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Special Issue
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This themed issue honors Professor Ekedimogu Eugene Nnuka, celebrating his distinguished career upon his retirement. His legacy of exemplary scholarship, mentorship, and commitment to advancing knowledge is commemorated in this collection of works.

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Investigating the mechanical behavior and microstructural properties of sustainable aluminum matrix composites reinforced with biodegradable coconut shell ash nanoparticles

Fakorede D. O.[1], Ajayi S. A.[2], Abioye A. G.[3], Adefajo A. A.[3], Oyewo A. T. [3]*
[1] Department of Mechanical Engineering, University of Cross Rivers State, Calabar, Nigeria.
[2] Department of Mechatronics Engineering, Bowen University, Iwo, Osun State, Nigeria.
[3] Department of Mechanical Engineering, Osun State University, Osogbo, Osun State, Nigeria
* Corresponding: abideen.oyewo@uniosun.edu.ng

Abstract
Aluminum's natural weaknesses, such as low strength, can be overcome by adding Coconut Shell Ash (CSA) particles, which enhance the composite's strength and toughness. CSA is a sustainable, eco-friendly, and locally available material that was studied for its potential to reinforce aluminum in automotive interior applications. The research aimed to determine the mechanical and microstructural properties of CSA-reinforced aluminum composites. The study used coconut shells, aluminum waste, and a mold to create composites with varying CSA content (0-16%). Thus, the mechanical properties increased with increasing CSA content. Notably, the results showed that the Al+12% CSA composite exhibited superior tensile strength (254 MPa), while Al+16% CSA impact strength (13.6 J) and hardness (67 BHN) were superior compared to the control and other composite samples. However, the Al+12% CSA composite displayed a uniform CSA distribution with minimal void, agglomeration and defects. Overall, the Al+12% CSA composite demonstrated significant improvements in mechanical properties, making it a promising material for high strength applications purposes.

Keywords: Aluminium matrix composite, mechanical properties, microstructural characteristics, coconut shell ash.

1. Introduction

A metal matrix composite (MMC) is a cutting-edge material that combines two or more reinforced materials with a metal matrix to enhance its properties. Composites have recently garnered significant attention as a highly promising material. The primary goal of designing metal matrix composites is to synergistically combine the desirable attributes of metals and ceramics, leveraging their unique strengths. By incorporating high-strength, high-modulus refractory particles into a ductile metal matrix, a material with mechanical properties that fall between the matrix alloy and ceramic reinforcement is created. Metals possess a valuable combination of properties, including high strength, ductility, and high-temperature resistance, although some may exhibit low stiffness. Ceramics, on the other hand, are stiff and strong but brittle. A striking example is the contrast between aluminum and silicon carbide, which exhibit vastly different mechanical properties, including Young's modulus, thermal expansion coefficients, and yield strength. By meticulously controlling the composite's ingredients, distribution, and processing conditions, these properties can be further optimized and enhanced (Dhanesh et al., 2021; Dubey & Gangwar, 2021).

Aluminium metal matrix composites (AMMCs) represent a cutting-edge class of materials that synergistically combine aluminium as the primary material (matrix) with various reinforcing elements. These reinforcements can encompass a range of materials, including silicon carbide, graphite, fly ash, alumina particles, red mud, cow dung, and rice husk, among others. Interestingly, aluminium and its alloys often exhibit a columnar solidification structure, characterized by coarse grain sizes, which can lead to compromised surface finish and mechanical properties.

Nevertheless, the strategic incorporation of copper (Cu) into commercially pure aluminium has been shown to markedly improve the material's microstructure, microhardness, grain refinement, impact resistance, strain tolerance, and overall mechanical performance (Dubey & Gangwar, 2021).

