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Carbonization Temperature and Its Effect on the Mechanical Properties, Wear and Corrosion Resistance of Aluminum Reinforced with Eggshell

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Abstract: In this paper, the effect of the carbonization temperature on the mechanical properties, density, wear and corrosion resistance of AA 6063 reinforced with eggshells was investigated. The selected fabrication route for this investigation was stir casting while the weight fraction of the eggshells was kept constant at 5 wt.%. The carbonization temperature was varied at 900, 1000, 1100, and 1200 °C. The microstructure revealed that the eggshells were fairly uniformly dispersed on the individual grains and along the grain boundaries of the base metal. It was also shown that the presence of agglomeration increased with increasing carbonization temperature. The densities of the eggshell-reinforced AMCs were lower than that of the base metal. The analysis of the microhardness showed an improvement of 40.79, 22.93, 25.70, and 29.43% for the 900, 1000, 1100, and 1200 °C carbonized eggshell samples, respectively. The compressive strength studies showed that the addition of carbonized eggshells improved the compressive strength of the composites compared to the base metal. The tribology studies showed that the wear resistance improved for the 900 and 1200 °C samples, while the electrochemical studies revealed that the corrosion resistance improved for the 900 and 1000 °C samples only.

Keywords: aluminum matrix composites; eggshells; carbonization temperature; mechanical properties; wear resistance; corrosion resistance

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1. Introduction

Aluminum matrix composites (AMCs) are being sought after owing to their attractive mix of properties not limited to good strength-to-weight ratio, high stiffness, and good corrosion resistance [1]. This has opened up potential areas of application not limited to aerospace, marine, and automobiles [2]. The advent of aluminum matrix composites highlighted the use of synthetic materials as reinforcements. Such synthetic materials include SiC, Al_2O_3, B_4C, and TiN [3]. These materials have successfully been applied in the fabrication of AMCs with recorded improvements in certain properties. Further research into the improvements of AMCs has led to the consideration of certain waste materials in the fabrication process. Citing the need for environmental sustainability and cost savings, studies in [4,5] have considered the use of waste materials for the fabrication of AMCs. Such materials that have been utilized include fly ash [6], red mud [7], rice husk ash [8], and coconut shell ash [9]. Among these materials, eggshells have also been considered for reinforcing aluminum and its alloys. Eggshells are by-products of the food industry, which is responsible for as much as 150,000 metric tons of waste per annum in the US alone [10]. This has made the disposal of the eggshells increasingly more difficult. Research into the use of eggshells as AMC reinforcements has shown that they have the advantages of lower density and the presence of prominently $CaCO_3$, which makes it viable for use as the dispersed phase in AMC fabrication. Research in [11] has shown that the fabrication of AMCs with eggshells is capable of saving up to 6% of the material cost compared to the base metal. It has been reported in Asuke et al. [12] and Oghenevweta et al. [13], that carbonizing eggshells is necessary to improve the mechanical properties, decrease the presence of voids, and promote good interfacial bonding between the reinforcements and the aluminum matrix. To this effect, several researches have been carried out to fabricate carbonized eggshell-reinforced AMCs with recorded improvements in weight reduction and mechanical properties. Such research includes that conducted by Dwivedi et al. [14] who investigated the effect of carbonized eggshells on the mechanical properties of AA 2014. The eggshells were carbonized at 500 °C. The density analysis revealed a decrease by up to 3.92%, whereas the hardness improved by 33%. The fatigue strength and tensile strength improved by 19.6 and 33%, respectively. An investigation recorded in [15] studied the effect of eggshells carbonized at 800 °C on the density, mechanical properties and corrosion resistance of AA 1050. The density decreased with increasing weight fraction of the carbonized eggshells. The tensile strength of the fabricated AMCs improved by up to 11%, while the hardness and compressive strength improved by up to 12.21 and 31.99%, respectively. The corrosion resistance of the reinforced AMCs improved compared to the base metal. An investigation carried out by Hassan and Aigbodion [16] stated that carbonizing of eggshells at 1200 °C improved the mechanical properties of Al-Cu-Mg compared to the samples reinforced with uncarbonized eggshells. It was also reported that the improvements brought about proper wettability between the reinforcements and the base metal, and grain refinement of the aluminum alloy. Research by Dwivedi et al. [10], recorded improvements of 80, 45.94, and 53.30% in hardness, tensile strength, and fatigue strength, respectively, with the addition of eggshells carbonized at 500 °C.

It is evident from the reviewed literature that there is an existing knowledge gap in the effect of varying the carbonization temperature on the eggshells applied as reinforcements for AMC application. To this effect, this work was carried out to investigate the effect of varying carbonization temperature on the density, mechanical properties, wear and corrosion resistance of AA 6063.

2. Materials and Methods

The proposed study was conducted by selecting AA 6063 as the base material. AA 6063 is a medium-strength aluminum alloy with excellent corrosion resistance. This alloy, commonly referred to as architectural alloy, is used in extrusions, window frames, doors, and irrigation tubing. The base metal was reinforced with 5 wt.% eggshell ash with varying carbonization times of 900, 1000, 1100, and 1200 °C.

