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#### **Research Paper**

### Experimental analysis, predictive modelling and optimization of some physical and mechanical properties of aluminium 6063 alloy based composites reinforced with corn cob ash.

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#### ABSTRACT

In the present work, aluminium alloy 6063 reinforced with various weight percentages of corncob ash (CCA) particles (2.5%, 5%, 7.5%, 10%, 12.5% and 15%) were prepared using stir casting process. Some physical and mechanical properties (density, % porosity, hardness, wear index, tensile strength and impact strength) of the produced composite were characterized and compared with that of the matrix alloy. Corncob ash reinforcements were observed to have been distributed homogeneously in the Aluminium Matrix with pockets of reinforcement particles agglomeration. The reinforcement of CCA particles improved the hardness, wear index and the density of the produced composite over that of the base alloy. There was a reduction in the tensile and impact strengths of the composites compared to the unreinforced matrix alloy. Statistical evaluation and optimization using mixture design of the Design-Expert software package (Stat-Ease) revealed the optimal concentrations of the matrix and the reinforcement mixture and their effects on the studied properties of the developed composites. Regression models were developed for predicting and optimizing the various physical and mechanical properties of the composites. Optimization solution indicates that optimal mixture components of 92.081 and 7.919% (matrix and reinforcement wt%) will yield optimal composite properties' responses. Based on the improved hardness, wear-resistance and low density of the CCA reinforced AA6063 base composites produced could be employed in the production of automotive components requiring lightweight, load-bearing and wearresistant composites.

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RESEARCH REVIEW of Sciences and Technologies

#### **1** Introduction

In the recent time, there have been increasing interests in researches on the use of naturally sourced and agro-waste based reinforcements in composite materials due to their ease of fabrication, availability, renewable, cost reduction, as well as the increasing global campaign on environmental protection and stiffer international sanctions for non-compliance [1]. Natural fibre and agro-wastes reinforced composites are environmentally benign materials with promising applications in various fields, especially where environmental and energy conservation are of great concern. They are considered as suitable materials to replace the expensive and highly dense synthetic materials, its allied products and other industrial wastes for lightweight, high strength and energy conservation applications. The real advantages of natural fibre and agro-wastes reinforcements in terms of flexibility and expanding the potential for the composite materials are the range of fibre reinforcing materials available. Naturally occurring fibres and agricultural wastes are promising options for the development of composite materials. Their environmental advantages such as, biodegradable, renewable, recyclable and the large number of them that are available as native, cultivated, or industrial residues stand over synthetic fibres [1-3]. It is worthy to note that, unlike synthetic fibres (such as glass, silicon carbide and carbon) extensively produced in highly industrialized countries; naturally occurring fibres of plants and animal origin are cultivated in abundance mainly in developing countries as agricultural by-products or agro-wastes. This is of socio-economic benefits and will represent a major source of income to sustain the economy of local populations in these developing nations. Nigeria is endowed with many mineral and agro-based resources whose exploitation leaves huge solid wastes such as palm kernel shell, coconut shell, wood charcoal, corncobs, periwinkle shell, rice husk, groundnut shell, etc. These solid agricultural residues could be used singly or as a hybrid in the development of environmental-friendly composite materials for various applications, such as Eco-pad that is employed in modern braking systems for vehicles [4].

Researches to develop aluminium metal matrix composites (AMMCs) have been a burgeoning field because of their various applications in industries such as aerospace, automotive, defence, marine, thermal management, electrical and electronic as well as sports goods. Aluminium alloys such as 2000, 5000, 6000 and 7000 series have been most frequently employed as a matrix in AMMCs fabrication [5]. AMMCs exhibit high strength to weight ratio, improved thermal conductivity, abrasion/wear resistance, creep resistance, dimensional stability, improved high-temperature performance and the exceptionally good stiffness-to-weight ratio [6-10]. Aluminium alloy AA6063 with silicon and magnesium as the major alloying elements are majorly employed in the automobile and aeronautic industries. Its good properties such as lightweight, high ductility, electrical and thermal conductivities are combined with the properties of reinforcements such as low coefficient of thermal expansion, high stiffness, and strength with abrasion resistance to produce composite material with tailored physical and mechanical properties [11]. The physical state of reinforcements used in the fabrication of AMMCs could be in the form of continuous and discontinuous fibres, whiskers or particulate [10, 12-14]. Particle-reinforced AMMCs, which are less expensive compared to fibre reinforced AMMCs are most often fabricated by either the solid-state (powder metallurgy processing route) or liquid state (stir casting, infiltration and *in-situ*) processes [6-8, 12, 15, 16]. The overall cost of AA6063 and its end products can be reduced by blending the alloy matrix with low cost and renewable particulate material fillers like coconut shell, periwinkle shell, palm kernel, charcoal, and corn cob, etc. both in the processed and the unprocessed states. The resulting composites could be employed for lightweight, load-bearing, wear resistance automotive and aeronautic applications.

