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Investigation and microstructural characterization of stir cast Rice husk ash (RHA) and Alumina reinforced Aluminium – Silicon hybrid composite.

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Abstract
Metal matrix composites combine the desirable properties of the constituent materials to enhance physical, thermal and mechanical properties and thus find application in almost all industries. In the current work, an Al – Si alloy was reinforced with 5.0 wt % RHA an agricultural waste derivative and 5.0wt % Al_2O_3 a ceramic to develop a hybrid composite. RHA was used in attempt to improve mechanical properties, reduce cost and enhance environmental sustainability. Cast composite samples were subjected to T6 heat treatment which involved solution heat treatment at $530^{O}C$, quenching in water at room temperature and ageing at $175^{O}C$ for 5hours. Scanning electron microscopy (SEM), Electron dispersion spectroscopy (EDS) and X – Ray diffraction (XRD) were used to study and characterize the micro structure of the developed composites. Heat treatment lead to improvement of mechanical properties and homogeneity in the distribution of the reinforcements in the matrix material. Result of chemical analysis showed that the main constituent of RHA was SiO_2 (93.8%) a hard substance with covalent bonds similar to those of diamond, hardness of 4500HRB and melting point of $1713^{O}C$ showing that RHA has similar properties to ceramic reinforcement materials. Microstructure of the composites showed a coarse α – Al dendritic structure, needle – like primary and acicular Si eutectic precipitates and intermetallics of Fe, Mg and fine particles of the reinforcements at the inter dendritic regions. Presence of the reinforcements was confirmed by the EDS profile. RHA can thus be used as a complimentary reinforcement alongside ceramics in the development of hybrid metal matrix composites.

[Keywords: Physical properties, Thermal properties, Mechanical properties, Metal matrix composites, Scanning electron microscopy, Electron dispersion microscopy, X- ray diffraction]

I. Introduction.

Metal matrix composites have been found to be the current solution to material property requirement such as low weight and density, high performance especially in the transportation industry (Macke et al. 2012). Weight carries a fuel penalty, modest for automobile, greater for trucks and trains, greater still for aircraft and enormous for space vehicles. Increased fuel combustion implies increased emission of CO_2 to the atmosphere consequently harm to the environment. (Ashata et al. 2016). Also, with the emergence of electric and hybrid vehicles the need to reduce weight has become tremendously necessary considering available power for propulsion.

So far, researched works have been carried out by incorporating hard ceramic particles such as Silica dioxide (SiO_2), Alumina (Al_2O_3), 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. (Alaneme and Bodunrin 2011; Rahmohan et al. 2013; Ramnath et al. 2014).This has been limited by high cost, and non-availability of the ceramic and industrial waste material 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 (Hpydi et al, 2013; Macke et al, 2012; Das et al, 2014).

Several approaches have been adopted to resolve 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 agricultural waste derivatives are some of the alternative reinforcing materials that have been investigated. (Bodurin et al, 2015; Madakson et al, 2012; Anilkumar, et al, 2012; Loh et al, 2013).

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. However, the poses inferior properties when compared to the aluminum matrix composites reinforcement with ceramic particulates. (Bodurin et al, 2015).

Another approach involved the development of aluminum matrix composite using two or more reinforcing materials known as Hybrid composites. 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 agricultural waste derivatives that have been used as complimenting reinforcing materials (Bodurin et al, 2015; Madakson et al, 2012; Anilkumar, et al, 2012). This approach gives room for possible reduction of cost of metal matrix composites alongside property optimization. This has put hybrid reinforced aluminium 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 agricultural 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 agricultural waste ashes for the development of low-cost high performance aluminium based hybrid composites.

Madakson et al,. (2012) identified the characteristics of coconut shell ash using spectroscopic and microscopic analysis. Density, particle size, refractoriness, SEM, XRD, XRF and FTIR methods were used for the characterization of the coconut shell ash. Results were compared and it was observed that the ash possessed similar properties with ceramic reinforcement materials.

