, relatively high GA loadings (5–20 wt. %) degraded hardness, flexural strength and fracture toughness, with deterioration roughly proportional to GA content (Alsadon et al., 2022; Khan et al., 2023).

Optimization studies on polypropylene/kenaf, bamboo/epoxy and banana fibre/epoxy systems consistently show that fibre content, fibre/particle dimensions and surface modification govern tensile strength, and that response surface methodologies (RSM) such as Box–Behnken and CCD can accurately capture these effects with R^2 typically above 0.95 (Taghipoor & Mirzaei, 2023; Yaghoobi & Fereidoon, 2018; Mohammed et. al., 2025; Hassan et al., 2019; Sultana et al., 2020).

Alkaline and thermal treatments have been shown to strongly modify the tensile properties of Pentaclethra-fibre composites by altering surface chemistry, cellulose/hemi-cellulose ratio and roughness, thereby improving fibre–matrix adhesion (Okechukwu et. al., 2025).

For powdered Pentaclethra macrophylla pod in bio-epoxy brake pads, CCD-based optimization identified intermediate reinforcement levels and fine particle sizes as yielding maximum hardness, confirming the sensitivity of mechanical performance to formulation parameters (Iloabachie et. al., 2023). Previous studies demonstrated that particle size, surface treatment, and reinforcement fraction strongly influence the mechanical and tribological properties of Pentaclethra-based composites (Iloabachie et al., 2023; Okechukwu et al., 2025).

Similar optimization trends have been observed in agro-waste reinforced composites, where moderate filler content and fine particle size maximize tensile properties before agglomeration and void formation occur (Daramola et al., 2023; Natrayan et al., 2024). This pattern emphasizes that there is an optimum GA content window that depends on matrix chemistry and microstructure.

Optimization studies across agro-waste reinforced composites consistently report peak mechanical properties at moderate filler loadings and fine particle sizes, beyond which agglomeration, void formation, and weak interfacial bonding dominate (Daramola et al., 2023; Natrayan et al., 2024). However, limited studies have systematically optimized the tensile behavior of Pentaclethra macrophylla pod–gum Arabic bio-composites using statistical design techniques.

Across a wide range of natural-fibre and hybrid composites, RSM and CCD have been successfully employed to optimize tensile and related properties by systematically varying fibre loading, particle size, orientation and surface treatment, routinely demonstrating distinct optima associated with agglomeration and interfacial failure at higher loadings (Taghipoor & Mirzaei, 2023; Mohammed et al., 2025; Hassan et al., 2019; Sultana et al., 2020; Arunachalam et al., 2025; Arunachalam et al., 2024; Lakshmi et al., 2024). However, there remains limited systematic optimization of the tensile behaviour of Pentaclethra macrophylla pod–gum Arabic composites using statistically designed experiments.

The present work therefore applies CCD to model and optimize the tensile strength of Pentaclethra macrophyllapod–gum Arabic bio-composites, focusing on the coupled effects of pod content, particle size and surface state (carbonized versus un-carbonized). The study aims to define an optimal formulation window for sustainable structural applications and to situate these materials within the broader class of statistically optimized, agro-waste reinforced bio-composites.

II. MATERIALS

The materials used in this study include powdered *Pentaclethra macrophylla* pod, gum Arabic resin, distilled water, and graphite, which served as lubricant and mould-release agent.

III. METHODS

3.1 Preparation of Reinforcement

Pentaclethra macrophylla pods were manually cleaned, washed with distilled water, sun-dried and subsequently oven-dried at 110 °C to constant mass, following drying protocols previously reported for *Pentaclethra*-based brake pad composites (Iloabachie et al., 2023). The dried pods were pulverized in a milling unit and sieved according to BS 1377:1990 to obtain 150µm and 210µm fractions, similar to particle-size ranges commonly used in optimization of natural-fibre particulates (Mohammed et al., 2025; Iloabachie et al., 2023). The sieved powder was split into two batches; one was carbonized at 950°C in a heat-treatment furnace, while the other remained un-carbonized.

