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Optimization Analysis of Hardness Test for Powdered *Pentaclethra macrophylla* Pod /Bio-Epoxy Resin Based Brake Pad Composite Using Central Composite Design

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

This work examined the optimization of hardness test for Powdered *Pentaclethra macrophylla* Pod /Bio-Epoxy Resin Based Brake Pad Composite Using Central Composite Design. The *Pentaclethra macrophylla* pod was manually cleaned and sun-dried for about three hours per day for three days. The sun-dried *Pentaclethra macrophylla* pod was later ovum dried at a temperature of about 110°C for three hours to achieve a constant weight and subsequently allowed to cool to room temperature. The ovum-dried *Pentaclethra macrophylla* pod was crushed to powder form using locally fabricated grinding machine and sieved. Part of the sieved powdered pod was carbonized in a heat treatment furnace at a temperature of about 950°C. Pre-impregnated process was used to prepare the brake pad composite samples. The weights of the powdered *Pentaclethra macrophylla* pods and bio-epoxy resin were varied while those of the lubricant, abrasives, friction modifier, catalyst and

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accelerator were kept constant The weights of the reinforcement i.e. powdered *Pentaclethra macrophylla* pods were varied between 10 wt. % and 50 wt. % at an interval of 10 wt. %. The formulation was poured into a wooden mould 50 mm × 50 mm × 8 mm placed in a hot platen press at temperature of about 180^oC, a moulding pressure of 15MPa and a curing time of 5 minutes. Post-heat treatment of the composites was performed in a hot air oven for a period of 4 hours at 180 °C. The produced brake pads were evaluated for hardness in accordance to ASTM D785 standard using Rockwell Scale K hardness testing machine. The results of the test showed that 150µm particle size reinforced brake pad sample had higher hardness values of 105.7 and 106.4 at 20wt. % and 30wt. % respectively. This result was also confirmed by the Central composite design (CCD) where maximum hardness values of 107.31 and 107.63 were obtained at 20wt. % and 30wt. % respectively.

Keywords: Pentaclethra macrophylla ; hardness test; epoxy resin; brake pad.

1. INTRODUCTION

The use of agro-waste as a friction material in the development of brake pad for automobile application has continued to gain the interest of researchers in recent times. Most agro-waste materials face poor disposal problems thereby constituting environmental and health challenges. Darius [1] identified materials composition, braking procedure and maintenance requirements as relevant factors in establishing the durability of any brake pad. Bijwe [2] and Subramanium et al. [3] reported "significant changes in the formulations of friction materials for the brake lining systems of automobiles". Mathur et al. [4] attributed "these changes to better heat resistance, higher coefficient of friction, and extended durability".

While Idris et al. [5] reported increasing researchers' global interest in utilizing industrial or agricultural wastes as friction materials in the development of brake pads due to foreign exchange earnings and environmental control, Sinha and Biswas [6] and Subramanium et al. [7] suggested Kevlar, glass fibre, steel wool, wollastonite, graphite fibres and a number of other types of mineral fibres as other possible replacements for asbestos in brake pads which Mathur et al. [4] observed met the requirements for better heat resistance, higher coefficient of friction, and extended durability. Mohan, [8] reported hardness of brake pad as one of its critical properties; however, Mathur et al. [4] expressed negative concerns about the hardness of these many non-asbestos materials. Therefore, Mathur et al. [4] opined that for a brake pad to overcome adverse braking conditions and offer durability to the braking system, the friction material should be hard.

Iloabachie et al. [9] observed "the presence of Al_2O_3, K_2O, CaO, MgO, SiO_2, Fe_2O_3, and P_2O_5 as the dominant oxides in *Pentaclethra macrophylla* Pod and concluded that the presence of hard metal oxides like silica-SiO_2, alumina-Al_2O_3 and hematite -Fe_2O_3 in *Pentaclethra macrophylla* Pod explains the hard nature of *Pentaclethra macrophylla* Pod, hence can be used as a friction material to develop a brake pad". Furthermore, Iloabachie et al. [9] hinted that "SiO_2, Al_2O_3 and Fe_2O_3 act as abrasive materials in brake pad by increasing friction between the pad and the disc and also control friction film build-up".

This work therefore explored the optimal design hardness of brake pad produced using powdered *Pentaclethra macrophylla* Pod.

