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**PALM KERNEL SHELL ASH PARTICLE REINFORCEMENT** 

### ABSTRACT

The demand for high-performance materials has increased particularly across all industries in recent times due to the various areas of applications. Industries now require materials with a high strength-toweight ratio, excellent anti-corrosion properties, good mechanical properties and good thermal conductivity. Given this, this study investigated the effect of palm kernel shell ash particle addition on the mechanical and thermal properties of Al-Mg-Si material. The mechanical properties of aluminium alloy (Al-Mg-Si)/palm kernel shell ash (PKSA) particles composites developed by powder metallurgy method were investigated. Also, Thermo-Gravimetric Analysis (TGA), and Differential Thermal Analysis (DTA) analysis were carried out to determine their thermal properties. The produced PKSA was characterized with XRF to determine its elemental composition and the result showed that the presence of phases such as SiO<sub>2</sub> K<sub>2</sub>O and Fe<sub>2</sub>O<sub>3</sub> in high proportions which are known to be hard with extreme thermal resistance indicates a better chance as reinforcement. The mechanical properties (hardness, impact strength and flexural deflection) and the thermal properties were used as criteria to access the PKSA reinforced composite. Results revealed that there is an improved mechanical property of the developed composites (with a 43.76% increase in the hardness value and 252.03% increase in the impact strength) which is not farfetched from the reinforcement effect on the reduction in the percentage of elongation.

Keywords: Composites; Reinforcements; Microstructure; Mechanical; Ash

# **INTRODUCTION**

The major persuasive reasons for utilizing composites materials particularly over the traditional materials are their high quality, lightweight, improved quality and overall general performance (Shagwira et al. 2021). Composites can be commonly assigned as polymer-matrix composites (PMCs), metalmatrix composites (MMCs) and ceramicmatrix composites (CMCs) based on the variety of matrix material used for development (Sharma et al. 2015; Shagwira et al. 2021). A metal-matrix composite (MMCs) alludes to assembling at least two materials where one matrix phase can be magnesium, aluminium, copper, iron, and so on and the other support phase can be carbides, oxides, nitrides and basic materials, intentionally added to include the

preferred properties (Kumar, 2017). Mostly, the support phase is responsible for the general performance of the created composite (Rohatgi, 1994). These support face materials could be in form of continuous or discontinuous fibres, whiskers or particles in the matrix to accomplish the blend of better-quality properties (Kumar, 2017). By controlling the addition of the reinforcement in a composite, the preferred properties, for example, strength, stiffness, damping capacity and wear can be additionally upgraded (Rohatgi, 1994; Miracle, 2005). However, the use of naturally sourced agro-waste has been found to be suitable for reinforcement particulate, due to its availability and ease of assessability. Fly ash, red mud, palm oil clinker, rice husk ash, coconut shell debris and sugarcane bagasse are the various sorts of mechanical agro-waste materials used in material reinforcing for the advancement of composites (Safiuddin et al. 2010; Alidokht et al. 2011; Debnath et al. 2013). Immense potentials are required to empower the use of composite materials in different sectors making them fit as aerospace, automotive, construction materials and other engineering applications (Ramachandra and Radhakrishna, 2005: Safiuddin et al. 2010: Alidokht et al. 2011: Debnath et al. 2013). The use of these wastes as fortifying materials can diminish the natural contamination and the space required for their initial disposal. A thorough literature review on metal-matrix composites using mechanical agro-waste as fortifying materials exists in terms of monolithic composites (single reinforcement) and hybrid composites (more than one fortification materials) (Jayeola et al. 2018; Patil et al. 2018; Venkatesh et al. 2019; Dang, 2019; Mahanta et al. 2019). Hence this work is aimed at the investigating the effect of addition of palm kernel shell (an agricultural by-product) ash particle reinforcement on Al-Mg-Si on the mechanical and thermal behaviour.

## MATERIALS AND METHODS

## **Preparation of Materials:**

## Palm Kernel Shell Ash (PKSA) Preparation

Fresh palm kernel shells (PKS) were obtained from a palm oil processing firm in Ikire, Osun State, South-West, Nigeria. The sample used was a mixture of palm kernel shells from Dura and Pisifera species since the varieties are not habitually sorted during palm oil processing. Before the commencement of the experiment, the shell was sun-dried in an open space at about 32 °C. The palm kernel shell was crushed with beater crushers that enable the production of small particles. This was further ground in the Pascal L9FS two-roll mill to aid the production of the particle size after which the fresh palm kernel shell was heated in a furnace to a temperature of about 850 °C. At this stage, the material turned into its carbon ash state which is a popularly known carbonization technique called 'ashing' (Oyedeji et al. 2021).