3.2 Composite Fabrication

Composite plates were fabricated using a pre-impregnation and hot-pressing route analogous to earlier *Pentaclethra*/bio-epoxy work (Iloabachie et al., 2023). The pod content was varied from 10 to 50 wt. % in 10 wt. % increments, with gum Arabic content adjusted accordingly and the levels of other additives (e.g. graphite as lubricant) kept constant. The mixtures were poured into 50 × 50 × 8 mm wooden moulds and cured in a hot press at 180 °C under 15MPa for 5 min, followed by post-curing at 180 °C for 4 h, conditions comparable to those used in epoxy–natural fibre composites (Mohammed et al., 2025; Iloabachie et al., 2023; Hassan et al., 2019).

3.3 Tensile Testing

Tensile tests were conducted following ASTM D638 using a TUE-C-100 universal testing machine. Dog-bone tensile specimens were machined from the plates and tested according to ASTM D638 using a universal testing machine. Ultimate tensile strength (UTS) was obtained from the ratio of maximum load to the initial cross-sectional area. A CCD-based RSM framework, similar to that employed for bamboo/epoxy, banana/epoxy and jute-reinforced systems, was used to establish a quadratic relationship between tensile strength and the three independent factors (reinforcement content, particle size and surface modification) and to evaluate model adequacy via ANOVA and regression diagnostics (Taghipoor & Mirzaei, 2023; Mohammed et al., 2025; Hassan et al., 2019; Sultana et al., 2020). Specimens were loaded axially in tension at a controlled cross-head speed, and tensile strength was calculated using:

$$UTS = \frac{UTL}{A \text{ (N/mm}^2\text{)}}$$

Where UTS is the ultimate tensile strength (N/mm²), UTL is the ultimate tensile load (N), and A is the cross-sectional area (mm²).

IV . RESULTS AND DISCUSSION

Tensile strength decreased monotonically with increasing pod content from 10 to 50 wt. % for both carbonized and un-carbonized composites, with the highest strengths obtained at 10 wt. % and 150µm. The un-carbonized composites consistently outperformed carbonized ones, reaching ≈16.4MPa compared to ≈14.2MPa at the optimum. This behaviour mirrors the optimized tensile performance observed in bamboo/epoxy and banana/epoxy systems, where low-to-moderate fibre loadings and fine particle sizes provide efficient stress transfer while avoiding excessive stress concentrations (Mohammed et al., 2025; Hassan et al., 2019).

ANOVA indicated that reinforcement content, particle size and carbonization state significantly affected tensile strength ($p \leq 0.05$), aligning with numerous RSM-based studies on kenaf, banana and jute fibre composites where filler fraction, fibre dimensions and surface treatments were the dominant determinants of tensile response (Taghipoor & Mirzaei, 2023; Yaghoobi & Fereidoon, 2018; Mohammed et al., 2025; Hassan et al., 2019; Sultana et al., 2020; Arunachalam et al., 2024). The CCD model yielded $R^2 \approx 0.996$ and close agreement between predicted and experimental strengths, comparable to R^2 values of 0.95–0.99 reported in similar optimization studies (Taghipoor & Mirzaei, 2023; Mohammed et al., 2025; Hassan et al., 2019; Sultana et al., 2020).

The reduction in strength at higher pod contents can be attributed to particle agglomeration, micro-voids and poor wetting, phenomena widely reported in agro-waste and nano-reinforced systems at elevated filler loadings (Taghipoor & Mirzaei, 2023; Mohammed et al., 2025; Iloabachie et al., 2023; Terán et al., 2024). In gum Arabic-modified binder sand matrices, analogous optima have been documented: low-to-moderate GA contents enhance stiffness and tensile-related properties, whereas higher levels introduce porosity and micro-cracking that degrade performance (Khan et al., 2023; Brzyski, 2021; Alsunbul et al., 2023; Alsadon et al., 2022; Terán et al., 2024). The superior strength of un-carbonized relative to carbonized pods is consistent with observations that aggressive thermal treatment can embrittle natural fillers and weaken interfacial adhesion, despite potential gains in thermal stability (Iloabachie et al., 2023; Okechukwu et al., 2025).

