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ORIGINAL RESEARCH ARTICLE

CHARACTERIZATION AND EXPERIMENTAL ANALYSIS OF AA7075 ALUMINIUM ALLOY AND RICE HUSK ASH REINFORCED HYBRID COMPOSITE

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ARTICLE INFORMATION ABSTRACT

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Keywords:

Energy Dispersive Spectroscopy Microstructural behaviour Stir casting Aluminium matrix hybrid composites Rice husk ash Mechanical properties The microstructural behavior along with the mechanical properties of Aluminium (AA7075) matrix hybrid composites reinforced with rice husk ash (RHA) and graphite were investigated. The RHA and graphite mixed in varied weight ratios were utilized to prepare samples of hybrid reinforced AA7075/RHA/Graphite alloy-based composite using the stir casting route. Impact, hardness and tensile tests were conducted to ascertain the mechanical properties of the developed composites, while scanning electron microscopy was used to characterize the composites produced. The results show a general decrease in hardness and tensile strength with increase in the weight ratio of RHA and graphite in the composites. The impact strength on the other hand increased from a 2.6 J value to 9.37 J (with the former being the base metal and the latter having 12 % RHA and 3 % graphite). Energy Dispersive Spectroscopy (EDS) profile displays peaks for silicon (Si), carbon (C), iron (Fe), aluminium (AI), and oxygen (O). The elements' presence verifies that graphite (C) and RHA were both utilized in the composite's production. The EDS results are consistent with the microscopy images and confirm the reinforcements' existence and dispersion inside the matrix.

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I.0 Introduction

The search for novel and sustainable solutions is more important than ever in the field of materials science and engineering. Scientists and engineers are focusing on developing advanced materials that can provide both high-performance qualities and environmental sustainability in response to the increasing demands on structural materials and the pressing need to lessen the environmental impact of industrial processes (Akadiri *et al.*, 2012). Because of their remarkable mechanical qualities and adaptability, aluminium alloys have continuously been the most popular materials among a wide range of options. Nonetheless, the search for even greater performance improvement and the use of sustainable materials has prompted research into innovative composites (Batz *et al.*, 2023). The choice of AA7075 aluminium alloy as a primary component in this hybrid composite is not arbitrary. AA7075 is renowned for its exceptional strength-to-weight ratio, corrosion resistance, and outstanding performance in demanding applications, particularly in the aerospace and automotive industries (Khalid *et al.*, 2023; Vikas *et al.*, 2021; Li *et al.*, 2008).

However, the energy-intensive industrial procedures associated with its application frequently result in significant carbon emissions. Therefore, there is a strong need to improve its qualities while also lowering its carbon footprint (Ali *et al.*, 2020; Reinsch and Benson, 2022; Adediran *et al.*, 2021). The incorporation of RHA, a waste product generated during rice processing, not only contributes to the sustainable utilization of agricultural by-products but also offers the potential to improve the overall mechanical and thermal properties of the composite.

For the fabrication of metal matrix composites (MMCs), aluminium-based matrices are also recognized to be the least expensive of the common metallic matrix materials (copper, titanium, and magnesium) (Sharma *et al.*, 2020). MMCs are essentially metallic alloys reinforced with ceramic materials, primarily silicon carbide (SiC), alumina (Al_2O_3), boron carbide (B_4C), tungsten carbide (WC), graphite (Gr), carbon nanotubes (CNT), and silica (SiO₂) (Selvam *et al.*, 2020; Nturanabo *et al.*, 2018). However, compared to other synthetic reinforcing particulates, silicon carbide and alumina are used more frequently. Similar to single reinforced aluminium matrix composites (AMCs), hybrid reinforced AMCs are typically made using either the liquid approach or the solid method (Garg *et al.*, 2019). Powder metallurgy techniques are used in the solid

approach, whereas stir casting, compo-casting, and squeeze casting are used in the liquid method (Ashebir *et al.*, 2022). Due to its simplicity, adaptability, and suitability for mass manufacturing, the liquid technique of processing is commonly preferred. One of the liquid techniques that is widely utilized to make AMCs is stir casting (Upadhyay and Saxena, 2021; Sahu and Sahu, 2017; Ramamoorthi *et al.*, 2021). The ceramic particles are slowly added to the molten aluminium after the entire aluminium matrix has been melted. A motorized stirrer is frequently used to create a vortex to speed up integration (Muhammad and Jalal, 2023).

