THE OPTIMIZATION OF ALUMINUM-BASED COMPOSITE OF COCONUT SHELL CHARCOAL AND COW BONE ASH USING D-OPTIMAL MIXTURE DESIGN

*[1]Oghenekevwe Oghoghorie and [2]Isaac Imariabe Ogiku

[1]Department of Mechanical Engineering, Benson Idahosa University, Benin City, Edo State, Nigeria.
[2]Department of Mechanical Engineering, University of Benin, Benin City, Edo State, Nigeria.

Corresponding authors' email: ooghoghorie@biu.edu.ng

ABSTRACT

Researchers now focus on new technologies in the development of automobile components with lightweight properties, good wear resistance, high hardness, and high tensile strength is one of the primary goals of designers and manufacturers. In this study, an Aluminium-Based Composite Material that comprises Coconut Shell Charcoal and Cow Bone Ash as reinforcement was developed. A stir casting method was used in the production of the specimens. The responses considered in this study are hardness, tensile strength, melting temperature, density, wear index, and creep rate. A total of 16 runs were generated by the D-Optimal Design Expert software. With the help of the D-Optimal design expert software, an optimized blend of the matrix and the reinforcement particles was obtained as follows; 81.75% weight of aluminium, 8.36% weight of coconut shell charcoal and 9.90% weight of cow bone ash with a desirability of 95.20%. The developed model in this study predicted the responses with their accuracy for hardness, tensile strength, melting temperature, density, creep rate and wear index as 99.88%, 98.70%, 99.56%, 99.69%, 99.10% and 94.23% respectively.

Keywords: Agro Waste Composite, Tensile Strength, Wear Rate, Hardness, Density, Melting Temperature, Optimization

INTRODUCTION

Currently, the understanding of new technologies in the development of automobile components with lightweight properties, good wear resistance, high hardness, and high tensile strength is one of the primary goals of designers and manufacturers, according to Khan *et al.,* 2025. To have a single material that will possess these properties, a composite material needs to be produced, which can be applied in developing automobile parts. To achieve high strength, metallic matrix materials often require high-modulus reinforcements, and the resulting composites can surpass most alloys in terms of strength-to-weight ratios according to Arulprasanna and Omkumar*,* 2023. Oghoghorie and Ebhojiaye (2024), studied the Effect of Coconut Shell Charcoal and Cow Bone Ash on the Tensile Strength and Melting Temperature of aluminum-based composite. The results obtained showed that the presence of coconut shell charcoal and cow bone ash particles in the aluminum matrix increased the tensile strength and melting temperature of the aluminum by 47.21% and 10.71%, respectively. Oghoghorie *et al.,* 2022 Studied the Assessment of The Wear Resistance and The Density Reduction of Aluminium When Reinforced with Coconut Shell Charcoal and Cow Bone Ash. Although from the composite produced by Oghoghorie *et al.,* 2022 and Oghoghorie and Ebhojiaye, 2024, Melting Temperature, Tensile Strength, Wear Index, and Density tests were conducted by them, but the data obtained were not Optimised, hence this study. Therefore, in this research, the Design of Experiment used by Oghoghorie *et al.,* 2022, and Oghoghorie and Ebhojiaye, 2024, will be used to develop a composite material with Aluminium as the matrix element while Coconut Shell Charcoal, Cone Bone Ash as the reinforcements. Two additional responses will be added to the responses considered by Oghoghorie *et al.,* 2022 and Oghoghorie and Ebhojiaye (2024) before the optimization. The additional responses are Hardness and Creep.

MATERIALS AND METHODS

Aluminium scrap is the matrix element, which was reinforced with coconut shell charcoal, Cow Bone Ash, and a magnesium metal powder was added to serve as a wetting agent to encourage proper bonding of the material. Other materials are, crucible furnace, an electric furnace, a digital mass balance, a green sand mould, stir casting rig machine.

Methods

Design of Experiment (DOE) using D-Optimal Mixture Design

A three variable mixture design was used to design the experimental plan for this work. Table 1 shows the Coded and actual levels of the factors and Table 2 shows the Experimental design matrix. These were adopted from the work of Oghoghorie *et al.,* 2022 and Oghoghorie and Ebhojiaye, 2024. Thus, for the three-component mixture investigated in this study, the constraint is defined as shown in Equation 1

$$0 \leq X_i \leq 100 \tag{1}$$

Where $i = 1, 2, 3$

The summability of the components is shown in Equation 2

$$X_1 + X_2 + X_3 = 100 \tag{2}$$

The Design Expert® software version 7.0.0 was used and it was also used to develop the statistical models that correlated the input factors to the chosen responses. A total of 16 runs were generated by the Design Expert software.

Table 1: Coded and Actual Levels of the Factors (Oghoghorie and Ebhojiaye, 2024)

Factors	Symbols	Unit	Variable levels	
			Low level	**High level**
Aluminum scraps	X_1	%	69	98
Coconut shell charcoal	X_2	%	1	20
Cow bone ash	X_3	%	1	20

Table 2: Experimental Design Matrix (Oghoghorie and Ebhojiaye, 2024)

Run	Actual Values of Factors		
	Aluminum Scraps (%)	**Coconut Shell Charcoal (%)**	**Cow Bone Ash (%)**
1	69	11	20
2	79	1	20
3	79	20	1
4	90	1	9
5	79	1	20
6	69	11	20
7	85	1	14
8	69	19	12
9	98	1	1
10	84	14	1
11	69	19	12
12	76	14	10
13	79	20	1
14	98	1	1
15	90	9	1
16	83	9	8

Composite development

The composite development was done stated by Oghoghorie et al. (2022). The developed composite is shown in figures 1 and 2.

Figure 1: Creep Test Samples Test

Figure 2: Developed Specimens for Hardness Test

Tests Conducted

The ASTM E384 standard Vickers hardness test was used in this research to obtain the hardness of the different composites developed.

RESULTS AND DISCUSSION

Test Results

The ASTM E139 and ASTM E139 standards were used for the creep and hardness tests respectively

Hardness and Creep Test Results

Table 3 shows Hardness and Creep Test results.