The rapid expansion of the global population and the enhancement of living standards, driven by technological advancements, have led to a significant increase in waste generation from agricultural activities. The disposal of these waste materials poses a substantial challenge, contributing to environmental pollution. However, harnessing these waste materials can mitigate contamination and reduce the need for disposal spaces. Consequently, converting waste into eco-friendly materials for applications in the automobile and construction sectors has become a pressing research focus. Various agricultural waste products, such as rice husk ash, coconut shell ash, sorghum husk ash, palm oil fuel ash, palm oil clinker, coconut husk, and sugarcane bagasse ash, hold immense potential for utilization in these industries. While extensive research has been conducted, ongoing developments aim to fully leverage waste materials as partial reinforcements in composite materials. The resulting eco-friendly, energy-efficient, and cost-effective materials derived from agricultural waste are expected to exhibit significant market potential, meeting the needs of both rural and urban populations (Ikubanni et al., 2021; Oyewo et al., 2022). The aluminium matrix composites (AMCs) have properties like light density, high stiffness, good strength, wear resistance, variable load resistance and good stability at a high temperature. Due to all these properties, these composites can be used for the design of different components for advanced applications (Chak et al., 2020).

Exploring biodegradable materials have extensive usage as reinforcement in metal matrix composites for applications such as shoe sole manufacturing and high strength applications (Kubendiran et al., 2024; Kumar et al., 2023; Kumar et al., 2024; Okafor, Okpe, et al., 2022; Okafor, Onovo, et al., 2022). Therefore, the advancements in technology and the exponential growth of the global population have led to an enhancement in living standards, resulting in a corresponding increase in waste generation from agricultural activities. However, the disposal of these waste materials poses a significant challenge, as they are often non-biodegradable and take an extended period to decompose, thereby contributing to environmental pollution. Harnessing these waste materials in any form can mitigate contamination and create alternative disposal solutions. In light of this, recycling waste materials into eco-friendly products for application in the automobile and construction sectors has become a pressing research priority. To address this, the current study focuses on investigating the mechanical and microstructural properties of aluminum reinforced with 0 (control), 4, 8, and 16 wt.% Coconut Shell Ash (CSA) composite samples, with the aim of converting waste into a valuable resource.

2. Materials and Methodology

2.1. Preparation of CSA and melting of aluminium scraps

A coconut shell was subjected to high-temperature processing in a furnace for 100 minutes, yielding a powdery ash that was then collected on a plate. Further refinement involved heating the powder in a specialized graphite crucible at an extremely high temperature of 900°C, resulting in the production of CSA powder. To achieve a precise fineness of 75 µm, the powder was then sifted through a carefully arranged sieve system for a duration of 2 hours. The scraps were heated to a temperature of 600°C, creating a molten compound from which impurities such as paint and additives were removed. The resulting molten aluminum was then poured into a pit, allowing it to solidify into a single ingot.

2.2. Aluminum metal matrix composite formation

The manufacturing process for Al-CSA-MMCs employed the stir casting method, which required preheating the aluminum matrix to a semi-solid state. A mechanically driven agitator was used continuously for 1 hour with a temperature of 500-600°C to ensure a homogeneous dispersion and less agglomeration of the reinforcing particles throughout the molten metal. Subsequently, the mixture was heated beyond its liquid temperature and poured into a handcrafted sand mold measuring 270 x 32.24 mm, created using refined clay soil. The first stage entailed allowing the molten aluminum alloy to cool and solidify within the mold. Then, a 4wt% CSA was added to the molten compound and stirred it together as done by Chak et al. (2020) to mix together and evenly circulate with this molten compound. The same process is repeated for 8, 12 and 12 wt.%. CSA reinforcement, respectively. Each sample was replicated three times and thee their standard deviations were recorded.

2.3. Tests on Aluminium matrix composite

After casting, tensile strength, impact strength, hardness test and the microstructure examination were conducted for the specimens produced.

2.3.1. Brinell Hardness Test

A sample was procured and cut to a specific length, followed by a surface hardening process involving manual filing to create visible file marks. The specimen then underwent successive stages of grinding and polishing using a grinding machine to achieve a high-quality surface finish. The prepared sample was then mounted in an extensometer and subjected to a brief compression test, applying a load of 250 kg for approximately 15 seconds. The resulting indentation was then measured using an eyescope, enabling the determination of the material's Brinell hardness number via a conversion table.

2.3.2. Tensile test

A comprehensive tensile testing protocol was implemented, wherein four separate samples were subjected to evaluation using Universal Testing Machines (UTM). The testing procedure strictly adhered to the guidelines set forth in the ASTM E8 standard. For each sample, a set of three identical test specimens was tested at room temperature, with a controlled strain/loading rate of 5 mm/min, utilizing a state-of-the-art, computerized Instron Testing Machine (model 3369). The results were meticulously recorded and subsequently used to determine critical mechanical properties (ultimate tensile strength).