This study is carried out to develop a novel hybrid composite along with investigation of its mechanical and microstructural properties, each aspect is scrutinized with precision. The ultimate goal is to provide an understanding of the hybrid composite's performance characteristics, elucidating the synergistic effects arising from the combination of AA7075 and RHA/graphite (reinforcements).

In addition, the environmental effects of using RHA as a reinforcing agent are examined, taking into account things like lower energy usage, less greenhouse gas emissions, and less waste production. An important step toward the creation of cutting-edge materials that are both environmentally friendly and technologically advanced is the examination of the characterization and experimental analysis of the hybrid composite reinforced with RHA/ graphite and aluminium alloy AA7075.

2. Materials and Methods

2.1 Materials

As the specimens must be cut to specific standard shapes, marking tools like scriber and steel rule were employed. Hacksaw and files were used for machining to bring out the necessary shape required. Principally three major machines were used to carry out the tests; they are underlisted.

- I. Electronic Universal Testing Machine
- 2. Charpy Impact Testing Machine
- 3. Indentec Universal Hardness Testing Machine

The hybrid MMC consists of matrix and reinforcement material. The AA 7075 is used as matrix material. The rice husk ash (RHA) and graphite are used as reinforcement material in this study.

2.1.1 Aluminium Alloy 7075

Aluminium alloy 7075 selected as the base metal for the composite fabricated and analysed in this study. Frequently denoted as AA7075, this high-strength aluminium alloy is renowned for having an exceptional strength-to-weight ratio. It is a member of the 7xxx family of aluminium alloys, which are mostly utilized in high-strength applications, especially in the aerospace and other sectors where lightweight materials with remarkable strength qualities are crucial (Sathishkumar, 2017; Narayana *et al.*, 2021). The sample required for work was procured from Obafemi Awolowo University IIe-Ife, Nigeria in extruded form. Chemical analysis of the samples was carried out at the Raw Materials Research and Development Council (RMRDC) Abuja to ascertain its purity.

2.1.2 Rice Husk Ash

Rice husk is the first reinforcement selected for the hybrid material fabricated. It is one of the most abundant agricultural byproducts in the world, as rice is a staple food for a large portion of the global population. Rice husk is abundant, and it is estimated that for every kilogram of rice produced, about 20% of the weight is rice husk. This makes it a readily available and cost-effective agricultural byproduct. The rice husk used for the study was procured from *Kpebegi* market local mills, located in Bida, Niger State, Nigeria. The rice husk was prepared and carbonised to produce the rice husk ash used.

2.1.3 Graphite

Graphite is a naturally occurring crystalline form of carbon. Because of its distinct structure and qualities, graphite is valuable in a variety of industrial applications (Narayana *et al.*, 2021). Graphite is resistant to most acids and bases, has a high melting point of around 3,550 °C, and is chemically stable. While natural graphite is abundant, synthetic graphite can be produced from carbon-containing precursors like petroleum coke or coal tar pitch (Narayana *et al.*, 2021). Synthetic graphite was obtained from Ahmadu Bello University for this study.

2.2 Methods

2.2.1 **Preparation of Rice Husk Ash**

The rice husk ash (RHA) was cleaned with water to remove dust and impurities and then left to dehydrate at room temperature for one day to eliminate moisture (Saiful *et al.*, 2022; Kaleli *et al.*, 2020). Following this, the dehydrated husks were heated at 200°C for one hour to remove any residual moisture (Gani *et al.*, 2023; Yiga *et al.*, 2023). The dried husks were then subjected to a high-temperature treatment at 600°C for 12 hours in a a Carbolite Sheffield muffle furnace, aiming to burn off organic matter and activate the silica within. This prolonged heat treatment promotes phase transformations, altering the crystalline structure of the ash to enhance its physical and chemical properties (Otaru *et al.*, 2013; Shengtai *et al.*, 2022). Finally, the carburized sample was analyzed using an EDXRF Analyzer at the Raw Materials Research and Development Council (RMRDC), Abuja.