The design of experiment (DOE) program is used effectively to conduct minimum experiments by varying input factors between their respective levels, analysing the factors' significance quantitatively, deriving model equations, and validating & confirming the models' adequacy and suitability based on the collected input-output data [17]. Optimization and predictive modelling of composite materials development process variables and properties have been carried out in the past by various researchers using experimental design tools and methodologies such as response surface design (central composite design (CCD), box Behnken design (BBD)), mixture design, factorial design, Taguchi method, etc. [18-24]. Besides, the results indicated that it is a great tool for composite materials design, modelling and optimization. The interaction of MMCs fabrication process factors and their effects on the physical and mechanical properties or the composites quality/properties (response variables) can be determined by studying input parameters that are varied simultaneously under the condition of experimentation and predictive modelling, using the factorial, response surface and mixture designs of the DOE. The factorial design method is used primarily for understanding if selected factors are important to the process. This can take the form of screening for a few important factors out of many possibilities or characterizing how known factors interact and individually affect the process. Mixture designs are useful when the response changes as a function of the relative proportions

of the components in a mixture such as composite materials. All mixture components are inputted in the same units of measure and each run summed up to the same total. The response surface method is a statistical and experimental design tool in which selected dependent variables respond to variations in one or more independent or factor variables [17, 25, 26]. These design types are useful techniques for predictive modelling and optimization of experimental process factors/variables and response properties [17, 26]. The aim is to optimize the response of the dependent variable (output/properties) based on variation in the design settings for one or more independent/factor variables (inputs/process parameters) [17, 26].

Several research efforts have been made at developing aluminium alloy-based composites with natural and agro-waste reinforcements such as periwinkle shell [14, 27], rice husk [28], sugarcane bagasse [29, 30], palm kernel shell [31-33], coconut shell [34, 35], etc. A few of such researches have investigated the potentials of corncob ash as suitable hybrid reinforcement and reported promising results and properties' improvements [36-39]. This research is concerned with developing an aluminium 6063-matrix composite using corncob ash (CCA) particles as single reinforcement. The objective of this work is to investigate the potency of processed corncob particles as reinforcement for the aluminium 6063 alloy matrix. The use of corn cob ash in this research is as a result of the improved properties obtained in using CCA as a complementing reinforcement in hybrid aluminium based composites and its very low density (1.96g/cm<sup>3</sup>) [36] in comparison to synthetic fillers like SiC (3.18g/cm<sup>3</sup>) and Al<sub>2</sub>O<sub>3</sub> (3.9g/cm). Corncob is also available in large quantity and widely distributed in most parts of the developing countries like Nigeria. It also studied the effects of variation of particulate compositions (weight fraction) of the corncob ash on some physical and mechanical properties (density, apparent porosity, hardness, impact strength, tensile strength and wear index) of the particulate reinforced AA6063 composite. The research will also undertake the predictive modelling and optimization of the experimentally evaluated properties using the mixture design method of the design expert software version 11. The utilization of this agro-waste as reinforcement in MMC will ameliorate the environmental challenges posed by this solid agricultural residue.

#### 2 Experimental techniques

The following experimental techniques and procedures were adopted in carrying out this research:

#### 2.1 Materials Preparation

#### 2.1.1 Matrix material

Aluminium based alloy AA6063 with the elemental chemical composition specified in table 1, was obtained from Nigalex Company in Ikeja, Lagos State, Nigeria.

			-			-		
Element	Al	Si	Mg	Fe	Cu	Mn	Zn	Cr
% Comp	98.61	0.5287	0.4741	0.2214	0.0009	0.0168	<0.0087	< 0.0029
Element	Ni	Ti	Sn	Zr	V	Ca	Be	Pb
% Comp	< 0.0034	0.0152	< 0.0070	0.0028	< 0.0056	>0.000	< 0.0001	< 0.0000

 Table 1 - Chemical Composition of AA6063 Aluminium Alloy Matrix.

#### 2.1.2 Processing of Reinforcing Material (corn cob ash)

Dried corncobs used for this research were obtained from Ogwashi-Uku, Delta State, Nigeria. The corncobs were placed in a metallic drum and burnt in the open air for thorough combustion. The ash obtained was conditioned in a muffle furnace at a temperature of 650°C for four hours to reduce the volatile constituents of the ash. The chemical composition of the corncob ash as obtained using the XRF technique is presented in table 2, while Figure 1b displays the processed corncob ash particles.

Table 2 - Chemical composition of corncob ash [39].

Compound	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O
Wt. %	66.34	7.48	4.44	11.57	2.06	1.07	0.41	4.92

#### 2.1.3 Fabrication of Composite

The AA6063 matrix composites were produced following double stir casting technique described elsewhere [16]. The molten composites were poured in a prepared sand mould to produce cast aluminium based composites reinforced with corn cob ash. Some cast composite samples are shown in Figure 1a.



Figure 1 - (a) Cast composites samples during fettling and (b) Corn cob ash.

#### 2.2 Measurement of Density and Apparent Porosity

Both experimental and theoretical densities of the unreinforced alloy and the developed composites were determined using the Archimedean principle and pycnometer method respectively. The procedure is described in [32]. The experimental densities were compared with the theoretical densities for each of the composites produced and the unreinforced AA6063 matrix (Table 3) and this was used as a basis to evaluate the apparent porosity of the composites. The following relation (Equation 1) was used to compute the apparent porosity [16].

Apparent porosity = 
$$\frac{\rho^T - \rho^{EX}}{\rho^T} \times 100$$
 (1)

where  $\rho^{T}$ : Theoretical Density,  $\rho^{EX}$ : Experimental density.

#### 2.3 Brinell Hardness Test

To evaluate the hardness properties of the developed metal matrix composite, the Brinell hardness testing machine is used per ASTM E10 standard. Three hardness tests were carried out for each sample and the average value taken as a measure of the hardness of the specimen. The results of the Brinell hardness test was obtained for both the base AA6063 alloy and the composites.