Table 3: Hardness and Creep Test Results

Samples	Average of Vickers Hardness Number HV	% Elongation/hr (Creep Rate)
1	106.0	1.75
2	87.3	1.14
3	99.7	2.27
4	86.5	1.40
5	93.3	1.35
6	102.1	2.00
7	85.6	1.54
8	103.3	1.32
9	97.6	1.77
10	98.9	1.83
11	103.4	1.38
12	104.6	1.65
13	99.3	2.27
14	90.0	2.14
15	101.4	1.83
16	80.7	1.41

Statistical Modelling
The development of statistical models for the five responses was done by assessing the suitability of the linear, quadratic, special cubic, and cubic models in the Design Expert model library. The linear model shown in Equation 4

$$Y = \sum_{i=1}^{N} b_i X_i \qquad (4)$$

Where Y is the dependent variable or predicted response, X_i is the independent variables, b_i is the regression coefficient
The quadratic model shown in Equation 5,

$$Y = \sum_{i=1}^{N} b_i X_i + \sum_{i,j=1}^{N} b_{ij} X_i X_j \qquad (5)$$

X_j is the independent variable or factor, while b_{ij} is the coefficient of the interaction terms.
For the special cubic and cubic models, the candidate points incorporate the vertices, the thirds of edges, the constraint plane centroids, the overall centroid, and the axial points as shown in Equations 6 and 7.

$$Y = \sum_{i=1}^{N} b_i X_i + \sum_{i,j=1}^{N} b_{ij} X_i X_j + \sum_{i=1}^{N} b_{ijk} X_i X_j X_k \qquad (6)$$

$$Y = \sum_{i=1}^{N} b_i X_i + \sum_{i,j=1}^{N} b_{ij} X_i X_j + \sum_{i=1}^{N} b_{ijk} X_i X_j X_k + \sum_{i=1}^{N} b_{ij} X_i X_j (X_i - X_j) \qquad (7)$$

Analysis of Formulated Models
The models formulated to predict the six responses were statistically analyzed to assess their level of fit with the actual experimental observations. This was done using the built-in analysis of variance (ANOVA) tool in the Design Expert

software. The ANOVA parameters used for this purpose were p-value, F-value, sum of squares, mean square, lack of fit, standard deviation, coefficient of variation, coefficient of determination (R^2), adjusted R^2, predicted (R^2), adequate precision, and predicted residual sum of squares (PRESS) (Amenaghawon and Amagbewan, 2017).

Numerical Optimization
The optimization process employed the Design Expert software, focusing on enhancing desirable responses such as hardness, tensile strength, and melting temperature, while reducing undesirable factors like density, wear index, and creep rate. Notably, Sahin (2016) references previous research on mixture design methods, including a tutorial by Cafaggi et al. (2003) demonstrating their application in pharmaceutical formulations. Bezerra et al. (2008) and Leardi (2009) detailed the utilization and benefits of Response Surface Methodology (RSM) in chemistry, with Leardi also presenting three practical examples of its application. Table 3 shows the summary of the factors and responses of the composite. The Melting Point, Density, Tensile Strength, and Wear Rate results were obtained from the works of Oghoghorie *et al.* 2022 and Oghoghorie and Ebhojiaye, 2024. The Hardness and Creep test results were obtained from the test carried out in this research. Table 4 shows the summary of the factors and responses of the composite.

Table 4: The Summary of the Factors and Responses of the Composite

	Factors			Responses					
Run	Aluminum (%)	Coconut Shell Charcoal (%)	Cow Bone Ash (%)	Hardness (HV)	Tensile strength (MPa)	Melting temperature (ºC)	Density (kg/m³)	Wear Index (10^{-4}) (mg/rpm)	Creep rate (%/hr)
1	69	11	20	106.0	163.84	598	2608.33	0.344	1.75
2	79	1	20	87.3	149.33	606	2477.78	0.428	1.14
3	79	20	1	99.7	159.78	602	2591.67	0.552	2.27
4	90	1	9	86.5	185.36	580	2350.00	0.336	1.40
5	79	1	20	93.3	142.58	585	2641.67	0.314	1.35
6	69	11	20	102.1	184.21	605	2691.66	0.492	2.00
7	85	1	14	85.6	170.59	595	2500.00	0.364	1.54
8	69	19	12	103.3	170.84	595	2669.23	0.316	1.32
9	98	1	1	97.6	165.50	620	2395.00	0.526	1.77
10	84	14	2	98.9	176.07	575	2485.71	0.510	1.83
11	69	19	12	103.4	170.94	595	2471.42	0.312	1.38
12	76	14	10	104.6	205.35	580	2482.35	0.334	1.65
13	79	20	1	99.3	157.13	602	2583.33	0.548	2.27
14	98	1	1	90.0	141.39	585	2676.92	0.446	2.14
15	90	9	1	101.4	159.83	607	2421.43	0.302	1.83
16	83	9	8	80.7	173.68	605	2500.00	0.316	1.41

Assessment of the Experimental Design
The standard error of the model terms was seen to be small, a situation that is desirable as shown in Table 5

Table 5: Estimated Standard Error of Design Model Terms

Term	Standard error	VIF	R_i^2	Power at 5 % alpha level for effect of		
				0.5 Std. Dev.	1 Std. Dev.	2 Std. Dev.
X_1	0.67	1.97	0.4932	5.7 %	7.7 %	16.3 %
X_2	2.43	14.55	0.9313	5.4 %	6.6 %	11.7 %
X_3	2.31	12.74	0.9215	5.4 %	6.8 %	12.2 %
$X_1 X_2$	5.95	7.86	0.8728	6.1 %	9.4 %	23.0 %
$X_1 X_3$	5.72	7.09	0.8590	6.2 %	9.7 %	24.5 %
$X_2 X_3$	8.01	15.25	0.9344	5.6 %	7.4 %	14.8 %

Table 6 shows the leverage values of the design space. The maximum leverage value was 0.45, which is less than unity and indicate that no experimental runs exhibit extreme leverage that could compromise the validity of the results according to MacKinnon *et al.,* 2023.

Table 6: Estimated Model Leverages

Standard order	Leverage
1	0.4000
2	0.4472
3	0.3860
4	0.3791
5	0.3882
6	0.4064
7	0.3402
8	0.3318
9	0.2685
10	0.3050
11	0.3195
12	0.4472
13	0.4064
14	0.4000
15	0.3882
16	0.3860

Tables 7 and 8 show the matrix of correlation coefficients and Pearson's r matrix of factors, respectively. These matrices show the correlation between the regression coefficients and the characteristics, and are thus used to compare a design's capability. The desirable situation is that the off-diagonal values of the matrices should be close to zero for the design to be deemed adequate. Indeed, that was the situation with the results obtained.

Table 7: Correlation Matrix of Regression Coefficients

	X_1	X_2	X_3	X_1X_2	X_1X_3	X_2X_3
X_1	1.0					
X_2	0.2	1.0				
X_3	0.1	0.2	1.0			
X_1X_2	-0.4	-0.9	-0.2	1.0		
X_1X_3	-0.4	-0.2	-0.9	0.2	1.0	
X_2X_3	-0.2	-0.8	-0.8	0.7	0.6	1.0

Table 8: Correlation Matrix of Factors

	X_1	X_2	X_3	X_1X_2	X_1X_3	X_2X_3
X_1	1.0					
X_2	-0.6	1.0				
X_3	-0.6	-0.2	1.0			
X_1X_2	0.1	0.5	-0.6	1.0		
X_1X_3	0.1	-0.6	0.5	-0.3	1.0	
X_2X_3	-0.8	0.5	0.5	-0.4	-0.4	1.0

Table 9 shows the degrees of freedom statistics for the design model. The lack of fit for the degree of freedom and pure error were both 5. It is generally recommended that the degrees of freedom for lack of fit and pure error should be a minimum of 3 and 4, respectively (Box et al., 2022). The value of 5 obtained for both shows that the design is very adequate.