2. MATERIALS

The following materials were used in this research work: powdered *Pentaclethra macrophylla* Pod, bio-epoxy with its hardener, distilled water, egg shell, waste car tire and graphite as lubricant, mould release agent.

3. METHODS

The *Pentaclethra macrophylla* Pod was manually cleaned by removing dirt and debris. This was followed by thorough washing with distilled water and sun-dying for about eighteen hours i.e. six hours per day for three days under atmospheric condition. The sundried *Pentaclethra macrophylla* Pod was ovum dried at a temperature of about 110^oC for three hours to achieve a constant weight and allowed to cool. The cooled *Pentaclethra macrophylla* Pod was crushed into powdered form using a locally fabricated pulverizing machine. This was followed by sieving using a set of sieves arranged in descending order of fineness in accordance with BS1377:1990 standard as was reported by Rajan et al. (2013) at the soil laboratory, Civil Engineering Department, Institute of Management and Technology, Enugu.

The sieved *Pentaclethra macrophylla* Pod was shared into two with one portion carbonized and the other un-carbonized.

Pre-impregnated process was used to prepare the brake pad composite samples. The weights of the powdered *Pentaclethra macrophylla* pods and bio-epoxy resin with its hardener were varied while those of the filler, lubricant and friction modifier kept constant. Powders of the *Pentaclethra macrophylla* pod reinforcement, waste car tire, graphite lubricant, and filler were mixed in a separate container and then poured into the bio-epoxy resin and the mixture stirred further to obtain a homogenous mixture. The reinforcement i.e. powdered *Pentaclethra macrophylla* pod was varied in the order 10wt.%, 20 wt.%, 30 wt.%, 40 wt.% and 50 wt.%. The formulation was poured into a wooden mould 50 mm × 50 mm × 8 mm placed in a hot platen press at temperature of about 180^{o}C, a moulding pressure of 15MPa and a curing time of 5 minutes. Post-heat treatment of the composites was performed in a hot air oven for a period of 4 hours at 180°C Kumar and Bijwe [10]. The post–cured brake pad composite was then tested for hardness. The above process was used for the carbonized (PMCp) and uncarbonized (PMUNp) powdered *Pentaclethra macrophylla* Pods.

3.1 Hardness Test

The machine model used was Type DVRB-M 220/240 V. The hardness tests were performed according to ASTM D785 standard using Rockwell Scale K hardness testing machine. The proper indenter ball 1/8” for scale K was installed. The indenter was put into the pressure shaft so that flat part of the indenter cylinder was in front of the Allen screw. The Allen screw was slightly tightened and the machine switched on by the main switch in front of the panel. Loading force of 100kg was selected using the lever force on the right side of the machine. The developed brake pads samples were placed on the anvil and lifted against the indenter. The anvil was lifted with the test specimen carefully until the green light in front of the panel comes on. The red light on the panel signals the completion of the test and the machine switches off automatically.

4. RESULTS AND DISCUSSION

4.1 Central Composite Design (CCD) Optimization

Mixture experiments are a special class of response surface experiments in which the product under investigation is made up of several components or ingredients. Designs for these experiments are useful because many product design and development activities in industrial situations involve formulations or mixtures. In these situations, the response is a function of the proportions of the different ingredients in the mixture.

Table 1a shows the table of coefficients as the subset of predictor terms chosen by the response surface **r**egression 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 (hardness in this instance) and also helps to minimize multi-collinearity (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. The smaller the values of standard error, the more precise the estimates for the term. From Table 1a it could be seen that the standard error (S.E) Coef for the powdered *Pentaclethra macrophylla* Pod, particle size and surface modification for hardness are 1.15, 0.810 and 0.810 respectively.

For a response surface **r**egression 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 1a that the p-values of 0.010, 0.012 and 0.023 confirms 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. hardness and the terms. The variance inflation factor (VIF) Table 1a 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. Montgomery and Peck (2015) suggested that "when VIF is greater than 5-10, then the regression coefficients are poorly estimated. Table 1a indicates that the VIF=1 which signifies that the regression coefficients were adequately estimated. This confirms the adequacy and desirability of the model".

These confidence intervals (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 1a 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 .

The model statistics summary table, Table 1b gives some important information about the model. It shows four important statistics that describes the model. These are S-value, R-squared, adjusted R-squared, predicted R-Squared. S represents the standard deviation of the distance between the data values and the fitted values. It is used to assess how well the model describes the response. The lower the value of S the better the model; for all four responses the value of S is sufficiently low and this indicates that the model describes the responses appropriately.