2.1. Reinforcement Preparations

The first step of the process was the preparation of the eggshells used as the dispersed phase in the composite fabrication. The eggshells were locally sourced from food stores and restaurants in the Auckland Park campus at the University of Johannesburg. The as-obtained eggshells were washed to remove all organic matter, which includes the membrane, lipids, and proteins. The washed eggshells were subsequently sun-dried for 1 week, before the carbonization process. The eggshells were further dried at 80 °C in an electric oven for 24 h to further remove any residual moisture present. The eggshells were subsequently milled using an electric ball milling machine with a rotational speed of 180 rpm. The milling process was done for 7 h to obtain particles in micro and nano ranges. For this study, the Flitra Vibracion S.L model FTL-0200 sieve shaker was used to sieve the milled eggshell particles. The eggshell particles selected for the studies were $\leq$75 µm. The already prepared eggshells were then charged in an electric muffle furnace to be carbonized at the stated varying temperatures (900, 1000, 1100, and 1200 °C) for 2 h.

2.2. Composites Fabrication

The selected fabrication route for this study was stir casting owing to its simplicity, low cost, and ability to produce a variety of geometries [17]. The elemental composition of the AA 6063 base metal is shown in Table 1. Since the aim of this investigation was to investigate the effect of the carbonization temperature on the aluminum alloy, the weight fraction of the carbonized eggshells was kept constant at 5 wt.%, while the carbonization temperature was varied. The metal sheets were cut and charged into a preheated graphite crucible and heated above its liquidus temperature to ensure uniform melting of the aluminum. The slag was removed and the melt was degassed with inert argon gas to reduce the tendency of the formation of an oxide layer. This is necessary to also reduce, or if possible avoid, the formation of intermetallic compounds. The varied carbonized eggshell samples were preheated in the muffle furnace for 1 h at 400 °C. This is important to improve the wettability and intermolecular bonding between the dispersed particles and the aluminum metal [18]. These properties were further improved by the addition of 1 wt.% magnesium to the melt [15]. The magnesium powder used for this work had a particle size distribution of 25 µm. The melt was stirred before casting to ensure uniform distribution of the reinforcements in the aluminum melt and before casting in the preheated steel die. The designation of the fabricated composites is summarized in Table 2.

Table 1. Elemental composition of the base metal.

Composition	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
%	0.5	0.07	0.45	0.02	1.02	0.14	0.6	0.02	Bal

Table 2. Designation of the cast samples.

Sample	Designation
AA 6063	C0
AA6063/5 wt.% (900 °C)	C1
AA6063/5 wt.% (1000 °C)	C2
AA6063/5 wt.% (1100 °C)	C3
AA6063/5 wt.% (1200 °C)	C4

2.3. Experimental Methodology

The mechanical properties considered for this investigation were microhardness and compressive strength. The Times Vickers microhardness tester was employed to determine the microhardness of the cast samples. A test force of 300 gF was applied using a diamond indenter for each indentation. The dwell time for each indentation was 15 s. There were 5 indentations 1 mm apart for each sample from which the average was then selected as the value of the microhardness for each sample. The compressive strength was measured using the Zwick/Roell Universal tensile testing machine. Three cylindrical specimens of 20 × 30 mm according to the ASTM-8 were employed for this study. All tests related to the determination of the mechanical properties of the samples under consideration were done at room temperature (23 °C).

The density of the cast samples was obtained via the Archimedes principle. This was done in 3 replicates. The theoretical density was obtained using the rule of mixtures, while the percentage porosity was obtained via the expression in Equation (1);

$$P = 1 - \frac{\rho_{experimental}}{\rho_{theoritical}} \times 100 \tag{1}$$

where P is the percentage porosity of the composites, $\rho_{experimental}$ is the experimental density, while $\rho_{theoritical}$ is the theoretical density.

The TESCAN model type VEGA 3 LMH scanning electron microscope was used to investigate the distribution of the dispersed particles in the aluminum matrix, the presence of defects that include the presence of pores, agglomeration, segregation, and formation of dendrites. The energy dispersive spectroscopy (EDS) analysis was conducted to ascertain the elemental composition of the cast composite samples. The X-ray diffraction analysis was used to perform phase identification analysis, and determine the position of the peaks and microstrain of the samples. The XRD analysis was conducted on the D8, Discover (Bruker, Billerica, MA, USA) with CuKα radiation of 1.54181 Â wavelength generated at 40 mA and 40 kV.

The tribology study was conducted using the Rtec MTF 5000 Universal ball-on-disc tribometer. This aspect of the characterization studies considered the effect of the carbonization temperature on the coefficient of friction (COF) and the volumetric wear rates. The selected wear ball was an E521000 steel alloy grade 25 of 6 mm diameter. The wear parameters selected for the study were 20 N, 120 mm, 120 s, and 1 mm/s for the applied load, sliding distance, sliding time, and sliding velocity, respectively. The volumetric wear rate was determined using the Archard equation shown in Equation (2).

$$K = \frac{V}{ws} \tag{2}$$

where K is the wear rate, V is the worn volume, w is the normal load, and s is the sliding distance.

