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Effect of Particulate Reinforcements on Mechanical, Microstructures and Wear Behaviours of Aluminium Alloy with Aquaculture Wastes Powder

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Abstract: This work aims to investigate the mechanical, microstructures and wear behaviours of aluminium alloy 6063 reinforced with aquaculture waste of 80 µm mussel shell powder (MSP) at different weight percentages (0 wt. % to 15 wt. %) at 3 wt. % interval. The 80 µm mussel shell powder was characterized by X-ray fluorescence (XRF). The matrix and the composite morphology were studied using a scanning electron microscope to determine the mussel shell powder particle distribution within the matrix. The wear behaviour of the alloy and composites produced at various reinforcements were carried out using a Taber abrasion wear-testing machine at different loads (25, 20, and 15 N respectively). The XRF showed the compositions of MSP to contain calcium oxide (95.70 %), silica (0.83 %), Fe_2O_3 (0.73%), Al_2O_3 (0.63%) and others. Mechanical properties showed that tensile values increase with increases in MSP to the optimum of 12 wt. %, and hardness value increases from 6 wt. % to 15 wt. % of MSP. The impact energy decreased from 36.5 J at 6 wt. % to 25.6 J at 15 wt. %, the percentage elongation also decreased from 32.4 % at 3 wt. % to 19.5 % at 15 wt. % MSP respectively. The morphologies revealed that MSP uniformly distributed within the matrix and resulted to the improvement in both mechanical and wear behaviours respectively. The wear resistance of the matrix and composites increases with an increase in load and decreases with an increase in the weight percentage of MSP. The minimum wear rate was obtained at the load of 15 N while highest at the load of 25 N. The specific wear rates of the alloy lost under loads 25, 20 and 15 N were 75, 62 and 55.4 mm^3 (Nm) while the specific wear rates of the composites at 15 wt. % were 44.1, 33.5 and 26.1 mm^3 (Nm) respectively. The composites produced can used for the production of brake pads, engine blocks, and insulators in both automobile and aerospace industries.

1. Introduction

Material development is to meet the requirement of industrial application properties in any industry. The growing demands in the automotive and aerospace industries for the reduction in energy consumption and producing more fuel-efficient vehicles continue to be a big challenge (Suleiman *et al,* 2019). Aluminum Metal Matrix Composites (AMMCs) are one such material to provide a combination of properties such as high strength, high stiffness, higher thermal conductivity, higher Young's modulus, and better tribological properties over unreinforced alloys. However, the AMMCs are mostly used in automobile, marine, and aerospace applications (Dieter, 1961).

Aluminium matrix composites (AMCs) have a unique combination of chemical, mechanical, and physical properties which cannot be attained with the use of monolithic materials (Sandeep et *al*, 2018). This is why AMCs were regarded as promising materials for the automotive and aerospace industries (Alaneme and Olubambi, 2018). The automobile parts made from these composites include connecting rods, brake drums, and cylinder heads and were relatively low in cost of processing when compared to others. However, the problem with unreinforced aluminium alloys is the poor tribological properties which can be resolved by reinforcing the alloys with other materials such as fly ash, Al_2O_3, S_iO_2, Fe_2O_3, T_iCT_iC, B_4C, and S_iC. With these reinforcements, their morphologies and tribological properties were greatly improved (Chak *et al*, 2020).

The 6063-aluminium alloy, known as architectural and decorative alloy, improves the casting, corrosion resistance property as well as the strength of the alloy; because of its easy extrudability property, distinctly superior finishing quality and strength (Siddiqui *et al*, 2000; Bazzi *et al*, 2003; Chak *et al*, 2020; Chhak *et al*, 2020; Ech-chihbi *et al*, 2023).

Despite their apparent widespread use, they are not produced in developing countries like Nigeria. The reliance on importation from overseas and the high foreign currency exchange involved imply that the synthetic reinforcements purchased locally are at a relatively high cost (Arunachalam *et al*, 2019). An alternative to the high cost of synthetic reinforcers in developing countries is to explore some other means such as using wastes from Animals and Agriculture. These wastes may be in the form of powders or ash particulates. Ashes and powders obtained from the wastes such as bamboo leaf, rice husk, bagasse, coconut shell, melon shell ash, and ground nut shell for the development of AMMCs had been investigated and proved successful in terms of mechanical and wear properties improvements (Netinger Grubeša *et al*, 2025; Hamed *et al*, 2021; Selvam *et al*, 2020; Chhak and Chattopadhyay (2020; Selvam and Dinaharan, 2020; Sozhamannan *et al*, 2012; Gomes *et al*, 2005; Howell and Ball, 1995). These ashes and powders are not just cost-effective but are also available and environmentally friendly and has been shown that some of these wastes contain a high percentage of refractory materials such as Al_2O_3, silica (S_iO_2), and hematite (Fe_2O_3) distributed in these wastes (Vencl *et al*, 2008).