The R-squared is the percentage of variation in the response that is explained by the model. The higher the R^2 value the better the model fits the data. The adjusted R^2 is the percentage of the variation in the response that is explained by the model, adjusted for the number of predictors in the model relative to the number of observations. The adjusted R-squared value should be close to the R-squared value the difference between the two values should not be more than 4%.

From Table 1b, it can be seen that the values for R- square, R-square adjusted and R-square predicted for the hardness mixture design model are 0.9795, 0.9562 and 0.8938 respectively. The independent variables in the model and the effect of each variable were evaluated. Therefore, to evaluate the adequacy of the selected model, several appraisal techniques were used. The coefficient of determination (R^2), the adjusted determination coefficient (adjusted R^2) and variation inflation factor (VIF) were used to weigh the adequacy of the model as has been used by some researchers, Chen et al. [11]. The predicted R^2 of 0.8938 is in reasonable agreement with adjusted R^2 of 0.9562. Also, the difference between the R-squared value and the adjusted R^2 two is 0.0233. The difference is less than 4% as suggested by the model. This therefore, confirms the adequacy of the model.

In addition, the predicted R-squared is used to determine how well a model predicts the response for new observations. The higher the value the better the model is in predicting accurate response for new observation. The predicted R^2 value of 0.8938 is high enough to indicate the good ability to predict and also reasonably close to the R-squared value to indicate that the model is not over-fit.

The Model F-value of 40.73 shown in Table 1c implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. Values of p-value less than 0.15 indicate that the model terms are significant. In this case A, B, C, AB, A^2, BC, AC are significant model terms. Values greater than 0.15 indicate the model terms are not significant.

Therefore the equation in terms of coded factors was developed which can be used to make predictions about the response for given levels of each factor. By default, the high levels of factors are coded as +1 and the low levels of the factors are coded as -1.

Table 1a. Coded coefficients for hardness of developed powdered *Pentaclethra macrophylla* pod/bio-epoxy resin based brake pad composite

Term	Coef	SE Coef	95% CI	T-Value	P-Value	VIF
Constant	94.60	1.26	(91.88, 97.33)	74.96	0.000	
A-Reinforcement	-3.44	1.15	(-5.91, -0.96)	-3.00	0.010	1.00
B-Particle Size	-5.840	0.810	(-7.590, -4.090)	-7.21	0.012	1.00
C-Surface Modification						
Carbonised	-9.420	0.810	(-11.170, -7.670)	-11.63	0.023	1.00
Uncarbonised	9.420	0.810	(7.670, 11.170)	11.63	0.023	*
A^2	-11.91	1.94	(-16.09, -7.72)	-6.15	0.000	1.00
A*B	1.87	1.15	(-0.60, 4.35)	1.64	0.126	1.00
B*C						
Carbonised	-2.230	0.810	(-3.980, -0.480)	-2.75	0.016	1.00
Uncarbonised	2.230	0.810	(0.480, 3.980)	2.75	0.016	*

Table 1b. Hardness model summary statistics of developed powdered *Pentaclethra macrophylla* pod/bio-epoxy resin based brake pad composite

S	R-sq	R-sq (adj)	PRESS	R-sq (pred)	AICc	BIC
3.62166	97.95%	95.62%	426.215	89.38%	128.71	123.59

PRESS = Predicted residual sum of squares

Table 1c. Analysis of variance for hardness of developed powdered *Pentaclethra macrophylla* pod/bio-epoxy resin based brake pad composite

Source	DF	Seq SS	Contribution	Adj SS	Adj MS	F-Value	P-Value
Model	6	3205.68	94.95%	3205.68	534.28	40.73	0.001
Linear	3	2574.83	76.26%	2574.83	858.28	65.44	0.000
A-Reinforcement	1	117.99	3.49%	117.99	117.99	9.00	0.010
B-Particle Size	1	682.11	20.20%	682.11	682.11	52.00	0.011
C-Surface Modification	1	1774.73	52.57%	1774.73	1774.73	135.31	0.014
Square	1	496.23	14.70%	496.23	496.23	37.83	0.000
A^2	1	496.23	14.70%	496.23	496.23	37.83	0.020
2-Way Interaction	2	134.61	3.99%	134.61	67.31	5.13	0.023
A *B	1	35.16	1.04%	35.16	35.16	2.68	0.126
B*C	1	99.46	2.95%	99.46	99.46	7.58	0.016
Error	13	170.51	5.05%	170.51	13.12		
Total	19	3376.19	100.00%				