# Al-Mg-Si/PKSA Composites Production Aluminium alloy (Al-Mg-Si) matrix

composites used for this study was obtained by powder metallurgy method carried out in Metallurgical and Materials Engineering Laboratory, Ahmadu Bello University, Zaria, Nigeria. The PKSA compositions of 0, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20 wt% on Al-Mg-Si powder was formulated which correlates to samples C1, C2, C3, C4, C5, C6, C7, C8, C9, C10, C11. The mixed samples were kept in the milling Machine, Pascal L9FS and ball milled with the aid of two roll mills. The operation was done by utilizing rotational cylindrical drum rollers operating in opposing pairs against a flat plate which crushed the materials. The milling was done slowly initially and regulated at a constant speed for about 60 hours, to enable the material to mix homogeneously. The samples were kept under loading for about 5 minutes before they were removed from the mould in preparation for sintering. Furthermore, the samples were compacted to the desired shape and some allowance was given to account for any shrinkage that might occur during the sintering process. The samples were heated to 560 °C in the furnace which is a temperature that is lower than the melting point of the powdered sample. Thereafter, the samples were left to cool in a controlled atmosphere (Oyedeji et al. 2021).

# Analytical Methods

# X-ray Fluorescence (XRF)

The chemical analysis of the palm kernel shell ash (PKSA) was carried out using an Xray fluorescence (XRF) machine. The X-ray Fluorescence (XRF) test was conducted with an XRF Machine, XSupreme 8000 by Oxford Instruments in Umaru Musa Yaradua University, Katsina. Katsina State. Nigeria.

# Mechanical Properties Determination Micro Hardness

The micro hardness test was conducted using the Rockwell hardness method. Each test samples which were formed to a size of 6.4 mm in size were used per the ASTM E18-79 standard. The indenter (diamond cone or hardened steel ball) of 1.56 mm in size was forced into the test sample with a load of 98.067 N (10 kgf). When equilibrium was reached the indicator device responded to the change in depth of penetration, this was followed by setting the indenter to a datum position. Thereafter, the value was taken directly from the semi-automatic digital scale and recorded (Aigbodion, 2019).

## **Impact Strength Determination**

The samples were prepared according to the ISO 8256 (2004) standard for DENT as specified (Fitri et al. 2020). The dimensions used to determine the impact strength was width of 10 mm, length of 80 mm and height of 64 mm. Notching was carried out with metal blades by a pneumatic notching tool on the thin sides of the sample up to an early crack length of 2 mm, which implies 1 mm on each side. The test was carried out under standard conditions according to ISO 291 (2008) at a temperature of 23 °C and relative air humidity of 50 % as specified (Fitri et al. 2020). The samples were fixed parallel between the stationary clamp and the crosshead, the pendulum hammer was set to hit the crosshead at the lowest point of the circular motion. Initially, at the gauge length of 30 mm, the notches were kept in the middle. This corresponded to the hammer speed between 2.9 and 3.7 m/s, then the hammer speed was set in between 1.0 m/s and 1.5 m/s which corresponded to the falling angle of  $40^{\circ}$  or  $60^{\circ}$ . The recorded values of load were analyzed with the aid of extension diagrams according to the service manual of the pendulum device (Odrobina et al. 2018).

# Determination of the Flexural Deflection Measurement

The sample was prepared in a rectangular shape with a length of 60.0 mm, a width of 30.0 mm and thickness of 5.0 mm based on the standard test method (ASTM D7028-07-2015) of the flexural strength test, using the Motorized Automatic Recording Tensometer in Mechanical Engineering Department, Laboratory, Ahmadu Bello University, Zaria. Nigeria. The autographic recording drum of the machine was removed and covered with the special graph sheet paper specified for the drum. The sample was installed on the Tensometer by using the flexural fixture and pins to keep it in place. The autographic recording drum was rotated to a suitable starting point and it was ensured that the perspex indicator was set to zero and it was also ensured that the pricker was on the zero axes of the graph sheet. As the load was applied to the samples the amount of elongation or deflection was transmitted via a rotating spindle continually until failure occurred. When the test was completed, the autographic recording drum was removed from the machine and the graph sheet paper was detached from it. On the graph sheet paper, the deflections were evaluated for analysis (Qu and Wang, 2018).