The various discontinuous dispersed utilizing mussel shell powder (MSP) is one of the aquaculture wastes by-product reinforcement available in large quantities in Nigeria. Hence, composites with aquaculture shell powder as reinforcement may likely succeed the cost, time, and hazards associated with the imported ceramic materials for better applications. The MSP is a great environmental threat causing damage to the land and the surrounding area where these wastes are being dumped. An effective way of utilizing the aquaculture shell was to subject it to treatment and convert it to powder under controlled conditions. The weight fractions of mussel shell powder at particle sizes of 80 and 100 μm were varied from 0 to 15 wt. % at 3wt. % interval. The MSP was characterized by X-ray fluorescence to ascertain the compositions and the morphologies of the alloy and composites, and mechanical and wear mechanisms were investigated by scanning electron microscopy (SEM).

2. Materials and Methods

2.1 Preparation of mussel shell powder (MSP)

The mussel shells were obtained from Port Harcourt Rivers State, South- South region of Nigeria, The shells were prepared by washing with water and detergent to remove the oil and dirt. It was sun-dried for 2 weeks and pulverized to form mussel shell powder (MSP). The mussel shell powder (MSP) prepared was subjected to sieve analysis. The arrangement of the sieves was done using a sieve scale in which the ratio of the aperture widths of adjacent sieves. Sieve sizes from 80 to 43 µm were arranged in a stack with the coarsest sieve on the top and the finest at the bottom. The arranged sieves were placed in a sieve shaker which vibrates the powder vertically. A particle size of 80 µm of mussel shell powder was obtained and utilized for this work. The Figure 1 below shows the mussel shell and powder respectively. The elemental analysis of the mussel shell powder was analyzed by an energy-dispersive X-ray spectrometer (Mini pal 4 ED-XRF machine, made by Panalytical, Netherlands, and was used). The sample was weighed and ground in a mortar and pressed in a hydraulic press to produce pellets. The pellets were loaded in the sample chamber of the spectrometer and a voltage of 30 kV and current of 1 mA were applied to the X-rays to excite the sample for 10 min. The spectrum from the sample was then analyzed to determine the concentration of the elements in the sample. The chemical compositions of the MSP and aluminium alloy (Al 0.7%Mg 0.4%Si) are presented in Tables 1 and 2 respectively.

Figures 1a and 1b. (A*) Mussel shell collections, and (B) mussel shell of particle size of 80 µm*

Table 1. The chemical compositions 80 µm mussel powder

Compounds	Mussel shell powder [%]
CaO	95.65
K_2O	0.35
SiO_2	0.83
SrO	0.26
Fe_2O_3	0.73
SO_3	0.86
MgO	0.48
Al_2O_3	0.67
LOI	Balance

Table 2. The chemical compositions of aluminium alloy 6063 (Al 0.7% Mg 0.4% Si) analysis

Compositions	Mg	Si	Fe	Mn	Cr	Cu	Al
Weight percent [wt. %]	0.7	.40	.01	.02	.01	.03	Bal.

2.2 Charge calculation

The summary of charge calculations in weight percent (wt. %) and in grams of the alloy and composites were presented in Tables 3 and 4 respectively.

Table 3. Summary of the charge calculations for the alloy and composites in weight percent (wt. %)

S/No:	Alloy (0%)	3wt.%MSP	6wt.%MSP	9wt.%MSP	12wt.%MSP	15wt.%MSP
(MSP)	0	3	6	9	12	15
Aluminium	98.9	95.9	92.9	89.9	86.9	83.9
Magnesium	0.7	0.7	0.7	0.7	0.7	0.7
Silicon	0.4	0.4	0.4	0.4	0.4	0.4
Total	100 wt.%	100 wt.%	100 wt.%	100 wt.%	100 wt.%	100 wt.%

Table 4. Summary of the charge calculation of the alloy and composites in gram (g)

S/NO	0 MSP (g)	3 MSP (g)	6 MSP (g)	9 MSP (g)	12 MSP (g)	15 MSP (g)
MSP	0	25.4477	50.8953	76.3430	101.7907	127.2383
Silicon	3.3930224	3.3930224	3.3930224	3.3930224	3.3930224	3.3930224
Magnesium	5.9377892	5.9377892	5.9377892	5.9377892	5.9377892	5.9377892
Aluminium	838.9258	813.4777884	788.0294884	762.58178	737.13408	711.68648
Total	848.2556g	848.2556g	848.2556g	848.2556g	848.2556g	848.2556g