Singh and Chauhan (2016) investigated the feasibility and viability of developing low cost - high performance hybrid composites for automotive and aerospace applications. The fabrication characteristics and mechanical behaviour of hybrid metal matrix composites fabricated through stir casting were reviewed. The optical micrographs indicated that the reinforcing particles were fairly distributed in the matrix alloy and the porosity levels were found to be acceptable. The density, hardness tensile behaviour and fracture toughness of these composites were found to either be comparable or superior to the the ceramic reinforced composites. They observed from literature that the direct strengthening of composites occurs due to the presence of hard ceramic phase while the indirect strengthening arises from the thermal mismatch between the matrix alloy and reinforcing phase during solidification.. They further concluded that hybrid composites offer more flexibility and reliability in the design of possible components depending upon the reinforcements combination and composition.

Ahamed et al., (2016) fabricated aluminium matrix composites reinforced with Rice Husk Ash particles. RHA particulates were incorporated into the aluminium matrix by melt stirring technique. 1wt.% magnesium was used as a wetting agent between the matrix and the reinforcement. 3, 6 and 9wt.% of RHA were added into the matrix. The microstructural analysis revealed the reinforcing particles were uniformly distributed inside the matrix indicating a successful fabrication of the composites.

Alaneme and Sanusi (2015) investigated the microstructural characteristics, mechanical and wear bebaviour of aluminium matrix hybrid composites reinforced with alumina rice husk ash and graphite. Alumina, rice husk ash and graphite were utilized to prepare 10wt.% hybrid Al - Mg - Si alloy based composite using two step stir casting technique. Result of microstructural analysis on tensile test samples showed that the tensile fracture morphology in all the composites produced were identical and characterized with the presence of the reinforcing particles housed in ductile dimples.

The current work is aimed at investigating the microstructure of an Al - Si hyper eutectic alloy reinforced with RHA and Al_2O_3 fabricated using two step stirring technique in other to ascertain the suitability of the two step stir casting technique and viability of using RHA as a complimentary reinforcement in the development of metal matrix composites

II. Methodology.

i. Preparation of Rice Husk Ash (RHA). 2.5 kg 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 2.0 kg. 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 1.2 kg. Loss on ignition was computed as:

$$\text{Loss on ignition} = \frac{2.5-1.2}{2.5} x\ 100\% = 52\% \qquad \ldots(6)$$

The 1.2 kg of the carbonized rice husk was then placed on a flat metallic container for preparation of the ash. A simple metallic drum with perforation to allow for air circulation to aid combustion filled with charcoal, which served as fuel source was used as the burner for the preparation of the RHA. The burner was ignited and carbonized rice husk was place on it and allowed to burn completely for 24 hours. The carbonaceous constituents were removed leaving the grayish white rice husk ash (RHA) to be used as the reinforcement as performed by Saravanan and Kumar (2013). Weight of RHA obtained was 450 g.

ii. Chemical composition of RHA

The chemical characterization of the RHA was determined using classical methods and instrumental techniques using PFP7 JENWAY (UK) flame photometer and UV-2201 SHI-MADZU (Japan) UV spectrometer as performed by Farooque *et al.* (2009).

iii. Composite fabrication

Two-step stir casting technique was used for the fabrication of the hybrid composite as performed by Alaneme and Aluko (2012) and Alaneme *et al.* (2013). The quantities of aluminum alloy, alumina ($A1_2O_3$), RHA and 0.l wt.% magnesium were evaluated using charge calculations.

The Al_2O_3 and RHA particles were initially preheated to a temperature of 250^0C for 5minutes to help improve wettability with the Aluminum alloy melt. Equally, the crucible pot was preheated to red-hot to ensure that the crucible pot was free from moisture and any volatile matter that could lead to contamination as reported by Alaneme and Aluko (2012).