Table 1d. Design factors and levels

Variable	Actual value		Coded value	
	Low level	High level		
Filler wt%	-	+	- 1	+ 1
Particle size	-	+	- 1	+ 1
Surface modification	-		- 1	+1

Table 2. Predicted results vs experimental results for hardness of carbonized and un-carbonized powdered *Pentaclethra macrophylla* pod/bio-epoxy resin brake pad composite

Reinforcement (%)	Particle Size (μm)	Surface Modification	Experimental Rockwell Hardness	Predicted Rockwell Hardness
10	210	Carbonized	65.5	66.766
20	210	Carbonized	69.6	74.917
30	210	Carbonized	81.0	77.114
40	210	Carbonized	73.7	73.357
50	210	Carbonized	66.0	63.646
10	150	Carbonized	89.5	86.656
20	150	Carbonized	92.0	92.932

Reinforcement (%)	Particle Size (µm)	Surface Modification	Experimental Rockwell Hardness	Predicted Rockwell Hardness
30	150	Carbonized	98.4	93.254
40	150	Carbonized	83.9	87.622
50	150	Carbonized	72.7	76.036
10	210	Un-carbonized	92.6	90.066
20	210	Un-carbonized	98.4	98.217
30	210	Un-carbonized	103.2	100.414
40	210	Un-carbonized	93.3	96.657
50	210	Un-carbonized	84.8	86.946
10	150	Un-carbonized	99.0	101.036
20	150	Un-carbonized	105.7	107.312
30	150	Un-carbonized	106.4	107.634
40	150	Un-carbonized	102.3	102.002
50	150	Un-carbonized	95.0	90.416

The equation in terms of coded factor is shown as;

Hardness equation for carbonised sample= $128.8 + 1.052\,A - 0.3628\,B - 0.02977\,A^2 + 0.00313A{*}B$

Hardness equation for un-carbonised sample= $120.9 + 1.052A - 0.2141\,B - 0.02977\,A^2 + 0.00313\,A{*}B$

A, stands for powdered *Pentaclethra macrophylla* pod (reinforcement) while B, stands for particle size of the reinforcement.

The equation in terms of actual factor is used to make predictions about the response for given levels of each factor. The levels are specified in original units for each factor [12,13].

The response values obtained by inserting the independent values are the predicted values of the model. These values were compared to the actual and experimental values. The result of the comparison is shown in Fig. 1.

The Pareto chart of Fig. 2 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. 2, the reference line is 1.53. On the Pareto chart of Fig. 2, bars that cross the reference line are statistically significant. It can be seen from Fig. 2 that all the terms are statistically significant. The Pareto chart is also used to determine the magnitude and the importance of the effects.

Four residual plots were generated by the model for hardness by the model. These are: Normal plot of residuals, Histogram of residuals, Residuals versus fits and Residuals versus order, Fig. 3. The points on the normal plot should generally form a straight line if the residuals are normally distributed. If the points on the plot depart from a straight line, the normality assumption may be invalid. The points in this plot for hardness response formed a straight line, hence, it can be said that the normality assumption is valid. 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 hardness 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. The residual versus fits plots for hardness response showed no pattern so the constant variance assumption is valid.

The Residual versus order plot of the hardness residual is in the order that the data was collected and can be used to find non-random error, especially of time-related effects [14,15]. The lack of pattern in this plot for the hardness response showed that the independence assumption is valid.

Contour plots display the 3-dimensional relationship in two dimensions, with x- and y-factors (predictors i.e. particle size of the powdered *Pentaclethra macrophylla* pod on the y-axis and percentage composition of the reinforcement on the x-axis) plotted on the x- and y-scales and response values i.e. hardness represented by contours Fig. 3a and Fig. 3b. A contour plot is like a topographical map in which x-, y-, and z-values are plotted instead of longitude, latitude, and elevation. The darker regions of the contour identify higher hardness values.

