

# Experimental Investigation of Microstructure and Phase Characterization of Rice Hush Ash (RHA) for Potential Utilization in Metal Matrix Composites

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## ABSTRACT

Recent trends in material research and development of composite materials are geared towards reducing cost, recycling and ensuring environmental sustainability. Agricultural waste derivatives have proven to be an alternative or complimentary reinforcement material in the development of metal matrix composites. The current work is an investigation of the microstructure and phase characterization of rice husk ash (RHA) obtained through open air burning. Scanning electron microscopy (SEM) equipped with electron dispersive spectrometry (EDS) was used for the microstructural examination, X – ray fluorescence (XRF) for the mineralogical composition and X – ray diffraction (XRD) for the determination of phases present. Results revealed that RHA is highly siliceous with 93.8% SiO<sub>2</sub>, highly porous and fibrous material with a wide surface area. EDS profile confirmed the presence of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>. The XRD pattern revealed the presence of quartz, cristobalite and anorthite phases in the RHA. Silica is a hard substance with strong covalent bonds similar to diamond with hardness rating of 7 on Mohs scale and melting point of 1713<sup>o</sup>C. Thus, RHA can be suitable used as a reinforcing material in the development of metal matrix composites for automotive components.

**Keywords:** Alumina, Electron dispersive spectroscopy, Scanning electron microscopy, Silica, X-Ray fluorescence.

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## 1.0 INTRODUCTION

Materials research and development have shifted from monolithic to composite materials adjusting to the need for reduced weight; improve mechanical properties, low cost and environmental friendliness of composite. So far, researched works have been carried out by incorporating hard ceramic particles such as Silica dioxide (SiO<sub>2</sub>), alumina (Al<sub>2</sub>O<sub>3</sub>), Silicon Carbide (SiC), Fly Ash (FA), graphite (Gr) and a host of synthetic materials to soft light matrix such as Aluminium, magnesium and titanium to mention a few, for the development of metal matrix composites. These composites have been discovered to be a suitable replacement to steel and cast iron in applications where the requirement for weight reduction and in pursuit of high efficiency and performance in the material is a design criterion [1].

This has been limited by high cost, and non-availability of the ceramic reinforcements especially in developing countries. Other challenges reported of interest in using ceramic particulate are inferior ductility, low fracture toughness and in ability to predict corrosion behavior [2-4]. Several approaches have been adopted to mitigate these problems. One such approach is finding alternative cheaper reinforcements which are aimed at providing solution to problems posed by high cost and limited availability of ceramic particulates. Industrial waste and agro waste derivatives are some of the alternatives reinforcing materials that have been investigated [5-8]. Results obtained from investigations carried using these alternative reinforcements have been promising as they showed significant improvement in the properties of the composite developed over the unreinforced metal or alloy. These wastes utilization would not only be economical, but may also result to foreign exchange earnings and environmental pollution control that way ensuring environmental sustainability as must of the agro waste are either burnt uncontrollably in open air which leads to generation of a high amount of CO<sub>2</sub> which is harmful [9]. Coconut shell ash, Rice husk Ash (RHA), Bamboo leaf Ash (BLA), Bagasse Ash (BA), Palm Kernel shell Ash (PKSA), Maize Stalk Ash (MSA), Corn Cob Ash (CCA) and Bean Shell Ash (BSA) are some of the agro waste derivatives that have been used as reinforcing materials in metal matrix composites [5-8]. This approach gives room for possible reduction of cost of metal matrix composites alongside with property optimization. This has put

metal matrix composites under the spotlight as many researchers forecast the huge promise of developing high performance and low cost metal matrix composite.