The corrosion study for this investigation was conducted using the HCH Instruments electrochemical analyzer. The samples were prepared by attaching an insulated copper wire of a 1.5 mm cross-sectional area using conducting tape. Each specimen was cold mounted and ground with the 320, 500, 800, 1200, and 4000 grit SiC papers and subsequently washed in distilled water, degreased in acetone, and left to dry in air. To ensure that the prepared working electrode was conductive, a multimeter was used. The selected corrosion medium for the study was 3.5% NaCl. Each test was carried out in the corrosion medium at a temperature of 23 °C. The electrochemical analyzer was equipped with a silver/silver chloride reference electrode stored in KCl and a graphite counter electrode. For potentiodynamic polarization analysis, a polarization range of −1.5 to 1.5 V was used, while a scan rate of 0.01 V/s was employed.

3. Results and Discussion

3.1. Microstructure

The examination of the microstructure of the fabricated AMC samples was done to understand the level of dispersal of the carbonized eggshells in the aluminum matrix. The SEM micrographs of the cast composites are shown in Figure 1. The AMC samples were all characterized by the fairly uniform distribution of the carbonized eggshells in the aluminum matrix. The carbonized eggshells for all the samples were dispersed on the individual grains and along the grain boundaries of the AMCs. There was also an indication of the segregation of the carbonized eggshell along the grain boundaries of the cast samples. The micrographs also indicate that the agglomeration of the eggshells increased with increasing carbonization temperature. This was more prominent in samples C3 and C4. The agglomeration of the eggshells for samples C3 and C4 increased due to the increasing carbonization temperature of the eggshell particles. In this case, increasing the carbonization temperature resulted in the agglomeration of the eggshell particles. This agglomeration increased the average particle sizes of the eggshell particles. The addition of these eggshell particles was responsible for the segregation of some of the particles in the samples. The formation of pores in the cast AMCs was attributed to the entrapment of air bubbles during the fabrication of the composites. The presence of the pores in the cast AMCs is also a result of the segregation of the carbonized eggshell particles along the grain boundaries of the aluminum alloy.

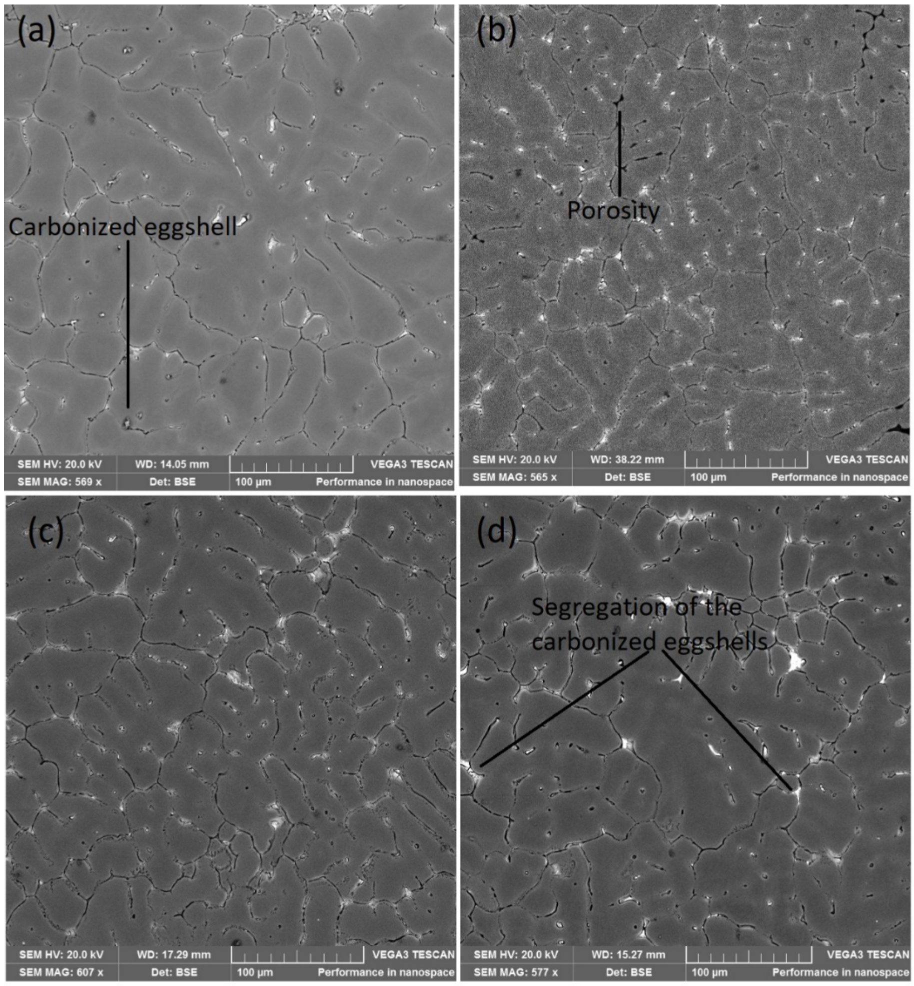

Figure 1. SEM morphology of the cast AMCs. (**a**) C1 (900 °C); (**b**) C2 (900 °C); (**c**) C3 (1100 °C); (**d**) C4 (1200 °C).

The EDS analysis was used to determine the elemental composition of the cast samples. The EDS analysis shown in Figure 2 shows that the cast AMC samples comprised of major elements including aluminum, carbon and oxygen in varying proportions. Carbon and oxygen were present due to the presence of the carbonized eggshells in the cast samples. Also recorded in the cast AMC samples were elements including copper, zinc, and magnesium, which were present as alloying elements present in the aluminum alloy. The EDS revealed the absence of calcium.