Table 9: Degrees of Freedom Statistics

Term	Value
Model	5
Residuals	10
Lack of fit	5
Pure error	5
Cor. total	15

Selection of Suitable Statistical Models
Tables 10 to 14 show the assessment of various statistical models for predicting six responses - hardness, tensile strength, melting temperature, density, creep rate, and wear index - based on input factors (aluminum, coconut shell charcoal, and cow bone ash) has yielded significant findings.

Table 10: Summary of Model Fit Results for Hardness

Source	Standard deviation	R^2	Adjusted R^2	Predicted R^2	PRESS	Remark
Linear	5.10	0.5930	0.5304	0.3713	523.18	
Quadratic	1.17	0.9837	0.9755	0.9622	31.43	
Special cubic	0.19	0.9996	0.9993	0.9988	0.97	Suggested
Cubic	0.22	0.9996	0.9991	0.9984	1.32	

Table 11: Lack of Fit Test Results for Hardness

Source	Sum of squares	Degree of freedom	Mean square	F-value	p value	Remark
Linear	338.41	8	42.30	714.06	< 0.0001	
Quadratic	13.29	5	2.66	44.88	0.0004	
Special cubic	0.040	4	9.96E-03	0.17	0.9456	Suggested
Cubic	7.86E-04	1	7.86E-04	0.013	0.9128	
Pure Error	0.30	5	0.059			

Table 12: Summary of Model Fit Results for Tensile Strength

Source	Standard deviation	R^2	Adjusted R^2	Predicted R^2	PRESS	Remark
Linear	13.96	0.1302	-0.0036	-0.2557	3656.73	
Quadratic	1.17	0.9953	0.9930	0.9879	35.16	
Special cubic	1.20	0.9956	0.9926	0.9870	37.98	
Cubic	0.80	0.9987	0.9967	0.9946	15.81	Suggested

Table 13: Lack of Fit Test Results for Tensile Strength

Source	Sum Of Squares	Degree Of Freedom	Mean Square	f-Value	p Value	Remark
Linear	2528.99	8	316.12	413.19	< 0.0001	
Quadratic	9.81	5	1.96	2.57	0.1622	
Special cubic	9.12	4	2.28	2.98	0.1310	
Cubic	2.97E-03	1	2.97E-03	3.88E-03	0.9527	Suggested
Pure Error	3.83	5	0.77			

Table 14: Summary of Model Fit Results for Melting Temperature

Source	Standard deviation	R^2	Adjusted R^2	Predicted R^2	PRESS	Remark
Linear	10.62	0.0356	-0.1128	-0.4054	2137.66	
Quadratic	10.24	0.3104	-0.0343	-0.5502	2358.03	
Special cubic	10.76	0.3152	-0.1413	-1.1198	3224.37	
Cubic	0.49	0.9990	0.9976	0.9956	6.65	Suggested

Table 15: Lack of Fit Test Results for Melting Temperature

Source	Sum of squares	Degree of freedom	Mean square	F-value	p value	Remark
Linear	1465.53	8	183.19	634.15	< 0.0001	
Quadratic	1047.43	5	209.49	725.17	< 0.0001	
Special cubic	1040.16	4	260.04	900.17	< 0.0001	
Cubic	4.89E-03	1	4.89E-003	0.017	0.9016	Suggested
Pure Error	1.44	5	0.29			

Table 16: Summary of Model Fit Results for Density

Source	Standard deviation	R^2	Adjusted R^2	Predicted R^2	PRESS	Remark
Linear	65.86	0.5927	0.5300	0.4160	80838.63	
Quadratic	4.03	0.9988	0.9982	0.9969	428.44	Suggested
Special cubic	4.18	0.9989	0.9981	0.9968	449.79	
Cubic	4.38	0.9992	0.9979	0.9967	461.40	

Table 17: Lack of Fit Test Results for Density

Source	Sum of squares	Degree of freedom	Mean square	F-value	p value	Remark
Linear	56268.31	8	7033.54	305.01	< 0.0001	
Quadratic	47.32	5	9.46	0.41	0.8246	Suggested
Special cubic	41.62	4	10.41	0.45	0.7696	
Cubic	1.81E-03	1	1.81E-03	7.84E-05	0.9933	
Pure Error	115.30	5	23.06			

Table 18: Summary of Model Fit Results for Creep Rate

Source	Standard deviation	R²	Adjusted R²	Predicted R²	PRESS	Remark
Linear	0.29	0.3260	0.2223	-0.0357	1.71	
Quadratic	0.22	0.7032	0.5548	0.2491	1.24	
Special cubic	0.23	0.7054	0.5090	0.1302	1.44	
Cubic	0.022	0.9982	0.9955	0.9910	0.015	Suggested

Table 19: Lack of Fit Test Results for Creep Rate

Source	Sum of squares	Degree of freedom	Mean square	F-value	p value	Remark
Linear	1.11	8	0.14	232.79	< 0.0001	
Quadratic	0.49	5	0.098	163.46	< 0.0001	
Special cubic	0.48	4	0.12	202.79	< 0.0001	
Cubic	1.63E-05	1	1.63E-05	0.027	0.8753	Suggested
Pure Error	2.99E-03	5	5.97E-04			

Table 20: Summary of Model Fit Results for Wear Index

Source	Standard deviation	R²	Adjusted R²	Predicted R²	PRESS	Remark
Linear	0.087	0.1793	0.0530	-0.2230	0.15	
Quadratic	0.071	0.5760	0.3641	-0.0514	0.13	
Special cubic	0.075	0.5772	0.2953	-0.3644	0.16	
Cubic	0.015	0.9889	0.9722	0.9423	6.93E-03	Suggested

Table 21: Lack of Fit Test Results for Wear Index

Source	Sum of squares	Degree of freedom	Mean square	F-value	p value	Remark
Linear	0.097	8	0.012	45.89	0.0003	
Quadratic	0.050	5	9.91E-03	37.45	0.0006	
Special cubic	0.049	4	0.012	46.68	0.0004	
Cubic	9.17E-06	1	9.17E-06	0.035	0.8596	Suggested
Pure Error	1.32E-03	5	2.65E-04			

