



Characterization, mechanical and wear behaviours of Aluminium alloy reinforced with sustainable agro-waste particulates for engineering applications

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Abstract: This paper aims to investigate the mechanical, microstructure and wear behaviours of aluminium-magnesium-chromium alloy reinforced with carbonated cow hoof ash (CCHA) at different weight percentages (0 wt.% to 20 wt.%) at 5 wt. % interval. The carbonated cow hoof ash was characterized by X-ray fluorescence (XRF). The matrix and the composites' morphology were studied using a scanning electron microscope to determine the distribution of carbonated cow hoof ash particulates within the matrix. The wear behaviour of variously reinforced alloys and composites was assessed using a Taber abrasion tester, while XRF analysis confirmed the presence of silica in the CCHA compositions: silica, iron oxide, magnesium oxide, zinc oxide, calcium oxide, potassium oxide, and others. Mechanical properties showed that tensile values increase with increases in CCHA, and the hardness value increases from 5 wt. % to 20 wt. % of CCHA. The impact energy exhibited a decreasing trend with increasing reinforcement content, while the flexural strength reached its optimum value at 10 wt. % CCHA. The morphological analysis revealed that the uniform distributions of CCHA within the matrix improved mechanical properties. The wear resistance of the composites improved with increasing applied load but declined as the weight percentage of CCHA increased. These wear behaviours make the aluminium alloy/CCHA composites suitable for various engineering applications where lightweight and wear resistance are critical. The areas include automotive components, such as brake rotors, pistons, and cylinder blocks, as well as lightweight structural components in the aerospace industry.

1. Introduction

The growth of the world's population and the increase in living standards resulting from agricultural activities, technological development, industrial, and mining activities have led to an increase in the quantity of waste materials generated (Li, 2020). The increasing demand in the automotive and

aerospace industries to minimize energy consumption and enhance fuel efficiency presents a major challenge. Aluminium matrix composites (AMCs) offer a distinctive blend of chemical, mechanical and physical properties unattainable in monolithic materials (Włodarczyk-Fligier *et al.*, 2008; Sandeep *et al.*, 2018; Sadashiva *et al.*, 2024). This is why AMCs were regarded as promising materials for both automotive and aerospace applications (Abdulwahab *et al.*, 2017).

These speedy advancements in industrial activities in recent times have resulted in an increased interest in composites containing low-density and cost effects (Selvam and Smart, 2020). This has made AMCs a strong competitor to steel in terms of versatility for use in a wide range of engineering applications (Higgins *et al.*, 2008; Azzaoui *et al.*, 2014; Alaneme and Olubambi, 2018; El Magri *et al.*, 2025). Another advantage of AMCs is the relatively low cost of processing in comparison to other matrix types. Also, simple processing techniques (such as casting and powder metallurgy) make the composites superior to other materials. However, one of the limitations peculiar to most unreinforced aluminium alloys is poor both mechanical and tribological behaviours (Suleiman *et al.*, 2018).

Different reinforcements such as aluminium oxide (Al_2O_3), silicon carbide (SiC), titanium carbide (TiC), tungsten (W), calcium oxide (CaO), silica (SiO_2), etc, have been used to enhance aluminium composite properties. Studies have also shown that hardness, toughness, tensile, and other properties were enhanced (Kumar *et al.*, 2016). These unnatural reinforcers, which are frequently used, were difficult to obtain in Nigeria, and the importation of such materials is at a high cost and with time constraints, which may impede the production of the composites. An alternative to these reinforcements in developing countries like Nigeria is to explore wastes such as agricultural, animal, and aquaculture, either in the form of ashes, fibres, or powders

The wastes of bagasse (Aigbodion, 2010), melon shell ash studied (Suleiman *et al.*, 2021), periwinkle shell powder (Ofem *et al.*, 2012), and oyster shells (Michele *et al.*, 2012), among others, for the development of AMCs have been investigated. The results also showed that waste ashes and powders generated contain high percentages of refractory materials, such as alumina (Al_2O_3), silica (SiO_2), hematite (Fe_2O_3), carbonate (CaCO_3) and calcium (Ca), among others, which can be explored for the production of composites. These ashes and powders are not just cost-effective but the available and environmentally friendly, and an alternative to the reinforcements in AMCs.

Cow hooves are animal waste by-products readily available across Nigeria and other parts of West Africa. Improper disposal of cow horns poses a serious environmental hazard, potentially degrading the land and contaminating surrounding areas where the waste is dumped. The utilization of this waste material can help minimize ecological contamination and conserve landfill space by transforming it into valuable resources for engineering applications (Khan *et al.*, 2025).

The research focused on the utilization of carbonated cow horn ash (CCHA) of the particulate size of 75 μm , dispersing into the Al-2.5%Mg-0.25%Cr alloy to produce matrix composites through the stir casting process which is cost-effective, sustainable, and environmentally friendly with engineering applications. CCHA was varied from 0 wt. % to 20 wt. % at 5 wt. % interval. The chemical analysis of carbonated cow hoof ash, characterizations, mechanical, and wear of the matrix and composites were carried out simultaneously by both X-ray Florescent (XRF) and Scanning Electron Microscope (SEM) attached with Energy Dispersive Spectroscopy (EDS).