2.3.3. Impact test

A 75 x 4 x 10 mm was produced for each sample and subjected to impact energy testing using a precision Izod impact tester, calibrated to ensure accuracy and precision. ASTM 3763 was used. Each specimen was then exposed to a high-velocity, double-sided impact event at a speed of 3.8 meters per second, and the resulting energy absorption was accurately measured and recorded from the testing apparatus.

2.4. Microstructure analysis

The internal structure and distribution of CSA powder within the metal matrix were scrutinized using SEM analysis. To achieve optimal surface quality and reveal intricate microstructural features, the composite specimens underwent a gradual polishing regimen employing a series of emery papers with progressively finer grit sizes (ranging from 180 to 2000) followed by a buffing step. This thorough surface preparation enabled a detailed examination of the specimen's microstructure using SEM technology.

3. Results and Discussion

3.1. Hardness Test Result

Figure 1 shows the result of Brinell Hardness Test done on the Aluminum at different CSA constituents. The base metal sample, without reinforcement, displayed a hardness value of 50 BHN, marking the lowest reading observed throughout the entire experiment. Introducing 4 wt% CSA into the aluminum alloy led to a slight yet notable enhancement in hardness, reaching 53 BHN, which became the baseline hardness value for the CSA-reinforced composite series. As the CSA content increased incrementally, a steady rise in hardness ensued, peaking at 68 BHN with the addition of 16 wt% CSA, and exhibiting intermediate values of 56 BHN and 59 BHN corresponding to 8 wt% and 12 wt% CSA additions, respectively.

In essence, the maximum hardness was attained by combining aluminum with 16wt% CSA, revealing a straightforward correlation between CSA content and hardness, which increased incrementally up to 16wt% CSA. This observation is attributed to the optimal SiO_2-to-Al_20 stoichiometry ratio, as earlier established by Dhanesh et al. (2021). The hardness values showed a remarkable rise from 50 BHN to 65 BHN with the introduction of 16wt% CSA, which can be ascribed to the increasing presence of hard and brittle CSA particles within the aluminum matrix. The inherent hardness of CSA particles stems from their chemical composition, comprising SiO_2, $NaAlSi_3O_2$, $CaCO_3$, and Al_4O_4C. Moreover, the incorporation of CSA into aluminum enhances dislocation density at particle-matrix interfaces due to thermal expansion coefficient disparities, leading to elastic and plastic incompatibility (Dhanesh et al., 2021). This pattern of hardness enhancement with reinforcement addition aligns with the findings of by Ranjan et al. (2022); Singh and Chauhan (2019).. The improved hardness is influenced by factors such as robust interfacial bonding, uniform dispersion, and strong filler-matrix interactions, indicating a direct proportionality between hardness and reinforcement loading, as previously reported by (Singh & Chauhan, 2019).