Another advantage is in the management of most agro waste which could be overwhelming and the best approach remains recycling. This approach provides applications where recycled waste can be productively utilized. Thus, current efforts are aimed at considering the potentials of utilizing a wide range of agro waste ashes for the development of low-cost high performance metal matrix composites: Alaneme and Adewale [2] investigated the influence of rice husk ash and silicon carbide weight ratios on the mechanical behavior behavior of Al –Mg – Si alloy matrix hybrid composites. RHA and SiC mixed in weight ratios of 0:1,1:3, 1:1, 3:1 and 1:3 were utilized to prepare 5, 7.5, and 10wt% of the reinforcement phase with Al – Mg – Si as the matrix material using two – step casting technique. The composites produced were characterized using density measurement, estimated percent porosity, tensile properties, fracture toughness and SEM examination. Results of the characterization showed that the composites were of good casting quality as the estimated porosity values were less than 2.5% in all grades of the composites produced. They observed that the tensile, yield and specific strength values decrease with increase of RHA in the RHA - SiC reinforcement. However, their results showed that composites with composition of 1:3 weight ratio offered comparable strength value with the single SiC reinforced composite. The strain to fracture values were invariant to the weight ratios of RHA/SiC for all weight percentages but the composite compositions containing RHA had improved fracture toughness compared to single reinforced composite. No microstructural examination of the RHA used undertaken to ascertain its suitability as a reinforcement. Ahamed et al.,[1] fabricated and characterized aluminium reinforced RHA composite prepared by stir casting method. Microstructure analysis revealed the reinforcements particle distribution inside the matrix which implied successful incorporation of the RHA inside the matrix. Density and mechanical properties were measured for both unreinforced metal and composites. Result showed a decrease in density with increasing reinforcement while increasing yield strength, ultimate strength and hardness of the composites with increasing reinforcement from the unreinforced condition. They concluded that RHA can be successfully incorporated into aluminium for the production of composites noting that this can solve the problem of disposal of RHA and utilization of agricultural waste. The composition and microstructure of the RHA was not analyzed. Prasad et al., [15] investigated the mechanical properties of an aluminium hybrid composite. Double stir casting process was used to fabricate aluminium composites reinforced with various volume fractions of 2, 4, 6, and 8wt% RHA and SiC particulates in equal proportions. Density, porosity and mechanical behavior of the unreinforced and reinforced hybrid composite were examined. Scanning electron microscopy (SEM) was used to study the microstructural characterization of the composites. It was observed that the hardness and porosity of the hybrid composite increased with increasing reinforcement volume fraction and density decreased with increasing reinforcement volume fraction. They observed that UTS and yield strength increased with an increase in the percent weight fraction of the reinforcement particles, whereas elongation decreased with the increase in reinforcement. The increase in strength of the hybrid composite was suggested to be probably due to the increase in dislocation density. They further undertook a systemic study of the base alloy and the composites using Brinell hardness measurement and corresponding age hardening curves were obtained. They observed that in comparison with the base alloy, the precipitation kinetics of the composites was accelerated by adding the reinforcements which effectively reduced the time for obtaining the maximum hardness by ageing heat treatment. Alaneme et al., [3] investigated the fabrication characteristic and mechanical behavior of rice husk ash and alumina reinforced Al – Mg – Si alloy hybrid composite. They concluded that RHA has great potential to serve as a complimenting reinforcement for the development of low cost high performance aluminium hybrid composites. Rice husk ash contains a quality of high surface area silica which can be purified by chemical leaching and fluidization furnace [15]. Usman et al., [17] noted that the chemical composition of RHA varies from one sample to another due to the difference in the type of paddy, crop harvest year, climate, property and constituent of the soil and geographical conditions though extensive study has shown that the major constituent of RHA is silica not withstanding variations in conditions. The current work is an investigation on the microstructure and phase analysis of RHA obtained through open air burning for utilization as reinforcement in the development of metal matrix composites.

## **2.0 MATERIALS AND METHODS**

### **2.1 Materials and equipment.**

2.5 kg of rice husk (obtained from a local rice mill in Mabudi, Langtang South local Government Area of Plateau state Nigeria), a flat metallic container, a metallic drum with perforations, an electronic balance, X – ray fluorescence (XRF) machine, Philips X – diffractometer (XRD) and a scanning electron microscopy (SEM) equipment equipped with energy dispersive spectroscopy (EDS) system

