Textural assessment of Aluminium Matrix Composites Developed from Si-Based Refractory Compounds of Selected Agro Wastes

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Received: 17-JUNE-2024; Reviewed: 03-AUGUST-2024; Accepted: 11-AUGUST-2024
https://dx.doi.org/10.4314/fuoyejet.v9i3.15

ORIGINAL RESEARCH

Abstract— The current work reports on the textural assessment and mechanical property of aluminium matrix composites developed from Si-based refractory compounds of selected agro wastes (rice husk, bamboo leaf, and coconut shell ash). The reliance on importation of refractory reinforcements motivated the need for a cost effective option. To this end, agro wastes materials are highly sought after because of their high silica content inherent in their structure. The Si-based refractory compounds were synthesized via a carbothermal process in a modified environment. The specific surface area, total pore volume, pore sizes, isothermal adsorption, and desorption isotherms were used to determine the textural assessment. Aluminium matrix composites were later developed using 10 wt.% formulation. From the results, a mesoporous type of distribution was observed in all the samples under investigation (the pores sizes were between 2 to 50 nm). The N2 adsorption-desorption isotherm shows the type IV nomenclature. This was also validated by the hysteresis loop showing a type H4 configuration. The BET surface area was at optimum for coconut shell ash (CCT) amounting to 293.754 m^2/g, while the least surface area showed a value of 26.49 m^2/g for rice husk (A1). An increase in microhardness value was observed in sample A1 (63.12 HV0.1) and a similar trend was noticed in sample B1, though with a reduction in hardness value having 6.73 % when compared with A1. The presence of silica polytypes partly appearing as cristobalite phase (a softer materials) might have led to the reduction in hardness value, as observed in BL2. Thus impeding the hardness level, hence having the least hardness value of 47.54 HV0.1. The findings showed that variations in the hardness values is due to the different elastic behaviour of the reinforcement and the matrix.

Keywords— Pores, Mechanical property, Refractory, Agro wastes, Silicon carbide, Composites, Surface area.

1 INTRODUCTION

Over the years, agro-waste materials have been used in the development of Si-based refractory compounds (Adediran & Sriariyanum, 2022; Shcherban, 2017). Their choice as refractory materials is based on the silica content in their structure. Different processing techniques have been reported in the literature for synthesizing Si-based refractory compounds from agro wastes. Few of these are, carbothermal processing (Adediran et al., 2018; Parrillo & Sanchez, 2024; Nayak et al., 2023); sol-gel (Aprillia et al., 2023; Li et al., 2014). The choice of carbothermal synthesing has been attributed to the high distribution network of carbon to silica interphase in these agro waste materials. An attempt has been made by authors to report the textural characteristics of agro wastes (Li et al., 2014). Parameters such as pore size distribution (Shen et al., 2014); surface area (Yuvakkumar et al., 2014); adsorption and desorption isotherm (Zhai et al., 2016) are essential elements in textural assessment of agro wastes ash. These refractory compounds, can serve as reinforcement as against the conventional synthetic reinforcing materials in the development of aluminium metal matrix composites (AMMC) with application in the automobile industry (Ghasali et al., 2017).
The merit of these materials has been attributed to the relatively lower strength and wear characteristics they possess (Prasad & Shoba, 2014; Alaneme & Sanusi, 2015). Aluminium matrix composites find application in several industries, such as; aerospace, structural and automobile. Their choice in these areas is attributed to the unique properties such as; low fabrication cost, good wear resistance, high strength-weight ratio and low density (Suthar & Patel, 2018; Singh & Chanhan, 2015; Shaikh et al., 2019). An extensive utilization of textural data in AMMC development with emphasis on the microhardness characteristics appears to the sparce to the best of our knowledge. The Majority of investigations of textural assessment of agro wastes have been limited to the quantitative and qualitative evaluation of either the pore size, pore volume, surface area or the adsorption isotherms. The present work attempts to investigate the effect of textural assessment of Si-based refractory compounds on the AMMC. The morphological characteristics of the AMMC were determined using a scanning electron microscope and the microhardness of the composites developed in a 10 wt.% reinforcement of the refractory compounds computed. Textural assessment of the Si-based powder was determined for the selected agro-wastes under examination.