Overall, the optimized *Pentaclethra* pod–gum Arabic composites follow the same mechanistic pattern as other statistically designed natural-fibre systems: there exists a narrow window of filler content, particle size and surface condition within which interfacial bonding and stress transfer are maximized and beyond which defects and incompatibility dominate (Taghipoor & Mirzaei, 2023; Mohammed et al., 2025; Hassan et al., 2019; Sultana et al., 2020; Okechukwu et al., 2025; Arunachalam et al., 2024; Lakshmi et al., 2024). Within this window, the measured strengths are suitable for non-structural and semi-structural applications where low density, renewability and low cost are prioritized over very high load-bearing capacity.

4.1 Effect of Reinforcement Content and Particle Size

Experimental and predicted tensile strength values for carbonized and un-carbonized composites are as presented in Table 1. Maximum tensile strength was achieved at 10 wt. % reinforcement and 150µm particle size for both composite types. The un-carbonized composites achieved (16.4MPa) exceeding the carbonized samples (14.2MPa). Increasing filler content beyond 20 wt. % resulted in strength reduction due to agglomeration, poor matrix wetting, and stress concentration. The observed trend aligns with prior studies on agro-waste reinforced composites, where moderate filler loading and finer particle size enhanced stress transfer and stiffness, while excessive filler content led to brittleness, agglomeration, and micro-void formation (Daramola et al., 2023; Natrayan et al., 2024). Daramola et al. (2023) reported peak tensile strength at 10–20 wt. % agro-waste filler content, and Natrayan et al. (2024) observed similar behavior in hybrid bio-composites. Similar optimum loading behavior has been reported for gum Arabic-modified systems, emphasizing the importance of matrix–filler compatibility (Brzyski, 2021; Khan et al., 2023). Fine particle size enhances surface area and interfacial bonding, thereby improving stress transfer efficiency.

4.2 Effect of Surface Modification

Although carbonization improves thermal stability (Iloabachie et al., 2022), it reduced tensile strength in this study. The increased brittleness and diminished interfacial adhesion likely contributed to reduced load transfer efficiency and this is consistent with findings reported for heat-treated natural fillers (Iloabachie et al., 2022).

Table 1: Predicted Results vs. Experimental Results for Compressive Strength of Carbonized and Un-carbonized powdered *Pentaclethra Macrophylla* pod/Gum-Arabic Resin Composite.

Reinforcement (%)	Size (µm)	Surface Modification	TS (MPa)	P TS (MPa)
10	210	Carbonized	13.7	13.8150
20	210	Carbonized	13.2	12.8675
30	210	Carbonized	11.2	11.9200
40	210	Carbonized	11.0	10.9725
50	210	Carbonized	10.5	10.0250

10	150	Carbonized	14.2	13.8150
20	150	Carbonized	12.6	12.8675
30	150	Carbonized	11.8	11.9200
40	150	Carbonized	11.3	10.9725
50	150	Carbonized	9.7	10.0250
10	210	Un-carbonized	15.8	16.0350
20	210	Un-carbonized	14.2	15.0875
30	210	Un-carbonized	13.7	14.1400
40	210	Un-carbonized	13.3	13.1925
50	210	Un-carbonized	12.5	12.2450
10	150	Un-carbonized	16.4	16.0350
20	150	Un-carbonized	15.6	15.0875
30	150	Un-carbonized	14.8	14.1400
40	150	Un-carbonized	13.3	13.1925
50	150	Un-carbonized	11.8	12.2450

TS – Tensile Strength, PTS – Predicted Tensile Strength

4.3 Central Composite Design (CCD) and Statistical Analysis

CCD was employed to model tensile strength as a function of reinforcement content (A), particle size (B), and surface modification (C). Model adequacy was evaluated using R^2 , adjusted R^2 , predicted R^2 , PRESS, and ANOVA statistics.