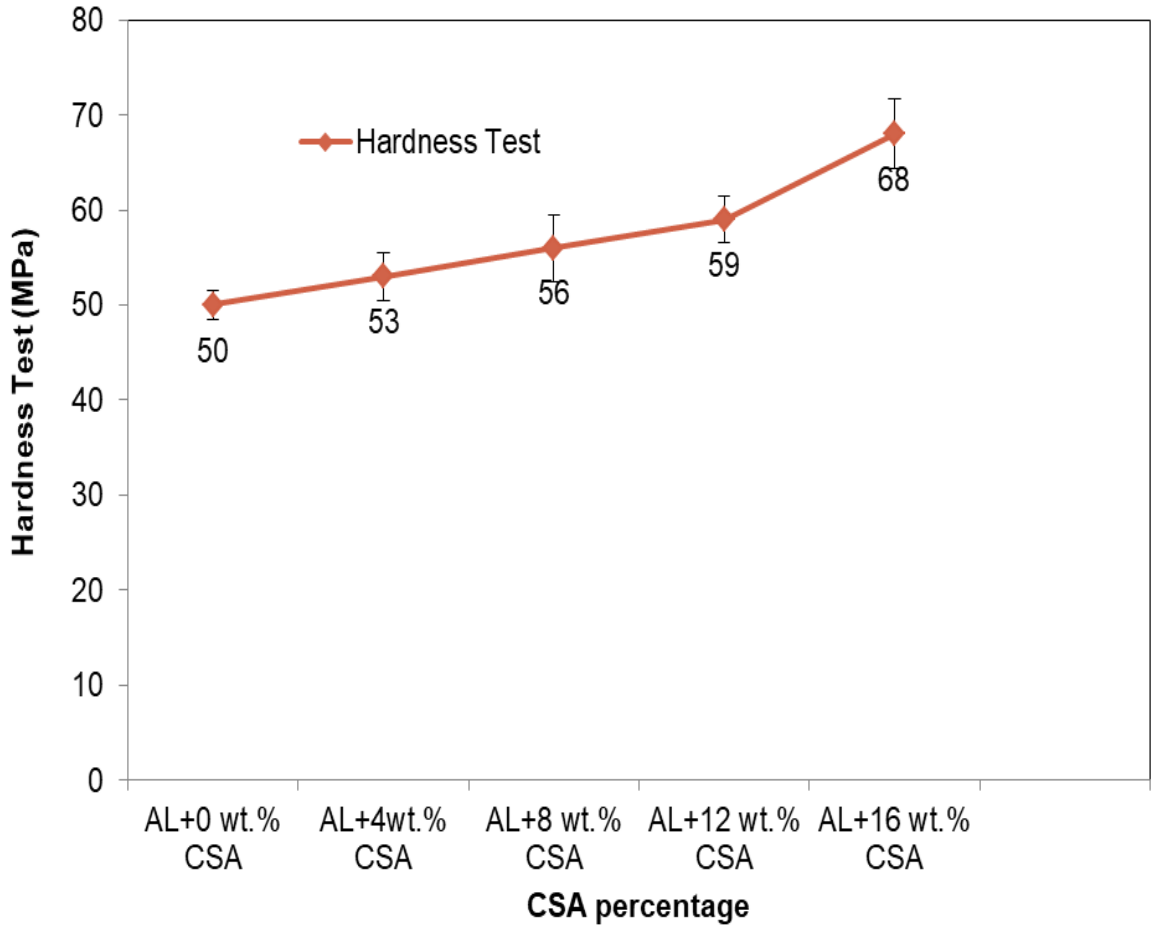

Figure 1: Effect of CSA on Hardness of aluminum composites

3.2. Tensile strength

The Ultimate Tensile Strength (UTS) results presented in Figure 2 show a significant enhancement in the composite's tensile properties across all CSA mass fractions, outperforming the unreinforced aluminum alloy matrix. The yield and tensile strength exhibit a similar trend, aligning with the findings of Vimalanathan et al. (2021) display a steady increase with escalating CSA content in the matrix. The highest tensile strength value was achieved with the addition of 12wt% CSA. This notable improvement in tensile properties can be attributed to a combination of factors, including strong interfacial bonding between particles and matrix, excellent wettability, the small size of reinforcement particles, and the reinforcing effect of CSA particles within the aluminum matrix, as previously reported by (Singh & Chauhan, 2019).

The CSA material acted as a reinforcement by bearing the bulk of the load through its crystalline fibrils, which caused the helically wound fibrils to extend in tandem with the matrix. Although the tensile properties of the composite increased in proportion to the fiber loading, a notable exception occurred at 8 wt.% CSA, where a decline in properties was observed. This irregularity can be attributed to defects introduced during fabrication, likely resulting from human error, which led to flaws, crazing, and stress concentration areas that reduced the composite's stiffness, as previously reported by Ranjan et al. (2022). Additionally, when the CSA loading exceeded 12 wt.% (as in the case of the 16 wt.% CSA composite), the resulting composite sample exhibited poor formation, non-homogeneity, hardness, and brittleness. This is due to the fact that high fiber volume fractions surpass the practical reinforcement limit, leading to an insufficient matrix material to effectively support the fibers, resulting in a compromised composite structure (Dhanesh et al., 2021; Dubey & Gangwar, 2021; Zuo et al., 2021).