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Section C- MECHANICAL/MECHATRONICS ENGINEERING & RELATED SCIENCES
Can be cited as:
Adediran A. A. (2024). Textural assessment of Aluminium Matrix Composites Developed from Si-Based Refractory Compounds of Selected Agro Wastes. FUOYE Journal of Engineering and Technology (FUOYE**JET), 9(3), 470-474.**
https://dx.doi.org/10.4314/fuoyejet.v9i3.15

2. MATERIALS AND METHOD

2.1 MATERIALS

Al-Mg-Si alloy (AA 6063) with chemical composition as obtained in Table 1 was used as the matrix for the composite production. The graphite used was of particle sizes (<50 µm), while an analytical grade of SiC of particle size were selected as reinforcing materials. The rice husk was sourced locally from a rice processing plant located at Igbemo Ekiti, Ekiti State, Nigeria. Coconut shell and bamboo leaves were obtained within Landmark

University Community.

Table 1. Elemental composition of Al-Mg-Si alloy

Element	wt%
Magnesium (Mg)	0.35
Silicon (Si)	0.59
Manganese (Mn)	0.35
Copper (Cu)	0.0117
Zinc (Zn)	0.002
Titanium (Ti)	0.0566
Iron (Fe)	0.47
Nickel (Ni)	0.0347
Aluminium (Al)	balance

2.2 METHODS

A carbothermal processing method was used for the synthesis of the Si-based refractory compounds as reported by Adediran et al. (2018). The specific surface area, pore volume and pore size distribution of the Si-based refractory compounds were determined from N2 adsorption-desorption isotherms at 77 K using a Micrometrics ASAP (2460). The surface area was calculated using the Brunauer–Emmett–Teller (BET) equation; pore size distribution and pore volume were evaluated using the BJH method. Before the adsorption measurement, samples were degassed under vacuum at 100 ^{o}C for 1 h to eliminate any physisorbed moisture. The liquid metallurgy method via two step stir casting route was employed for the production of the composites. Firstly, the determination of the quantities of Si-based refractory compounds (SiRC) from the agro wastes required to produce 10 wt.% particle reinforced composites was computed. Prior to the production of the cast products, the SiRC particles were preheated in accordance with Alaneme et al (2013). Charge calculations were later done using the procedures stated by Adesina et al (2023). Fettling process was later carried out on the cast products and machined for microhardness test. The microhardness test was evaluated on the composites developed using a standard procedures. The samples were machined and polished to obtain a smooth plane surface before the test was conducted following ASTM E92-17 standard (ASTM E92, 2017). A microhardness tester FM-800 was used at 100 gf loads at 15 s dwell time an average of ten indentations were taken within the margin of ±2%. Figure 1 shows the representative micro hardness trace on the composite during testing.

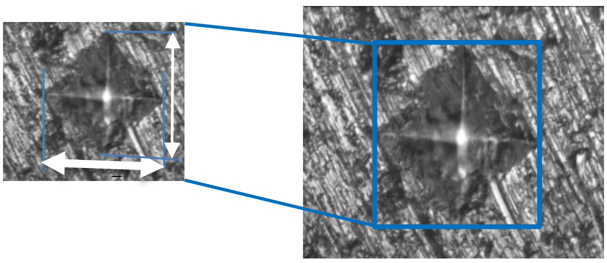

Figure 1. Representative micro-hardness of composite

The morphological features and elemental compositions of the composites were examined using a field emission scanning electron microscope (JEOL JSM-7800F) equipped with energy dispersive spectroscope (EDS). Samples were metallographically prepared and polished to mirror like surface finish. They were subsequently etched in Keller's reagent (95 ml water, 2.5 ml HNO_3, 1.5 ml HCl, 1.0 ml HF) by swabbing 10-20 s prior to microstructural examination.

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Section B- MATERIALS/METALLURGICAL ENGINEERING AND RELATED SCIENCES
Can be cited as:
A.A. Adediran (2024): Textural assessment of Aluminium Matrix Composites Developed from Si-Based Refractory Compounds of Selected Agro Wastes. FUOYE Journal of Engineering and Technology (FUOYE**JET), 9(3), 1-3.**
https://dx.doi.org/10.4314/fuoyejet.v9i3.1

3. RESULTS AND DISCUSSIONS

3.1 TEXTURAL EVALUATION

Figure 2 shows the representative variations in the adsorption-desorption isotherms. Pores size distribution of the agro wastes are presented in Figures 3 and 4 show the variation in the specific surface area against the diameter.