## Thermo-Gravimetric Analysis (TGA/DTA)

The thermo-gravimetric analysis and differential thermal analysis (TGA/DTA) of the samples were conducted using the Perkin Elmer TGA 4000 in the Mechanical Engineering Department, Laboratory, Federal University of Technology, Minna. Nigeria. The TGA measures the change in weight of the sample in relation to change in the controlled temperature and these curves showed the thermal scan between 30 °C and 950 °C at 10.00 °C/min during combustion. DTA and Thermo Gravimetric Analysis (TGA) were carried out on the control sample, developed composites and carbonized palm kernel shell at a heating rate of 10 °C/min in a nitrogen atmosphere (Qu and Wang, 2018).

# **Statistical Analysis**

This study employed a descriptive statistical analysis in studying the effect of PKSA reinforcement on Al-Mg-Si alloy. The percentage variation in the mechanical properties were computed using equation (1) (Anderson, 2019) which used the values of the mechanical properties of the samples (C2-C11) and the mechanical properties of the control sample (C1).

% variation =  $\frac{\text{property of samples - property of control}}{\text{property of control}} \times 100$  (1)

Furthermore, in obtaining the optimal sample, this sample used the data transformation statistical which transformed the mechanical properties obtained into ranking

for both the reinforced samples (C2-C11) and the control (C1). For the hardness and the impact strength, the "higher the better" data transformation technique was utilized while for the deflection, the "lower the better" data transformation technique was used.

### **RESULTS AND DISCUSSION**

The chemical analysis of the palm kernel shell ash (PKSA) result is as given in Table 1.

Table 1:	Composition	of PKSA	(wt.%)	using XRF
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$Al_2O_3$	SiO <sub>2</sub>	$P_2O_5$	SO <sub>3</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	$Cr_2O_3$	MnO	Fe <sub>2</sub> O <sub>3</sub>	NiO	CuO	Yb <sub>2</sub> O <sub>3</sub>
7.40	25.30	2.10	1.30	14.90	11.80	0.95	0.28	0.77	12.3	0.20	1.40	0.89 <sup>1</sup>

From Table 1, it is observed that major peaks of the chemical composition of the PKS ash were 25.3 wt% Silicon Oxide (SiO<sub>2</sub>), 14.9 wt% Potassium Oxide (K<sub>2</sub>O), 12.3 wt% Iron Oxide (Fe<sub>2</sub>O<sub>3</sub>), 11.8 wt% Calcium Oxide (CaO), 7.40 wt%, Aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) and 2.1 wt% Phosphorus pentoxide  $(P_2O_5)$  with very notable elements in small proportions. The presence of these elements such as SiO<sub>2</sub>, K<sub>2</sub>O and Fe<sub>2</sub>O<sub>3</sub> in high proportion indicates a better chance as reinforcement (Oyedeji et al. 2021).

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### Influence of the Reinforcement on the Mechanical Properties



Figure 1: Variation of hardness due to the effect of PKSA particulates addition

An Official Publication of Enugu State University of Science & Technology ISSN: (Print) 2315-9650 ISSN: (Online) 2502-0524 This work is licenced to the publisher under the Creative Commons Attribution 4.0 International License. As observed in Figure 1, sample C3 which corresponds to 4 wt% PKSA, 96 wt% Al-Mg-Si recorded the highest hardness value of 93 HRB which indicates a 47 % increase in the hardness value of the base sample with no PKSA percentage weight reinforcement (63.3 HRB) as denoted by C1. Similarly, samples C4 and C5 which corresponds to 6 wt% PKSA, 94 wt% Al-Mg-Si and 8 wt% PKSA, 92 wt% Al-Mg-Si both experienced an increase in hardness value due to the effect of PKSA to the matrix. With this occurrence, it is evident that the addition of the reinforcement to powdered alloy showed excellent improvement in the hardness value.