The composites' tensile properties displayed a steady enhancement with increasing fiber content, but a notable deviation occurred at 16 wt.% CSA loading, where the values decreased compared to the 12 wt.% CSA loading. This slight reduction in tensile properties at 16 wt.% CSA suggests that the sample became overly rigid and lost its ability to withstand additional stress at the same strain level. The CSA material served as a reinforcing component by absorbing the primary share of the load through its crystalline fibrils, causing the helically wound fibrils to extend in conjunction with the matrix, thereby contributing to the enhancement of the composite's tensile properties (Bachchan et al., 2021; Gupta et al., 2021; Yigrem et al., 2022). Poornesh et al. (2017) compares. The mechanical properties of coconut shell ash powder (CSA) and coconut shell powder (CSP) aluminum composites. CSA composites display superior hardness (67-85 BHN) and tensile strength (250 MPa) compared to CSP composites, which show lower hardness (50-75 HRB) and tensile strength (180-200 MPa). This was due to due to the ceramic nature of the CSA, whereas CSP was softer, more porous powder. The maximum tensile strength (244 MPa) and hardness (67 BHN) of this study was correlated with the study of Poornesh et al. (2017) at 250 MPa and 67-85 BHN, respectively. Overall, CSA composites demonstrate improved mechanical properties, making them suitable for high-performance applications, while CSP composites may be more suitable for general-purpose uses.

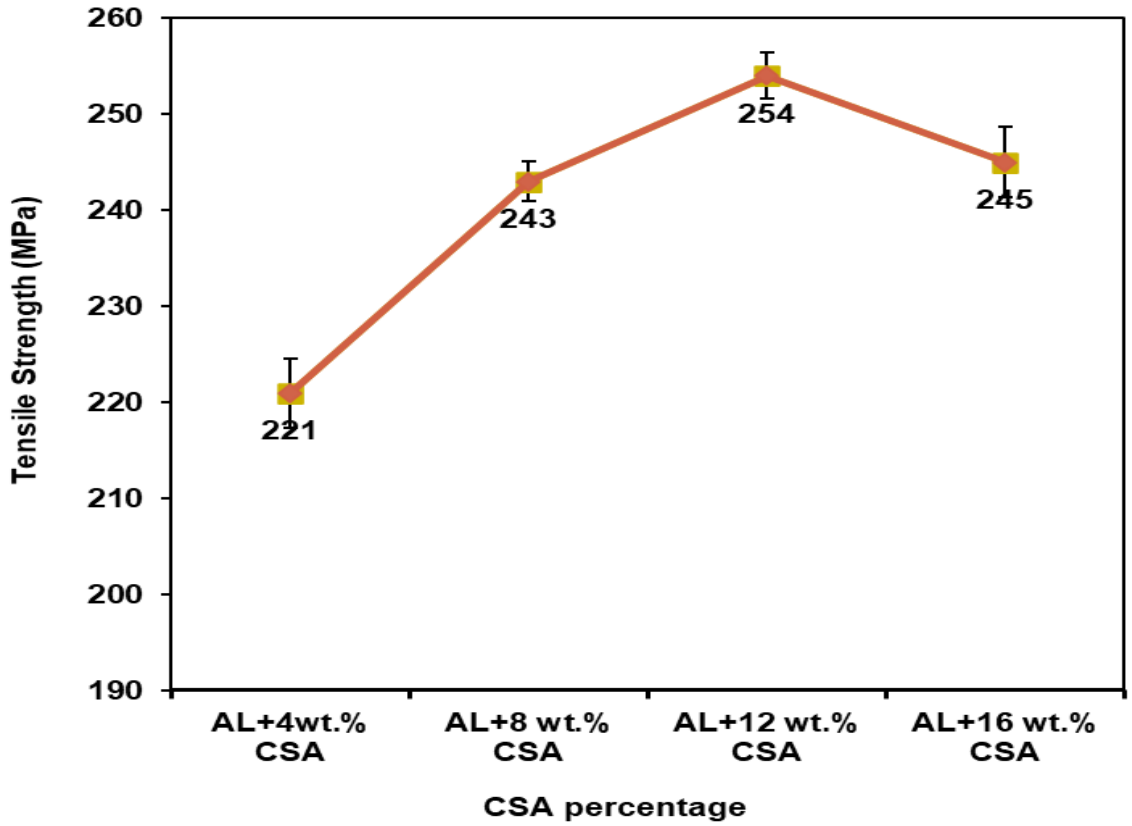

Figure 2: Effect of CSA on Tensile Strength of Aluminum Composite

3.3. Impact strength

The graph in Figure 3 illustrates the impact strength of the composite material reinforced with CSA particles, which signifies the material's ability to absorb energy before breaking. The total energy absorbed by the composite before failing is a direct measure of its capacity to resist impact, a vital property for applications requiring high strength. The impact strength of a material is a critical safety factor, as it determines its ability to withstand high-energy impacts without sustaining damage or shattering. In other words, a material's capacity to absorb impact energy without failing is directly proportional to its impact strength, making it an essential property for materials subjected to high-energy impacts (Senthilkumar et al., 2022).