#### 2.4 Charpy Impact Test

Through cutting and grinding of the samples from the material, a test piece of dimension  $55 \times 10 \times 10 \text{ mm}^3$  was produced. At half the length of the test piece, a 45-degree notch, 2 mm deep was produced. The specimen is supported as a beam on the pedestal impact testing machine base with the north centrally located and backing the hammer mass of the machine. The specification of the pendulum length of the machine is 0.7486 kg, hammer mass is 21.9 kg. The pendulum with the hammer lifted (swung up backwards) and locked, the nominal energy scale set to a maximum of 300 *joules* and the specimen north backing the hammer mass, the pendulum released at a velocity of 5.23 ms<sup>-1</sup> to load the specimen directly behind the notch. The impact energy of the specimen which reduced the swinging of the pendulum before the fracture is read off the energy scale deflected backwards in a clockwise movement. To compute the impact strength of the material the relation given in equation 2 is used.

impact strength = 
$$\frac{impact energy}{cross \sec tional area of the material at the notch}$$

(1)

#### 2.5 Tensile test

The tensile test was carried out to determine the ability of the developed composite material to withstand loads before elongation. The testing is conducted using digital JPL-100k tensile testing machine. The metal matrix composites were machined to the required dimension for the test. Ultimate tensile strength is calculated as it is the maximum stress that a material can withstand under tensile loading.

#### 2.6 Wear Behaviour

The wear behaviour of the composite was evaluated using a rotary platform abrasion tester commonly referred to as Taber Abrasion Machine. The composite samples produced were machined in accordance with the specification for wear and then placed on the turntable platform of the wear machine and gripped at a constant pressure by two abrasive wheels lowered on to the sample surface. The turntable platform rotates at a fixed speed of 1000 *rpm* for 20 *min*. The rubbing action between the surface and the abrasive wheel during the rotary motion of the machine results in the generation of abraded loose composite debris from the surface of the sample. The initial weight and final weight of the sample were measured using a digital weighing balance, and the wear index is calculated using equation 3 [39].

$$WearIndex(WI) = \frac{(initial weight - final weight)}{number of \ test \ cycle} \times 1000$$
(1)

where the initial and final weights are measured in grams, and the number of wear test cycle measured in rpm.

#### 2.7 Microstructural Examination

Preparation of samples for optical microscopic examination was done by sectioning the parent material in the length of 15 *mm*, using the series of grinding with various emery papers of different grades (220, 320, 400, 800, and 1200). The workpiece is placed on the emery paper and it is moved against the cutting lines until they are completely wiped out and the surface of the piece free from any scratch. It is then polished with gamma-alumina powder to attain mirror-like surface, it is then etched by immersing in 200 *ml* of Keller's reagent (190 *ml* of water, 2 *ml* of hydrofluoric acid, 5 *ml* of nitric acid, 3 *ml* of hydrochloric acid) for 20 *secs* and it is then rinsed in acetone and allowed to dry before it is observed under a metallurgical microscope.

#### 2.8 Predictive Modelling and Optimization of Composites Properties Using Design of Experiment

An attempt is here made by applying experimental design software packages and mixture design techniques in modelling and optimizing the composites fabrication process variables and predicting the responses of the properties of the composites. The studied physical and mechanical properties of the composites were optimized by mixture design tool of Design-Expert software version 11. Simplex lattice design type, which requires the difference between low and high levels to be the same for all the mixture components, and quadratic design model were applied during the analysis. Matrix and Reinforcement weight concentrations (%) were selected as the independent variables (components 1 & 2), while density, apparent porosity, hardness, tensile strength, impact strength and wear index were the dependent/response variables (Responses 1 to 6). Twelve runs of experiments were performed to obtain the effects of the factor on the properties studied as well as responses of the dependent variables/properties.

#### **3** Results and discussion

#### 3.1 Physical Properties

The experimental results for physical properties (density and porosity) are presented in Table 3. From the table, it could be observed that both the theoretical and the experimental densities decreased progressively as the reinforcement concentration is increased, while the matrix component is reduced. This reduction in density could be attributed to the influence of low density of the reinforcement particles. Reduction in the density of AMMCs reinforced with natural and agro-

based fillers have been reported by several investigations [14, 32, 40]. This is a desirable trend, as it motivates their employment in lightweight load-bearing applications.

The apparent porosity is used to estimate the percentage of voids in the fabricated composites. The porosity of the composite as shown in Table 3 increased from 1.717% at 2.5wt% CCA to 2.066% at 15wt% CCA. The maximum porosity of the lightest composite sample is 2.066%, corresponding to the lowest density of 2.4367g/cm<sup>3</sup>. Thus, the porosity level of the fabricated composite is below the maximum limit of 4% reported as tolerable for cast aluminium metal matrix composites [28, 32, 33, 36]. It can further be noted that the composite fabrication route (stir-casting) was necessary and efficient in minimizing to the barest minimum the porosity level of the developed composites.

Sample	Theoretical density $\left( \rho^T \right) \left[ g/cm^3 \right]$	Experimental density $\left( ho^{EX} ight)\left[g/cm^3 ight]$	Apparent Porosity (%)
Unreinforced Al	2.69	2.6605	1.097
Al - 2.5 wt% <sub>CCA</sub>	2. 6564	2. 6108	1.717
Al – 5wt% <sub>CCA</sub>	2. 6227	2.59091	1.212
Al - 7.5 wt% CCA	2.5891	2.5612	1.078
Al-10wt% <sub>CCA</sub>	2.5554	2.5113	1.726
Al-12.5wt% cca	2.5218	2.4914	1.205
Al-15wt% cca	2.4881	2.4367	2.066

Table 3 - Density and Apparent Porosity of the AA6063/CCA Composites and the Unreinforced AA6063

#### 3.2 Mechanical Properties

The influences of the reinforcement content on the hardness, wear resistance, impact strength and tensile strength of the AA6063 based composites reinforced with corn cob ash particles are presented in Figures 2 to 5.