### **2.2 Methods**

The 2.5kg of rice husk was washed thoroughly with water to remove any grainy or stony matter. It was dried at room temperature for 24hours. Weight of the husk now reduced to 2.0kg. The husk was then place in a flat metallic container and then carbonized by

burning in open atmosphere. This helped in removing any moisture and, organic constituents. The color now changed from yellowish to black due to charring of organic matter. Weight of carbonized rice husk reduced to 1.2kg. Loss on ignition was computed as;

$$\text{Loss on ignition} = \frac{2.5-1.2}{2.5} \times 100\% = 52\%$$

The 1.2kg of the carbonized rice husk was then placed on a flat metallic tray for preparation of ash. A simple metallic drum with perforations to allow for air circulation to aid combustion filled with charcoal which served as fuel for the preparation of the RHA was used as the burner. The burner was ignited and carbonized rice husk was placed on it and allowed to burn completely for 24hours. The carbonaceous constituents were removed leaving the grayish white rice husk ash (RHA). Weight of RHA obtained was 450g. The ash was further conditioned by heat treating at a temperature of 650°C for 3hours to further reduce the carbonaceous and volatile constituent of the ash as performed by Alaneme et al., [3] and Saravanan and Kumar [16].

20g of the RHA sample was prepared for X – ray fluorescence (to determine the mineral content) and X – ray diffraction (to determine the crystallinity) by grinding to powder form. It was mixed with 0.4g of stearic acid as flux and binder. The prepared sample was used to produce pellets using a Herbol pelletizing machine using a 20KN force. The samples were then subjected to XRF and XRD analysis. A SEM machine equipped with EDS was used for the microstructural examination.

### 3.0. RESULTS AND DISCUSSION

#### 3.1 Chemical composition

Result of chemical composition of the RHA sample presented in table 1.0. It shows that the RHA used in this study contains SiO<sub>2</sub> (93.8%), Al<sub>2</sub>O<sub>3</sub> (1.36%), P<sub>2</sub>O<sub>5</sub> (1.53%) and MgO (1.01%). SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> are known hard substances. SiO<sub>2</sub> is a hard substance with strong covalent bonds similar to diamond, hardness of 4500HRB and melting point of 1713°C. The presence of hard elements like silica dioxide and alumina suggest that RHA can be used as a particulate reinforcement in the development of metal matrix composites.[12]. Other compounds are in trace quantities as presented in table 1.0.

**Table 1 Chemical Composition of RHA**

Oxide	SiO <sub>2</sub>	CaO	P <sub>2</sub> O	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	SO <sub>3</sub>	Cl	Mn <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	ZnO	LOI
%	93.8	0.78	0.68	0.07	1.01	1.36	1.53	0.36	0.04	0.26	0.26	0.01	5.17

LOI; Loss on ignition.

From the results, it is evident that RHA contains mainly silica. A survey of literatures [4,8,12] reveals that 79.5% – 97.6% Silica is found in RHA and 90% of this Silica is present in gel form and the remaining is in the form of metallic silicates or in fine colloidal form.

The RHA underwent 5.17% weight loss on ignition (LOI). Loss on ignition indicates that some free carbon is retained in the RHA due to insufficient combustion of the husk. According to ASTM C 311 – 77, loss on ignition should not exceed 12%. This shows that the process used for the derivation of the RHA is acceptable.

#### 3.2 Microstructural characterization

A SEM image of the RHA is presented in figure 1.0 (a). The SEM micrograph reveals the siliceous nature of the RHA which is confirmed by the presence of quartz in the XRD analysis. Close examination of the SEM micrograph also suggest that RHA is highly porous and fibrous material with high surface area and honey comb structure which is in agreement with Farooque et al. [8].