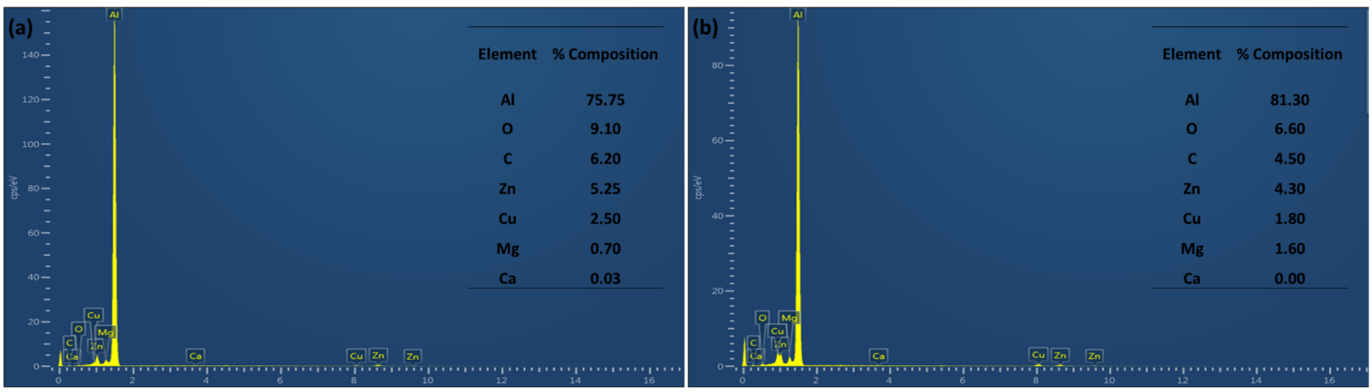

Figure 2. *Cont.*

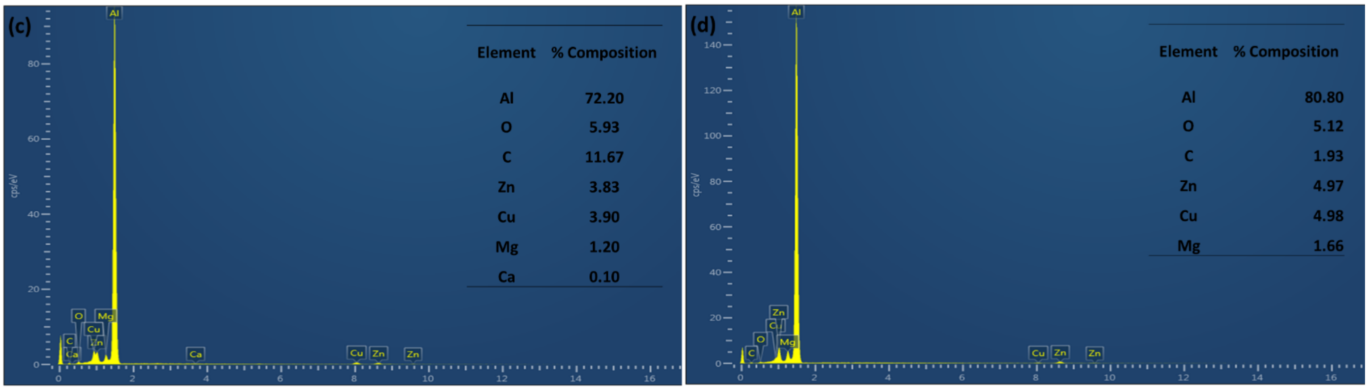

Figure 2. EDS analysis of the cast AMCs. (**a**) C1 (900 °C); (**b**) C2 (900 °C); (**c**) C3 (1100 °C); (**d**) C4 (1200 °C).

The X-ray diffraction spectrum of the cast samples is shown in Figure 3. The plots revealed mostly peaks of aluminum in all the samples under consideration. The phase analysis of the AMCs showed no formation of intermetallic compounds. The absence of intermetallic compounds and Ca peaks shows that there were no new relevant peaks formed in any of the cast samples due to the addition of the carbonized eggshell to the aluminum matrix. The XRD plot was characterized by the formation of five major peaks of aluminum formed at approximate peak positions of 38°, 44°, 65°, 78°, and 82°. Using Scherrer's equation, the crystallite size of the cast AMCs was obtained. The results show that the crystallite size for samples C1, C2, C3, and C4 was 689, 663, 548, and 637 nm respectively. Compared to the crystallite size of the unreinforced alloy which was 501.78 nm, the addition of the carbonized eggshell resulted in the increase in the crystallite size, which is an indication of grain structure refinement of the aluminum alloy.