Figure 2 - Hardness of the CCA reinforced composites and the unreinforced aluminium AA6063 alloy.

Figure 4, shows that AA6063 matrix composite reinforced with corn cob ash has lower impact strength than the base aluminium alloy, the reason being that as the concentration of the corn cob ash is increased, the number of hard oxides (SiO<sub>2</sub>, CaO, Al<sub>2</sub>O<sub>3</sub>) and carbon content of the composite is increased [36], the hardness of the composites (Figure 2) also increased progressively, thereby resulting in a corresponding decrease in the impact strength of the CCA reinforced composites, as it becomes more brittle.

Very low impact and tensile strengths were obtained for the 12.5 and 15wt% CCA reinforcement content because of the high volume of the dispersed phase (CCA) in the Al-Si-Mg alloy matrix. The analysis of the effect of the reinforcement weight fraction on the wear resistance of the aluminium alloy revealed that as the corn cob ash increases, the tendency of the

composite material to resist wear increased, this is indicated by the progressive decrease in wear index with the addition of the reinforcement particles (Figure 3). This may be explained to be as a result of good bonding between the aluminium alloy matrix and the particles of the reinforcements [5]. While in Figure 5, there was an abysmal deterioration in the tensile strength, as the amount of CCA in AA6063 matrix increased gradually. This is expected as a trade-off between hardness and strength. Most hard materials are low in tensile and impact strengths. This was why the optimization of the experimental results was carried out to ascertain the optimal composition of the reinforcement and matrix mixture that will yield optimal physical and mechanical properties.



Figure 3 - Wear index of the CCA reinforced composites and the unreinforced aluminium AA6063 alloy.



Figure 4 - Impact strength of the CCA reinforced composites and the unreinforced aluminium AA6063 alloy.



Figure 5: Tensile strength of CCA reinforced composites and the unreinforced aluminium AA6063 alloy.

#### 3.3 Optical Microscopy

The microstructure of the unreinforced alloy is shown in the optical micrograph of Figure 6a. The structure of unreinforced aluminium 6063 alloy is normally made up of essentially an array of Al12Mg7, AlFe2Mn, Mg2Si and plates of Al-Si-Mg compound in an alpha-aluminium matrix [32]. Micrographs of Figure 6b-g, show the microstructures of the CCA reinforced AA6063 alloy. The structures reveal the dispersal of particulate CCA at the grain boundaries. The microstructures reveal the homogeneous distribution of the CCA particles in the grain boundaries with a few agglomerates of the reinforcement. There was good interfacial bonding between the corncob ash particles and Al-Si-Mg matrix. The good interfacial bonding may be as a result of the presence of magnesium in the matrix which helped in enhancing wettability of the reinforcement phase in the metal matrix [41].



Figure 6: Optical micrographs of (A) unreinforced AA6063, (B) AA6063/2.5wt%CCA, (C) AA6063/5wt%CCA, (D) AA6063/7.5wt%CCA, (E) AA6063/10wt%CCA, (F) AA6063/12.5wt%CCA, & (G) AA6063/15wt%CCA.

#### 3.4 Results of Predictive Modelling and Optimization Using Mixture Design

The results from the mixture design study show that the density, apparent porosity, hardness, tensile strength, impact strength and wear index are functions of the mixture composition (reinforcement and matrix concentrations). The design

summary for the mixture components (matrix and reinforcement) and the responses are given in Tables 4 & 5 respectively. The tables indicate the analysis type, minimum and maximum values for the mixture components and the responses, mean, standard deviation, factor coding and the models, etc.

Component	Name	Units	Туре	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
А	Matrix	%	Mixture	85	100	$+0 \leftrightarrow 85$	$+1 \leftrightarrow 100$	92.50	6.03
В	Reinforcement	%	Mixture	0	15	$+0 \leftrightarrow 0$	$+1 \leftrightarrow 15$	7.50	6.03
				Total = 100.00		L_Pseud	o Coding		

Table 4 - Design Summary for Mixture Components.

		Table 5 - Des	ign summary i	or responses.		
Response	<b>R</b> 1	R2	R3	R4	R5	R6
Name	Density	Porosity	Impact Strength	Hardness	Tensile Strength	Wear Index
Units	$g/cm^3$	%	$J/mm^2$	BHN	MPa	
Obs	12	12	12	12	12	12
Analysis	Polynomial	Polynomial	Polynomial	Polynomial	Polynomial	Polynomial
Minimum	2.4367	1.078	0.125	26	64.6682	7.5
Maximum	2.6605	2.066	1.125	58.58	241.188	20
Mean	2.55	1.46	0.5760	41.34	151.90	13.46
Std. Dev.	0.0882	0.4319	0.4012	13.63	70.89	5.27
Ratio	1.09	1.92	9.00	2.25	3.73	2.67
Transform	None	None	None	None	None	None
Model	Linear	Linear	Quadratic	Linear	Quartic	Quartic

Table 5 - Design summary for responses.