The aluminum alloy ingots were charged into a charcoal fired crucible furnace and heated to a temperature of 750 ± 30^0C (above the liquidus temperature of the alloy) which is the casting temperature of aluminium. At this temperature, the slag was scooped and removed from the surface of the molten alloy. The alloy was then allowed to cool in the furnace to a temperature of 600^0C as reported by Alaneme and Aluko (2012).

The preheated Al_2O_3, RHA and 0.1 wt. % magnesium were then added at this temperature and stirring of the slurry was performed manually for 5 to 10 minutes. The composite slurry was then superheated to 720^0C and a second stirring operation was performed using a mechanical stirrer at a speed of 200r.p.m for 10minutes to help improve distribution of the alumina and RHA particles. The molten composite was then cast into prepared sand mould and allowed to cool. After cooling, hybrid composite samples were then obtained. Temperature measurements were performed using a hand-held double laser infrared thermometer model 201506011240.

iv. Heat treatment

The developed hybrid composite samples were then subjected to T6 heat treatment condition. This involved solution heat treatment at 530^0C for 1hour, quenching in water at room temperature and age hardening at 175^0C for 4 hours as performed by Sridlar, Bhat and Mehesh (2014). This was performed using an electric resistance furnace model 5 - x - 12.

v. Micro structural analysis of cast composites.

To study the microstructures of the composites, test samples were cut from the castings. They were belt grinded with abrasive papers of grades 200, 400, 600, 800 and 1000 respectively as performed by Sharma et al., (2015). They were then polished with emery cloth , washed and dried after which they were etched with Keller's reagent (95ml water, 2.5ml HNO_3, 1.5ml HCL, 1.0ml HF) by swabbing 10 - 20 seconds before microstructural examination.

The microstructural and morphological analysis of the composites were carried out using Joel JSM 5900LV scanning electron microscope (SEM) equipment equipped with an Oxford INC^{TM} energy dispersive spectroscopy (EDS) system. The SEM equipment is an instrument that produces a largely magnified image by using electrons instead of light to form an image. A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope which is held within a gun. SEM relies on the detection of high energy electrons emitted from the surface of a sample after being exposed to a highly focused beam of electrons from the electron gun. This beam of electrons is focused on a small spot on the sample using the SEM objective lens.

Elemental analysis of surfaces in SEM was performed using Energy Dispersive Spectroscopy (EDS) which measured the energy and intensity distribution of X – ray signals generated by the electron beam striking the surface of the sample.

As a general rule, elements with atomic number below 11(Na) were not realistically detected by using SEM/EDS (The atomic number is the number of protons in the nucleus of atom, and the number of protons define the identity of an element). Also, elements such as carbon, oxygen, nitrogen and Sulphur were detected for their presence, however they were not be quantified.

Gold coating was applied to the samples to avoid charge effects so that clear images could be obtained. The sample to be tested was placed on the sample holder and images were captured under various magnifications. The SEM was operated at an accelerating voltage of 5KV to 20KV (Aigbodion et.al, 2010).

III. Results And Discussion.

i. Chemical composition of Rice husk ash (RHA).

Table 1.0 Chemical composition of RHA

Oxide	SiO_2	CaO	K_2O	Na_2O	MgO	Al_2O_3	P_2O_5	SO_3	Cl	TiO_2	Cr_2O_3	MnO_3	Fe_2O_3	ZnO
%	93.79	0.78	0.68	0.07	1.01	1.36	1.53	0.36	0.04	0.03	0.09	0.26	0.01	0.01

Result of chemical composition of the RHA sample presented in Table 1.0 shows that the RHA used in this study contains SiO_2 (93.8%), Al_2O_3 (1.36%), P_20_5 (1.53%) and MgO (1.01%). SiO_2 and Al_2O_3 are known hard substances. SiO_2 is a hard substance with strong covalent bonds similar to diamond, hardness of 4500HRB and melting point of 1713^0C. 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 (Madakson *et al.*, 2012). Other compounds are in trace quantities as presented.