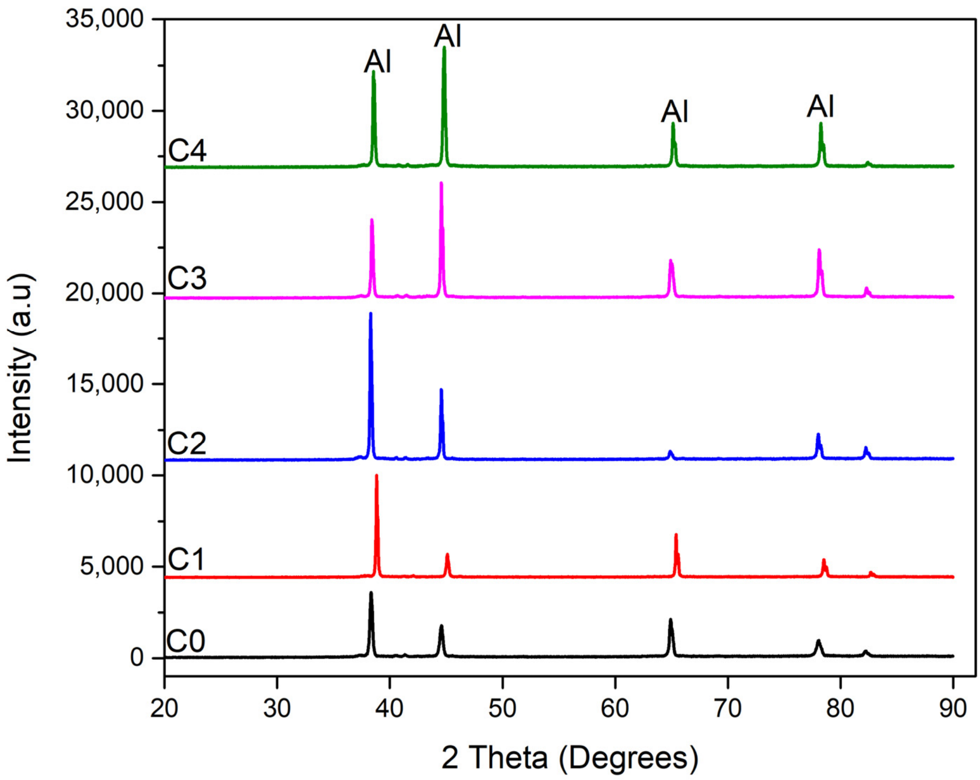

Figure 3. X-ray diffraction analysis of the base metal and AMCs.

3.2. Density and Porosity

The density of the eggshell ash at the different carbonization temperatures was obtained with the gas pycnometer. The densities obtained were 2.97, 2.88, 2.88, and 3.70 g/cm^3 for the eggshell samples carbonized at 900, 1000, 1100, and 1200 °C, respectively. The incorporation of the carbonized eggshell particles into the aluminum matrix was capable of decreasing the weight of the resulting AMCs as evident in the reduced density compared to the base metal. The results are summarized in Figure 4. From the analysis, it was concluded that the density of the carbonized eggshell is directly proportional to the density of the corresponding AMC. This statement is highlighted by the results of the density measurements. The lowest density for the cast AMCs was obtained as 2.56 g/cm^3 for sample C2, while sample C4 recorded the highest density of 2.67 g/cm^3. The porosity analysis was used to obtain the percentage volume of pores in respect to the total volume of the investigated sample. The results of the porosity analysis summarized in Figure 4 shows that the presence of the carbonized eggshell samples resulted in the formation of pores which are present as void formed due to trapped gases during fabrication and segregation/agglomeration of the carbonized eggshell samples. The percentage porosity was 3.45, 5.50, 4.39, and 2.91% for samples C1, C2, C3, and C4, respectively.

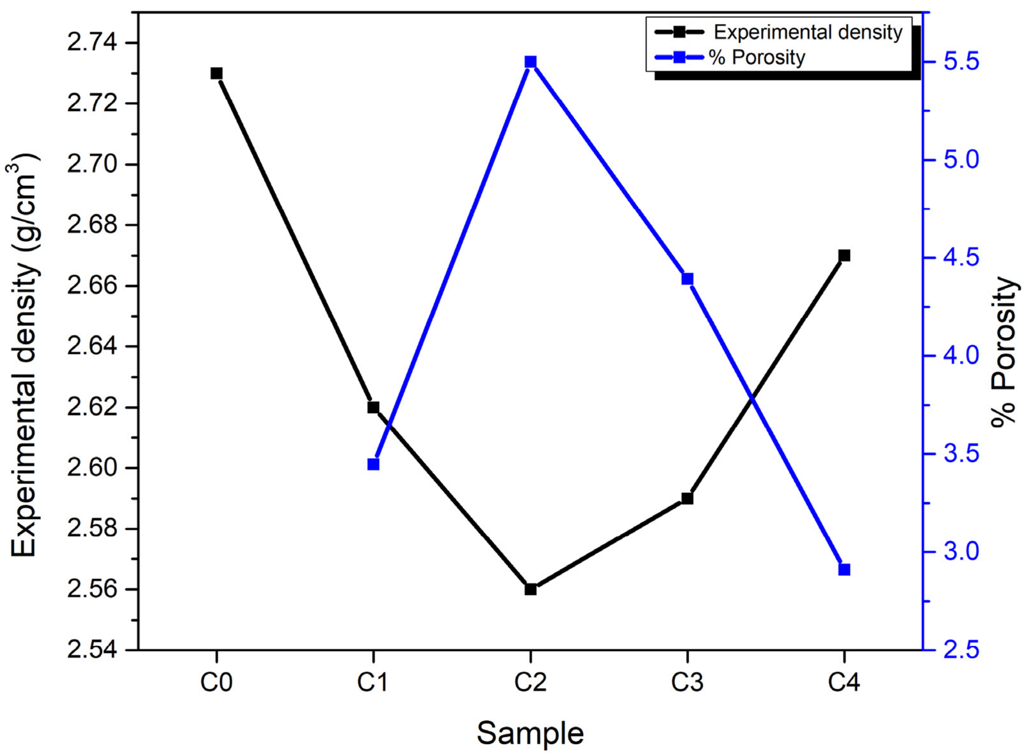

Figure 4. Experimental density and % porosity of the cast samples.