Analysis of Variance of the Statistical Models

The D-Optimal mixture design was used to develop the models for all the responses in terms of aluminum (X_1), coconut charcoal (X_2) and cow bone ash (X_3) as shown in Equations 1 to 6 respectively. These equations were then used to predict their corresponding responses and the results are shown in Table 22 to 28. The results predicted by the models were very similar to those of the actual experiments. This is an indication of the accuracy of the model predictions.

$$Hardness = 0.95X_1 - 1.31X_2 + 5.32X_3 + 0.032X_1X_2 - 0.060X_1X_3 + 0.39X_2X_3 - 0.0052X_1X_2X_3 \quad (1)$$

$$Tensile\ strength = 1.44X_1 - 12.62X_2 - 22.10X_3 + 0.18X_1X_2 + 0.29X_1X_3 + 0.54X_2X_3 - 0.0012X_1X_2X_3 \quad (2)$$

$$Melting\ temperature = 6.09X_1 + 387.01X_2 - 455.15X_3 - 6.34X_1X_2 + 7.70X_1X_3 + 0.53X_2X_3 +$$

$$0.0011X_1X_2X_3 + 0.026X_1X_2(X_1 - X_2) - 0.033X_1X_3(X_1 - X_3) - 0.066X_2X_3(X_2 - X_3) \quad (3)$$

$$Density = 25.53X_1 + 112.66X_2 + 142.02X_3 - 1.058X_1X_2 - 1.40X_1X_3 - 1.76X_2X_3 \quad (4)$$

$$Creep\ rate = 0.023X_1 - 0.75X_2 - 5.41X_3 + 0.015X_1X_2 + 0.091X_1X_3 + 0.084X_2X_3 - 0.00061X_1X_2X_3 - 0.000080X_1X_2(X_1 - X_2) - 0.00039X_1X_3(X_1 - X_3) - 0.00068X_2X_3(X_2 - X_3) \quad (5)$$

$$Wear\ index = 0.0059X_1 - 1.91X_2 - 0.28X_3 + 0.033X_1X_2 + 0.0047X_1X_3 + 0.035X_2X_3 - 0.00029X_1X_2X_3 - 0.00014X_1X_2(X_1 - X_2) - 0.000022X_1X_3(X_1 - X_3) + 0.000015X_2X_3(X_2 - X_3) \quad (6)$$

Table 22: Experimental and RSM Predicted Results for Tensile Strength and Hardness

Run	Actual values of factors			Hardness (HV)		Tensile strength (MPa)	
	Aluminum (%)	Coconut shell charcoal (%)	Cow bone ash (%)	Actual Experiment	RSM Prediction	Actual Experiment	RSM Prediction
1	69	11	20	106.3	106.2	163.8	163.0
2	79	1	20	87.3	87.3	143.1	145.2
3	79	20	1	99.3	99.7	157.8	156.4
4	90	1	9	85.5	85.6	175.7	174.9
5	79	1	20	87.5	87.3	145.6	145.2
6	69	11	20	106.0	106.2	164.0	163.0
7	85	1	14	84.2	84.2	170.1	169.3
8	69	19	12	104.5	104.3	176.8	177.6
9	98	1	1	94.0	94.0	153.1	153.1
10	84	14	1	99.6	99.5	169.7	170.4
11	69	19	12	104.3	104.3	176.7	177.6
12	76	14	10	95.2	95.1	188.9	189.0
13	79	20	1	99.9	99.7	156.6	156.4
14	98	1	1	94.0	94.0	153.1	153.1
15	90	9	1	98.7	98.7	168.5	169.5
16	83	9	8	90.3	90.3	190.2	190.0

Table 23: Experimental and Rsm Predicted Results for Melting Temperature and Density

Run	Actual values of factors			Melting temperature (ºC)		Density (kg/m³)	
	Aluminum (%)	Coconut shell charcoal (%)	Cow bone ash (%)	Actual Experiment	RSM Prediction	Actual Experiment	RSM Prediction
1	69	11	20	605.4	605.4	2700	2706
2	79	1	20	583.8	584.4	2642	2633
3	79	20	1	600.8	601.4	2592	2593
4	90	1	9	577.8	577.8	2438	2440
5	79	1	20	585.0	584.4	2629	2633
6	69	11	20	605.4	605.4	2708	2706
7	85	1	14	602.5	602.5	2488	2491
8	69	19	12	595.0	594.9	2660	2658
9	98	1	1	602.4	602.4	2514	2514
10	84	14	1	579.7	579.7	2484	2484
11	69	19	12	594.9	594.9	2659	2658
12	76	14	10	582.7	582.6	2500	2499
13	79	20	1	602.0	601.4	2592	2593
14	98	1	1	602.4	602.4	2514	2514
15	90	9	1	607.0	607.0	2456	2454
16	83	9	8	597.7	597.8	2421	2421

Table 24: Experimental and RSM Predicted Results for Creep Rate and Wear Index

Run	Actual values of factors			Creep rate (%/hr)		Wear index (mg/rpm) ×10⁻⁴	
	Aluminum (%)	Coconut shell charcoal (%)	Cow bone ash (%)	Actual Experiment	RSM Prediction	Actual Experiment	RSM Prediction
1	69	11	20	1.94	1.97	0.48	0.48
2	79	1	20	1.32	1.32	0.32	0.32
3	79	20	1	2.26	2.26	0.55	0.53
4	90	1	9	1.37	1.37	0.36	0.36
5	79	1	20	1.32	1.32	0.32	0.32
6	69	11	20	2.00	1.97	0.48	0.48
7	85	1	14	1.55	1.55	0.34	0.34
8	69	19	12	1.35	1.37	0.31	0.31
9	98	1	1	1.97	1.97	0.48	0.48
10	84	14	1	1.90	1.89	0.48	0.48
11	69	19	12	1.40	1.37	0.31	0.31
12	76	14	10	1.58	1.58	0.38	0.38
13	79	20	1	2.26	2.26	0.50	0.53
14	98	1	1	1.97	1.97	0.48	0.48
15	90	9	1	1.76	1.77	0.33	0.34
16	83	9	8	1.48	1.48	0.27	0.27