From the results, it is evident that RHA contains mainly silica. A survey of literature (Farooque *et al.*, 2011; Alaneme and Sanusi, 2015; Prasad and Krishna) 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. 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 1713^0C. This shows that RHA can be suitably used as a reinforcing material in the development of metal matrix composites (Usman *et al*., 2014; Mangai *et al*., 2023).

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 ASTMC 311 – 77, loss on ignition should not exceed 12%. This shows that the process used for the derivation of the RHA is acceptable.

ii. Chemical composition of the matrix material.

Table 2.0. Chemical composition of Al - Si alloy

Element	Na	Mg	Si	P	Cl	K	Ca	Ti	Cr	Mn	Fe	Zn	S	Al
%	0.98	0.97	18.79	0.02	0.49	0.03	0.14	0.05	0.02	0.14	1.36	0.78	0.02	Rest

Table 2.0 is the result of chemical characterization of the matrix material. Al in the secondary (recycled) alloy material is 76.42% while Si is 18.79%, Fe is 1.36% others are in trace quantities. Thus the alloy is a hyper eutectic Al - Si alloy. Hyper eutectic alloys (alloys with greater than 12.6% Si, the eutectic composition) contain primary Si particles that provide improved mechanical properties. According to Asensio – Lozano and Voort (2015) elements such as Si are Fe are common impurities found in aluminium alloys but silicon is a deliberate alloying addition in both cast and wrought Al alloys. Cast Al alloys can contain Si in amounts from 5 – 22%. At this level, Si improves the fluidity and cast ability of aluminium.

iii. Micro-structure of composite.

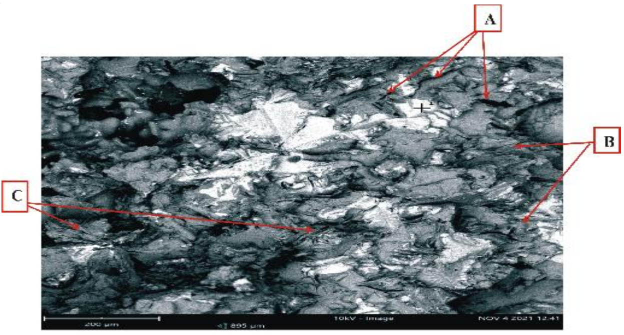

Figure 1.0. SEM micrograph cast Al - Si reinforced RHA/ Al_2O_3 composite

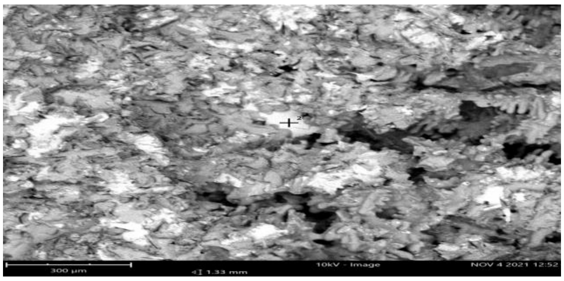

Figure 2.0. SEM Micrograph heat treated Al - Si reinforced RHA/Al_2O_3 composite

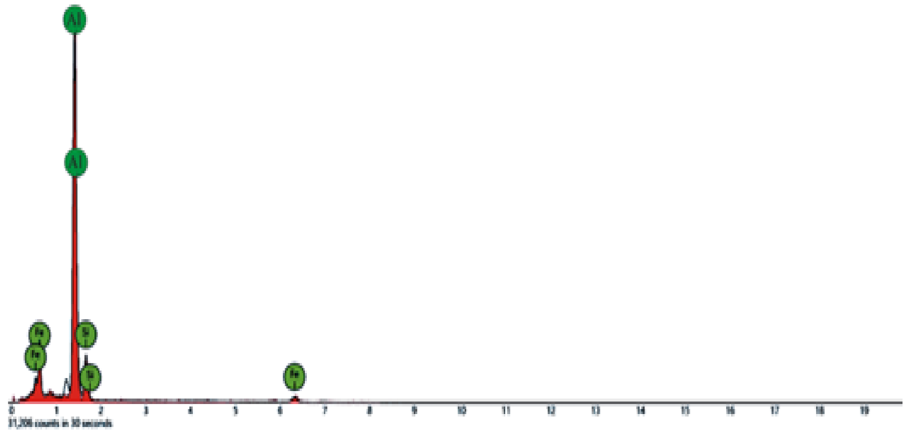

Figure 3.0. EDS Profile of composite.