2. Materials and Methods

2.1 Preparation of cow horn ash

The cow hooves were thoroughly washed with water, soaked in an acetone-based solution for one week, and then sun-dried for another week to eliminate fats and other impurities. The cow hooves were

then crushed into smaller pieces, as shown in [Figure 1a](#), to enable carbonization to occur evenly. The pieces of the hoofs were packed in a metal crucible and fired in an electric furnace to a temperature of 250 °C for 3 hours. The carbonized hoofs (carbon-rich) were crushed using a pestle and mortar, ground to powder in a conventional mill, and sieved to a size of 75µm in accordance to the findings ([Suleiman et al., 2018](#)). [Figure 1b](#) shows the ash sieved at 75µm. The particle size analysis of the carbonized cow hoof ash was carried out by ASTM using XRF.



Figures 1(a). Cow hoofs collections [and 1b.](#) CCHA particle size of 75 µm

2.2 Equipment

The equipment used in this research includes the following: Electric crucible furnace, Heating oven, Steel crucible pot, Hand shank ladle, Metal mould, Moulding sand, Wood patterns, Rammer, Grinding machine, Sieve, Saw, Bench and vice, Universal testing machine (Tensometric), Rockwell hardness machine, Izod impact machine, electronic weighing balance, Genius-IF Xenomerix XRF, and Pin on Disc machine.

2.3 Charge Calculations

These calculations consist of estimating the weight in percentage (%) and in grams (g) of the composition of the various materials that were charged into the furnace. This study was restricted to five distinct samples, each prepared with varying weight percentages of Al-Mg-Cr alloy and carbonated cow hoof powder mixtures, as outlined below. The charge calculations of the aluminium alloy and composites were carried out in the course of this research.

2.4 Production of Al-Mg-Cr/ Carbonated Cow hoof ash Particulate Composite

The present study utilized aluminium alloy (Al2.5Mg0.25Cr) and cow hoof ash (CCHA) with a particle size of 75 µm as base matrix and reinforcement, respectively. The chemical composition of the alloy and the amounts of carbonated cow hoof ash (CCHA) used as reinforcers were determined using charge calculations. The aluminium alloy was superheated to 830°C after being charged into a crucible furnace. The stainless-steel stirrer was used to stir the molten alloy/composites manually. The reinforcement particles were preheated to 200°C for 30 minutes. After preheating, CCHA particles were consolidated into the melt to exclude moisture. The magnesium content of the melt enhanced wettability. This magnesium improves the wettability between the matrix alloys, reinforcement, thus,

0 wt. % to 20 wt. % at 5 wt. % interval by equal CCHA proportions was used. Preheated moulds were set before casting the alloy and the composite. The mixture was poured into the mould, and after solidification, the alloy and the composite were knocked off from the sand mould using manual method by breaking the mould with a hammer and a pry bar. The released alloy and composite were fettled using a hand file to remove and clean the composites, respectively. After the casting process, five samples were produced for each test—one serving as the control sample and the remaining four as the composite specimens. Chemical analyses were conducted to determine the composition of the alloy and the composites as seen in [Figure 2](#).



Figure 2. The casting of the finished products

2.5 Mechanical properties

2.5.1 Determination of tensile strength

The samples were tested in tension at room temperature (30 °C) in accordance with ASTM E8 using a universal testing machine. (Instron). This standard ASTM E8/E8M is one of the most commonly referenced standards for metallic materials. It provides detailed guidelines on the dimensions and preparation of tensile test specimens. The standard specifies that the gauge length, width, and thickness of the specimen must be carefully controlled to ensure consistency in testing ([Beckert et al, 2025](#)). The test was conducted using a strain rate of 2mm/min. Tensile test specimens of the as-cast Al alloy and composite were prepared using a lathe and a shaper, following the specified dimensions shown in [Figure 3](#) ([Mu'azu et al., 2022](#); [Suleiman et al, 2021](#)). [Figure 3](#) shows the dimensions of the standard test bar used in these tests. The specimens were machined to the standard diameter size of 5 mm as specified in BS 2789:2002. A Tensometric universal testing machine (Tensometric) at the Department of Metallurgical and Materials Engineering, University of Nigeria was used for the tests.

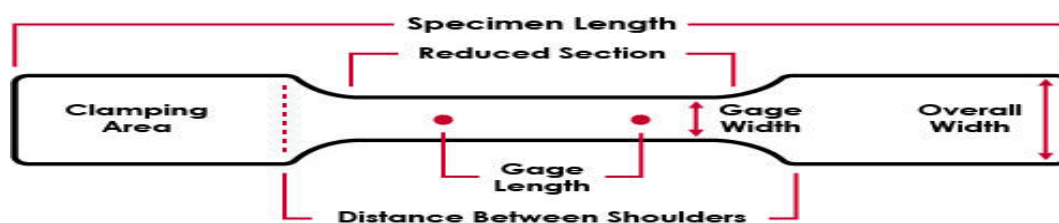


Figure 3. Dimensions of tensile test specimen

2.5.2 Determination of the hardness values

The hardness test was carried out using a Rockwell hardness machine. The hardness specimen was placed on a flat horizontal stand with a preload of the diamond cone indenter used to indent on the surface of the specimen. Its hardness value was reflected on a dial gauge of the machine and the readings were read from the calibrated C-scale of the gauge as carried out in (Abdullahi *et al*, 2022).