2.2.2 Fabrication Method for Composite

The aluminium 7075 hybrid composite material was processed through stir casting process. RHA reinforcement was added in 3, 6, 9 and 12 wt.% respectively while Graphite was fixed at 3 wt.% as shown in table 1. The average particle size of RHA was 0.191 microns and Graphite was 1.04 microns. At a temperature of around 750 °C Aluminium 7075 alloy ingot was melted in a mild steel crucible. Succeeding to melting, degassing was carried by adding hexachloroethane tablets. The mixture was stirred by using zirconium coated mild steel stirrer at about 400 rpm for 15 min. Simultaneously during the time of stirring Graphite and RHA were added which were earlier pre-heated at 250 °C. Finally, the molten hybrid composite material was poured into preheated metal mould and allowed to solidify. Once solidified Aluminium 7075 with various weight percentage of reinforcement hybrid composites were ready to prepare test specimens. Test specimens were machined in accordance to ASTM standards.

| Samples | Composition | | |
|---------|-------------------------------|----------|---------|
| | Aluminium Matrix (AA7075) (%) | Graphite | RHA (%) |
| А | 100 | 0 | 0 |
| В | 94 | 3 | 3 |
| С | 91 | 3 | 6 |
| D | 88 | 3 | 9 |
| Е | 85 | 3 | 12 |

2.2.3 Impact Test

Charpy impact testing is a widely used method to evaluate the toughness or impact resistance of materials, including aluminium composites. The procedure involves striking a notched specimen with a pendulum and measuring the energy absorbed by the material during fracture (Ikumapayi *et al.*, 2021). The prepared samples A, B, C, D and E in three sets were machined from the aluminium composite material fabricated. The specimens had the specific size and shape according to relevant standards (ASTM E23). A V-notch was machined into the centre of the specimen. The size and depth of the notch are also defined by the testing standards (Figure 1).



Figure 1: ASTM E23 proportions of Charpy V-notch specimen (Source: Badiger et al., 2017)

The test was carried out at the Department of Metallurgical and Materials Engineering, Ahmadu Bello University, Zaria using Cat.Nr.412 Charpy Impact Testing Machine of 15 J-25J capacity. Three tests were conducted for each sample to ensure consistency and reliability of the results.

2.2.4 Hardness Test

The hardness test was conducted using an Indentec Universal Hardness Testing Machine (model 81875 LKV Model B, serial no. 053158) to assess the material's hardness. This method can be applied to various hardness scales, such as Rockwell, Brinell, and Vickers. In this study, the test took place at the Department of Metallurgical and Materials Engineering, Ahmadu Bello University, Zaria. The setup of the experiment is shown in Plate 1.



Plate I: Experimental setup for the Hardness test using the universal testing machine.

The hardness test was conducted according to ASTM E18 standards. The specimen surface was cleaned, flattened, and aligned perpendicularly to the indenter. A 1/16" (1.588 mm) hardened steel ball indenter was used for the test. The Universal Hardness Testing Machine was stabilized, and a pre-load of 10 kgf (98.1 N) followed by a main load of 100 kgf (980.7 N) was applied. After a 5-second dwell time, the main load was removed while maintaining the pre-load, allowing for depth-sensitive measurement (Wang, 2012; Zubairu et al., 2020). The Rockwell hardness (HRB) value was determined based on the depth of indenter penetration, calculated using the machine's measuring system and displayed on a computer screen. The Rockwell hardness (HRB) was determined using Equation I

$$HRB = 100 - \frac{h}{0.002} \tag{1}$$

Where, h is the depth of the indentation in mm. The hardness value was recorded in HRB, along with any relevant information such as the applied load and dwell time.

2.2.5 Tensile Test

The Electronic Universal Testing Machine, Model; WDW-1000KN was used for the tensile study and the test was carried out at Department of Metallurgical and Materials Engineering, Ahmadu Bello University,

Zaria. Plate 2 shows the setup of the experiment. The procedure for conducting a tensile test using a Universal Testing Machine (UTM) involves a series of steps to determine the mechanical properties of a material.



Plate 2: Tensile Universal Testing Machine setup

A representative specimen according to ASTM E8 for metallic materials selected. The specimen dimensions, particularly gauge length and cross-sectional area was ensured to comply with the specified standard as shown in Figure 2.



Figure 2: ASTM E8 standard tensile test specimen (Source: Badiger et al., 2017)

The specimen was mounted securely in the grips of the UTM and the grips aligned with the longitudinal axis of the specimen. The UTM was calibrated according to the manufacturer's guidelines and relevant standards while the crosshead speed was set as per the testing standard. Plate 3 shows the failed specimens tested.