Equally, it can be inferred from this result that the processed PKSA reinforced Almatrix phase was better than the based sample

and this can be attributed to a greater force of adhesion between the processed PKSA and the Al-matrix phase. Similarly, a previous study shows that the addition of frit particles into the Al6061 alloy matrix enhances the hardness of the composites (Ramesh et al. 2010). Though the addition of frit particles at various proportions was from 0 to 10 wt% but in this study, the proportions were up to 20 wt%. It was however noticed that at 20 wt% PKSA addition, the hardness value of the sample which is denoted by C11 was not obtained due to the drastic drop in the hardness of the sample at this point. This can be attributed to the decrease in the interaction of the particles with each other leading to clustering of particles.



Figure 2: Variation of impact strength due to the addition of PKSA particulates

Result obtained from the impact strength of the samples (Figure 2) showed that sample C4 which consists of 6 wt% PKSA and 94 % wt Al-Mg-Si have the highest impact strength of 0.433 J with a 250% increase in the impact strength as against the base sample. Samples C9 and C10

with 16 wt% PKSA, 84 wt% Al-Mg-Si and 18 wt% PKSA, 82 wt% Al-Mg-Si respectively also had a noticeable increase in impact strength. This could also be a result of the high force of adhesion between palm kernel shell ash and aluminium matrix. Similar to the case of the

hardness result, at 20 wt% PKSA addition, the impact strength of the sample which is denoted by C11 reduced drastically. This can be

attributed to the decrease in the interaction of the particle with each other leading to the clustering of particles.





Figure 3 shows the flexural deflection of the samples (C1 to C11) based on the variation in the PKSA percentage weight reinforcement. The result shows that the highest value of flexural deflection is 1.8 mm for the base sample with no PKSA percentage weight reinforcement. This is followed by sample C11 with 20 wt% PKSA with a flexural deflection of 1.4 mm. However, a drastic decrease could be noticed in the transition from sample C1 to C2 which was averagely maintained up to sample C6. A low value of flexural deflection corresponds to an increase in flexural strength of the material. This implies that the addition of PKSA from 2 wt% to 10 wt% resulted in an

increase in material strength in comparison with the unreinforced sample. The reinforcement within the particle distribution of the samples could be the contributing factor. Beyond 10 wt%, a reduction in flexural strength which corresponds to an increase in the flexural deflection was noticed. This might have been caused as a result of grain boundaries, crack formation and difficulty in machining.

### **Statistical Analysis of Mechanical Properties**

This study employed a descriptive statistical analysis in studying the effect of PKSA reinforcement on Al-Mg-Si alloy. Table 2 shows the percentage variation in the mechanical properties of the Al-Mg-Si samples reinforced with PKSA in different variation with the control sample C1. This result showed that for the hardness of the developed composites, there was an increase by about 46.92% for sample C3, while sample C4 had an increase in hardness of 43.76% in comparison with the unreinforced Al-Mg-Si. Also, the percentage variation in impact strength of the samples showed that sample C4 had an increase

in impact strength by about 252.03% in comparison with the unreinforced sample, although samples C5, C7, and C11 experienced a decrease in impact strength. The variation of deflection of the samples with respect to the unreinforced sample C1 showed that samples C2, C3, and C4 had a decrease in deflection by about 66.67% which corresponds to increase in flexural strength of the samples.

Samples	% Variation in Hardness	% Variation in Impact Strength	% Variation in Deflection
C2	15.32	2.44	61.11
C3	46.92	12.20	66.67
C4	43.76	252.03	66.67
C5	39.02	-37.40	66.67
C6	43.76	12.20	63.89
C7	43.13	-46.34	61.11
C8	15.32	9.76	50.00
C9	40.60	143.90	38.89
C10	29.54	70.73	27.78
C11	-	-89.43	$22.22^{1}$

Table 2: Variation in the mechanical properties of the reinforced sa	amples (C2 – C11)
compared with the unreinforced sample (C1).	

Furthermore, a data transformation statistical analysis (as given in Table 3) was carried out to determine the optimal sample based on the mechanical properties considered for both the reinforced samples (C2-C11) and the control (C1). For the hardness and the impact strength, the "higher the better" data transformation technique was utilized while for the deflection, the "lower the better" data transformation technique was used. The overall ranking result obtained showed that sample C4 representing composition (6:94) wt% of PKSA/Al-Mg-Si was the optimal sample, this was followed by sample C3 and C6.