2.5.3 Determination of impact toughness

The impact specimen was placed on a horizontal stand of the Izod Impact Machine. It was arranged such that the notch was directly opposite to the point of impact of a heavily suspended mass. With the gauge set properly, the suspended mass was released from a height to hit the specimen. The energy absorbed by the specimen was reflected on a calibrated scale (Chhab *et al*, 2020).

2.5.4 Flexural testing

The flexural test was conducted to assess the flexural properties of the alloy and the composite, respectively. This test was carried out at the NLNG laboratory of the Metallurgical and Materials Engineering Department, University of Nigeria. A tensometric universal testing machine was used. The capacity and behaviour of the specimens when they were each subjected to a simple bending load of 2500 Kgf were recorded. The specimen was prepared as per ASTM: A-370 standard using a Tensometric universal machine, shown in Figure 4.



Figure 4. Testometric universal testing machine.

2.6 Wear test

The wear behaviours of the tested samples (alloy and composites) were determined using the Taber abrasion wear-testing machine under dry conditions as per ASTM G99-95 standards. The wear test specimens were 8 mm in diameter and 30 mm. The counterpart disc materials were made up of En-31 steel heat-treated with 8 mm and a surface roughness of 10 and Ra of 0.1. The initial weight of the specimen was measured using an electronic weighing machine with an accuracy of three digits (0.001g). During the experiment, the pin (specimen) was pressed against the rotating disc with a distance (500 mm). The experiment was conducted at different loads (15 and 20 N) to investigate the wear behaviour of the investigated alloy and composites. Mass loss (ΔM), wear loss (W), and coefficient of friction (μ) between the particles were determined by using the pin-on-disc wear test unit.

The sliding distance (L) was calculated to detect the wear rate as given in Eq. 1 (Subramani *et al.*, 2021):

$$L = 2\pi R^2 nt \quad \text{Eq. 1}$$

Where R is the radius of the counterpart disc (20 mm), n is the number of revolutions (200 rpm), t is the testing time (20 min), and π is a constant ($22/7$).

However, the volume of worn material (ΔV) was obtained from Eq. (2) (Suleiman *et al.*, 2021). Where Δm and ρ are the mass loss and density of the alloy and composites, respectively.

$$\Delta V = \frac{\Delta m}{\rho} \quad \text{Eq. 2}$$

From Eqs. (1) and (2), the wear rate (W) of the alloy and composites is calculated in Eq. (3) (Kumar *et al.*, 2016).

$$\text{Wear Rate} = \frac{\Delta v}{\rho \times h}, \frac{mm^2}{Nm} \quad \text{Eq. 3}$$

Where, Δv , P , and L are the worn material, applied load, and sliding distance. The characterization was carried out by using a scanning electron microscope (SEM).

2.7 Microstructural examination

The morphologies of the alloys and composites produced were examined using a scanning electron microscope (SEM). Specimens of the alloys and composites were polished on emery papers of different grades. The polishing was carried out on a circular cloth pad on its surface. Rough polishing was performed using silicon carbide paste, and the final polishing operation was conducted using alumina polishing paste. Etching of the specimen was carried out using cotton wool soaked in nital to wipe the specimen's polished surface to give a dull reflection surface (Chauhan *et al.*, 2017; Abdulwahab *et al.*, 2017). A Phenom Pro desktop scanning electron microscope (SEM) was employed for the characterization. The accelerating voltage was varied from < 1 to 30 kV on the specimens. Increasing the accelerating voltage decreased lens aberrations and thus better the resolution. BSE energy range was wide (from 50 eV to that of the incident beam energy). The morphologies of the alloy and composites, before and after wear tests were also carried out.

3. Results and discussion

XRF analysis of Carbonized cow hoof ash compositions (CCHA)

The chemical compositions of the carbonized cow hoof powder (CCHA) as described in the experimental procedure are presented in Tables 1-4. From the table 1, it is evident that the chemical analysis comprises various refractory materials, including Fe_2O_3 , SiO_2 , CaO , ZnO , K_2O , and P_2O_5 , with others present in trace amounts. However, the refractory materials played vital roles when used as fillers in the aluminium matrix composites for industrial applications. The presence of hard elements like Fe_2O_3 , SiO_2 , and CaO suggested that the carbonated cow hoof ash can be used as particulate reinforcement in various metal matrices according to the previous findings (Rao *et al.*, 2021; Kumar *et al.*, 2022).