3.3. Mechanical Properties

The evaluation of the microhardness of the cast samples summarized in Figure 5 shows that the inclusion of carbonized eggshells is capable of improving the hardness of aluminum and its alloys. The average microhardness for the base metal, C1, C2, C3, and C4 was 83.85, 118.05, 103.08, 105.40, and 108.53 HV, respectively, indicating that all carbonized eggshell particles improved the hardness of the aluminum alloy by more than 20%. The improvement in the microhardness was brought about by several factors including the dispersal of the reinforcement in the aluminum matrix, adequate wettability, and interfacial bonding between the aluminum matrix and the eggshell particles. The presence of the carbonized eggshell particles works to resist the application of localized load on the fabricated composite. The hard reinforcing eggshell particles also improves the hardness of the matrix by working to resist the movements between the individual grain during the application of localized load. This resistance is brought about due to the of the distribution of the reinforcements between individual grains. The compressive strength analysis revealed that the carbonized eggshell particles improved the property in comparison to the base metal. This improvement was brought about by hardening of the aluminum alloy due to the presence of the reinforcing particles and effective load transfer from the ductile aluminum alloy to the hard-brittle eggshell phase. The carbonization temperature has no significant effect on the compressive strength of the AMCs, although the highest compressive strength of 132.81 MPa was recorded for the 900 °C carbonized eggshell sample.

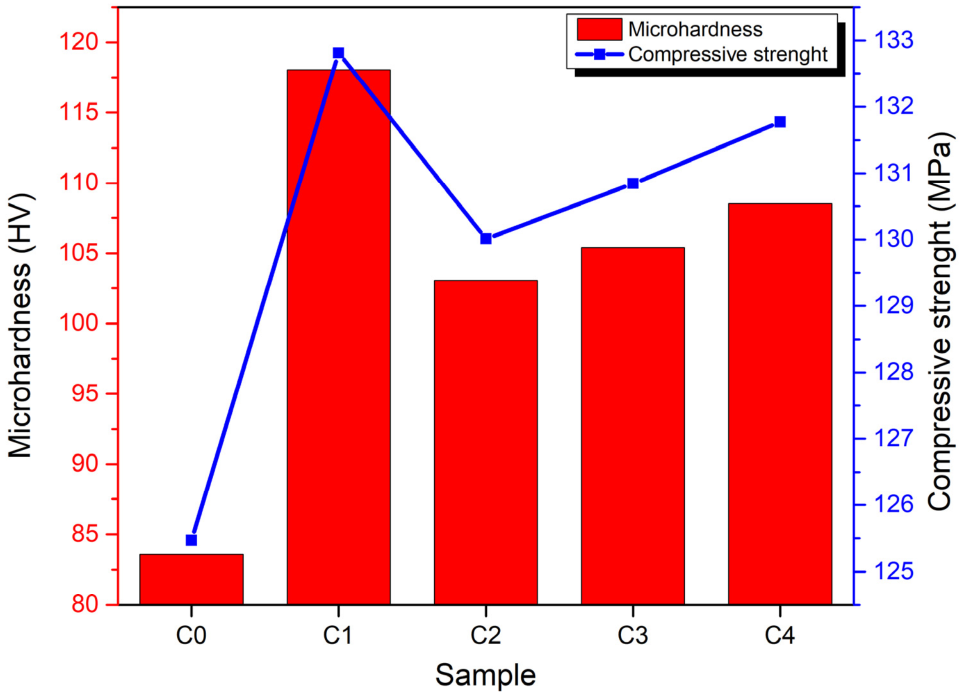

Figure 5. Microhardness and compressive strength of the cast samples.

3.4. Wear

The tribology study of the cast samples under consideration was done by analyzing the coefficient of friction (COF) and the volumetric wear rates. The wear rates were obtained using Ackards's equation shown in Equation (2). Results from the COF depicted in Figure 6 show that the wear-resistant properties of samples C1 and C4 (900 and 1200 °C, respectively) were improved relative to the base metal. However, the wear resistance of samples C2 and C4 was lower than that of the base metal. These results are summarized in the volumetric wear rates shown in Figure 7, which has also shown that the lowest wear rate of 2.70×10^{-4} mm^3/Nmm was obtained for sample C4. The second-best sample was sample C1 with a wear rate of 4.46×10^{-4} mm^3/Nmm. These results translate to a decrease in wear rate of 19.98 and 51.35%, respectively, for samples C1 and C4. The COF curves were used to analyze the mechanics of the wear activities during the wear experiment. All the cast samples under consideration experienced a rapid spike in the coefficient of friction owing to the initiation of the siding movement of the steel counterface against the surface of the samples. The improvement in the wear resistance of samples C1 and C4 was due to the presence of the hard reinforcing carbonized eggshell particles, which work to resist the flow of the ductile aluminum alloy during the sliding motion of the steel counterface. This is attributed to the relatively lower porosity and the strong interfacial bonds formed between the aluminum alloy and the reinforcements. Another reason for the improved wear resistance is the reduction of the contact area on the aluminum alloy brought about by the presence of the carbonized eggshell for all the cast samples. The COF was relatively stable after the sudden spike at the commencement of the wear activities due to the formation of an Al_2O_3 barrier, which reduced the contact between the counterface and the surface of the samples, thereby stabilizing the COF. The increased wear rate of samples C2 and C3 is a combination of increased porosity owing to the segregation of the carbonized eggshell particles and the possibly weaker interfacial bonding brought about by the lower wettability between the matrix and the reinforcing particles.