2.3 *Production of Al–Mg-Si/mussel shell powder composites*

The chemical composition of the alloy is presented in Table 2. The amounts of mussel shell powder (MSP) used as reinforcers were determined using a charge alloy that was superheated to 800 °C after being charged into a crucible furnace. The stainless-steel stirrer was used to stir the molten alloy/composites manually. The reinforcement particles, MSP of 80 μm were preheated to 300°C for 30 minutes. After preheating, MSP particles were consolidated into the melt to exclude moisture. The degassing tablet was added after the alloy/composites were completely melted to reduce the porosity. The wettability was enriched by the composition of magnesium in the melt. This magnesium improves the wettability between the matrix alloy and reinforcement; thus, 3 wt. % to 15 wt. % at 3 wt. % interval by equal MSP proportions used. Preheated moulds were set before casting the alloy and the composite of 20 mm by 50 mm, allowed to solidify, and presented in Figure 2. The same procedures were repeated for 6, 9, 12, and 15 wt. % MSP reinforcements, respectively.

Figure 2. Finished products of the alloy and composites, respectively.

2.4 Determination of Mechanical behaviours of alloy and composites

2.4.1 Determination of tensile strength

The tensile tests were carried out on the samples according to ASTM E08-95 at room temperature (30°C), using a Universal testing machine (INSTRON). The test was conducted using a strain rate of 2mm/min. As cast Al alloy and composites, tensile test specimens were prepared using a lathe machine and shaper machine. The specimens were machined to the standard diameter size of 2 mm by the works of (Gireesh *et al,* 2018; Sharma *et al,* 2017).

2.4.2 Determination of the hardness values

The hardness test was carried out using a Rockwell hardness machine. The hardness specimen was placed on a flat horizontal stand, with a preload of the diamond cone indenter used to indent on the surface of the specimen its hardness value was reflected on a dial gauge of the machine and the readings read from the calibrated C-scale of the gauge. Three indentations were made on each sample, and the average of the hardness readings were taken as carried out in (Nirmal *et al,* 2015; Chauhan *et al,* 2017; Aichouch *et al.,* 2025). The Hardness test was carried out in the NLNG laboratory of the Metallurgical and Materials Engineering department, University of Nigeria, Nsukka.

2.4.3 Impact testing

The impact specimen was placed on a horizontal stand of the Izod Impact Machine. It was arranged such that the notch was directly opposite to the point of impact of a heavily suspended mass. With the gauge set properly, the suspended mass was released from a height to hit the specimen. The energy absorbed by the specimen was reflected on a calibrated scale (Chauhan *et al,* 2017).

2.5 Wear analysis

Wear test specimens with a diameter of 25 mm and a thickness of 5 mm were machined from the as-cast produced composites. The surfaces of each specimen were prepared with 600-grade SiC abrasive papers. A total number of specimens (alloy and composites) were used for the whole experiment, as for each composition, three different loads of 15, 20 and 25 N were used. The wear test was carried out on the surface of the specimens using an Anton Paar TRN Tribometer (asper ASTM G99-95 standards). The abrasive medium used was made of stainless steel ball. An applied load of 15, 20 and 25 N at 153 rev/min wheel speeds and a dwell time of 3.26 min were used. The sliding speed used was 2 m/s. Weight loss method was adopted to study the wear behavior. The weight of the specimen before and after each test was measured using a digital weight balance. The mass loss was determined for each specimen by finding the difference between the initial and final mass. The weight loss method was used to calculate the wear rate (Tugiman *et al,* 2017; Francis *et al,* 2016).

2.6 Microstructural examination

The morphologies of the alloys and composites produced were investigated using a Scanning Electron Microscope (SEM). Samples were polished on emery papers of different grades. The polishing was carried out on a circular cloth pad on its surface. Rough polishing was done using silicon carbide paste, and the final polishing operation was carried out using alumina polishing paste. Etching of the specimen was carried out using a ball of cotton wool soaked in nital to wipe the specimen's polished surface to give a dull reflection surface (Abdulwahab *et al,* 2017; Phanibhushana *et al,* 2017). This study used an XL30SFEG high-resolution field emission scanning electron microscope. The surface morphology of the alloy and composites was their fore analysed.