Table 1. *XRF Result of carbonized cow hoof ash (CCHA)*

Compounds	Carbonated cow hoof powder (CCHP) (%)
SiO ₂	3.56
V ₂ O ₅	0.03
MnO	0.17
Fe ₂ O ₃	21.30
CuO	0.40
Nb ₂ O ₃	0.12
P ₂ O ₅	1.99
SO ₃	28.73
CaO	10.60
K ₂ O	7.46
Al ₂ O ₃	5.14
Ta ₂ O ₅	0.06
MgO	11.00
TiO ₂	0.66
ZrO ₂	0.06
WO ₃	0.07
ZnO	1.58
Cl	6.77

Table 2. *XRF Result of Al-Mg-Cr Alloy*

Compositions	Al-Mg-Cr Alloy (%)
Si	0.28
V	0.02
Cr	0.26
Mn	0.86
Fe	0.61
Cu	0.14
S	0.06
Ca	0.11
Mg	2.48
Al	96.50
Ta	0.09
Ti	0.03
Zn	0.04
Ag	0.01
Cl	0.64

Table 3. *Summary of charge calculations in weight percent [wt. %]*

S/NO:	0 wt. % CCHA	5 wt. % CCHA	10 wt. % CCHA	15 wt. % CCHA	20 wt. % CCHA
carbonized Cow hoof ash (CCHA)	0	5	10	15	20
Chromium	0.25	0.25	0.25	0.25	0.25
Magnesium	2.5	2.5	2.5	2.5	2.5
Aluminium	97.25	92.25	87.25	82.25	77.25
Total	100%	100%	100%	100%	100%

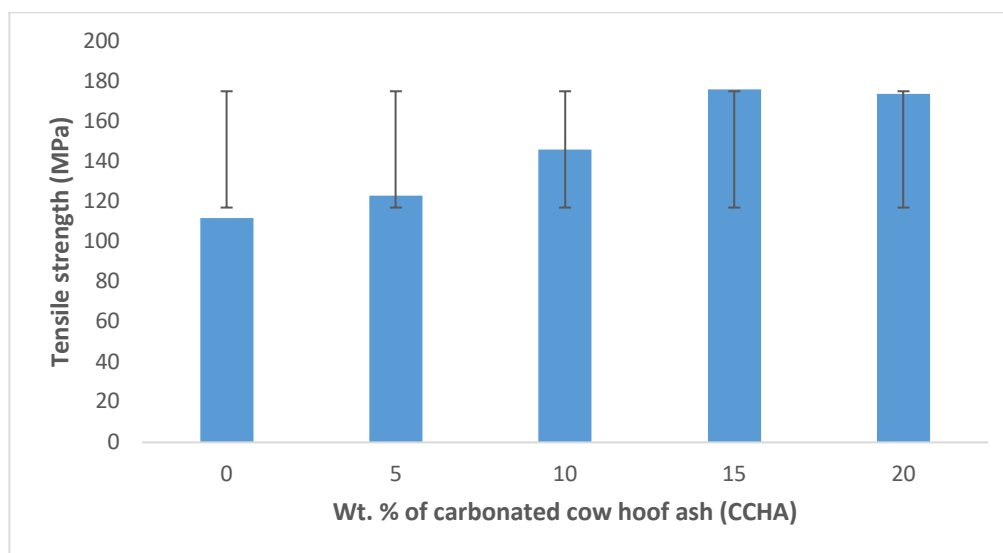
Table 4. Summary of the composition in grams (gm)

S/NO:	Alloy (0%)	5% CCHP	10% CCHP	15% CCHP	20% CCHP
Cow hoof powder (CHP)	0	7.37	14.74	22.11	29.48
Chromium	0.3685	0.3685	0.3685	0.3685	0.3685
Magnesium	3.685	3.685	3.685	3.685	3.685
Aluminium	143.3465	135.9765	128.6065	121.2365	113.8665
Total	147.4g	147.4g	147.4g	147.4g	147.4g

3.2 Mechanical Behaviours of Alloy and Composites

3.2.1 Tensile strength

The relation between the tensile strength of the Al-2.5%Mg-0.25%Cr alloy and composites with the different weight percentages of carbonated cow hoof ash particles is presented in Figure 5. It can be seen that the tensile strength increased with an increase in the weight percentage of carbonated cow hoof ash content. Hence, the CCHA particles acted as barriers to the dislocations when taking up the load applied. A similar observation was found in (Srinivas *et al*, 2020). The improvement observed in the tensile strength of the composite was attributed to the fact that the carbonated cow hoof ash acted as filler and possessed higher strength, which provided more resistance. Although there was a decrease in the tensile strength of the samples with carbonated cow hoof ash weight fraction beyond 15 wt.% % and can be estimated to be a 1.136 % reduction in the tensile strength from the ultimate to 20 wt.%. % CCHA. The decrease in tensile strength at 20 wt. % CCHA reinforced was also due to the effect of segregation of the oxide particles in tensile testing specimens and may be attributed to the poor wettability of the reinforcement with the matrix (Singh and Belokar 2020).