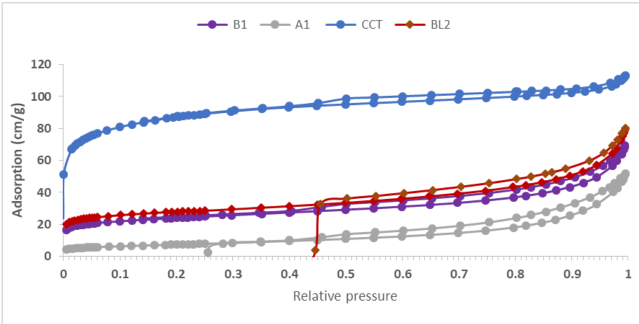

Figure 2. Variation in adsorption against relative pressure of agro wastes.

It appears there are similarities in the trend of the isotherm presented in Figure 2. This is likely due to the treatment sequence adopted in the production process for the Si-based refractory compounds. There appears to be a partial desorption of BL2 at a relative pressure of 0.45. A similar observation of partial desorption behaviour was reported by authors Zhai et al., (2016). From Figure 2, the N2 adsorption-desorption isotherm under investigation was of type IV isotherm when compared with reference Sing et al., (1985). This also validates the Si-based refractory compounds as mesoporous. The hysteresis loop of type H4 was observed for all samples as presented in Figure 2. This was in accordance with the International Union of Pure and Applied Chemistry (IUPAC) classification for adsorption isotherms.

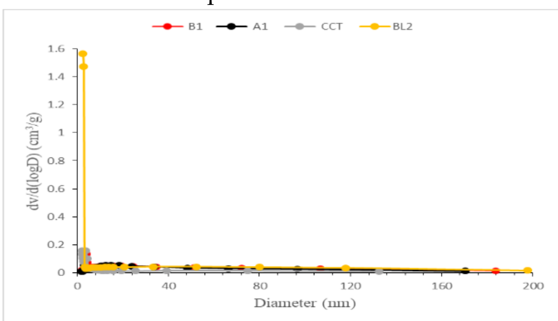

Figure 3. Pores size distribution of the agro-wastes

Figure 3 shows a mesoporous type of distribution as observed in all samples under investigation. Pores are grouped into three generic categories according to IUPAC; macropores- for pores having a width greater than 50 nm, mesopores-for pores with width sizes between 2 nm and 50 nm while micropores are for pores with a width not exceeding 2 nm (Sing et al., 1985). From the foregoing, the pores sizes of the agro wastes materials under investigation is classified as mesopores. A similar report was observed by An et al., (2019) and Soltani et al., (2015).

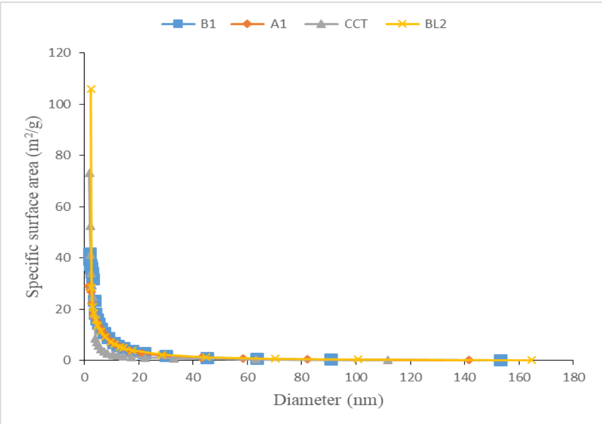

Figure 4. Variations in the specific surface area and the diameter.

Figure 4 shows the variations in pores sizes. There appear to be a peak value when the pore size was between 2-3 nm. With a decline in the pore size, the specific surface area of the pores smaller than 20 nm tend to increased significantly.

Table 2: Variation in BET surface area for different agro-wastes materials.