**Figure 1.0 (a). SEM micrograph of RHA.**

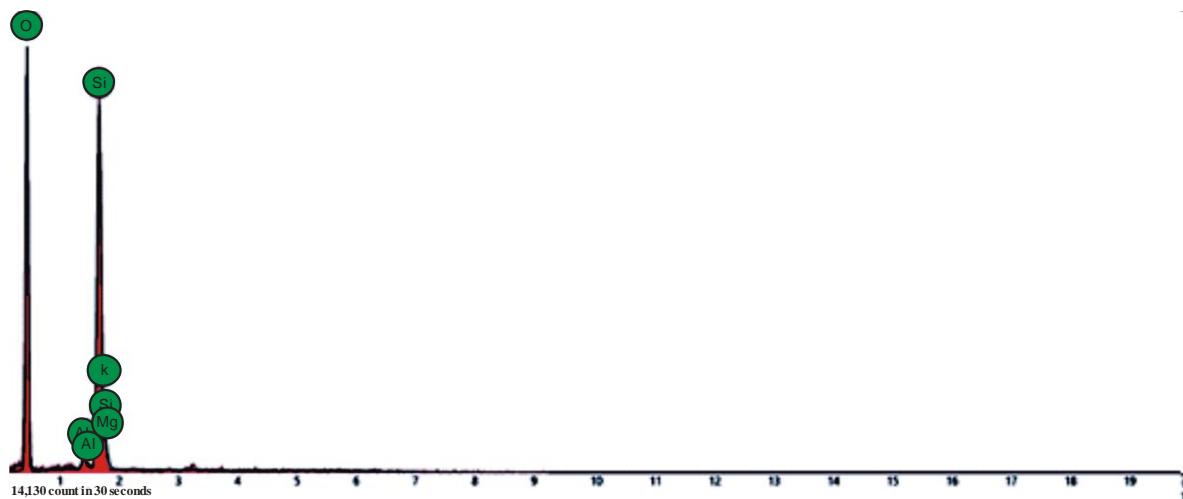


Figure 1b. EDS profile of RHA.

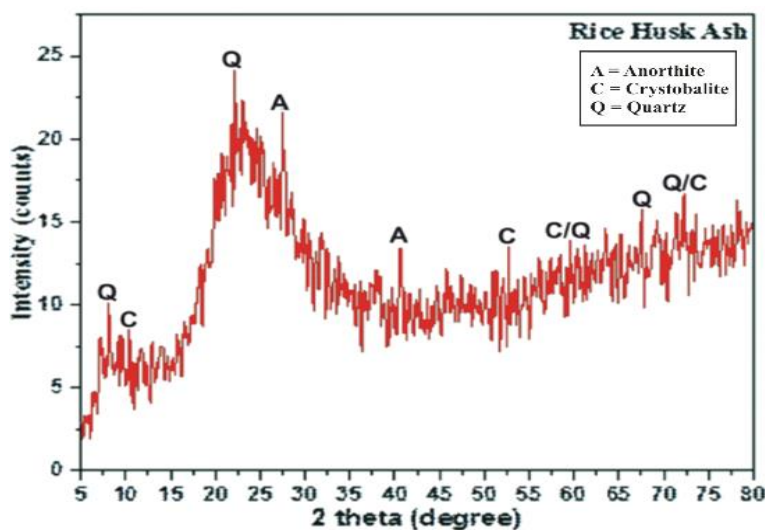


Figure 2. XRD pattern of RHA.

The EDS profile figure 1b confirms the result of chemical analysis in table 1.0. It confirms the presence of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  as major constituents of RHA.

The XRD pattern figure 1b indicates the presence of quartz ( $8.21^\circ$ ,  $21.40^\circ$ ,  $52.83^\circ$ ,  $67.50^\circ$  and  $72.50^\circ$   $2\theta$  peaks) and crystobalite ( $10.25^\circ$ ,  $52.50^\circ$ ,  $55.80^\circ$ , and  $72.50^\circ$   $2\theta$ ). Anorthite phase is also found ( $27.91^\circ$  and  $41.65^\circ$   $2\theta$ ). Farooque *et al.*, [8] reported that at low temperatures ( $600^\circ\text{C}$  -  $700^\circ\text{C}$ ) silica in RHA is amorphous and crystallization occurs when temperature goes above  $700^\circ\text{C}$ . The presence of crystallite phase indicates that RHA was produced above  $700^\circ\text{C}$ . At high temperatures quartz transforms to tridymite which is favored in presence of impurities in the RHA. [8]. Crystallinity was obtained as 56.8%.

#### 4.0 CONCLUSION

Microstructural and compositional characterization confirms that silica is the major oxide present in RHA and the two most important phases are quartz and crystobalite. Silica is a hard substance with strong covalent bonds similar to diamond with hardness rating of 7 on Mohs scale (a scale used to measure hardness of mineral relative to each other) and melting point of  $1713^\circ\text{C}$ . This shows that RHA can be suitably used as a reinforcing material in the development of metal matrix composites.