Fig. 3a and Fig. 3b represent the carbonized and un-carbonized forms of the powdered *Pentaclethra macrophylla* pod reinforcement in the brake pad formulation. From Fig. 3a and Fig. 3b, it is evident that maximum hardness value of greater than 90 for the carbonized sample and greater 100 for the un-carbonized brake pad samples was obtained at 150 μm between 20wt. % and 30wt. % of the reinforcement. It could also be observed that the bigger particle sizes of the reinforcement had lower hardness values for the both carbonized and un-carbonized samples of the developed brake pads.

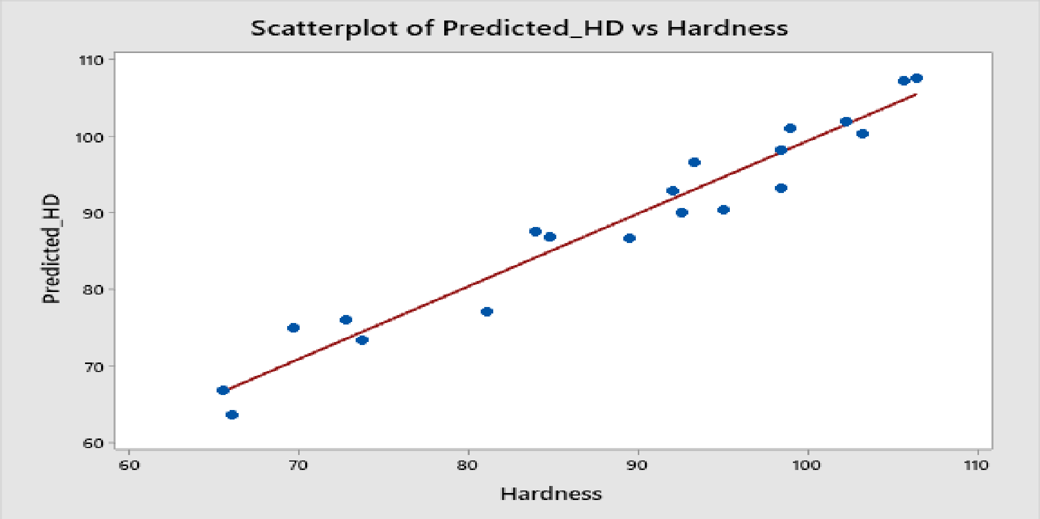

Fig. 1. Hardness pareto chart analysis of developed powdered *Pentaclethra macrophylla* pod/bio-epoxy resin based brake pad composite

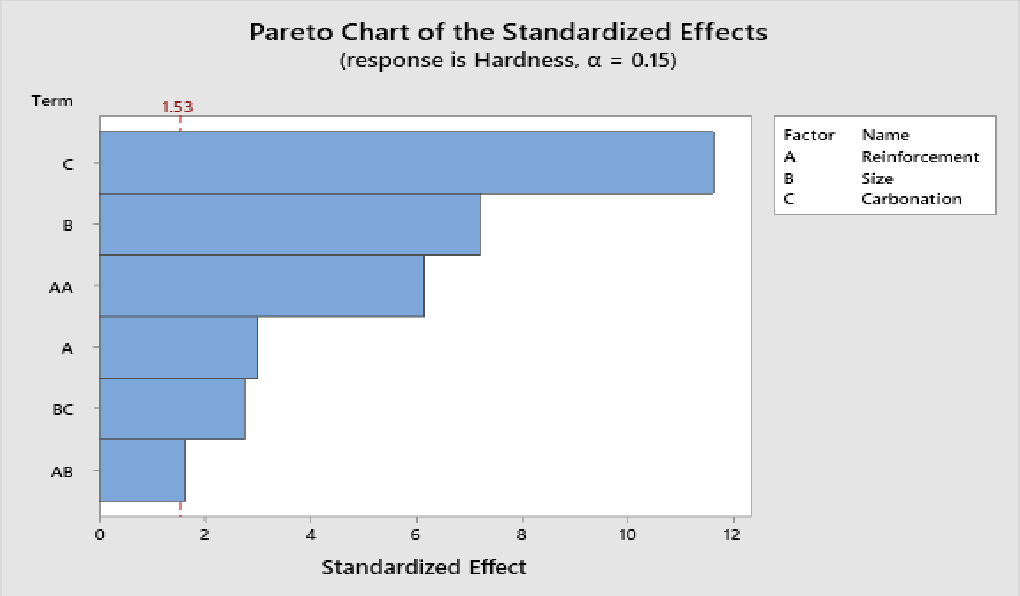

Fig. 2. Hardness residual plots analysis of developed powdered *Pentaclethra macrophylla* pod/bio-epoxy resin based brake pad composite