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а

b

Plate 3: a. Tensile test samples b. Collated failed samples Grouped according to RHA percentages

3 Results and Discussion

3.1 Chemical composition of AA7075 and RHA

The Energy-dispersive X-ray fluorescence (EDXRF) is a non-destructive, rapid, multielement, highly accurate, and environment friendly analysis compared with other elemental detection methods. The EDXRF results for the AA 7075 aluminium sample is given in Table 2.

| Element | Concentration (%) | |
|---------|-------------------|--|
| Fe | 0.27 | |
| Si | 1.02 | |
| AI | 90.4 | |
| Mg | 7.47 | |
| P | 0.01 | |
| S | 0.04 | |
| Ti | 0.006 | |
| Mn | 0.30 | |
| Cu | 4.30 | |
| Zn | 7.24 | |
| Cr | 0.004 | |
| Ni | 0.034 | |
| Others | Balance | |

 Table 2: Chemical Composition of AA 7075 Aluminium Sample

The EDXRF results for the carburized RHA are presented in Table 3.

The elemental analysis was accessed by X-ray fluorescence to determine the inorganic chemical composition of the samples (AA7075 and RHA samples), as shown in Tables 2 and 3. From these results, Table 2 shows that the Zn % is 7.24 which is well within the range for AA7075 (Ouler *et al.*, 2019; Khalil *et al.*, 2019). Silica content in the RHA sample along with ferric oxide, Aluminium oxide and other compounds were detected. In Table 3, the silica content was detected to be 92.92 % which is considerable and consistent with literature (Joharudin *et al.*, 2020; Yekinni *et al.*, 2020). This outcome can be attributed to the calcination temperature of the rice husk. Burning the rice husk at a higher temperature gave room for the loss of impurities in the sample, which maximized the silica content in the sample.

| Element | Concentration (%) |
|--------------------------------|----------------------|
| SiO ₂ | 92.92 |
| Fe ₂ O ₃ | 0.26 |
| Al_2O_3 | 1.18 |
| MgO | 0.30 |
| SO ₃ | 0.17 |
| CaO | 0.72 |
| K ₂ 0 | 1.52 |
| PbO | 0.01 |
| Others | Balance |

Table 3: Chemical Composition of RHA

3.2 Impact Test

Impact energy is a measure of energy or work done to fracture a specimen. Impact energy increased as percent weight fraction of reinforcements increase except in sample D (9:3 of RHA to graphite) which reduced as seen in Figure 3. Maximum impact strength of 9.37 J was obtained at 12 wt.% RHA and 3 wt.% graphite corresponding to 360 % increase when compared with pure aluminium with impact strength of 2.6 J. This result mimics what was reported by Yekinni *et al.* (2019). The increase in impact strength was attributed more to the influence of hard RHA.



Figure 3: Variation of the impact tests

3.3 Hardness Test

In Figure 4 the base metal, sample A has the highest Rockwell HRB hardness value at 27.43, indicating that it is the hardest among the samples. Sample B has an intermediate hardness value at 10.23. Samples D and E have relatively lower hardness values at 8.07 and 8.97, respectively. Sample C falls between the higher and lower values when Figure 12 is studied closely. The significant difference in hardness values point to the variations in the composition and/or manufacturing process of the aluminium composite samples.



Figure 4: Variation of the hardness tests

Even with the variations in the results of the hardness (Figure 12) there is clearly a reduction in the hardness of the composites compared with the base metal (A) which is 27.43 (HRB). Sample C with ratio of RHA to Graphite as 6:3 respectively had 26.03 (HRB). The reduction in hardness is as a result of the presence of silica polytypes partly appearing as cristobalite phase (softer material) resulted in the reduction in hardness value. Rice Husk Ash containing mainly SiO_2 which is a softer ceramic compared to alumina, will result in composites having lower hardness value. The reduction in hardness of Aluminium composites has been reported by Yekinni *et al.* (2019), Adediran *et al.* (2021), Alaneme and Sanusi (2015) and Saini *et al.* (2020), even though contrary behaviour is also available. Alaneme and Sanusi (2015) further explained the reduction in hardness with increase in RHA and graphite content is as a result of the lower hardness possessed by both reinforcing materials

3.4 Tensile Test

The tensile strength and yield strength values (Figure 5) are also observed to follow to some degree the same trend as the hardness results (Wholly). It is observed that the yield strength, tensile strength and break strength decreases with increase in the RHA content (from A - E sample series). This is consistent with reported literature (Yekinni *et al.*, 2019; Adediran *et al.*, 2021; Alaneme and Sanusi, 2015; Saini *et al.*, 2020). Also, there is a slight decrease in tensile values of C sample (9:3 of RHA to graphite) in comparison to the remaining samples that had averagely similar values. However, there is a general decrease in tensile test values compared with the base metal. The presence of a greater amount of graphite and RHA reduces potential direct strengthening effect from load transfer from the matrix to the reinforcing particles since Al matrix and the dominant RHA particles have the same elastic modulus of 70 GPa while graphite has a lower elastic modulus value of approximately 27 GPa (Alaneme and Sanusi, 2015).