3. Results and discussion

3.1 XRF Analysis of 80 µm Mussel shell powder

The results revealed by the elemental analysis of 80 µm mussel shell powder from X-ray fluorescence (XRF) are presented in Table 1. It could be observed from the table that, calcium oxide (CaO) has the highest percentage composition of 95.65 wt. % followed by SiO_2 (0.82 wt. %), Fe_2O_3 (0.73 wt. %), MgO (0.47 wt. %), Al_2O_3 (0.67 wt. %) and the remaining balance was lost on ignition (LOI) respectively. Calcium oxide and Silicon oxide played vital roles when used as fillers in the aluminium matrix composites for industrial applications. The presence of hard elements like CaO and SiO_2, Al_2O_3, Fe_2O_3 suggested that the mussel shell powder can be used as particulate reinforcement in various aluminium metal matrixes according to the previous findings (Chandla *et al.,* 2017; Lancaster *et al,* 2013).

3.2 The morphologies of the aluminium alloy 6063/reinforced composites

The reinforcement contributed to the refinement of the AA6063 microstructure, reducing grain size and enhancing the interfacial bonding between the matrix and reinforcement, which played a crucial role in the observed improvements in both mechanical and wear behaviours (Meena *et al,* 2013). The physical properties of the composites however depend on the microstructure, reinforcement particle size, shape and distribution in the alloy. Prepared samples were examined using a scanning electron microscope (SEM) of magnification (× 1000) to study the distribution pattern of 80 µm mussel shell powder as a reinforcer in the matrix. The micrograph shown in Fig. 3a depicts the microstructure of as-cast Al 0.7% Mg 0.4% Si alloy. In Fig. 3a, the structure was completely uniform with the solubility of silicon in the Al matrix. Figure 3b revealed the volumes of the reinforcer at the optimum concentration of 12 wt. % of MSP. In Fig. 3b, the volume of the MSP shows the metallic phase seems to be white while the light black portion of carbon oxide (CaO) and CaO was more in the XRF results presented in Table 1 (95.65 %). It was also observed that there was good bonding between the Al matrix and the filler (MSP) particles and no gap was observed between the particle and matrix (Apasi *et al,* 2016). The microstructures of the composites revealed small discontinuities and reasonably uniform distribution, good retention and interfacial bonding of mussel shell powder particles in the aluminium alloy matrix. The addition of magnesium in the alloy 6063 also improved the wettability of mussel shell powder particles. The results obtained also supported with the previous works (Deuis *et al,* 1996).