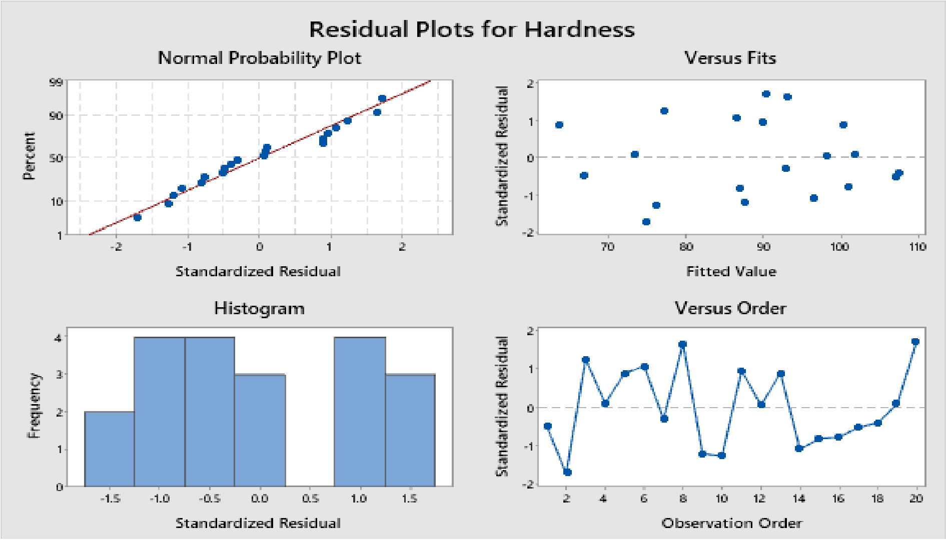

Fig. 3. Hardness contour plots analysis of developed powdered *Pentaclethra macrophylla* pod/bio-epoxy resin based brake pad composite

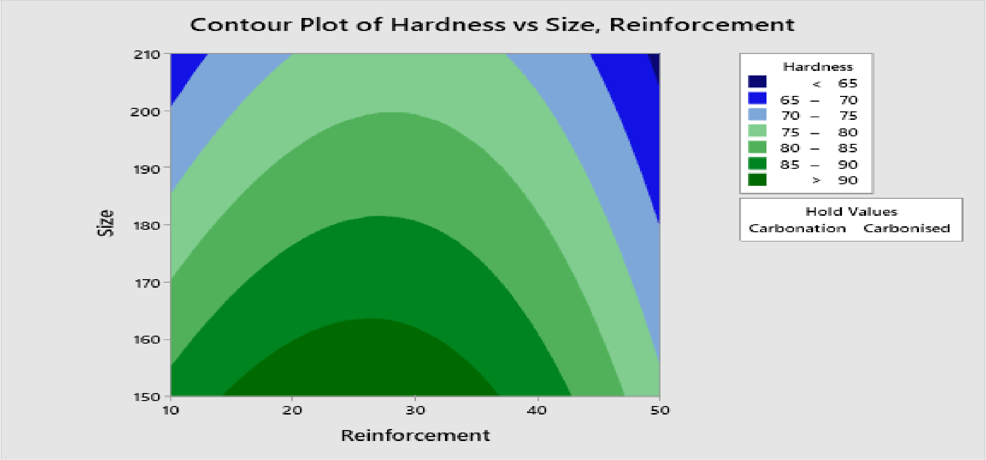

Fig. 3a. Contour plots for hardness of developed carbonized powdered *Pentaclethra macrophylla* pod /bio-epoxy resin based brake pad composite