#### 3.4.1 Analysis of Variance (ANOVA), Fit Statistics and Model Equations

The detailed results of the polynomial analysis of the various factors are discussed in terms of the analysis of variance (ANOVA), modelling and numerical optimization. The diagnostics plots and model graphs of predicted response values versus actual/observed response values for all the factors showed a linear graph (Figure 7a-f) with the data points split evenly by the 45-degree line, indicating that the design models are sufficient to predict the factors' responses.

The analysis of variance (ANOVA) and the summary of fit statistics for the composites response properties are presented in Tables 6 -11. The descriptive statistics are used as a secondary check for the usefulness of the models. The probability values (**P-value**) obtained in the regression for all the responses were less than 0.05, which is a good indication that the model terms (matrix and reinforcements) influenced significantly those response variables. In other words, the model's terms are significant for all the responses. The **model F-value** is as well significant for all the six responses. Conversely, **the lack-offit F-values** for all the models are non-significant relative to the pure error, and since the model should fit, non-significant lack of fit is good. The **predicted R<sup>2</sup>** and **adjusted R<sup>2</sup>** values are in reasonably good agreement for all the responses analyzed because their differences are less than 0.2 [42]. The **Adeq Precision** (adequate precision), is a measure of the signal to noise ratio. Adequate precision values greater than 4, indicate models that have signals that are strong enough and suitable to be used for optimization. The ratios obtain are greater than 4 for all the responses, showing that the signals are adequate and that the models can be used to navigate the entire design space.

Source	Sum of Squares	df	Mean Square	<b>F-value</b>	p-value	
Model	0.0850	1	0.0850	1309.43	< 0.0001	significant
Linear Mixture	0.0850	1	0.0850	1309.43	< 0.0001	
Residual	0.0006	10	0.0001			
Lack of Fit	0.0006	5	0.0001			
Pure Error	0.0000	5	0.0000			
Cor Total	0.0857	11				
Std. Dev.	0.0081		R <sup>2</sup>	0.9924		
Mean	2.55		Adjusted R <sup>2</sup>	0.9917		
C.V. %	0.3158		Predicted R <sup>2</sup>	0.9897		
			Adeq Precision	66.4780		

Table 6 - ANOVA and fit statistics for the density of AA6063/CCA composites.

Table 7 - ANOVA and fit statistics for apparent porosity of AA6063/CCA composites.

Source	Sum of Squares	df	Mean Square	<b>F-value</b>	p-value	
Model	1.05	1	1.05	10.55	0.0087	significant
Linear Mixture	1.05	1	1.05	10.55	0.0087	
Residual	0.9983	10	0.0998			
Lack of Fit	0.9983	5	0.1997			
Pure Error	0.0000	5	0.0000			
Cor Total	2.05	11				
Std. Dev.	0.3160		R <sup>2</sup>	0.5135		
Mean	1.46		Adjusted R <sup>2</sup>	0.4648	-	
C.V. %	21.66		Predicted R <sup>2</sup>	0.3398	-	
			Adeq Precision	5.9683	-	

Table 8 - ANOVA and fit statistics for impact strength of AA6063/CCA composites.

Source	Sum of Squares	df	Mean Square	<b>F-value</b>	p-value	
Model	1.74	2	0.8706	270.18	< 0.0001	significant
Linear Mixture	1.71	1	1.71	532.08	< 0.0001	
AB	0.0267	1	0.0267	8.28	0.0183	
Residual	0.0290	9	0.0032			
Lack of Fit	0.0290	4	0.0072			
Pure Error	0.0000	5	0.0000			
Cor Total	1.77	11				
Std. Dev.	0.0568		R <sup>2</sup>	0.9836		
Mean	0.5760		Adjusted R <sup>2</sup>	0.9800		
C.V. %	9.85		Predicted R <sup>2</sup>	0.9742		
			Adeq Precision	34.6003		

Source	Sum of Squares	df	Mean Square	<b>F-value</b>	p-value	
Model	1986.15	1	1986.15	345.02	< 0.0001	significant
Linear Mixture	1986.15	1	1986.15	345.02	< 0.0001	
Residual	57.57	10	5.76			
Lack of Fit	57.57	5	11.51			
<b>Pure Error</b>	0.0000	5	0.0000			
Cor Total	2043.72	11				
Std. Dev.	2.40		R <sup>2</sup>	0.9718		
Mean	41.34		Adjusted R <sup>2</sup>	0.9690	- -	
C.V. %	5.80		Predicted R <sup>2</sup>	0.9646		
			Adeq Precision	34.1239		

Table 9 - ANOVA and fit statistics for the hardness of AA6063/CCA composites.

 Table 10 - ANOVA and fit statistics for tensile strength of AA6063/CCA composites.

Source	Sum of Squares	df	Mean Square	<b>F-value</b>	p-value	
Model	55192.57	4	13798.14	1147.74	< 0.0001	significant
Linear Mixture	54665.57	1	54665.57	4547.13	< 0.0001	
AB	122.15	1	122.15	10.16	0.0153	
AB(A-B)	6.86	1	6.86	0.5708	0.4746	
<b>AB</b> ( <b>A-B</b> ) <sup>2</sup>	509.64	1	509.64	42.39	0.0003	
Residual	84.15	7	12.02			
Lack of Fit	84.15	2	42.08			
<b>Pure Error</b>	0.0000	5	0.0000			
Cor Total	55276.73	11				
Std. Dev.	3.47		<b>R</b> <sup>2</sup>	0.9985		
Mean	151.90		Adjusted R <sup>2</sup>	0.9976		
C.V. %	2.28		Predicted R <sup>2</sup>	0.9923		
			Adeq Precision	78.7172		

Table 11 - ANOVA and fit statistics for wear index of AA6063/CCA composites.