**Figure 5.** Tensile strength of Al-0.25%Mg-2.5%Cr/5–20 wt. % CCHA particulates

3.3.2 Hardness values

The hardness values of the developed composites increased with an increasing percentage of carbonated cow hoof ash particles from (5– 20%) additions. The hardness value of the Al-2.5%Mg-0.25%Cr% / (5–20 wt. % % CCHA) is illustrated in Figure 6. It could be observed that the hardness

values increase with an increase in the weight percent of CCHA in the composites. The increments in the hardness value of the composites can be attributed to the increase in the weight percentage of hard and brittle phases in the carbonated cow hoof ash particles within the Aluminium matrix alloy. The XRF analysis revealed the presence of major ceramic materials in the carbonated cow hoof ash, including K_2O , Mg_2O , CaO , SiO_2 , Al_2O_3 , and Fe_2O_3 , as presented in Table 1. In addition to the above, carbonated cow hoof ash particles in the alloy increase the dislocation density at the particle-matrix interfaces as a result of differences in the coefficient of thermal expansion (CTE) between the hard and brittle reinforced particles. This resulted in elastic and plastic incompatibility between the matrix and the reinforcement of the CCCHA (Ogheneme *et al*, 2022; Selvam *et al*, 2020).

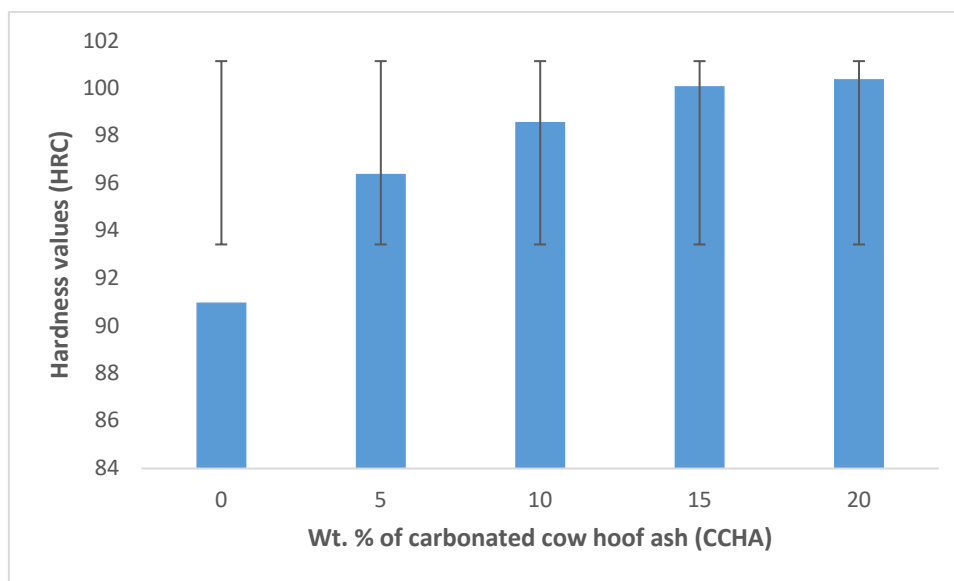


Figure 6. Variation of hardness of Al-2.5%Mg-0.25%Cr/5–20 wt. % CCHA particulates

3.3.3 Impact toughness

Figure 7 shows the result of the impact fracture of the alloy and the composites, respectively. It was observed that the fracture toughness of the composites containing 5, 10, 15, and 20 wt. % CCHA had lower fracture toughness than the Al-2.5%Mg-0.25%Cr alloy. The fracture mechanisms were attributed to particle cracking, interfacial cracking, and particle–matrix debonding (Dinaharan *et al*, 2019). It was also established that ceramic particulates are hard and brittle and tend to resist rapid crack propagation poorly. In this research work, a decrease in fracture toughness was observed in the new materials with 5, 10, and 15 wt.%. At 15 and 20 wt. %, the toughness values became virtually constant. However, it is clear that the addition of 5–20 wt. % CCHA deteriorated the fracture toughness of the Al-2.5%Mg-0.25%Cr reinforced with CCHA matrix composites (Rao *et al.*, 2021).

3.3.4. Flexural Strength

Figure 8 also presents the flexural test results for both the alloy and the newly developed materials. The results revealed that increasing the weight percentage of CCHA led to a noticeable improvement in flexural strength. Furthermore, with the incorporation of these reinforcements, the newly developed composites exhibited a significant enhancement in flexural strength. However, a reduction in flexural strength was observed at the reinforcement level of 15 wt. % CCHA. This increase in flexural strength from 5- 10 is attributed to the improved tensile strength resulting from the incorporation of CCHA.

Hence, the new composites within this range exhibit greater resistance to deformation and fracture, as they can endure higher flexural and bearing-induced stresses. This suggests that composites with lower weight percentages are better able to withstand higher loads without significant plastic deformation (Srinivas et al., 2020). This enhanced performance is due to the improved tensile strength from incorporating the elements present in the carbonated cow hoof ash. This change can be attributed to the particles and interfacial cracking of the produced composites, which is in line with the previous findings (Selvam et al., 2020).