Regression analysis confirmed that reinforcement content, particle size, and surface modification were statistically significant factors affecting tensile strength ($p \leq 0.05$). The developed CCD model exhibited excellent adequacy, with $R^2 = 0.9957$, adjusted $R^2 = 0.9932$, and predicted $R^2 = 0.9858$, Table 2, indicating strong predictive reliability and minimal over fitting.

Table 2: Model Statistics Summary

S	R-sq	R-sq(adj)	PRESS	R-sq(pred)	AICc	BIC
0.136343	0.9957	0.9932	0.736314	0.9858	2.84	-6.20

Table 3 below shows the table of coefficients as the subset of predictor terms chosen by the response surface regression model. It shows the constant value of the model equation and the coefficients of the predictor terms. For each of these terms it shows the coefficient, Coef, confidence intervals (CI), standard error (S.E) Coef, the T-value, the P-value and the variation inflation factor (VIF). The coefficient describes the size and direction of the relationship between a term in the model and the response variable (tensile strength) and also helps to minimize multi-co linearity (i.e. is the prediction of one model term from the others) among the terms. The coefficient for a term represents the change in the mean response associated with an increase of one coded unit in that term, while the other terms are held constant. The sign of the coefficient indicates the direction of the relationship between the term and the response.

The standard error of the coefficient is used to measure the precision of the estimate of the coefficient. From Table 3, it could be seen that the standard error (S.E) Coef for the powdered *Pentaclethra Macrophylla* Pod, particle size and surface modification for tensile strength are 0.0431, 0.0305 and 0.0305 respectively.

For a response surface regression model the components must be in the model. The t-value which measures the ratio between the coefficient and its standard error is used to determine the p-value, which tests whether the coefficient is statistically significant or not. The p-value α (test value) for these analyses is 0.15 and any term whose value is less than or equal to the p-value is statistically significant while any term with a value greater than the p-value is statistically insignificant. Therefore, it could be seen from Table 3 that the p-values of 0.003, 0.010 and 0.005 confirm that the powdered *pentaclethra macrophylla* pod, the particle size of the powdered *pentaclethra macrophylla* pod and the surface modification of the powdered *pentaclethra macrophylla* pod were all statistically significant in the model showing that there is a statistically significant association between the response variable i.e. tensile strength and the terms. The variance inflation factor (VIF) Table 3 indicates how much the variance of a coefficient is inflated due to correlations among the predictors in the model. Also, the variance inflation factor VIF is used to detect whether one predictor has a strong linear association with the remaining predictors (the presence of multi-co linearity among the predictors). VIF measures how much the variance of an estimated regression coefficient increases if the predictors are correlated (multi collinear). If VIF = 1, it indicates no relation; however, if VIF > 1, it indicates otherwise. The largest VIF among all predictors is often used as an indicator of severe multi co linearity. Iloabachie et al. (2023) suggested that when VIF is greater than 5-10, then the regression coefficients are poorly estimated. Table 3 indicates that the VIF=1 which signifies that the regression coefficients were adequately estimated. This confirms the adequacy and desirability of the model.

The confidence intervals Table 3 (CI) are ranges of values that are likely to contain the true value of the coefficient for each term in the model. The confidence interval helps to assess the practical significance of the results. The percentage of these confidence intervals that contain the parameter is the confidence level of the interval. The confidence interval is for assessing the estimate of the coefficient for each term in the model. Table 3 shows a 95% confidence level for the model. With a 95% confidence level, it can be said that the model is 95% confident that the confidence interval contains the value of the coefficient

Table 3: Coded Coefficients.