## REFERENCES

1. Ahamed, A.A., Ahmed, R., Hossain, M.B and Billah M (2016). Fabrication and characterization of aluminium- Rice husk ash composite prepared by stir castin. Rajshabi university journal of science and engineering, Vol. 449-18.
2. Alaneme, K.K and Adewale, T.M (2013). Influence of Rice Husk Ash – Silicon Carbide weight ratios on the mechanical behavior of Al – Mg – Si alloy matrix hybrid composites. Tribology in industry, Vol.35, No. 2 Pp. 163 – 172.
3. Alaneme, K.K., Akintunde, I.B., Olubambi, A.P and Adewale, M.T (2013). Fabrication characteristics and mechanical behavior of Rice Husk Ash – Alumina reinforced Al – Mg –Si alloy matrix hybrid composites. Journal of materials research and technology, 2(1): 60 – 67.
4. Alaneme, KK and Sanusi,O.K (2015). Microstructural characteristics, mechanical and wear behavior of aluminium matrix hybrid composites reinforced with alumina, rice husk ash and graphite. Engineering science and technology, An international journal. 18(2015)60 – 67.
5. Anilkumar, H.C Hebbar H.S and Ravishankar, K.S (2012). Mechanical Properties of Fly ash reinforced aluminum alloy 6061 composites. International journal of mechanical and materials Engineering. 6 (1), 41 - 45.
6. Bodurin. M.O, Alaneme, K.K. Chown, L.H (2015). Aluminum hybrid composites. A review of reinforcement philosophies, mechanical, corrosive and tribological characteristics, Journal of materials research and technology, 4(4), 434-445.
7. Das, D.K., Mishra, P.C., Singh, S and patfanaik, S. (2014): Fabrication and heat treatment of Ceramic reinforced aluminum matrix composites. A review. International journal of Mechanical and material Engineering, 1 (6), 1-15.
8. Farooque, K.N., Zaman, N., Halim,E., Islam,S., Hossain,M., Mollah, AY and MahmoodA.J (2009). Bangladesh journal of science and industrial research, 44(2). 152 – 162.
9. Hpydi, R., Adhithan, B and Bakrudeen, A. (2013): Microstructure exploration of aluminum tungsten carbide composite. International journal of soft computing and Engineering, 2(6). 210-225
10. Loh, Y.R., Sujan d., Rahman, M.E and Das, C.A (2013): Sugar cane bagasse the future composites material; A Literature review. Journal of Resources Conversation and Recycling, 2013; 75: 14 – 22.
11. Macke, A. Schultz, B.F, Rohatgi, P. (2012): Metal Matrix Composites: Offer to the automotive Industry an Opportunity to reduce vehicle weight and improve performance. Journal of Advance materials, 170(3), 19-23.
12. Madakson, P.B., Yawas D. S., Apasi A. (2012): Characterization of coconut shell ash for potential utilization in refal matrix composites for automotive applications. International journal of engineering Science and Technology, 3(4), 1190 – 1198.
13. Mavhungu, S.T., Akinlabi, E.T., Onitiri, M.A and Varachia, F.M (2017). Aluminium matrix composites for industrial use: Advances and trends. Procedia Manufacturing 7(2017). 178 – 182.
14. Prasad, S.D and Krishna, R.A (2011): production and mechanical properties of A356.2/RHA Composite. International Journal and Advanced Science and Technology. 33, 51 – 58.
15. Prasad, D.V., Shoba, C. and Ramanaiah,N (2014). Investigation on mechanical properties of aluminium hybrid composites (2014),. Journal of materials research and technology, 3(1); 79 - 85.
16. Saravanan, S.D., and Kumar,M.S (2013). Effect of mechanical properties on rice husk ash reinforced aluminium alloy (AlSi10Mg) matrix composites. Procedia Engineering 64(2013). 1505 – 1513.
17. Usman, A.M., Raji, A and Waziri,N.H (2014). Characterisation of Girei rice husk ash for silica potential. IORS journal of environmental science, toxicology and food technology, 8(1). 68 – 71.