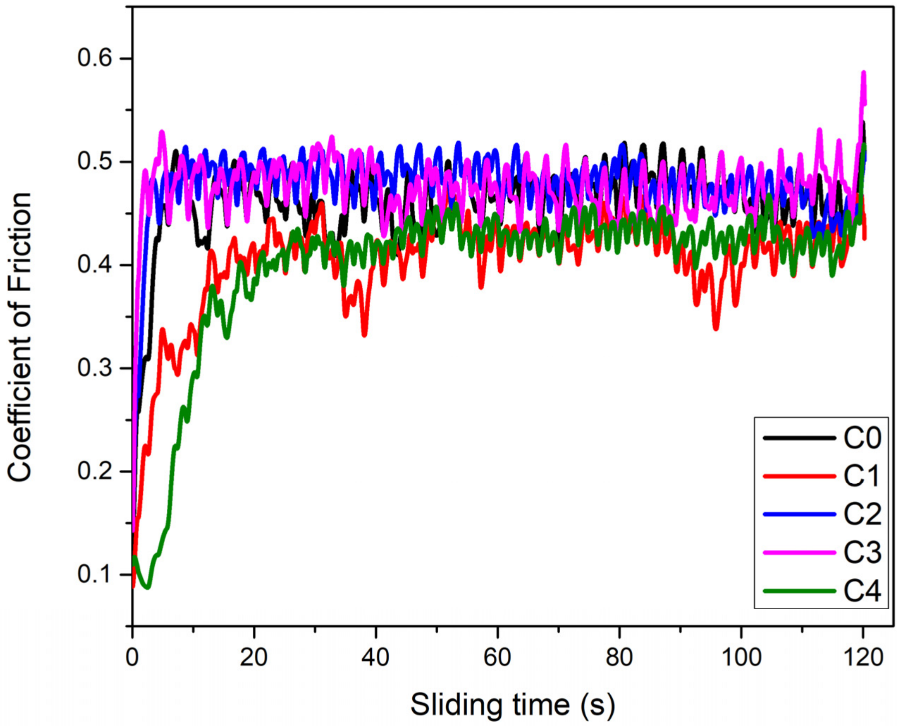

Figure 6. Coefficient of friction of the cast samples.

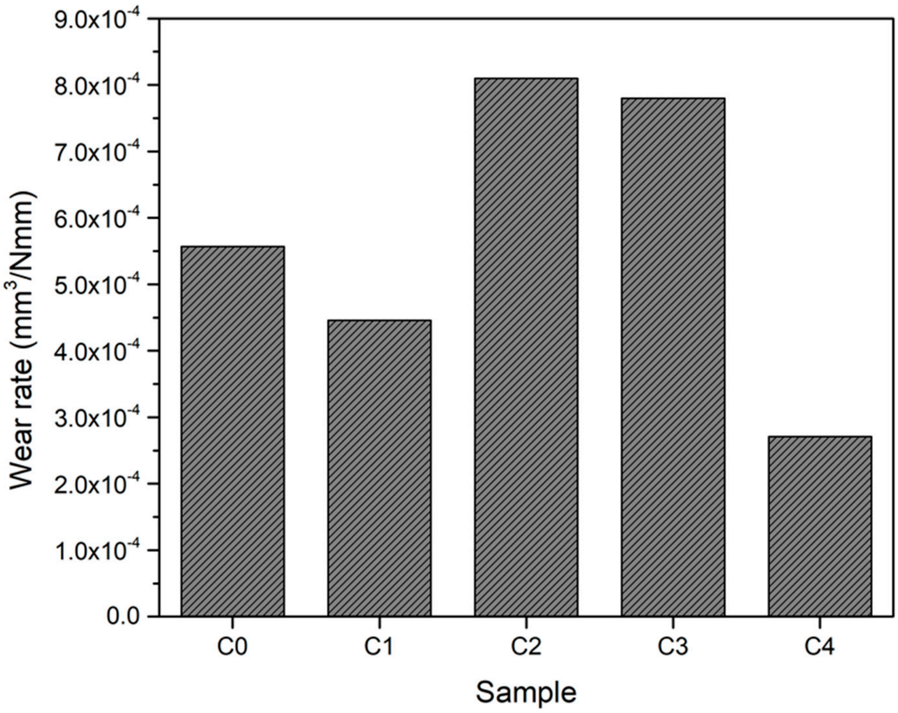

Figure 7. Volumetric wear rate of the cast samples.