Source	Sum of Squares	df	Mean Square	<b>F-value</b>	p-value	
Model	305.65	4	76.41	7042.63	< 0.0001	significant
Linear Mixture	299.72	1	299.72	27623.92	< 0.0001	
AB	3.05	1	3.05	280.70	< 0.0001	
AB(A-B)	2.58	1	2.58	237.49	< 0.0001	
<b>AB</b> ( <b>A-B</b> ) <sup>2</sup>	1.30	1	1.30	119.60	< 0.0001	
Residual	0.0760	7	0.0109			
Lack of Fit	0.0760	2	0.0380			
<b>Pure Error</b>	0.0000	5	0.0000			
Cor Total	305.73	11				
Std. Dev.	0.1042		<b>R</b> <sup>2</sup>	0.9998		
Mean	13.46		Adjusted R <sup>2</sup>	0.9996	- -	
C.V. %	0.7740		Predicted R <sup>2</sup>	0.9980	- -	
			Adeq Precision	185.6083	- -	



Figure 7 - Model graphs of predicted response values against observed response values of (a) Density, (b) Apparent porosity, (c) Impact strength, (d) Hardness, (e) UTS, and (f) Wear index.

ANOVA for mixture design normally generates three model equations for each response. One for the pseudo model (factor coding) taken from the model coefficient table, one in the real coding scale, and the third one in the actual coding scale. The default analysis computes and tests the pseudo model which is then converted to the real model and finally to the actual model. The predictive equations of density, apparent porosity, hardness, impact strength, tensile strength and wear index for the composites as a function of the mixture components are given in a coded form and outlined in equations (4a, b, c, d, e & f). The model equations expressed in terms of coded can be used to predict the responses of the composite properties at specified weight concentrations (wt%) of the mixture components (A & B). By default, the high levels of the mixture components are useful for identifying the relative impact of the factors (mixture components) by comparing the factor coefficients [17].

 $density = 2.66088 \times A + 2.44219 \times B$   $apparent \ porosity = 1.07379 \times A + 1.84364 \times B$   $impact \ strength = 1.11186 \times A + 0.129833 \times B - 0.439925 \times AB$   $hardness = 24.6268 \times A + 58.0515 \times B$   $tensile \ strength = 241.115 \times A + 64.9369 \times B + 31.8465 \times AB + 16.9371 \times AB(A - B) - 303.073 \times AB(A - B)^{2}$   $wear \ index = 19.9901 \times A + 7.51031 \times B - 5.0286 \times AB + 10.379 \times AB(A - B) + 15.2934 \times AB(A - B)^{2}$ (4)

#### 3.4.2 Design Numerical Optimization

Mixture design of the DOE enables the optimization of response values by manipulating the mixture components. Optimization of the independent variable (mixture components) was performed to minimize the density, porosity, wear index and maximize hardness, impact and tensile strengths responses of the composites. During numerical optimization, target criteria constraints were specified for both the mixture components and the responses. The matrix and the reinforcement concentrations were set to be in range. Conversely, the responses of density, apparent porosity and wear index were set to minimum, while that of hardness, impact and tensile strengths were set to maximum. The ultimate goal is not to maximize the desirability value, but the factor settings that result in the highest desirability value indicate there is an acceptable outcome of the numerical optimization. One numerical optimization solution of the mixture components and responses for the CCA reinforced aluminium alloy AA6063 composites with the degree of desirability is presented in Table 12. The results of numerical optimization indicate that at a matrix and reinforcement concentrations of 92.081 and 7.919% respectively, optimal responses will be obtained for the developed composite.

No	1
Matrix	92.081
Reinforcement	7.919
Density	2.545
Porosity	1.480
Impact Strength	0.484
Hardness	42.273
Tensile Strength	156.040
Wear Index	12.016
Desirability	0.512

#### 4 Conclusion

From this investigation, the following conclusions can be drawn.

- AA6063 alloy/CCA composites were successfully produced using stir casting technique.
- The hardness of the composite is more when compared to the base AA6063 alloy and increases as the reinforcement weight fraction is increased.
- Addition of corn cob ash increases, the tendency of the material to resist wear.
- AA6063 alloy/CCA composites produced have a lower density than AA6063 base alloy. This means that lightweight load-bearing composites can be produced using corn cob ash as reinforcement.
- The porosity of the composite is low and within an acceptable level of 0-4% due to filler matrix compatibility and stir casting process.
- The application of this solid agricultural waste in AMMC production, especially for automotive components requiring lightweight and wear resistance, will help to rid our neighbourhood of this agricultural solid waste residue and maintain a green environment.

Mixture design modelling revealed the optimal reinforcement and matrix composition that yield optimal responses
of composite physical and mechanical properties.