Term	Coef	SE Coef	95% CI	T-Value	P-Value	VIF
Constant	12.9036	0.0475	(12.8001, 13.0071)	271.59	0.000	
A	-1.5750	0.0431	(-1.6689, -1.4811)	-36.53	0.003	1.00
B	-0.4100	0.0305	(-0.4764, -0.3436)	-13.45	0.010	1.00
Surface Modification						
Carbonised	-1.0700	0.0305	(-1.1364, -1.0036)	-35.10	0.005	1.00
Uncarbonised	1.0700	0.0305	(1.1364, 1.0036)	35.10	0.000	*
A ²	-0.3071	0.0729	(-0.4659, -0.1484)	-4.21	0.001	1.00
A*B	-0.0750	0.0431	(-0.1689, 0.0189)	-1.74	0.108	1.00
A*C						

Carbonised	-0.0850	0.0431	(-0.1789, 0.0089)	-1.97	0.072	1.00
Un carbonised	0.0850	0.0431	(0.1789, 0.0089)	1.97	0.072	*
A*C						
Carbonised	0.0900	0.0305	(0.0236, 0.1564)	2.95	0.012	1.00
Un-carbonised	0.0900	0.0305	(0.0236, 0.1564)	2.95	0.012	*

4.4 Pareto Chart Analysis

The Pareto chart of Fig. 1 shows the absolute values of the standardized effects from the largest effect to the smallest effect. The standardized effects are t-statistics that test the null hypothesis that the effect is 0. The chart also plots a reference line to indicate which effects are statistically significant. From Fig. 1, the reference line is 1.54. On the Pareto chart of Fig. 1, bars that cross the reference line are statistically significant. It can be seen from Fig. 1 that all the terms are statistically significant. The Pareto chart is also used to determine the magnitude and the importance of the effects.

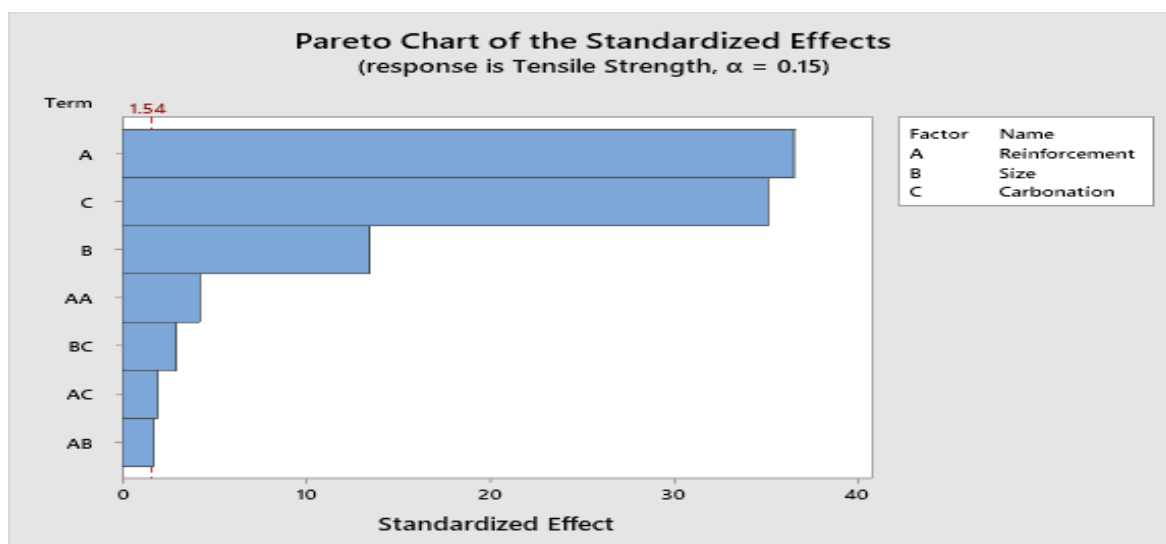


Fig. 1: Pareto Chart of Tensile Strength in Gum-Arabic Resin.