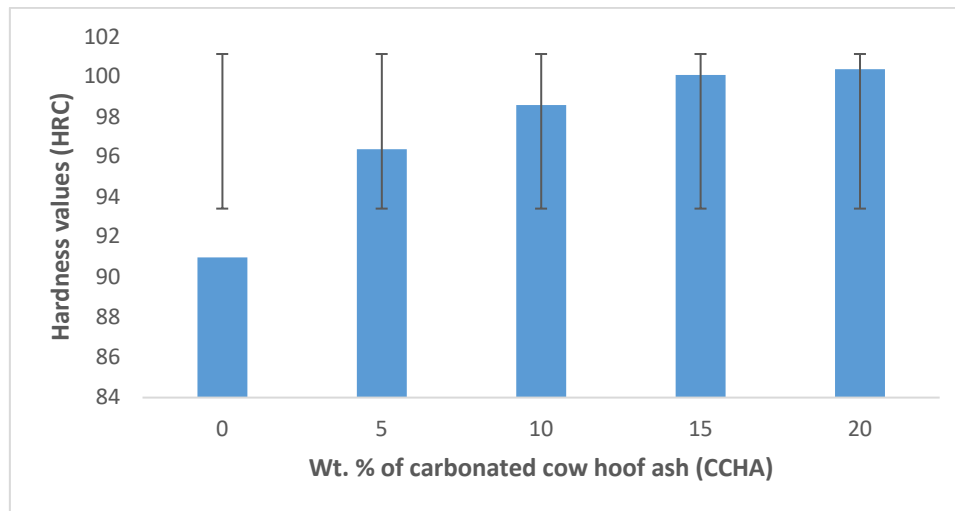


Figure 7. Variation of impact strength of Al-2.5%Mg-0.25%Cr/5–20 wt. % CCHA particulates

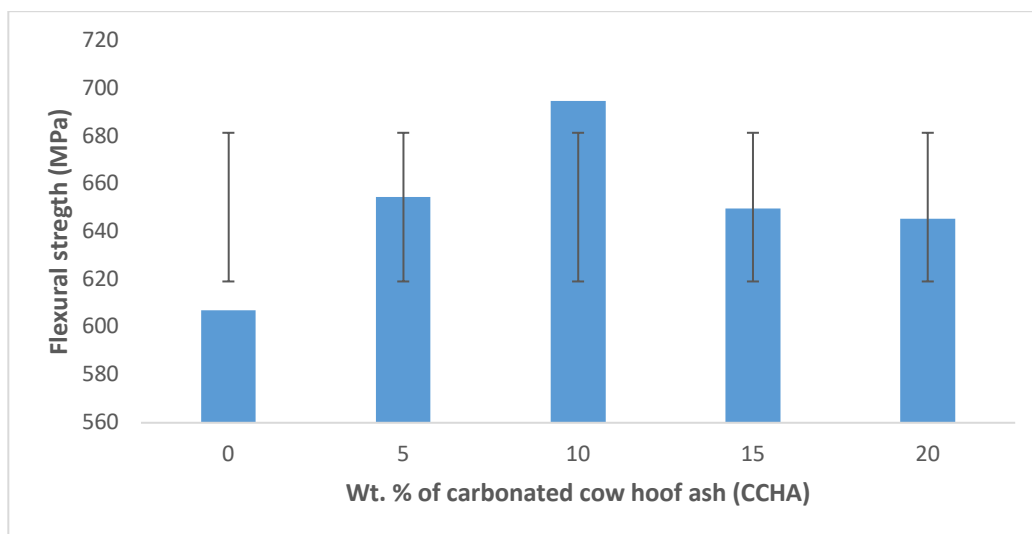


Figure 8. Flexural strength of Al-2.5%Mg-0, 25%Cr/5–20 wt. % CCHA particulates

3.4 Wear behaviours of aluminium alloy/composites

The wear behaviour of aluminium alloy reinforced with carbonated cow hoof ash (CCHA) particulates was investigated. Standards for tribological tests were carried out according to ASTM G99. These standard covers wear testing using a pin-on-disk apparatus. It involves a stationary pin or ball sliding against a rotating disk, with controlled load and speed. The test was used to evaluate wear rate only. The wear rate of the Al-Mg-Cr /carbonated cow hoof ash (CCHA) particulates composite increases when the load changes from 15 to 20 N. CCHA particulates. These reinforcements improve the mechanical and tribological properties of aluminium alloys by enhancing the hardness of the

composite and acting as a solid lubricant in certain conditions, reducing friction. Wear resistance increases with the increase in CCHA. The beneficial effect of the reinforcement on the wear resistance of the composites was observed to be the best at a low load of 15 N and is influenced by the unique properties of the reinforcement material and its interaction with the matrix (Chhab *et al*, 2020). The composites exhibited significantly higher wear resistance than the Al-Mg-Cr alloy due to the addition of CCHA, which has higher refractory materials that might have acted as a load-bearing constituent, as shown in Figure 9. Figure 10a and 10b showed the morphologies of the alloy and the composite at 20 wt. % CCHA particulates at minimum wear rate at an applied load of 15 N. It was observed that larger plastic deformation was noticed on the composites under the load of 20 N compared with 15 N. For tribological tests using the ASTM G99 standard, the number of repetitions typically depends on the specific objectives of the study and the desired level of statistical confidence. However, a common practice is to repeat the tests three times to ensure reproducibility and gather reliable data. From the results, specimens under the load of 20 N experienced greater weight loss when compared with that of the composite under 15 N (Liu *et al*, 2021).