Analysis of Variance of Models

The results of the analysis of variance (ANOVA) for the models assessing various properties such as hardness, tensile strength, melting temperature, density, creep rate, and wear index are summarized in Tables 25 to 30. Table 25 indicates that the hardness model was statistically significant with a p value of less than 0.0001, confirming all model terms as significant. The lack of fit was not significant either, as shown by a p value of 0.9456. Table 26 presents results for tensile strength, where all model terms, except for the cubic term, were significant, demonstrating that the components— aluminum, coconut charcoal, and cow bone ash— substantially affect hardness. This model also exhibited a low p value ($p < 0.0001$) and did not show a lack of fit. Similarly, Table 27 reveals the melting temperature model's results, confirming all terms significant apart from the cubic term, with a low p value and no lack of fit, indicating strong model performance in fitting the experimental data. The ANOVA results presented in Table 28 indicate that both the quadratic model for density and the linear mixture are significant ($p < 0.0001$), confirming that variations in the values of aluminum (X_1), coconut charcoal (X_2), and cow bone ash (X_3) significantly impact the density of the composite material. The model p value further corroborates its effectiveness in predicting tensile strength (Qi et al., 2009), while the lack of fit p value (p=0.8246) suggests no significant lack of fit, which is favorable (Montgomery, 2017). Table 29 illustrates that the cubic model for creep rate is also significant ($p < 0.0001$), with all single effect terms demonstrating significance, except for the interaction between aluminum and coconut charcoal (X_1X_2). The lack of fit in this model is not significant (p=0.8753), as noted for the cubic model assessing wear index in Table 30, which remains significant ($p < 0.0001$) without significant lack of fit ($p > 0.05$).

Table 25: ANOVA Results for Model Representing Hardness

Source	Sum of Squares	Degree of freedom	Mean square	F value	p value
Model	831.83	6	138.64	3713.10	< 0.0001
Linear mixture	493.46	2	246.73	6608.13	< 0.0001
X_1X_2	8.63	1	8.63	231.04	< 0.0001
X_1X_3	53.29	1	53.29	1427.13	< 0.0001
X_2X_3	6.33	1	6.33	169.58	< 0.0001
$X_1X_2X_3$	13.25	1	13.25	354.96	< 0.0001
Residual	0.34	9	0.037		
Lack of fit	0.040	4	9.96E-03	0.17	0.9456
Pure error	0.30	5	0.059		
Cor. Total	832.16	15			

Table 26: ANOVA Results for the Model Representing Tensile Strength

Source	Sum of Squares	Degree of freedom	Mean square	F value	p value
Model	2899.04	6	483.17	335.79	< 0.0001
Linear mixture	379.17	2	189.59	131.76	< 0.0001
X_1X_2	375.63	1	375.63	261.05	< 0.0001
X_1X_3	1058.76	1	1058.76	735.80	< 0.0001
X_2X_3	1217.73	1	1217.73	846.28	< 0.0001
$X_1X_2X_3$	0.69	1	0.69	0.48	0.5062
Residual	12.95	9	1.44		
Lack of fit	9.12	4	2.28	2.98	0.1310
Pure error	3.83	5	0.77		
Cor. Total	2911.99	15			

Table 27: ANOVA Results for the Model Representing Melting Temperature

	Sum of Squares	Degree of freedom	Mean square	F value	p value
Model	1519.62	9	168.85	699.03	< 0.0001
Linear mixture	54.10	2	27.05	111.99	< 0.0001
X_1X_2	528.21	1	528.21	2186.79	< 0.0001
X_1X_3	393.71	1	393.71	1629.96	< 0.0001
X_2X_3	8.76	1	8.76	36.27	0.0009
$X_1X_2X_3$	0.087	1	0.087	0.36	0.5702
$X_1X_2(X_1-X_2)$	501.40	1	501.40	2075.82	< 0.0001
$X_1X_3(X_1-X_3)$	452.28	1	452.28	1872.45	< 0.0001
$X_2X_3(X_2-X_3)$	1002.34	1	1002.34	4149.71	< 0.0001
Residual	1.45	6	0.24		
Lack of fit	4.889E-003	1	4.889E-003	0.017	0.9016
Pure error	1.44	5	0.29		
Cor. Total	1521.07	15			

Table 28: ANOVA Results for the Model Representing Density

Source	Sum of Squares	Degree of freedom	Mean square	F value	p value
Model	1.38E+05	5	27652.33	1700.38	< 0.0001
Linear mixture	82040.66	2	41020.33	2522.40	< 0.0001
X_1X_2	22346.95	1	22346.95	1374.15	< 0.0001
X_1X_3	42587.92	1	42587.92	2618.79	< 0.0001
X_2X_3	34165.79	1	34165.79	2100.90	< 0.0001
Residual	162.62	10	16.26		
Lack of fit	47.32	5	9.46	0.41	0.8246
Pure error	115.30	5	23.06		
Cor. Total	1.38E+05	15			

Table 29: ANOVA Results for the Model Representing Creep Rate

Source	Sum of Squares	Degree of freedom	Mean square	F – value	p value
Model	1.65	9	0.18	366.72	< 0.0001
Linear mixture	0.54	2	0.27	538.93	< 0.0001
X_1X_2	9.88E-04	1	9.88E-04	1.97	0.2096
X_1X_3	0.041	1	0.041	82.84	< 0.0001
X_2X_3	0.026	1	0.026	51.31	0.0004
$X_1X_2X_3$	0.028	1	0.028	56.26	0.0003
$X_1X_2(X_1-X_2)$	4.60E-03	1	4.60E-03	9.19	0.0231
$X_1X_3(X_1-X_3)$	0.065	1	0.065	129.06	< 0.0001
$X_2X_3(X_2-X_3)$	0.11	1	0.11	216.56	< 0.0001
Residual	3.00E-03	6	5.00E-04		
Lack of fit	1.63E-05	1	1.63E-05	0.027	0.8753
Pure error	2.99E-03	5	5.97E-04		
Cor. Total	1.65	15			

Table 30: ANOVA Results for the Model Representing Wear Index

Source	Sum of Squares	Degree of freedom	Mean square	F value	p value
Model	0.12	9	0.013	59.38	< 0.0001
Linear mixture	0.022	2	0.011	48.44	0.0002
X_1X_2	6.54E-03	1	6.54E-03	29.46	0.0016
X_1X_3	7.61E-06	1	7.61E-06	0.034	0.8593
X_2X_3	3.51E-03	1	3.51E-03	15.80	0.0073
$X_1X_2X_3$	6.49E-03	1	6.49E-03	29.22	0.0017
$X_1X_2(X_1-X_2)$	0.015	1	0.015	67.28	0.0002
$X_1X_3(X_1-X_3)$	2.03E-04	1	2.03E-04	0.91	0.3760
$X_2X_3(X_2-X_3)$	5.45E-05	1	5.45E-05	0.25	0.6380
Residual	1.33E-03	6	2.22E-04		
Lack of fit	9.17E-06	1	9.17E-06	0.035	0.8596
Pure error	1.32E-03	5	2.65E-04		
Cor. total	0.12	15			

Tables 31 shows the goodness of fit statistics for the models representing hardness, tensile strength, melting temperature, density, creep rate and wear index, respectively. The models demonstrate exceptional fits with R^2 values exceeding 0.99, indicating strong alignment with experimental data. The hardness model achieved an R^2 of 0.9996, with high reliability reflected in a low coefficient of variation (CV) of 0.2 and an adequate precision value of 172.0180. Similarly, the tensile strength model showed R^2 values around 0.995, with a CV of 0.72 and an adequate precision of 56.4790. The melting temperature model also performed well, with an R^2 of 0.9990 and a CV of 0.0830, supported by an adequate precision value of 75.037. The density model further confirmed its robustness with an R^2 of 0.9988, a low CV of 0.1600, and an adequate precision of 115.4910, underlining the high reliability and accuracy of all models in design exploration (Khuri and Mukhopadhyay, 2010).

