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Effects of waste eggshells and SiC addition in the synthesis of aluminum hybrid green metal matrix composite

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Abstract: The mechanical behavior, physical behavior, microstructural characteristics, and corrosion behavior of AA2014/silicon carbide (SiC)/carbonized eggshell hybrid green metal matrix composites (MMCs) were investigated. Twenty-five samples of hybrid composite with different combinations of SiC and carbonized eggshell particles in AA2014 matrix alloy were prepared. Microstructure presents that the reinforcement particles (SiC and eggshells) are uniformly distributed in the matrix AA2014 alloy. Transmission electron microscope image shows proper wettability between SiC, carbonized eggshell, and AA2014 aluminum alloy. The tensile strength and the fatigue strength for the composites containing 2.5 wt.% SiC up to 7.5 wt.% carbonized eggshell were observed to be higher than that of the other selected composites. The hardness values for the composites containing 12.5 wt.% SiC and 2.5 wt.% carbonized eggshell were in all cases higher than that of the other composites. The results show that toughness decreases with the increase in the weight ratio of SiC and carbonized eggshell in the composites. The results reveal that the sample of AA2014/2.5% SiC/12.5% carbonized eggshell shows minimum corrosion rate among all the selected samples. Density, porosity, and overall cost of hybrid metal matrix composites were also calculated to see the effects of carbonized eggshell and SiC addition in AA2014 matrix alloy.

Keywords: corrosion; density; green hybrid MMC; waste eggshells; wettability.

1 Introduction

The modern development in the field of science and technology demands the developments of advanced engineering materials for various engineering applications, especially in the field of transportation, aerospace, and military engineering related areas. These area demands light weight high strength having good properties. Such demands can only be met by the development and processing of aluminum metal matrix composite (MMC) materials. The main challenge in the development and processing of engineering materials is to control the microstructure, mechanical properties, and cost of the product by optimizing the chemical composition, processing method, and heat treatment [1–5].

A lot of environment pollution due to the waste of industries/societies encourages our societies to use these waste products in research areas. More recent advancements involved the use of waste or recycling materials such as fly ash, red mud, rice hull ash, bagasse ash, basalt fiber, breadfruit seed hull ash, maize stalk waste, and eggshell waste particle. These raw materials offer great opportunities because synthesized reinforcements can be produced *in situ* economically. Chicken eggshell waste is an industrial by-product, and its disposal constitutes a serious environmental hazard. Chicken eggshell can be used in commercial products to produce new materials, and it has been highlighted in recent investigation because of its renovation prospective. Although there have been several attempts to use chicken eggshell components for a variety of applications, its chemical composition and accessibility make chicken eggshell a probable source of biofiller-reinforced composites, giving additional or improved thermal and mechanical properties. The other advantages of using chicken eggshell are its availability in bulk quantity with lightweight and being economical and environmental friendly [6–8]. Reviews of various hybrid metal matrix composites with different reinforcements are shown in Table 1 [9–19].

In the previous work [20], AA2014/carbonized eggshell green MMCs were fabricated. Mechanical properties of aluminum MMCs with reinforced eggshells (AA2014/

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Table 1: Hybrid metal matrix composites with different reinforcements [9–19].

Reference	Matrix material (theoretical density in g/ml)	Hybrid MMCs (theoretical density in g/ml)
[9]	Al2219 = 2.84	Al2219/15% SiC/3% graphite composites = 2.87
[10]	AlSi12CuMgNi = 3.3	AlSi12CuMgNi/12% Al ₂ O ₃ /4% carbon fiber composites = 3.22
[11]	Al6061 = 2.70	Al6061/5% Al ₂ O ₃ /5% Graphite = 2.74
[12]	A356 = 2.68	A356/10% SiC/5% B ₄ C MMC = 2.73
[13]	Al6061 = 2.70	Al6061/7.5% fly ash/10% SiCp = 2.74
[14]	A413 = 2.8	A413/4.5% fly ash/4.5% B ₄ C = 2.78
[15]	Al2024 = 2.78	2024Al/(5% Al ₃ Zr + 5% Al ₂ O ₃ np) = 2.90
[16]	Al6063 = 2.70	Al6063/5 wt% SiC/2.5 wt% Al ₂ O ₃ /2.5 wt% Gr composite: 2.75
[17]	Al = 2.7	Al/15 wt.% fly ash/1.5 wt.% graphite (Gr) = 2.66
[18]	LM25Al = 2.68	LM25Al + 7.5% SiC + 5% TiO ₂ = 2.79
[19]	A356 = 2.68	A356/10% SiC/5% RHA MMC = 2.70

Table 2: Chemical composition of AA2014 alloy (wt.%).