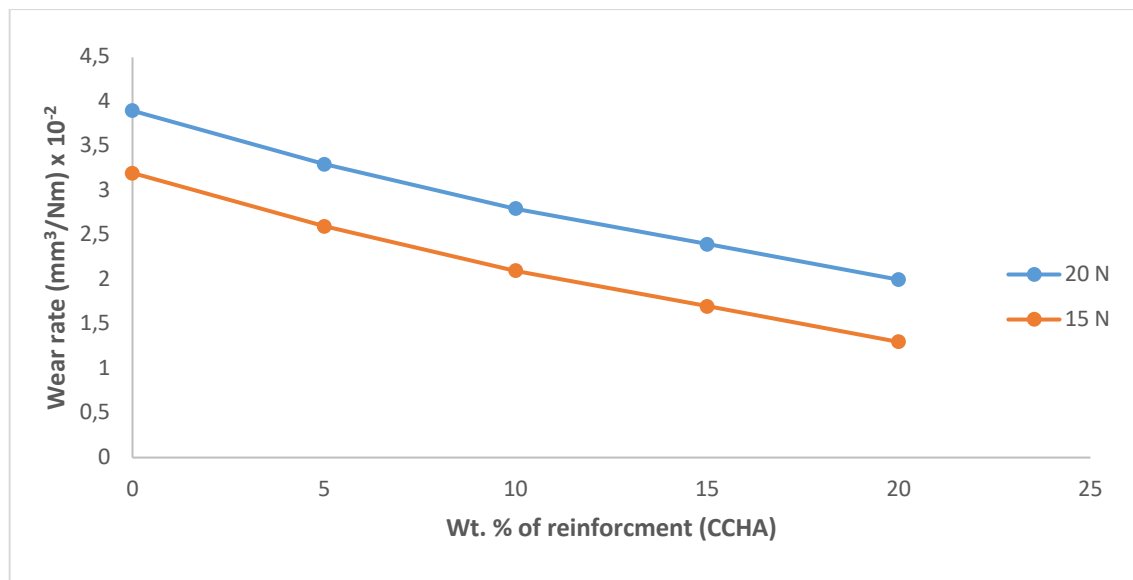


Figure 9. Effect of reinforcement on specific wear rate

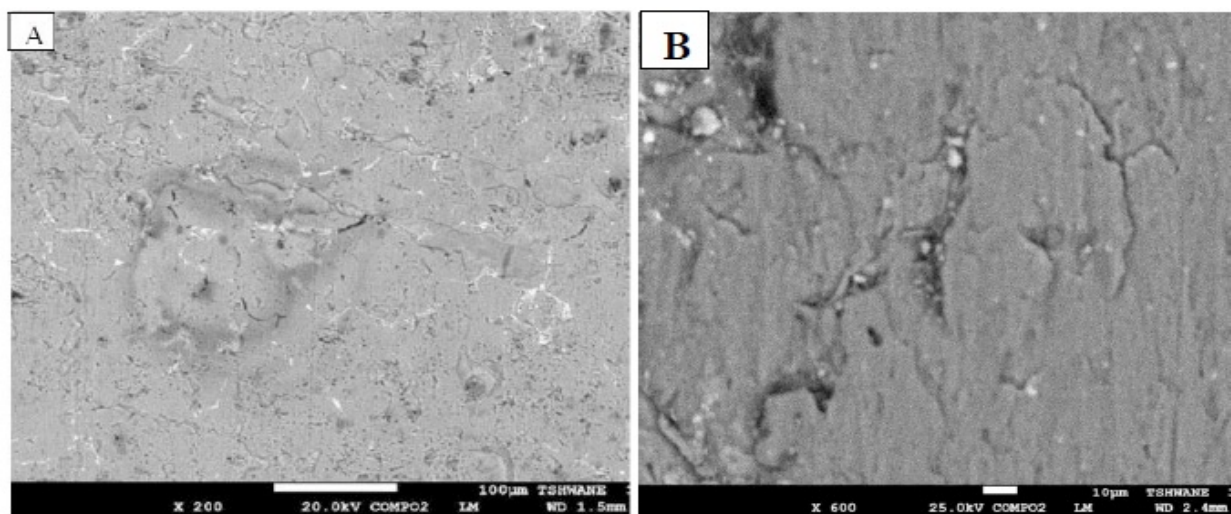


Figure 10. SEM morphology of Al-2.5%Mg-0.25%Si alloy (a) without load (b) reinforced with 20 wt. % CCHA particles at minimum applied load

3.5 Characterisations of the Alloy/Composites

As established earlier, microstructures of materials play an important role in the overall performance of engineering materials such as composites (Ogheneme *et al*, 2022). The physical properties of the composites, however, depend on the microstructure, reinforcement particle size, shape, and distribution within the matrix. **Figure 11** (a-e) presents the morphologies of the composite matrix from 0–20 wt. % at 5 wt. % interval, respectively. It was also found that there was good bonding between the matrix and carbonated cow hoof ash particulates at different weight percentages. The microstructures of the composites revealed no discontinuities and a reasonable uniform distribution of carbonated cow hoof ash within the matrix.

There was good retention and strong interfacial bonding of the carbonated cow hoof ash particles within the composites. The results were also in agreement with the previous findings (Abdullahi *et al*, 2022).