The addition of agro-waste reinforcement, as previously observed by (Haider et al., 2019), generally leads to a increase in the elongation and ductility of aluminum composites. Similarly, the impact strength of the Al matrix exhibited a

increase with the introduction of CSA, from 0 (control) to 8 wt.% CSA composites. However, a notable continues till 16 wt.% CSA. The effective dispersion of CSA particles within the Al matrix likely contributed to a robust interfacial bond and enhanced mechanical properties. Conversely, Biradar et al. (2020) attributed the increasing strength in their study to a enhanced interfacial interaction between polypropylene (PP) resin and short pineapple fibers, which created defects that concentrated stresses and facilitated crack initiation with minimal energy input. Tay et al. (2021) elucidated that the energy absorption mechanism in composites involves the utilization of energy to debond fibers and pull them out of the matrix, facilitated by a weak fiber-matrix interface. In practical terms, a substantial portion of energy absorption during impact loading occurs through the fiber pull-out mechanism.

Afzaluddin et al. (2019) research suggests that the toughness of composite materials is largely determined by the stress-strain behavior of their fiber components. The addition of robust reinforcements like CSA, which exhibit high failure strain, can substantially enhance the work to fracture in composites, due to the high density and volume fraction of strong particles in these materials. However, this finding seems to contradict the tensile properties, specifically tensile modulus and tensile strength. A logical explanation for this inconsistency is that composites can resist rapid impact loads, but are vulnerable to reinforcement slippage from the matrix under slow tensile stress, leading to weak points and stress concentration areas. This results in reduced elongation at break and lower toughness. Furthermore, an increase in impact properties can be attributed to plastic deformation of the epoxy matrix, interface debonding, and better interfacial reaction, which are common issues in fiber composites with moderate reinforcement.

The impact strength of composites is dictated majorly by two factors: the ability of the filler to absorb energy that can stop crack propagation and interfacial bonding which induces micro-spaces between the filler and the matrix, culminating into crack propagation (Matlakala et al., 2019; S. Vieira et al., 2018). Moulded-in stresses, additives, polymer orientation, weak spots – such weld lines or gate areas – and part geometry will influence the impact performance.