3.5. Corrosion

Results of the potentiodynamic polarization studies conducted on all the samples under consideration are shown in Figure 8. The summary of the results from the corrosion studies shown in Table 3 was obtained by extrapolating the cathodic and anodic Tafel curves at the corrosion potential for studied samples. The Tafel plots indicate the presence of passive and active corrosion activities. The excellent corrosion resistance of aluminum and its alloys is due to the formation of an oxide passive layer on its surface, which works to resist the oxidation of the metal during corrosion activities. However, increased exposure of the metal to the corrosion environment eventually leads to the deterioration and eventual disintegration of the formed passive layer to initiate localized pitting. The corrosion rates obtained from the Tafel plots indicate that the carbonization temperature is an important factor in the corrosion resistance improvement of the carbonized eggshell reinforced AA 6063 matrix. The corrosion resistance for samples C1 and C2 were 4.08×10^{-8} g/hr and 1.74×10^{-6} g/hr respectively, which were lower than that of the base metal. It was also shown that the corrosion rate increased with increasing carbonization temperature up to 1000 °C. The improvement in the corrosion resistance for samples C1 and C2 was due to the even distribution of the reinforcement. The corrosion resistance is also improved due to the chemical inertness of the carbonized eggshell particles, which decreases the effect of the chloride ions during exposure in the corrosion medium. The corrosion density (Icoor), which signifies the tendency for the initiation of corrosion activities was also used as an indicator for the corrosion behavior of the studied samples. The corrosion density of the samples followed the same trend as the corrosion rates already discussed. The current density was lowest for sample C1 at 1.21×10^{-7} A/cm^2.

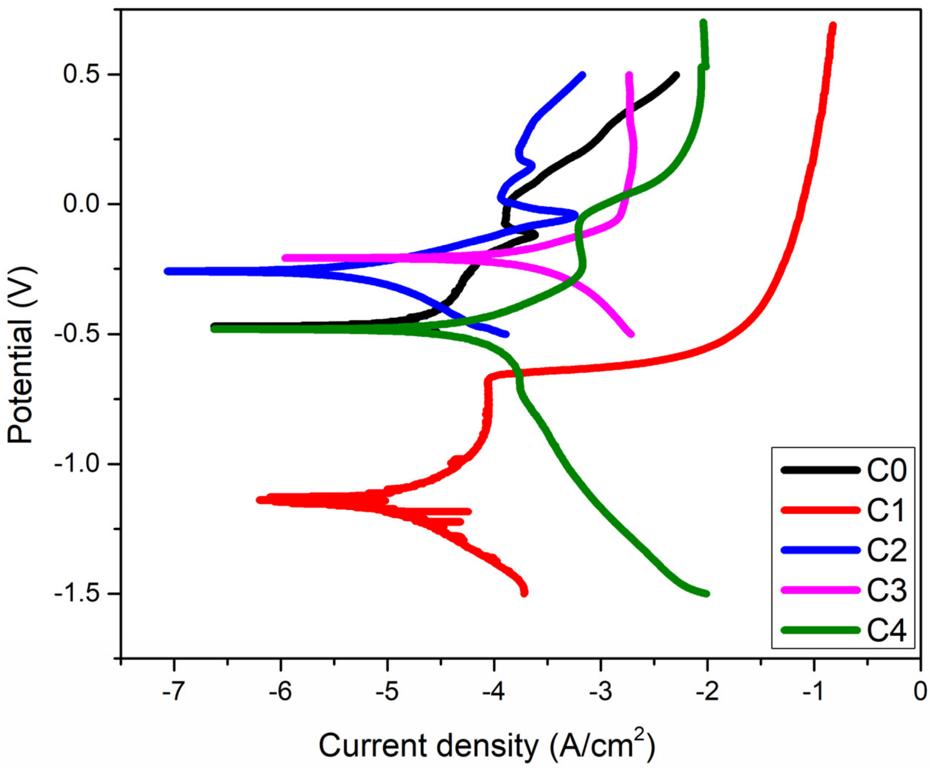

Figure 8. Potentiodynamic polarization curves for the cast samples.

Table 3. Summary of the results from the potentiodynamic polarization test.

Sample	Linear Polarization Resistance (Ω)	Corrosion Rate (g/hr)	Corrosion Density (Icoor)	Potential (Ecoor)
C0	1144	5.53×10^{-6}	1.64×10^{-5}	−0.471
C1	331,405	4.08×10^{-8}	1.22×10^{-7}	−1.144
C2	5461	1.74×10^{-6}	5.17×10^{-6}	−0.259
C3	165	6.92×10^{-5}	2.06×10^{-4}	−0.208
C4	574	2.59×10^{-5}	7.72×10^{-5}	−0.481

4. Conclusions

This paper investigated the effect of varying carbonization temperature of eggshells on the mechanical properties, physical properties, wear and corrosion resistance of AA 6063. The results of the investigation showed that the reinforcements were distributed along the grain boundaries of the aluminum matrix. The increased carbonization temperature increased the segregation of the eggshells in the aluminum matrix. The density was lowest for the sample carbonized at 1200 °C. The porosity was also lowest for that sample. The addition of the carbonized eggshell to the aluminum matrix improved the microhardness by up to 20%. The compressive strength was highest for the 900 °C sample. The corrosion and wear resistance were lowest at 4.08×10^{-8} g/hr for the 900 °C sample and 2.70×10^{-4} mm^3/Nmm for the 1200 °C, respectively.

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