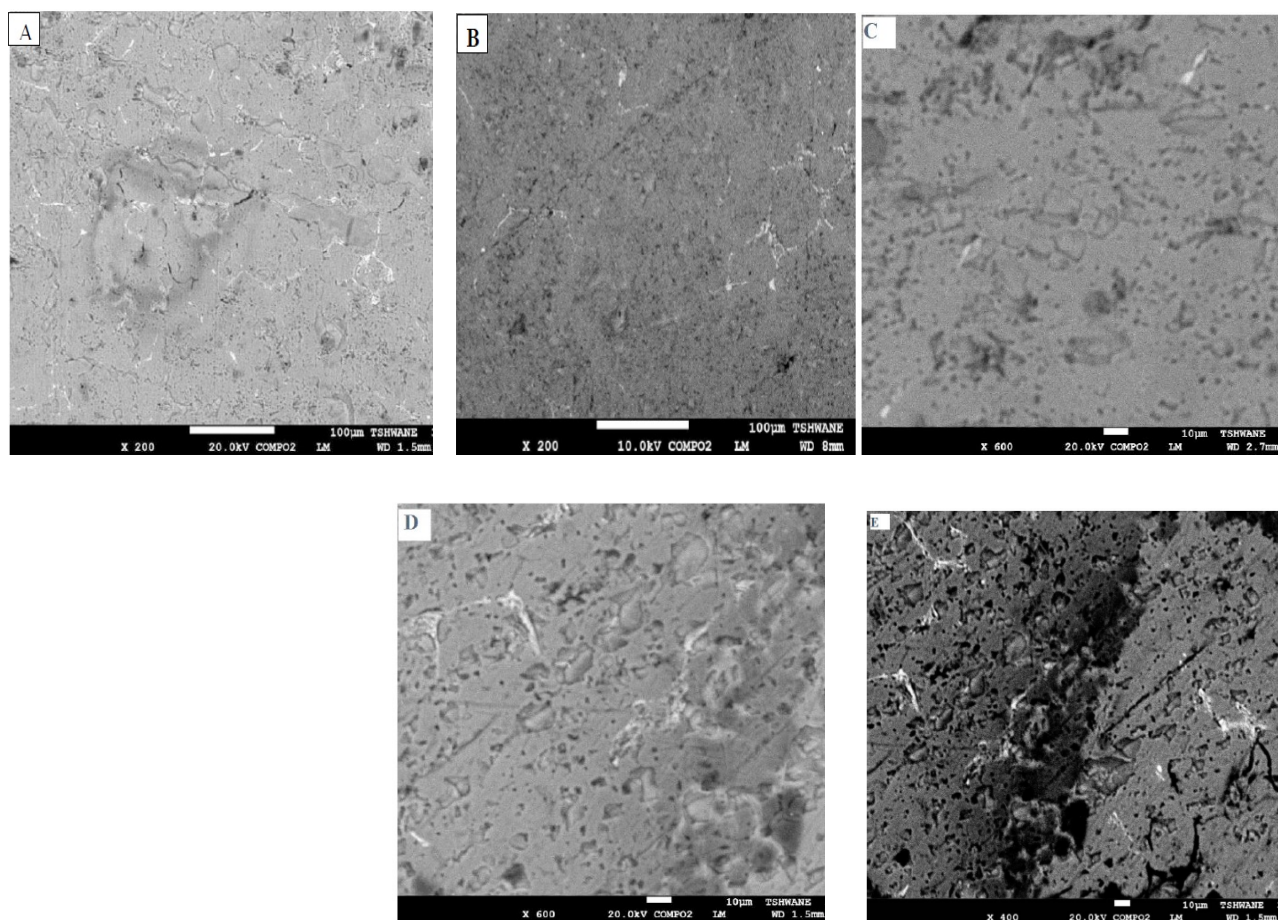


Figure 11. (A-E) a) the morphology of Al-2.5%Mg-0.25%Cr. b) Al-2.5%Mg-0.25%Cr/5 wt. % CCHA. c) Al-2.5%Mg-0.25%Cr/10 wt. % CCHA. d) Al-2.5%Mg-0.25%Cr/15 wt. % CCHA. e) Al-2.5%Mg-0.25%Cr/20 wt. % CCHA

Conclusions

From the investigation carried out on the aluminum alloy reinforced with (5–20) wt. % of CCHA, the following conclusions can be drawn:

1. The microstructural examination demonstrated that the reinforcements of CCHA were consistently dispersed throughout the new composite produced.

2. Incorporating carbonated cow hoof ash particulates enhances the mechanical properties of the composite, attributed to the hard ceramic characteristics of the agro-waste particles.
3. The reinforced composites demonstrate improved wear resistance owing to the hard and abrasive characteristics of the CCHA particles. Moreover, incorporating CCHA particulates provides a sustainable and eco-friendly alternative by transforming waste materials into useful resources, thereby reducing environmental pollution, supporting a circular economy, and lessening reliance on conventional ceramic reinforcements such as silicon carbide and aluminium oxide, which are resource-intensive.
4. The superior mechanical and wear characteristics make these composites appropriate for lightweight and moderate-stress applications, including automotive parts like brake pads and pistons, as well as in aerospace and structural contexts.
5. The lower cost of agro-waste reinforcements relative to traditional reinforcements can make these composites appealing for budget-conscious applications.

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