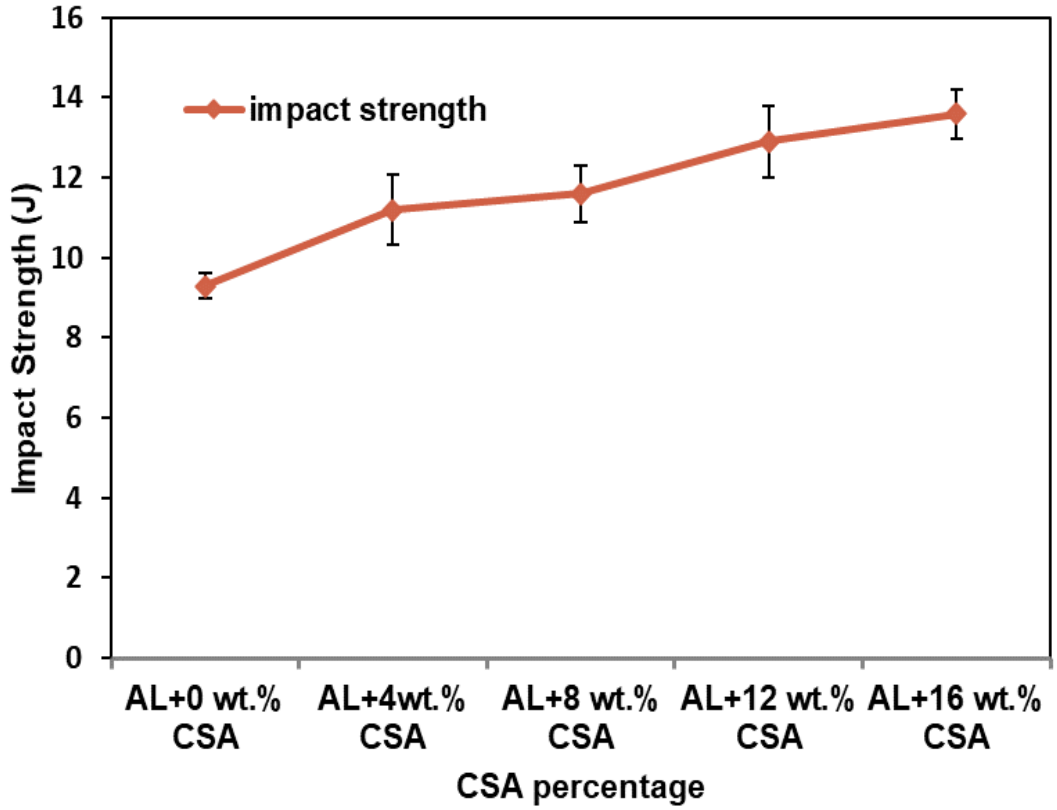

Figure 3: Effect of CSA on Impact Strength of Aluminum Composite

3.4. Scanning Electron Microscope

SEM analysis results for the Aluminum-CSA reinforced specimens, consisting of 700g of pure aluminum and aluminum mixed with varying CSA weights (4wt%, 8wt%, 12wt%, and 16wt%), are presented in Figure 4 (a-e). The SEM images reveal irregular, roughly spherical shapes, showcasing the progressive addition of CSA reinforcement up to 12wt% in aluminum. The micrograph for 12wt% CSA (Figure 4d) stands out, displaying a uniform distribution of reinforcement within the aluminum particles, which is indicative of enhanced mechanical properties and consistent with the highest tensile strength observed in the tensile test results. Additionally, Figure 4d exhibits minimal pores, agglomeration, and fiber pull-out, signifying a homogeneous interface between the matrix and reinforcement. Conversely, the micrograph for the highest CSA reinforcement (Figure 4e) shows an uneven distribution, likely due to excessive CSA loading that exceeded the aluminum matrix's capacity. This finding is in agreement with previous research (Alaneme & Sanusi, 2015; Dwivedi et al., 2020; Singh & Chauhan, 2019), which demonstrated that a uniform distribution between reinforcement and matrix occurs when the reinforcement is within the matrix’s capacity, as exemplified by the Al+12wt% CSA sample.