Samples	Average Hardness Values (HRB)	Ranking of Average Hardness Values	Average Impact Strength (J)	Ranking of Average Impact Strength	Deflection, (mm)	Ranking of Deflection	Overall Ranking
C1 (Control)	63.3	10th	0.123	7th	1.8	11th	10th
C2	73	8th	0.126	6th	0.7	5th	6th
C3	93	1st	0.138	4th	0.6	1st	2nd
C4	91	2nd	0.433	1st	0.6	1st	1st
C5	88	6th	0.077	9th	0.6	1st	4th
C6	91	2nd	0.138	4th	0.65	4th	3rd
C7	90.6	4th	0.066	10th	0.7	5th	6th
C8	73	8th	0.111	8th	0.9	7th	9th
C9	89	5th	0.3	2nd	1.1	8th	4th
C10	82	7th	0.21	3rd	1.3	9th	6th
C11	-		0.013	11th	1.4	10th	$11 \text{th}^1$

Statistical ranking of the samples with control inclusive. The table showed the effect of PKSA addition to Al-Mg-Si on its mechanical properties.

## Influence of the PKSA on Thermal Analysis of the Composite



**Fig 4: TGA/DTA curves for (6:94) wt.% of PKSA/Al-Mg-Si** An Official Publication of Enugu State University of Science & Technology ISSN: (Print) 2315-9650 ISSN: (Online) 2502-0524 This work is licenced to the publisher under the Creative Commons Attribution 4.0 International License. From the earlier results obtained from hardness. impact strength and flexural deflection (Section 3.1), thermal analysis was carried out on the control sample (C1) and the samples with promising results (C2, C3, C4, and C5). Figure 4 shows the TGA and DTA curves for these samples which denote compositions of 0, 2, 4, 6, and 8 wt% PKSA/Al-Mg-Si powder. The results show that for all formulations, there was an initial release of moisture at around 150 °C. This observation is in agreement with the result presented by Wang et al. (2009) who observed that the magnitude of the decomposition depends on the material properties and the contact area between the sample and the pan and that the heating rate has a major effect on the results. From the result, the sample with composition (6:94) wt% of PKSA/Al-Mg-Si uniquely peaked at about 770 °C corresponding to the decomposition of the ingredients in the aluminium melt. The first decomposition was noticed within the range of the onset temperature 300 °C and 370 °C with a corresponding mass depletion of 58 %. The second stage of decomposition was around 370-440 °C with a corresponding mass depletion of 21 % while the third stage of decomposition was found about 440 °C onwards, a plateau region exhibiting almost constant weight corresponding to about 9%. The decomposition of samples was a three-step process involving the weight loss in the first stage due to water molecules associated with the crystals corresponding to the drying period. The second step was a rapid loss of mass due to the formation of metal oxide during degradation at which major step in all thermochemical translation process involving biomass occurred and the third stage in which the degression cause a mass loss due to the loss of molecular fragments of the sample or as a result of particles of smoke released from burning caused by inconsistencies in the material itself, beyond the oxidation of carbon.

### **CONCLUSION**

The study revealed that Palm kernel shell ash (PKSA) particle was homogenously mixed in the aluminum alloy matrix and from results, the additions of PKSA particles increased the hardness (by 43.76%), increased the impact strength (by 252.03%) and considerably decreased the elongation of the composites (by 66.67%) for the optimal sample (C4). The strengthening was sustained at high temperatures at a weight fraction of PKSA and decreases hastily when further increased in the composition. The sample with composition (6:94) wt.% of PKSA/Al-Mg-Si uniquely peaked at about 770 °C and another occurred in the range of 600-650 °C which corresponds to the decomposition of the ingredients in the aluminum melt. The flexural elongation decreases with increasing constituent composition as well as with the increased aging time in the heat treatable alloys. Furthermore, the results showed that PKSA/Al-Mg-Si based composites have enhanced mechanical properties and can be recommended for use at high temperature, even though doubling the heating rate caused the mass depletion to about 50%. However, the temperature regime for this application area is achieved at the onset temperature of 300 °C.

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**CONFLICTS OF INTEREST:** The authors declare no conflicts of interest.

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