#### References

- M. Sanjay, G. Arpitha, L.L. Naik, K. Gopalakrishna, B. Yogesha, Applications of natural fibers and its composites: An overview. Natural Resources 7(3) (2016) 108-114. doi:10.4236/nr.2016.73011.
- [2]- S.N. Monteiro, K. Satyanarayana, A. Ferreira, D. Nascimento, F. Lopes, I. Silva, A. Bevitori, W. Inácio, J. Bravo Neto, T. Portela, Selection of high strength natural fibers. Matéria 15(4) (2010) 488-505.
- [3]- S.N. Monteiro, K.G. Satyanarayana, F.P.D. Lopes. High strength natural fibers for improved polymer matrix composites. Mater. Sci. Forum 638-642(2010) 961-966. doi:10.4028/www.scientific.net/MSF.638-642.961.
- [4]- U. Shehu, O. Aponbiede, T. Ause, E. Obiodunukwe, Effect of particle size on the properties of polyester/palm kernel shell (PKS) particulate composites. J. Mater. Environ. Sci, 5(2) (2014) 366-373.
- [5]- V.R. Rao, J.R. Chowdary, A. Balaji, D.S. Krishna, B. Bhavabhuthi, G. Sreevatsava, K. Abhiram, A review on properties of aluminium based metal matrix composites via stir casting. Int. J. Sci. Eng. Res. 7(2) (2016).
- [6]- R. Dasgupta, Aluminium alloy-based metal matrix composites: a potential material for wear resistant applications. Int. Schol. Res. Not. (2012). doi: 10.5402/2012/594573.
- [7]- P. Babalola, C. Bolu, A. Inegbenebor, K. Odunfa, Development of aluminium matrix composites: a review. Online Int. J. Eng. Technol. Res. 2 (2014) 1-11.
- [8]- A.V. Muley, S. Aravindan, I. Singh, Nano and hybrid aluminum based metal matrix composites: an overview. Manufact. Rev. 2 (2015) 15. doi:10.1051/mfreview/2015018
- [9]- M. Dinesh, D.R. Ravindran, Aluminium AA 7075 properties enhanced by Zirconium and Chromium nano particle. Int. J. Chem. Tech. Res. 9(1) (2016) 296-301.
- [10]- M.O. Bodunrin, K.K. Alaneme, L.H. Chown, Aluminium matrix hybrid composites: a review of reinforcement philosophies; mechanical, corrosion and tribological characteristics. J. Mater. Res. Technol. 4(4) (2015) 434-445. doi:10.1016/j.jmrt.2015.05.003.
- [11]- R. Akaluzia, F. Edoziuno, A. Adediran, B. Odoni, S. Edibo, T. Olayanju, Evaluation of the effect of reinforcement particle sizes on the impact and hardness properties of hardwood charcoal particulate-polyester resin composites. Mater. Today Proceedings (2020). doi:10.1016/j.matpr.2020.02.980.
- [12]- M. Surappa, Aluminium matrix composites: Challenges and opportunities. Sadhana, 28(1-2) (2003) 319-334. doi:10.1007/BF02717141.
- [13]- S. Adeosun, L. Osoba, O. Taiwo, Characteristics of aluminum hybrid composites. World Academy of Science, Engineering and Technology, Open Science Index 91, Int. J. Mater. Metal. Eng. 8(7) (2014) 731-738.
- [14]- R. Umunakwe, D. Olaleye, A. Oyetunji, O. Okoye,I. Umunakwe, Assessment of the density and mechanical properties of particulate periwinkle shell-aluminium 6063 metal matrix composite (PPS-ALMMC) produced by two-step casting. Acta Technica Corviniensis-Bul. Eng. 10(1) (2017) 83.
- [15]- D. Srinivasan, R. Kadadeveramath, S. Rajendran, E. Ganesh, State-of-art review of ceramic reinforced aluminum metal matrix composite and it's machining characteristics. J. Chem. Pharm. Sci. 11 (2017) 154-164.
- [16]- K. Alaneme, M. Bodunrin, Mechanical behaviour of alumina reinforced AA 6063 metal matrix composites developed by two step-stir casting process. Acta Tech. Corviniensis-Bul. Eng. 6(3) (2013) 105.
- [17]- F. Edoziuno, A. Adediran, B. Odoni, A. Akinwekomi, O. Adesina, M. Oki, Optimization and development of predictive models for the corrosion inhibition of mild steel in sulphuric acid by methyl-5-benzoyl-2-benzimidazole carbamate (mebendazole). Cogent Eng. 7(1) (2020) 1714100. doi:10.1080/23311916.2020.1714100.
- [18]- A. James, M.B. Davanageri, H. Rangaswamy, B.K. Chacko, D.M. Shanthappa, R. Kumar, D.S. Akkunji. The study of wear behaviour of glass/epoxy composites reinforced with titanium dioxide and graphite filler materials. in AIP Conference Proceedings, 2019. doi:10.1063/1.5092894.
- [19]- M.M. Kandar, H. Akil, Application of design of experiment (DoE) for parameters optimization in compression moulding for flax reinforced biocomposites. Proceedia Chem. 19(2016) 433-440. doi:10.1016/j.proche.2016.03.035.
- [20]- C. Homkhiew, T. Ratanawilai, W. Thongruang, The optimal formulation of recycled polypropylene/rubberwood flour composites from experiments with mixture design. Composites Part. B-Eng. 56 (2014) 350-357. doi:10.1016/j.compositesb.2013.08.041.
- [21]- C. Homkhiew, T. Ratanawilai, W. Thongruang, Optimizing the formulation of polypropylene and rubberwood flour

composites for moisture resistance by mixture design. J. Reinf. Plast. Compos. 33(9) (2014) 810-823. doi:10.1177/0731684413518362.