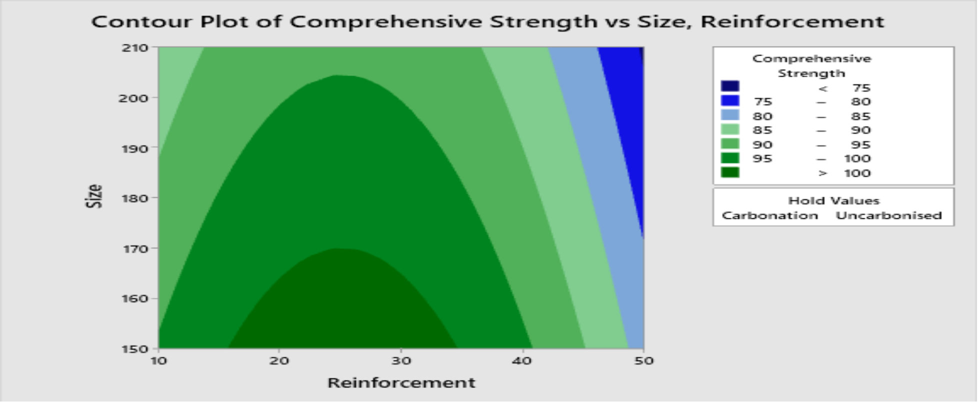

Fig. 3b. Surface plots analysis of developed powdered *Pentaclethra macrophylla* pod/bio-epoxy resin based brake pad composite

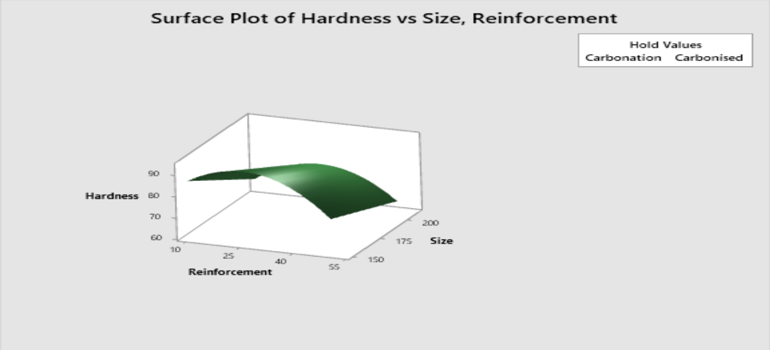

Fig. 4a. Surface plot (3-D) for hardness of developed carbonized powdered *Pentaclethra macrophylla* pod /bio-epoxy resin based brake pad composite

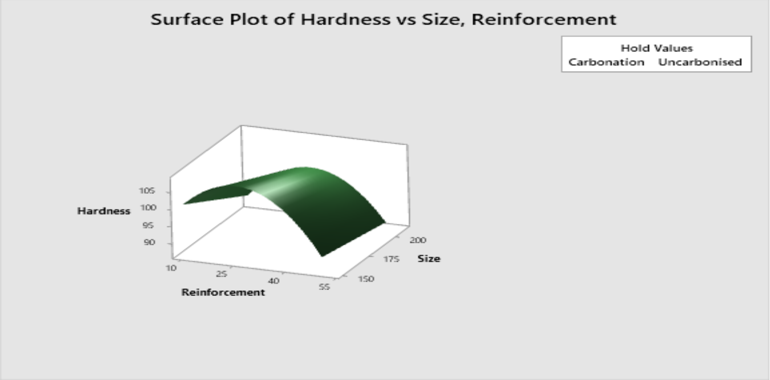

Fig. 4b. Surface plot (3-D) for hardness of developed un-carbonized powdered *Pentaclethra macrophylla* pod /bio-epoxy resin based brake pad composite

3D surface plots are used to explore the potential relationship between three variables. Fig. 4a and Fig. 4b showed the 3D surface plots of the carbonized and un-carbonized powdered *Pentaclethra macrophylla* pod reinforcement in bio-epoxy resin brake pad formulation The predictor variables i.e. particle size of the powdered *Pentaclethra macrophylla* pod on the y-axis and percentage composition of the reinforcement on the x-axis are displayed on the x- and y-scales, and the response (z) variable hardness represented by a smooth surface (3D surface plot).

From Fig. 3a and Fig. 3b, it can be observed that the 150 µm particle size of the reinforcement had higher hardness value than the 210 µm particle size of the reinforcement in both the carbonized and un-carbonized samples of the developed brake pad samples.

5. CONCLUSION

Powdered *Pentaclethra macrophylla* pod can be used as a suitable replacement for asbestos to improve the hardness of asbestos-free brake pad for automobile application.

Better hardness value of 107.634 was recorded by the 150µm particle size of the reinforcement for the un-carbonized brake pad sample at 30wt. % of the reinforcement as a result of the optimization.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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