Table 31: Goodness of fit Statistics for all Six Responses

Parameter	Value for Each Response					
	Hardness	Tensile Strength	Melting Temperature	Density	Creep Rate	Wear Index
R^2	0.9996	0.9956	0.9990	0.9988	0.9982	0.9889
Adjusted R^2	0.9993	0.9926	0.9976	0.9982	0.9955	0.9722
Predicted R^2	0.9988	0.9870	0.9956	0.9969	0.9910	0.9423
Mean	96.0400	165.8500	595.28	2562.29	1.7100	0.4000
Standard deviation	0.1900	1.2000	0.4900	4.0300	0.0220	0.0150
C.V %	0.2000	0.7200	0.0830	0.1600	0.0150	3.7200
Adequate precision	172.0180	56.4790	75.0370	115.4910	53.0380	21.7620

Model Diagnostics

Model diagnostics were performed to evaluate the developed models. Figures 3 to 8 show normal probability plots for all responses. The data points clustered closely around a straight line, indicating that the residuals have a normal distribution, which is favorable. (Telford, 2007).
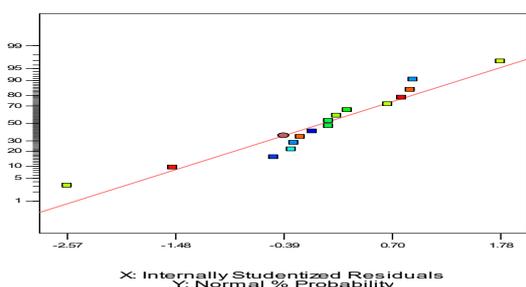
Figure 3: Normal probability plot for model representing tensile strength
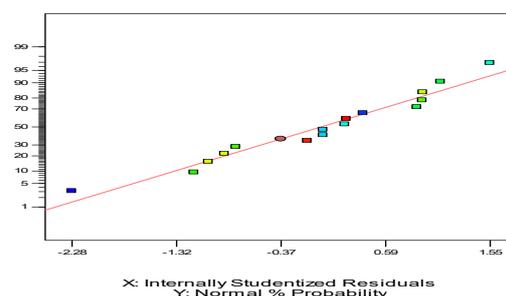
Figure 4: Normal probability for model representing hardness
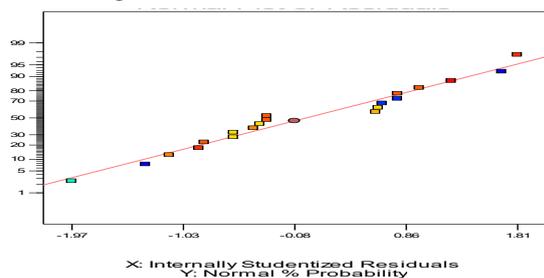
Figure 5: Normal probability plot for model representing density
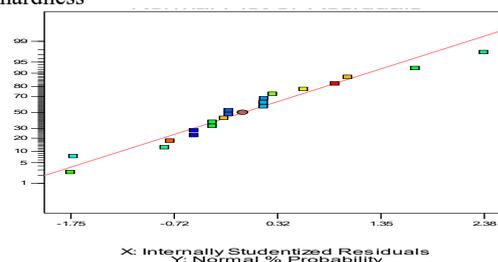
Figure 6: Normal probability plot for model representing melting temperature
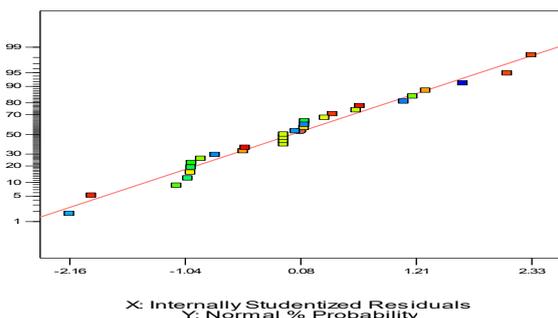
Figure 7: Normal probability plot for model representing wear index
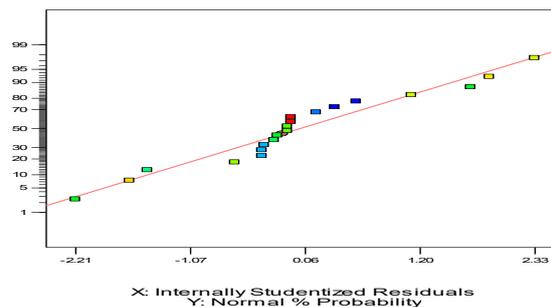
Figure 8: Normal probability plot for model representing creep rate

Response Surface Plots

Figure 9 shows the 3D response surface displaying the effect of the amount of aluminum (A), coconut shell charcoal (B), and cow bone ash (C) on the hardness of the composite material. This is seen in the fact that as the composition of coconut charcoal and cow bone ash were increased, the hardness of the material also increased, suggesting a protagonist effect as noted by Ademoh and Adeyemi (2015). However, the composition of aluminum had an antagonistic effect on the hardness of the material as seen in the fact that as the level of aluminum was increased, the hardness of the material was observed to decrease. This could be as a result of the poor mechanical strength of the aluminum which necessitated the incorporation of the coconut shell charcoal and cow bone ash. The red regions of the contour plot indicate regions of high values of hardness while the blue regions indicate regions of low values of hardness. The green region shows intermediate values of hardness. Figure 10 shows the 3D response plot displaying the effect of the amount of aluminum, coconut charcoal and cow bone ash on tensile

strength of the composite material. The trend observed in indicates that intermediate levels of all the components were needed to maximize the tensile strength of the composite material. This is because as the composition of the components was increased, the tensile strength was observed

to increase until it reached a maximum value of 190 MPa which then began to reduce as the amount of the components were further increased. Similar trends have been reported by other researchers (Adeyemi et al., 2015).
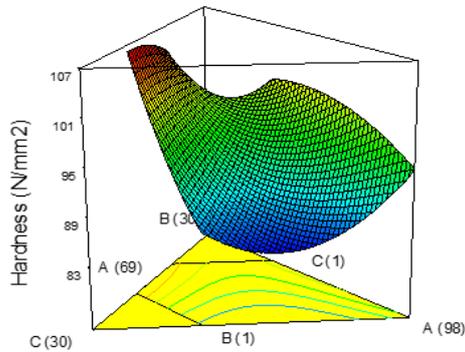