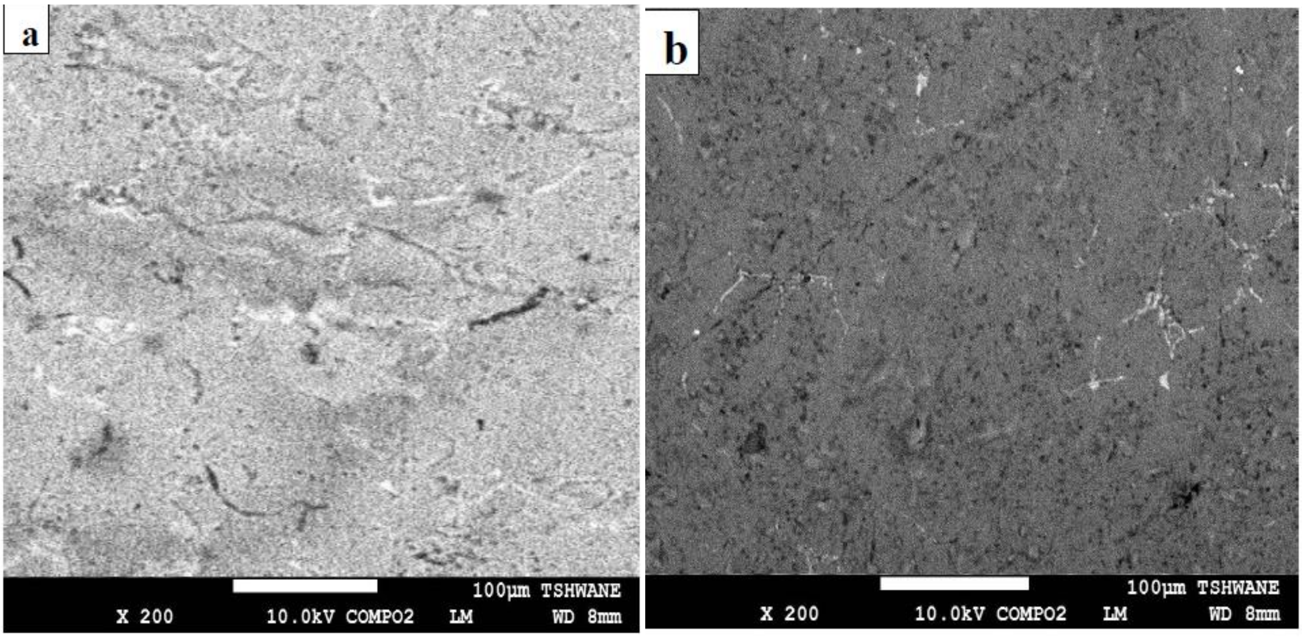

Figure 3. The morphologies of (**a**): Al-0.7% Mg-0.4 % Si, (**b**): Al-0.7% Mg-0.4 % Si/12 wt. % MSP

3.3 Mechanical Properties of Alloy/Composites

3.3.1 Tensile values

The incorporation of 80-micron particle size of mussel shell powder into Aluminium Alloy 6063 has shown a significant increase in tensile strength compared to the unreinforced alloy. Composites with higher weight percent fractions of mussel shell powder exhibited superior strength due to enhanced load transfer capabilities between the matrix and the reinforcement (Meena *et al,* 2013; Apasi *et al,* 2016). Optimal reinforcement percentages (typically between 3 wt. % to 12 wt. %) have been found to yield the highest tensile strength, while excessive reinforcement may lead to agglomeration and reduced performance. The reinforcement helps in load transfer, which enhances the tensile strength of the composite (Figure 4). Studies have shown that smaller and more uniformly shaped mussel shell particles are expected to lead to greater strength improvements due to more uniform stress distribution and enhanced interfacial bonding with the matrix (Vencl *et al,* 2008). Higher volume fractions of mussel shell powder generally lead to higher strength, up to a point where agglomeration and other detrimental effects may occur. The incorporation of mussel shell powder into Aluminium Alloy 6063 resulted in a noticeable increase in tensile strength. Optimal formulations showed improvements of up to 12 wt. % of MSP compared to the unreinforced alloy (Deuis *et al,* 1996).

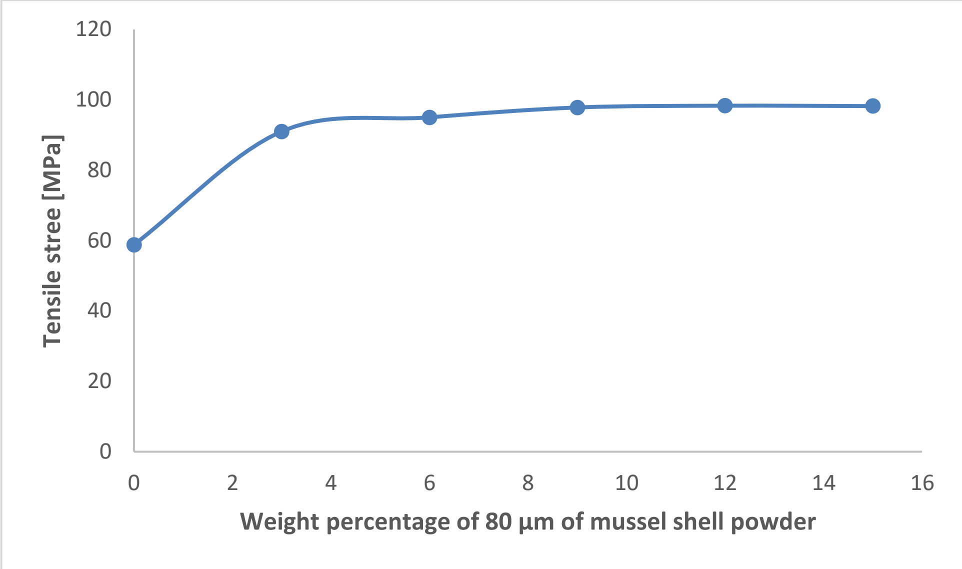

Figure 4. Variations of tensile stress of Alloy and composites against wt. % MSP

3.3.2 Hardness values

Hardness tests reveal that the addition of mussel shell powder markedly improves the hardness of the composite. The hardness increase is due to the inherent toughness and abrasiveness of the shell powder, which inhibits dislocation movement in the aluminium matrix. The results indicate that the hardness values increase with the percentage of mussel shell powder until reaching a plateau, where further additions do not significantly enhance hardness. The hardness of the composite is also significantly improved due to the presence of the hard mussel shell particles, which impede dislocation movement within the aluminium matrix (Figure 5). The micro hardness values increased as the reinforcement content rose, attributed to the hard nature of the shell particles, which impede dislocation movement within the aluminium matrix (Thakur, & Dhindaw, 2001).