Figure 1.0 shows the representative SEM micrograph of the cast Al - Si reinforced RHA and Al_2O_3 composite. The SEM analysis of the cast composite showed a needle like primary and acicular hyper-eutectic Si at the interdendritic regions (A), coarse primary α - Al dendrites (B), acicular eutectic Si phase and stacked Fe rich γ phase having a skeletal shape (C). The rice husk ash and alumina were uniformly distributed due to their fine particle sizes. The α - Al dendrites pushed these reinforcements into the interdendritic regions. Even distribution of particles with little segregations and clusters suggested to be the rice husk ash and alumina reinforcements which were also observed along the the dendritic boundaries due to high cooling rate as suggested by Xin Li et al, (2019) which often occurs during solidification of metal matrix composites having components with different densities and wettability characteristics.

Figure 2.0 shows representative SEM micrograph of the heat treated Al - Si reinforced RHA and Al_2O_3 composite. From the micrograph, it was observed that the microstructure of the composite showed more dispersion of the reinforcements in the matrix. Heat treatment process lead to dissolution of the eutectic needle like Si into spherical precipitates . The dendritic structure decomposed into equiaxed grain sizes as ageing time increased. (Viswanatha et al., 2021). From the micrograph, there is little or no needle like morphology instead ageing at elevated temperature changed the morphology of Si,Fe and α - Al into near sphericity, scattered and distributed respectively. During ageing, RHA and Al_2O_3 showed more dispersion through out the matrix due to their fine particle sizes. Reinforcement addition and dispersion increases the rate of diffusion of solid solution elements and reduce the nucleation activation energy of the precipitated phase. It stabilizes the cellular structure, which accelerates ageing reaction and increases the rate of work hardening. (Viswanatha el al.,2021).The EDS profile figure 3.0 confirms the presence of the Al_2O_3, SiO_2, Fe_2O_3 and CaO.

IV. Conclusion.

This work is a contribution to the development of metal matrix composites as a replacement of monolithic materials such as non - ferrous metals, iron and steel in the fabrication of components for the transportation industry . To meet requirement for reduced weight, enhanced properties, reduced cost and enhanced environmental sustainability a secondary (recycled) Al alloy was reinforced with 5.0wt Al_2O_3, and 5.0wt% RHA. In order to enhance uniformity and homogeneity in the microstructure of the composite two - step stir casting technique was used to fabricate the hybrid composite. Samples were subject to T6 heat treatment condition which involved solution treatment at 530^{O}C for 2hours, quenching in water at room temperature and ageing at 175^{O}C for 4hours.

Microstructural examination of the developed composite showed dispersion of the reinforcements into the interdendritic regions of the α – Al dendritic structure signifying that the two step stir casting technique adopted was effective in obtaining homogeneity in the microstructure. Heat treatment process dissolved the eutectic needle – like Si precipitates andthe α – Al dendritic structure decomposed into equiaxed grain size.

More agricultural waste derivatives can be investigated and further research should be directed towards optimizing the production process in order to determine the optimum processing parameters. This will form a basis for developing metal matrix composites in developing countries like Nigeria leading to industrialization of such countries considering the vast amount of agricultural waste generated annually.Further work on thermal behavior of aluminium alloy reinforced agricultural waste derivatives should be undertaken as most of these metal matrix composites are used in applications where high temperatures are attained.

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