4.5 Residual Plot Analysis

Four residual plots were generated by the model for tensile strength by the model. These are: Normal plot of residuals, Histogram of residuals, Residuals versus fits and Residuals versus order, Fig. 2. The points on the normal plot should generally form a straight line if the residuals are normally distributed (Iloabachie et. al., 2025). If the points on the plot depart from a straight line, the normality assumption may be invalid. The points in this plot for tensile strength response formed a straight line, hence, it can be said that the normality assumption is valid (Iloabachie et. al., 2025). Furthermore, the histogram of residuals plot usually resembles a normal (bell-shaped) distribution with a mean of zero. Substantial clusters of points away from zero may indicate that factors other than those in the model may be influencing the result. There was no substantial cluster in the histogram of residuals plot for tensile strength response; therefore, it is safe to say that no factor other than those in the model is influencing the result.

Nevertheless, the Residuals versus fits plot should show a random pattern of residuals on both sides of 0. There should not be any recognizable patterns in the residual plot (Iloabachie et. al., 2025). The residual versus fits plots for tensile strength response showed no pattern so the constant variance assumption is valid.

The Residual versus order plot of the tensile strength residual is in the order that the data was collected and can be used to find non-random error, especially of time-related effects. The lack of pattern in this plot for the tensile strength response showed that the independence assumption is valid.

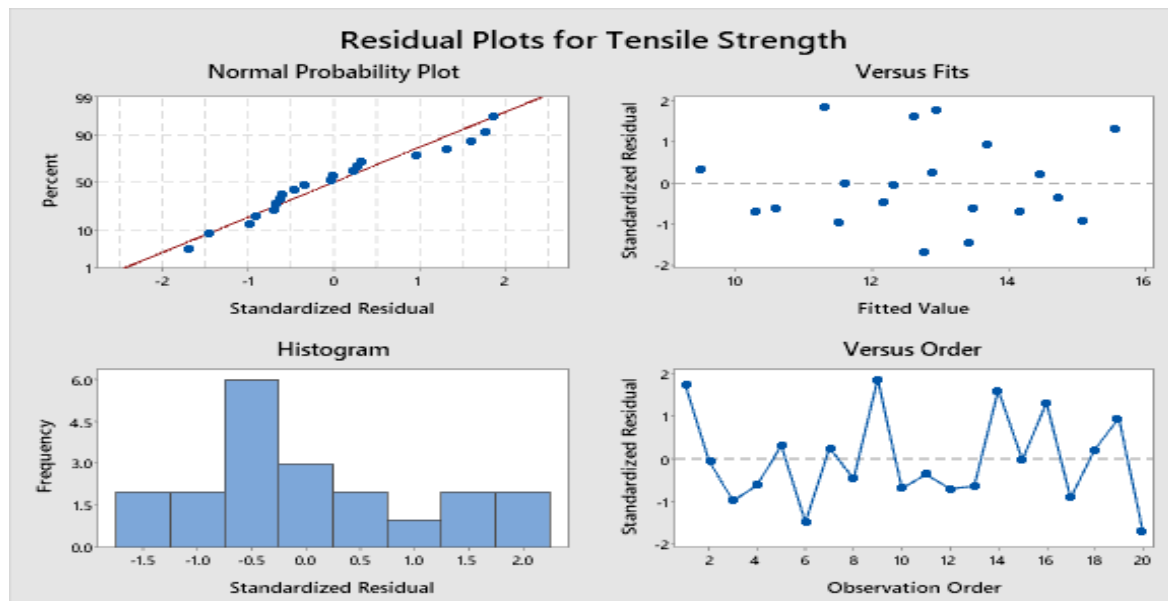


Fig. 2: Residual Plot of Tensile Strength in Gum-Arabic Resin.