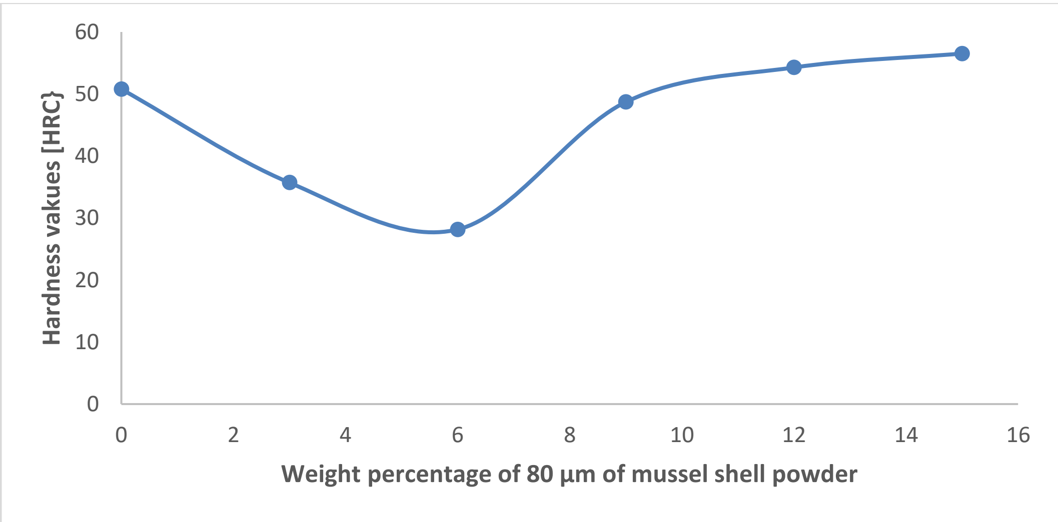

Figure 5. Variations of Hardness values of Alloy and composites against wt. % MSP

3.3.3 Impact values

The result of the impact fracture of the alloy and composites is presented in Figure 6. It was observed that the fracture toughness of the composites containing 3, 6, 9, 12, and 15 wt. % MSP had lower fracture toughness values in comparison with the 6063 AlMgSi alloy. The mechanisms of fracture had been attributed to particle cracking, and interfacial cracking as observed in the previous studies [28]. It was also established that ceramic particulates are hard, brittle and always have a poor tendency to resist rapid crack propagation (Thakur and Dhindaw, 2001). In this research work, the fracture toughness decrease was observed in 9, 12, and 15 wt. %. However, it is clear that the addition of 9–15 wt. % MSP deteriorated the fracture toughness of the AlMgSi alloy reinforced with MSP matrix composites. Similar findings were also found in research works (Francis and Paramasivam, 2016).