Figure 5: Variation of the tensile tests

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3.5 SEM Results

Representative SEM micrographs and EDS analysis of composites produced are presented in Figure 6. It is observed from the Figure 6 (B, C, D and E) that the reinforcing articles are visible and fairly well distributed in the alloy matrix. This suggests that the stir casting method used to produce the composite is dependable. The hybrid composite's elemental mappings' EDS profile displays peaks for silicon (Si), carbon (C), iron (Fe), aluminium (AI), and oxygen (O). The elements' presence verifies that graphite (C), aluminium, alumina, silica (SiO₂), ferric oxide (Fe₂O₃), and both were utilized in the composite's production. Alaneme and Sanusi (2015) have reported similar elemental compositions and microstructural characteristics. An EDS (Energy Dispersive Spectroscopy) study was performed to confirm the reinforcements' existence and dispersion inside the matrix. The EDS results (Figure 6 (B, C, D and E)) are consistent with the microscopy images.

The presence of 2.02% titanium (Ti) is only noticed in the EDS results of sample E as seen in Figure 6(E). In the AA7075 composite it can significantly influence its impact strength through various mechanisms. Ogunmefun *et al.* (2023) mentioned that titanium promotes the formation of fine and uniform grain structures within the composite matrix, enhancing its mechanical properties, including impact resistance. Moreover, during processing, titanium can precipitate or form intermetallic phases, which function as powerful strengthening agents to prevent deformation and fracture under impact loading (Ogunmefun *et al.*, 2023). By experiencing plastic deformation or microstructural changes upon impact, phases containing titanium also contribute to toughening mechanisms by efficiently absorbing energy and limiting the spread of cracks (Jiang *et al.*, 2020).



Figure 6: EDS profile and SEM photomicrograph of the base sample (A) and hybrid composites B, C, D and E

The method by which titanium is processed to ensure appropriate dispersion and distribution of titanium particles throughout the matrix has an impact on the mechanical properties of the composite. Furthermore, titanium's interaction with copper, magnesium, and zinc, among other alloying elements, fortifies the composite's resistance to impact damage.

(Jiang et al., 2020). Overall, the incorporation of titanium in the AA7075 composite holds promise for improving its impact strength, making it suitable for demanding applications requiring high-performance materials. The results obtained from the impact strength test shows great improvement in the impact strength of E (Graphite 3: RHA 12).

Apart from the presence of silica polytypes partially appearing as cristobalite phase (softer material), samples B, D, and E exhibit the greatest drop in hardness, correspondingly. Additional explanations are given from the SEM study. Changes in the microstructure of the composite, such as grain size, distribution of phases, and presence of defects, can influence hardness. Variations in the samples' microstructure caused by processing could be a factor in their varying hardness. Inclusions, contaminants, or processing-related flaws can serve as stress concentrators in the composite, lowering its hardness. Reduced hardness may also be caused by the occurrence of porosity or the production of unwanted phases. Sample B and E show some defects with some voids present in both samples which may have contributed to the reduced hardness of the samples. Sample D on the other hand reveals the availability of graphite in clusters that lead to hardness reduction.

4. Conclusion

In this study, the properties of a developed hybrid composite have been investigated. The findings show that the mechanical characteristics of the hybrid composite were considerably impacted by the addition of RHA/Graphite to the aluminium alloy AA7075. The findings also show that the material qualities may be tailored to certain applications by adjusting the RHA content, which balances the properties of toughness, hardness, and tensile strength. The tests show that as RHA increases the hardness and tensile characteristics of the composite decreases.

Sample E was found to be with highest impact energy of 9.37 J, this suggests that it could be particularly promising for applications requiring high toughness. Notably, the microstructural study verified that the RHA reinforcements were appropriately distributed throughout the AA7075 matrix, guaranteeing even dispersion and strong bonding. This distribution highlighted the potential of this hybrid composite for applications needing customized toughness, hardness, and tensile strength and contributed to the observed improvements in mechanical properties. The study's overall findings show that adding RHA to the aluminium alloy AA7075 can greatly improve its qualities, opening the door for the creation of high-performing, environmentally friendly composite materials.

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