4.6: Contour Plots Analysis

Contour Figs. 3&4 and surface Figs. 5&6 plots revealed that tensile strength decreased with increasing particle size and reinforcement fraction beyond 20 wt. %, with optimum values concentrated at 10–20 wt. % and 150 μ m particle size. Residual and Pareto analyses further validated the robustness and statistical soundness of the model.

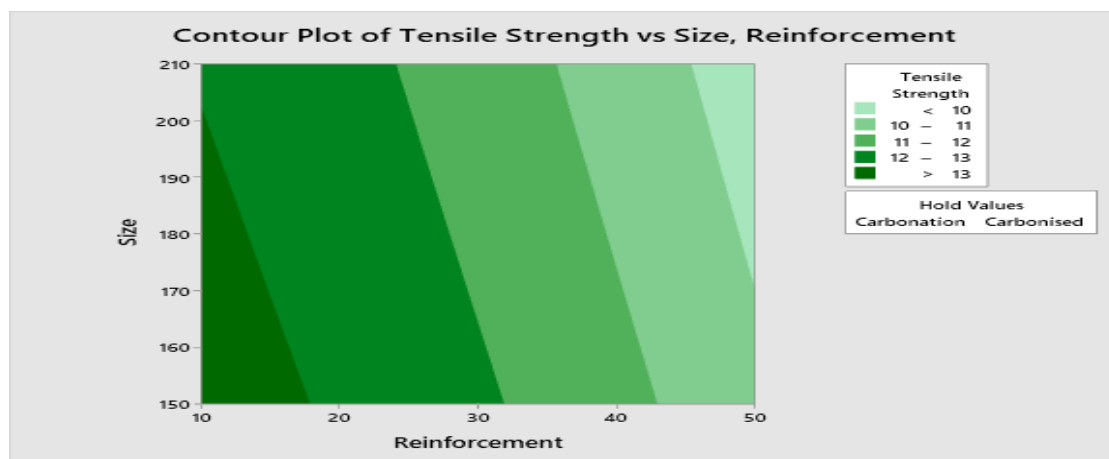


Fig. 3: Contour Plot of Tensile Strength vs. Particle Size, Reinforcement in Gum Arabic Resin (Carbonized)

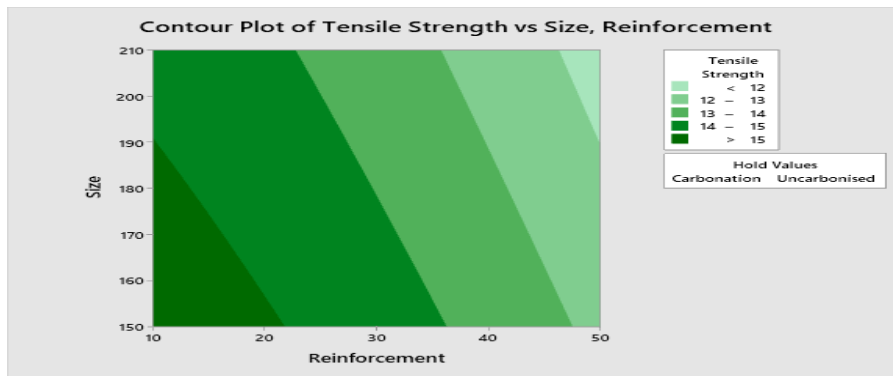


Fig. 4: Contour Plot of Tensile Strength vs. Particle Size, Reinforcement in Gum Arabic Resin (Un-Carbonized)