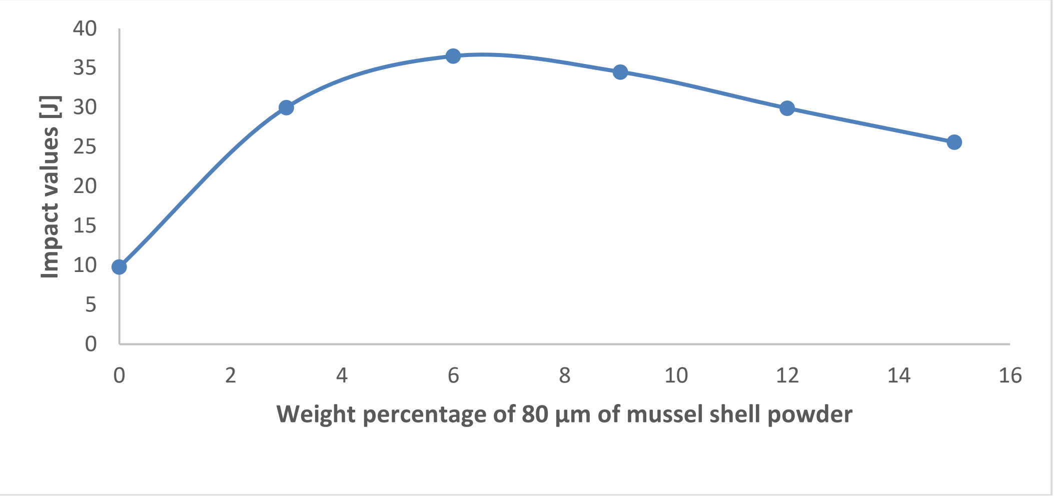

Figure 6. Variations of Impact values of Alloy and composites against wt. % MSP

3.3.4 Percentage elongation

While tensile strength and hardness improved, ductility showed a varying trend sd presented in Figure 7. Composites with lower percentages of mussel shell powder maintained reasonable ductility, but higher concentrations resulted in increased brittleness. This suggests a trade-off between strength and ductility that must be carefully balanced in applications. While the tensile strength and hardness improved, a decrease in ductility was observed at higher reinforcement levels. Excessive mussel shell powder led to brittleness, highlighting the importance of optimizing the reinforcement ratio to balance strength and ductility (Suleiman *et al*, 2018).

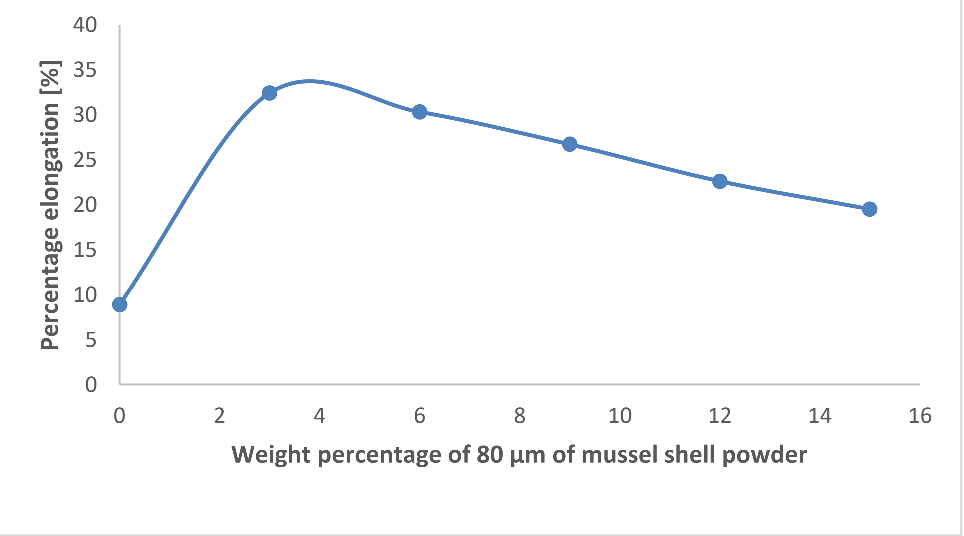

Figure 7. Variations of percentage elongation of Alloy and composites against wt. % MSP

3.4 Wear behaviours of the alloy/composites

The wear tests demonstrated that composites reinforced with mussel shell powder exhibited lower wear rates compared to the unreinforced alloy as seen in Figure 8. The hardness of the mussel shell particles contributed significantly to the overall wear resistance, making these composites suitable for applications where abrasion is a concern. Wear is a gradual loss of material due to relative motion between a surface and the contacting substance and the wear damage may be in the form of micro-cracks or localized plastic deformation (Francis and Paramasivam, 2016; Suleiman *et al,* 2018), .

The wear mechanisms were analysed, revealing that the presence of mussel shell powder alters the wear process. The harder particles such as Calcium oxide (CaO) reduce the depth of wear tracks and minimize material loss during the sliding tests (Figure 9). This was evident under varying loads and sliding speeds, where reinforced composites consistently outperformed the base alloy and agreed with the findings (Srinivas *et al,* 2020).