Samples	BET surface area (m^2/g)
BL2	94.074 ± 1.027
CCT	293.754 ± 3.274
A1	26.491 ± 0.059
B1	81.722 ± 0.652

From the results in Table 2, CCT has an optimum value of surface area amounting to 293.754 m^2/g. This shows that the silica polytypes contain a high specific surface area, detailed phases in this material have been reported elsewhere by Adediran *et al.*, (2019). Also, BL2 and B1 show a significantly high surface area, BL2 has 94.074 m^2/g, whereas B1 having 81.722 m^2/g. The least value of surface area amounting to 26.491 m^2/g was observed in A1, this was higher than the one reported previously by Li et al., (2013); Mohammed et al., (2015); Yalcin & Sevinc (2000).

3.2 MICROSTRUCTURES

The representative scanning electron microscopy image of the Si-based refractory compounds of selected agro wastes materials is as presented in Figure 5(a-d).

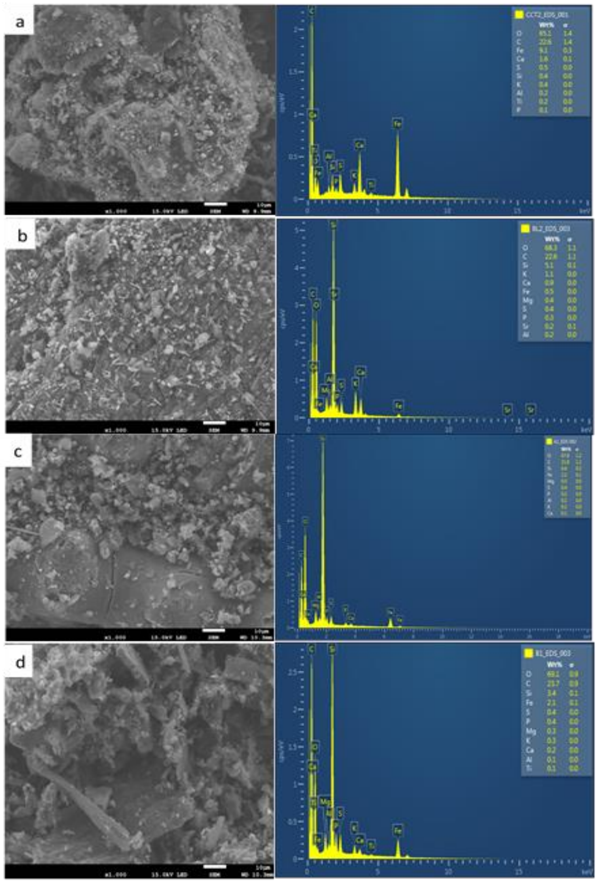

Figure 5. The morphology and the EDS spectra of powder materials used for the composite development denoted as (a) CCT (b) BL (c) A1 (d) B1.

From the Figure 5(a), it appears there are few dispersion of SiC within the structure. The EDX confirm the constituent materials present in the coconut shell (CCT). Figure (5b) present a representative morphological features of the Si-based refractory compounds from bamboo leaf (BL). The presence of residual elements as observed in the EDS spectra is evident. In Figure 5(c), we noticed a well dispersion of SiC polytypes within the network architecture of the rice husk (A1). The impurity element contained in the husk were revealed in the EDS spectra. A similar observation was noticed in Figure 5(d). However, there appears to be a clustering of phases around the structure (Omodara *et al.* 2020).