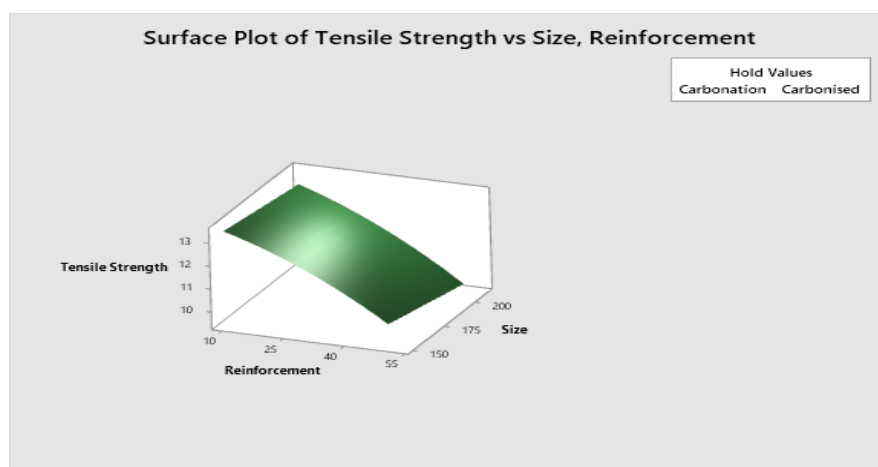


Fig. 5: Surface Plot of Tensile Strength vs Size, Reinforcement in Gum Arabic Resin (Carbonized)

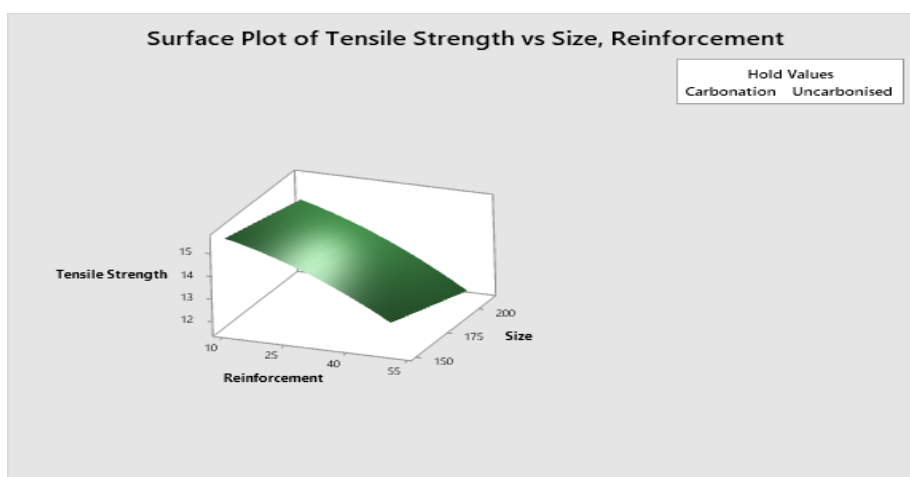


Fig. 6: Surface Plot of Tensile Strength vs. Size, Reinforcement in Gum Arabic Resin (Un-Carbonized)

4.7 CCD Model Adequacy

The regression model demonstrated excellent adequacy ($R^2 = 0.9957$; $R^2_{adj} = 0.9932$; $R^2_{pred} = 0.9858$). ANOVA confirmed that reinforcement content ($p = 0.003$), particle size ($p = 0.010$), and surface modification ($p = 0.005$) significantly influenced tensile strength. Variance Inflation Factor ($VIF = 1$) indicated absence of multi-co linearity.

Pareto and residual analyses validated statistical significance, normality, and independence assumptions, confirming model robustness.

Contour and surface plots indicated optimal tensile performance within 10–20 wt. % reinforcement and 150 μ m particle size, consistent with literature on agro-waste composite optimization (Daramola et al., 2023; Wang et al., 2023).

V. CONCLUSIONS

The tensile strength of powdered *Pentaclethra macrophylla* pod–gum Arabic bio-composites is strongly dependent on reinforcement content, particle size, and surface modification. Optimal tensile performance was achieved at approximately 10 wt. % reinforcement and 150 μ m particle size, yielding peak strengths of about 16.4MPa for un-carbonized and 14.2MPa for carbonized composites. Beyond this optimum, tensile strength declined due to filler agglomeration and matrix degradation. The integration of experimental testing with CCD-based optimization proved effective for identifying formulation windows and can be extended to the design of other sustainable bio-composite systems.

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