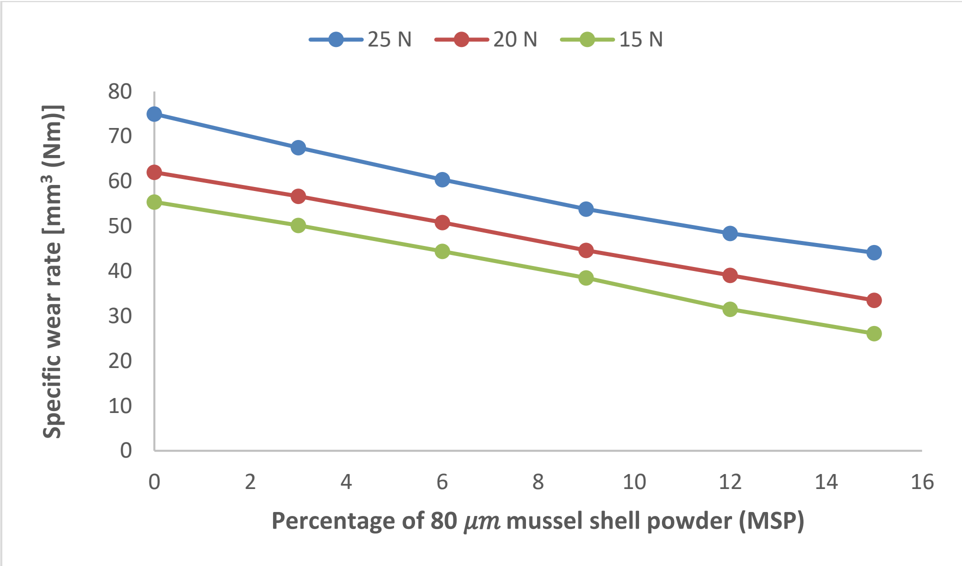

Figure 8. Effect of reinforcement on specific wear rate

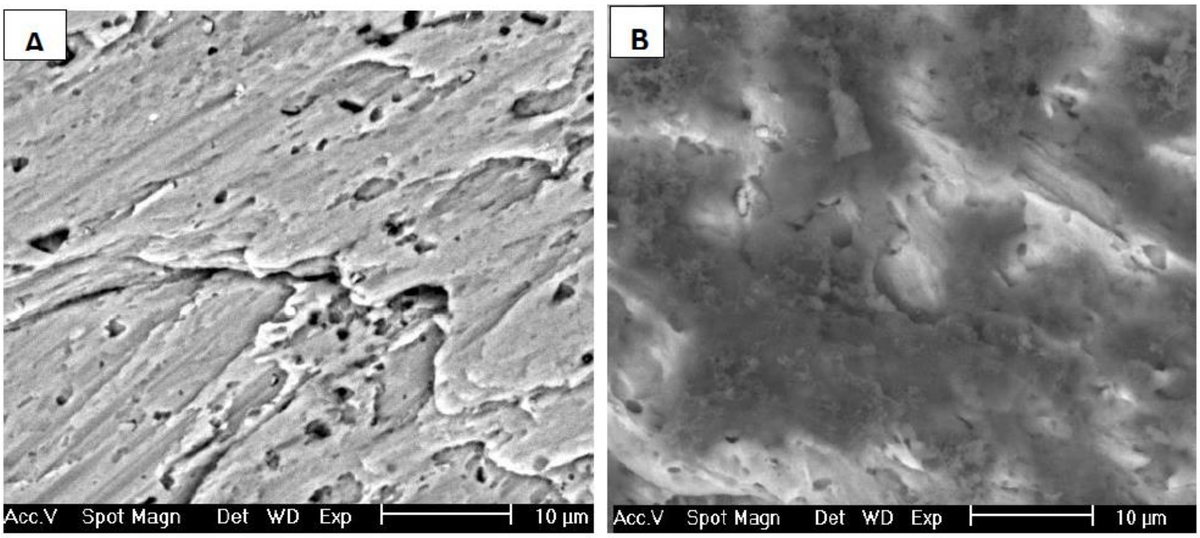

Figure 9. (a)-Morphology of Aluminium alloy 6063 without loads (b) – Alloy 6063 reinforced with optimum 15 wt. % of 80 μm MSP particles at different loads

The study identified optimal reinforcement levels for achieving the best wear performance. Excessive powder content may lead to agglomeration and a decrease in overall performance, highlighting the importance of careful formulation (Figure 9). Utilizing mussel shell powder as a reinforcement not only improves material performance but also contributes to sustainability by recycling wastes from the seafood industry. This aligns with growing trends toward eco-friendly materials in manufacturing (Bharath et *al*, 2022).

Conclusions

From the investigation carried out on the alloy 6063 and the reinforced composite with 80 µm (3–15) wt. % of MSA, the following conclusions can be drawn:

1. By optimizing the particle size and distribution of mussel shell powder, a composite produced balances strength, hardness, and wear resistance, making it a promising candidate for various engineering applications
2. MSP being aquaculture wastes is a good substitute to imported reinforcers such as Al_2O_3, TiC, SiC, and B_4C which are available, cheap, environmentally friendly, sustainable, and crucial for developing materials for engineering applications.
3. The addition of mussel shell powder increased hardness, and tensile, and enhanced wear resistance through various mechanisms of strength due to effective particle dispersion, improved load transfer, and grain refinement.
4. The wear resistance of the composites increases with increase in the applied load and decrease with increase in the weight percentage of 80 µm of MSP.
5. The improved wear performance is attributed to the hard nature of the mussel shell particles, which provide a protective effect against wear.

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