- [22]- P.S. Kumar, S.P. Reddy, G. Kartheek, Study of Hardness and Optimization of Wear Properties of AL 7075 Hybrid Composite. Int. J. Eng. Res. Technol. 6(6) (2017). doi:10.17577/IJERTV6IS060401.
- [23]- G. Agarwal, A. Patnaik, R.K. Sharma, Parametric optimization and three-body abrasive wear behavior of sic filled chopped glass fiber reinforced epoxy composites. Int. J. Compos. Mater. 3(2) (2013) 32-38. doi:10.5923/j.cmaterials.20130302.02.
- [24]- A. Vedrtnam, A. Kumar, Fabrication and wear characterization of silicon carbide and copper reinforced aluminium matrix composite. Mater. Discovery 9(2017) 16-22. doi:10.1016/j.md.2018.01.002.
- [25]- I.Y. Mohammed, Y.A. Abakr, S. Yusup, F.K. Kazi, Valorization of Napier grass via intermediate pyrolysis: Optimization using response surface methodology and pyrolysis products characterization. J. Cleaner Product. 142 (2017) 1848-1866. doi:10.1016/j.jclepro.2016.11.099.
- [26]- C.R. Kothari, Research methodology: Methods and techniques. New Age International, 2004.
- [27]- R. Umunakwe, D. Olaleye, A. Oyetunji, O. Okoye, I. Umunakwe, Assessment of some mechanical properties and microstructure of particulate periwinkle shell-aluminium 6063 metal matrix composite (PPS-ALMMC) produced by two-step casting. Nigerian J. Technol. 36(2) (2017) 421-427. doi:10.4314/njt.v36i2.14.
- [28]- A. Yekinni, M. Durowoju, J. Agunsoye, L. Mudashiru, L. Animashaun,O. Sogunro, Automotive application of hybrid composites of aluminium alloy matrix: A review of rice husk as based reinforcements. Int. J. Compos. Mater. 9(2019) 44-52. doi:10.5923/j.cmaterials.20190902.03.
- [29]- J. Hernández-Ruiz, L. Pino-Rivero, E. Villar-Cociña, Aluminum Matrix Composite with Sugarcane Bagasse Ash as Reinforcement Material. Revista Cubana de Física 36(1) (2019) 55-59.
- [30]- N. Srivastava, S.P. Dwivedi. Development of Green Hybrid Metal Matrix Composite using Agricultural Waste Bagasse as Reinforcement-A Review. Mater. Sci. Eng. Conf. Series (2019) doi:10.1088/1757-899X/691/1/012051.
- [31]- F.O. Edoziuno, B.U. Odoni, F.I. Alo,C.C. Nwaeju, Research paper dry sliding wear and surface morphological examination of an aluminium matrix composite reinforced with palm kernel shell. Acta Metal. Slovaca 26(2) (2020) 54-62. doi:10.36547/ams.26.2.537.
- [32]- F.O. Edoziuno, A.A. Adediran, B.U. Odoni, O.G. Utu, A. Olayanju, Physico-chemical and morphological evaluation of palm kernel shell particulate reinforced aluminium matrix composites. Mater. Today Proc. (2020). doi:10.1016/j.matpr.2020.03.641.
- [33]- I.O. Oladele, A.M. Okoro, The effect of palm kernel shell ash on the mechanical properties of as-cast aluminium alloy matrix composites. Leonardo J. Sci. 15 (2016) 15-30.
- [34]- A. Apasi, D. Yawas, S. Abdulkareem, M. Kolawole, Improving mechanical properties of aluminium alloy through addition of coconut shell-ash. J. Sci. Technol. 36(3) (2016) 34-43. doi:10.4314/just.v36i3.4.
- [35]- O.O. Daramola, A.A. Adediran, A.T. Fadumiye, Evaluation of the mechanical properties and corrosion behaviour of coconut shell ash reinforced aluminium (6063) alloy composites. Leonardo Electron. J. Pract. Technol. 27(2015) 107-119.
- [36]- O.B. Fatile, J.I. Akinruli, A.A. Amori, Microstructure and mechanical behaviour of stir-cast Al-Mg-Sl alloy matrix hybrid composite reinforced with corn cob ash and silicon carbide. Int. J. Eng. Technol. Innov. 4(4) (2014) 251.
- [37]- O.O. Joseph,K.O. Babaremu, Agricultural Waste as a Reinforcement Particulate for Aluminum Metal Matrix Composite (AMMCs): A Review. Fibers 7(4) (2019) 33. doi:10.3390/fib7040033.
- [38]- K.R. Garadimani, G. Raju, K. Kodancha, Study on mechanical properties of corn cob particle and e-glass fiber reinforced hybrid polymer composites. Am. J. Mater. Sci. 5(3C) (2015) 86-91. doi:10.5923/c.materials.201502.18.
- [39]- K. Alaneme, A. Babajide, A. Omotoyinbo, J.O. Borode, M. Bodunrin, Corrosion and wear behavior of stir-cast aluminium-based hybrid composites reinforced with silicon carbide and corn cob ash. African Corr. J. 1(2) (2015).
- [40]- V. Aigbodion, I. Ezema, Multifunctional A356 alloy/PKSAnp composites: Microstructure and mechanical properties. Defence Technol. 16(3) (2020) 731-736. doi:10.1016/j.dt.2019.05.017.
- [41]- M.N. Nwabufoh. Development and characterization OF Al-3.7%Cu-1.4%Mg alloy/periwinkle ASH (Turritella communis) particulate composites. Thesis. Ahmadu Bello University, Zaria, Nigeria, 2015.
- [42]- F.O. Edoziuno, R.O. Akaluzia, B.U. Odoni,S. Edibo, Experimental study on tribological (dry sliding wear) behaviour of polyester matrix hybrid composite reinforced with particulate wood charcoal and periwinkle shell. J. King Saud Univ-Eng. Sci. (2020). doi:10.1016/j.jksues.2020.05.007.