Figure 9: Response surface plot showing aluminium, coconut shell, charcoal and cow bone ash composition on hardness
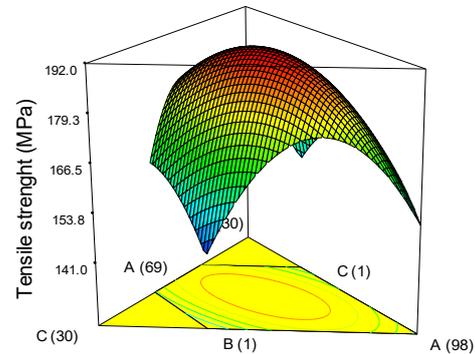
Figure 10: Response surface plot showing the effect of the effect of aluminium, coconut shell charcoal and cow bone ash composition tensile strength

Figure 11 shows the 3D response surface plot displaying the effect of the amount of aluminum, coconut charcoal and cow bone ash on melting temperature of the composite material. The trend observed shows that the introduction of coconut charcoal and cow bone ash increased the melting point of the composite material. This is probably attributable to the fact that these materials are made of carbon which impacts the higher melting point. Conversely, the lower melting point of aluminum compared to the coconut charcoal could explain the inverse relationship observed between the amount of aluminum and the melting temperature of the composite material. Figure 12 shows the 3D response surface plot displaying the effect of the amount of aluminum, coconut charcoal and cow bone ash on the density of the composite material. The trend observed shows that the density of the composite material decreased as the amount of aluminum was increased. This is possibly because aluminum is a light metal indicating that it will have a low density. On the other hand, increasing the level of coconut charcoal and cow bone ash had

the opposite effect as the density of the composite material was found to increase. Figure 13 shows the 3D response surface plot displaying the effect of the amount of aluminum, coconut charcoal and cow bone ash on creep rate of the composite material. It was desired to minimize the creep rate of the material and thus, the response surface shows that the minimum creep rate was obtained in the blue regions which is characterized by a low value of coconut charcoal and high value of cow bone ash as well as relatively high levels of aluminum (around 80%). Figures 14 shows the 3D response surface plot displaying the effect of the amount of aluminum, coconut charcoal and cow bone ash on wear index of the composite material. Just like the case of creep rate, it was also desired to have a wear index that is as small as possible; thus, the response was minimized. It can be seen from the figures that minimum values of wear index were obtained at low values of coconut charcoal and cow bone ash as well as high values of aluminum.
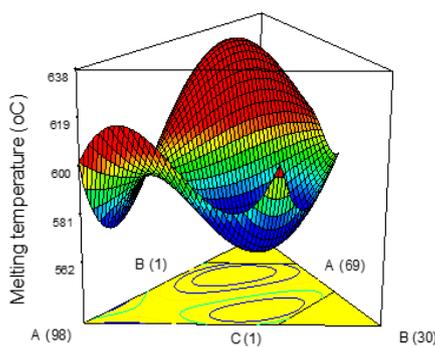

Figure 11: Response surface plot showing the effect of aluminum, coconut shell charcoal and cow bone ash composition on melting temperature
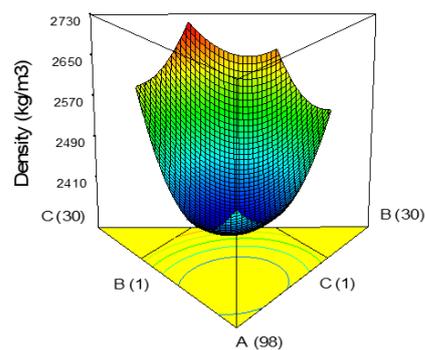

Figure 12: Response surface plot showing the effect of aluminium, coconut shell charcoal, and cow bone ash composition on density
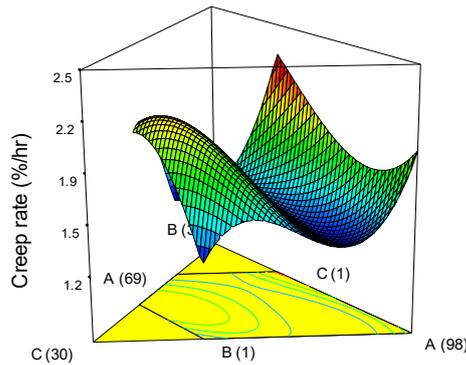
Figure 13: Response surface plot showing the effect of aluminum, coconut shell charcoal and cow bone ash composition on creep rate
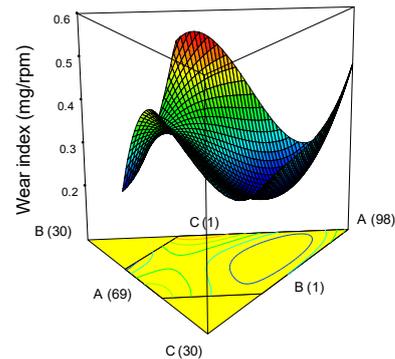

Figure 14: Response surface plot showing the effect of aluminium, coconut shell charcoal, and cow bone ash composition charcoal on wear index

Numerical Optimization of Input Factors and Responses

Numerical optimization was carried out using the built-in optimization algorithm of the Design Expert software, as shown in Table 32 for all the responses.

Table 32: Table of Constraints for Numerical Optimization

Variables	Symbols	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
Input variables/factors							
Aluminium (%)	X_1	is in range	69	98	1	1	3
Coconut Shell Charcoal (%)	X_2	is in range	1	20	1	1	3
Cow Bone Ash (%)	X_3	is in range	1	20	1	1	3
Output variables/responses							
Hardness (HV)	Y_1	maximize	84.22	106.34	1	1	3
Tensile strength (MPa)	Y_2	maximize	143.10	190.22	1	1	3
Melting temperature (°C)	Y_3	maximize	577.85	607.02	1	1	3
Density (kg/m³)	Y_4	minimize	2421.49	2707.82	1	1	3
Creep rate (%/hr)	Y_5	minimize	1.32	2.26	1	1	3
Wear index (mg/rpm)	Y_6	minimize	0.27×10^{-4}	0.55×10^{-4}	1	1	3