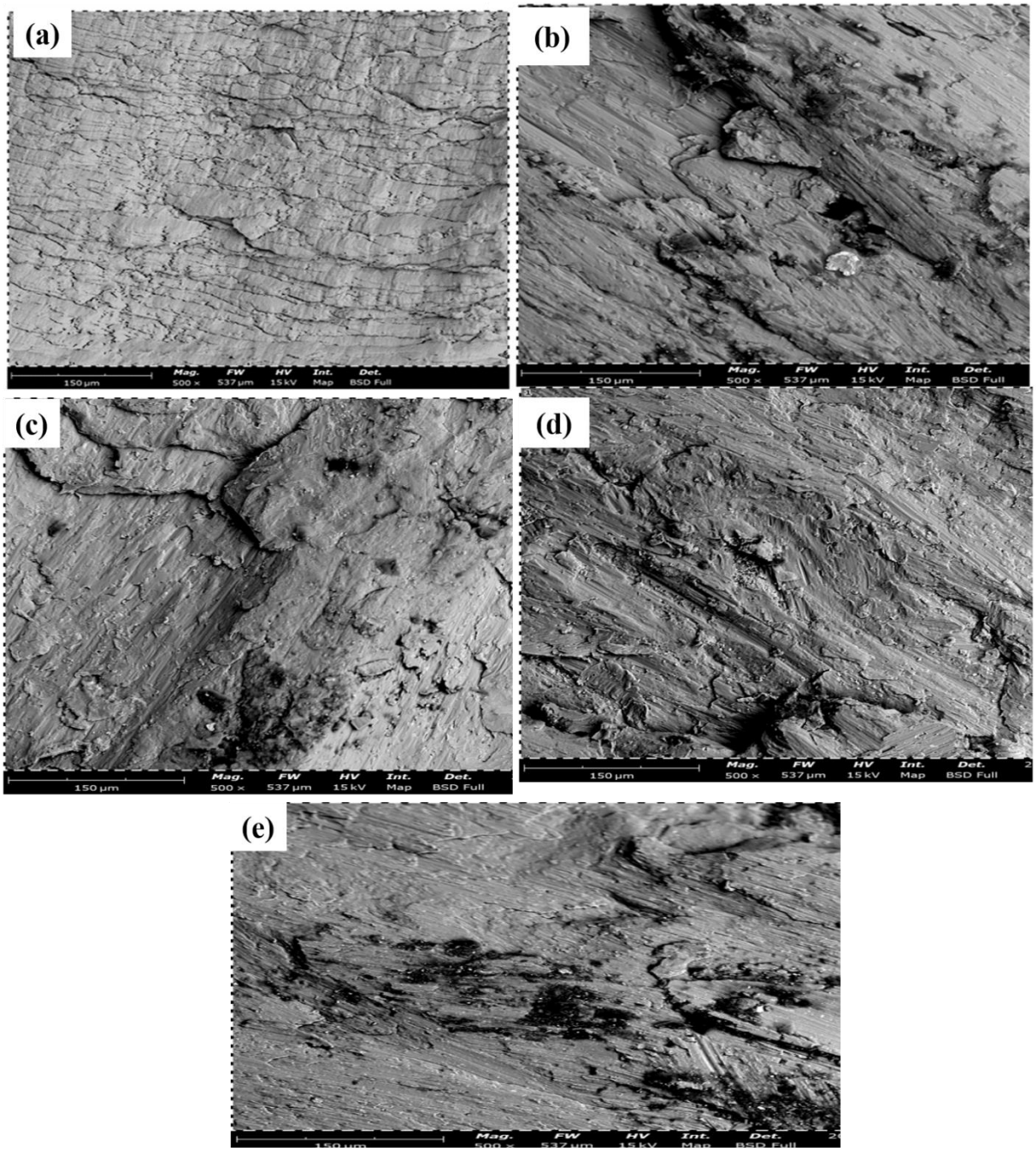

Figure 4: SEM analysis for (a) Control (b) Al/4 wt.% CSA sample (c) Al/8 wt.% CSA sample (d) Al/12 wt.% CSA sample (e) Al/16 wt.% CSA sample

4. Conclusions

This research examined the effects of incorporating biodegradable coconut shell ash nanoparticles on the mechanical behavior and microstructural properties of sustainable aluminum matrix composites. The study used a stir casting method to reinforce aluminum with varying weights (0, 4, 8, 12, and 16%) of coconut shell ash nanoparticles. The investigation revealed the following key findings:

i. The control sample demonstrated the lowest hardness value of 50 HB in the BHN Hardness test, while the addition of CSA to the aluminum composites at varying weights (4, 8, 12, and 16 wt%) resulted in a gradual

increase in hardness, culminating in a maximum hardness value of 65 BHN for the Al+12 wt% CSA composite.

ii. The Al+12 wt% CSA composite exhibited the highest tensile strength, but this value decreased when the CSA content was increased to 16 wt%, likely due to the insufficient matrix capacity to support the excessive CSA load.

iii. The impact test revealed that the control sample had a lowest impact strength than the CSA-reinforced composites (4, 8, 12, and 16 wt%), which was attributed to the CSA particles increasing the ductility and elongation of the composites. The Al+16 wt% CSA composite showed the highest impact strength, with a value of 13.6 J.

iv. The SEM analysis results also revealed a uniform distribution of CSA particles within the matrix, without little voids and agglomeration, particularly in the Al+12 wt% CSA composite, indicating effective reinforcement.

However, it is recommended that optimization of the synthesis process to achieve uniform nanoparticles should be performed as well as exploring other biodegradable reinforcements like rice husk ash or bamboo leaf ash, and investigating the effects of different sintering techniques. Further research should also focus on interfacial reactions, hybrid reinforcements, and corrosion behavior. Scaling up the synthesis process and conducting mechanical testing under various conditions will help assess industrial applicability. Additionally, exploring new applications in the automotive, aerospace, and energy sectors could reveal new potential for these sustainable composites.

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