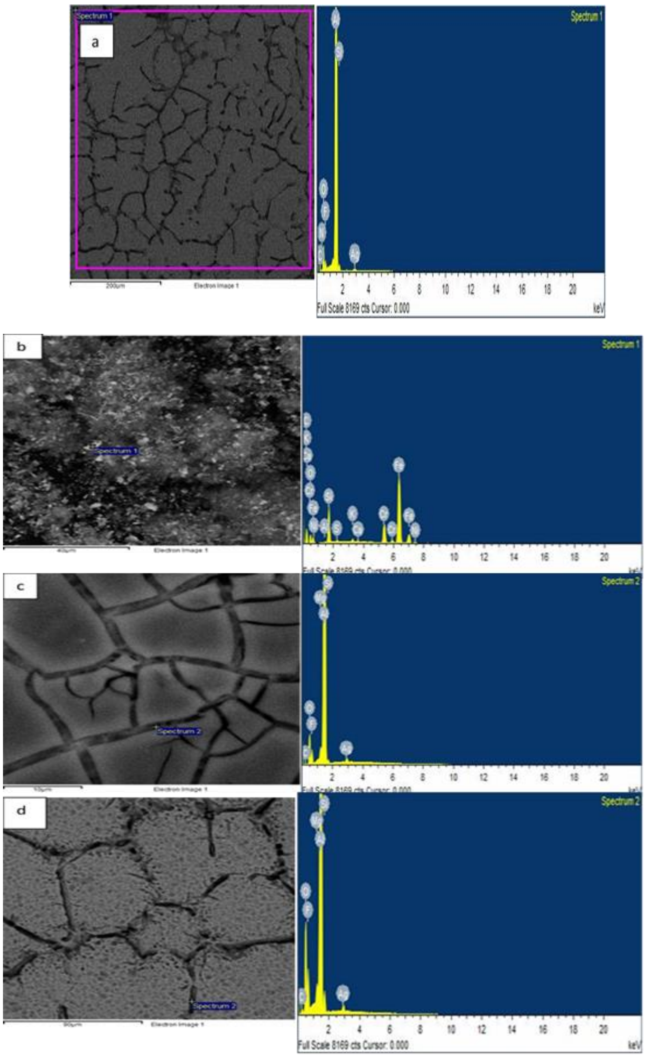

Figure 6. Showing the morphology and the EDS spectra of aluminium matrix composites developed from the Si-based refractory compounds of selected agro wastes; (a) CCT composite (b) BL composite (c) A1 composite (d) B1 composite.

The representative scanning electron image (SEI) presented in Figure 6 shows a well distribution of Si-based refractory as present in the reinforcement materials. The EDS spectra show a high peak in Al, Mg, and Si elements (Figure 6a-d). These are attributed to the matrix alloy, while the peak showing the presence of Si, C, and O respectively confirms the presence of Si-based refractory compounds in the reinforcement.

3.3 MICROHARDNESS

As presented in Figure 7, it is evident that an increase in microhardness value observed in sample A1 (63.12 $HV_{0.1}$) is likely to be attributed to the high proportion of SiC polytypes in the refractory compounds used as reinforcement for the composites (Adediran et al., 2018). A similar trend was however notice in sample B1, though with a reduction in hardness value amounting to 6.73 % when compared with A1.

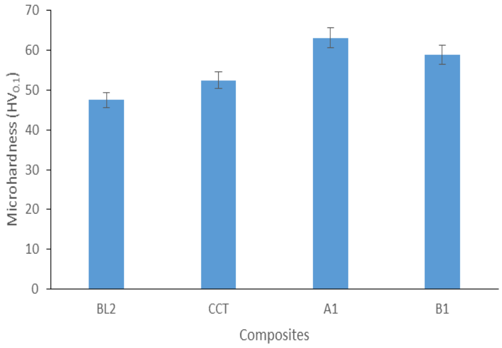

Figure 7. The variation in microhardness value of the composites developed.

The presence of silica polytypes partly appearing as cristobalite phase (a softer materials) might have led to the reduction in hardness value, as observed in BL2, thus impeding the hardness level (Adesina et al. 2023; Shaikh et al. 2019). The earlier stated reason accounts for the least hardness value of 47.54 $HV_{0.1}$. It is obvious that the variations in the hardness values is due to the different elastic behaviour of the reinforcement and the matrix.

4 CONCLUSION

The textural assessment of Aluminium Matrix Composites Developed from Si-Based Refractory Compounds of Selected Agro Wastes was investigated in the current study. From the results obtained, the following conclusions are drawn:

- ❖ a mesoporous type of distribution was observed in all the samples under investigation (the pores sizes were between 2 to 50 nm).
- ❖ the N_2 adsorption-desorption isotherm shows the type IV nomenclature. This was also validated by the hysteresis loop showing a type H4 configuration.
- ❖ the BET surface area was at optimum for coconut shell ash (CCT) amounting to 293.754 m^2/g, while the least surface area amounting to 26.49 m^2/g was observed in rice husk (A1).
- ❖ an increase in microhardness value was observed in sample A1 (63.12 $HV_{0.1}$). A similar trend was also noticed in sample B1, though with a reduction in hardness value amounting to 6.73 % when compared with A1. The presence of silica polytypes partly appearing as cristobalite phase (a softer materials) might have led to the reduction in hardness value, as observed in BL2.

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