Scanning Electron Microscope (SEM) Analysis

Figures 15 and 16 show SEM Micrography the EDS of the produced sample. The result shows that the granules of the reinforcements are well embedded in the space between the aluminium matrix. Although a small porosity was seen which may also reduce the mechanical properties of the optimum composite material.
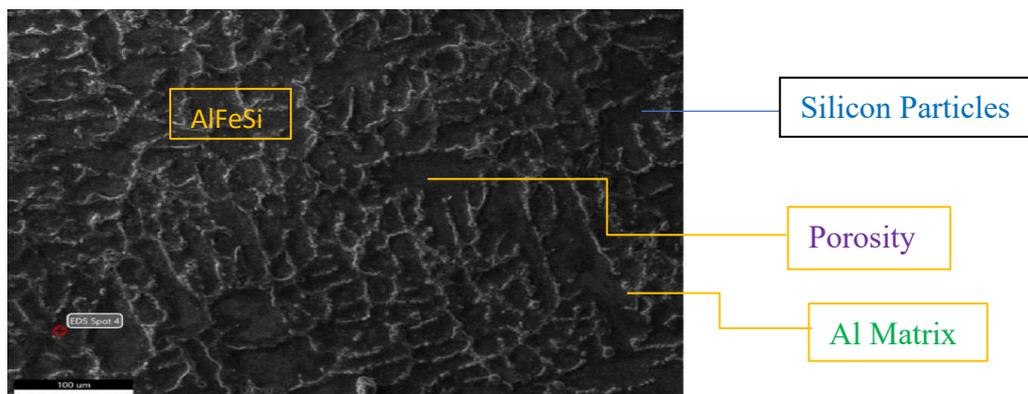

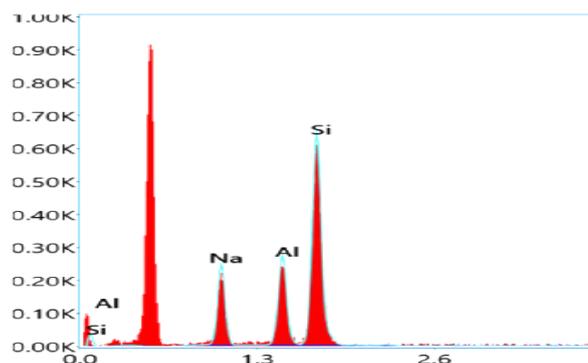Figure 15: SEM Micrography of the Produced Sample

Figure 16: EDS Micrography of the Produced Sample

CONCLUSION

With the help of the D-Optimal design expert software, an optimized blend of the matrix and the reinforcement particles was obtained as follows; 81.75% weight of aluminium, 8.36% weight of coconut shell charcoal and 9.90% weight cow bone ash with a desirability of 95.20%. The developed model in this study predicted the responses with their accuracy for hardness, tensile strength, melting temperature, density, creep rate and wear index as 99.88%, 98.70%, 99.56%, 99.69%, 99.10% and 94.23% respectively. It has also shown that the agro – waste materials have improved the mechanical properties of the aluminium as stated by Oghoghorie and Eboigbe, 2025.

REFERENCES

Ademoh, A.N. and Adeyemi, I.O. (2015). Development and Evaluation of Maize Husks (Asbestos Free) Based Brake Pad. *International Institute for Science, Technology and Education (IISTE): Industrial Engineering Letters –IEL*. 5(2), pp. 67 – 80.

Amenaghawon, N.A. and Amagbewan, E. (2017). Evaluating the effect of acid mixtures and solids loading on furfural production from sugarcane bagasse: optimization using response surface methodology and artificial neural network. *Nigerian Research Journal of Engineering and Environmental Sciences*, 2(2), pp. 578-587

Arulprasanna, A. and Omkumar, M (2023) "A review on composites: Selection and its applications" *Elsevier*, https://doi.org/10.1016/j.matpr.2024.06.014

Bezerra, M.A, Santelli, R.E, Oliveira, E.P, Villar, L.S and Escaleira, L.A. (2008) "Response surface methodology (RSM) as a tool for optimization in analytical chemistry". *Talanta*, 76(5), 965- 977

Box, G. E., Hunter, J. S., and Hunter, W. G. (2022). Statistics for Experimenters. In Wiley series in probability and statistics. Hoboken, NJ: Wiley.

Cafaggi, S., Leardi, R., Parodi, B., Caviglioli, G. and Bignardi, G. (2003) "An example of application of a mixture design with constraints to a pharmaceutical formulation" *Chemometrics and Intelligent Laboratory Systems* 65(1):139-147, http://dx.doi.org/10.1016/S0169-7439(02)00045-X

Fardin Khan, Nayem Hossain, Juhi Jannat Mim, SM Maksudur Rahman, Md. Jayed Iqbal, Mostakim Billah and Mohammad Asaduzzaman Chowdhury (2025), Advances of composite materials in automobile applications – A review. Journal of Engineering Research, ELSEVIER, 13(1), Pp. 1001–1023

Leardi, R. (2009) "Experimental design in chemistry: A tutorial". *Analytica Chimica Acta,* 652(1-2), 161-172

MacKinnon, J. G., Nielsen, M. Ø. and Webb, M. D. (2023) "Leverage, Influence and the Jackknife in Clustered Regression Models: Reliable Inference Using Summclust". *The Stata Journal*, 23(4), 942-982

Montgomery, D. C. (2017) "Design and analysis of experiments". *John wiley & sons.*

Oghoghorie, O. and Eboigbe, C. I. (2025) "Optimization of Agro-Waste Reinforced Aluminium - Based Composite Using D-Optimal Mixture Design", *Nigerian Journal of Technology*, 44(4), pp. 1 – 17. https://doi.org/10.4314/njt.2025.5184

Oghoghorie O., Ebhojiaye R. S. and Amiolemhen P. E. (2022) "Assessment of The Wear Resistance and the Density Reduction of Aluminium When Reinforced with Coconut Shell Charcoal and Cow Bone Ash" *World Journal of Engineering Research and Technology (WJERT)*. Vol. 8, Issue 4, 27- 40.

Oghoghorie, O. and R.S. Ebhojiaye (2024). "Effect of Coconut Shell Charcoal and Cow Bone Ash on the Tensile Strength and Melting Temperature of Aluminum Based Composite". *Pacific Journal of Science and Technology*. 25(1): 5-9

Sahin, B.Y., Demirtas, E.A. and Burnak, N. (2016) "Mixture design: A review of recent applications in the food industry" *Pamukkale University Journal of Engineering Sciences,* https://doi.org/10.5505/pajes.2015.98598, 22(4), 297-304

Telford J.K. (2007). A brief Introduction to Design of Experiments. *John Holpkins APC Technical Digest,* 27(3), pp. 224-232.

Qi, B., Chen, X., Shen, F. and Wan, Y. (2009). "Optimization of Enzymatic Hydrolysis of Wheat Straw Pre-treated by Alkaline Peroxide Using Response Surface Methodology". *Industrial and Engineering Chemistry Research*, 48, pp. 7346-7353.