Si	Fe	Cu	Mn	Mg	Zn	Ti	Ni	Cr	Al
0.5–0.9	0.5	3.9–5.0	0.4–1.2	0.2–0.8	0.25	0.2	0.1	0.1	Balance

carbonized eggshells) can be further improved by adding silicon carbide (SiC) particles with low density, as the density of SiC is lower than Al₂O₃ and higher than B₄C. SiC is composed of tetrahedra of carbon and silicon atoms with strong bonds in the crystal lattice. This produces a very hard and strong material. The high thermal conductivity coupled with low thermal expansion and high strength gives these material exceptional thermal shock-resistant qualities.

On the basis of literature review (Table 1), it is found that no researcher has investigated the mechanical and physical properties of AA2014/SiC/carbonized eggshell green hybrid MMC. From the literature, it was observed that in most of cases, densities and cost of hybrid composites increased. Hence, in view of the above facts, an investigation was conducted to find the effects of SiC and carbonized eggshell addition on the physical and mechanical properties of green hybrid metal matrix composite.

particularly poor. Chemical composition and mechanical properties are shown in Tables 2 and 3, respectively [20].

2.2 Primary reinforcement material

In the present investigation, carbonized eggshell powder was selected for primary reinforcement material. Figure 1A shows the photograph of hen eggshells. Hen eggshells naturally consist of ceramic materials. The chemical composition (by weight) of a by-product eggshell has been reported as follows: calcium carbonate (94%), magnesium carbonate (1%), calcium phosphate (1%), and organic matter (4%). The hen eggshell was cleaned and sun dried to eliminate the covering layer of eggshell [20]. The dried eggshell was ball milled to obtain eggshell powder as shown in Figure 1B. It was then carbonized to 500°C for 3 h to remove the carbonaceous materials, as shown in Figure 1C. The densities of uncarbonized and carbonized eggshell particles are 2.47 and 2.0 g/cm³, respectively.

2.3 Secondary reinforcement material

SiC is selected as a secondary reinforcement to further improve the mechanical properties of composites. The introduction of SiC

2 Materials and methods

2.1 Matrix material

In the present investigation, AA2014 aluminum alloy was selected as a matrix material. AA2014 aluminum alloy is an aluminum-based alloy often used in the aerospace industry. It is easily machined in certain tempers and among the strongest available aluminum alloys. It also has high hardness. The corrosion resistance of this alloy is

Table 3: Measured properties of AA2014 alloy.

Melting point	640°C
Density (g/cm ³)	2.8
Tensile strength (MPa)	185
Hardness (BHN)	60
Toughness (J)	12
Ductility (percentage elongation)	13
Fatigue strength (MPa) for 1 × 10 ⁷ cycles	90

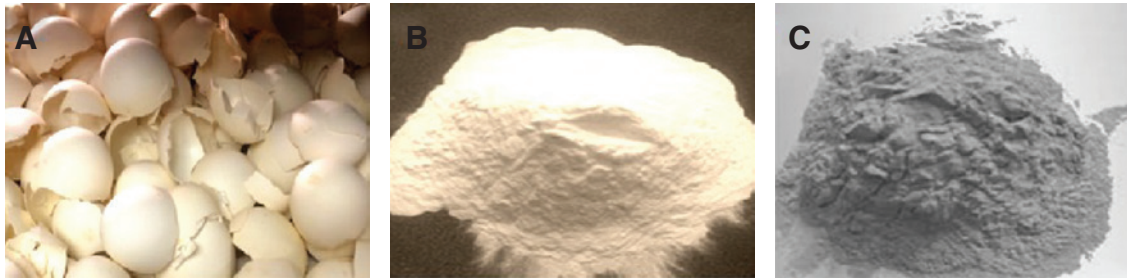


Figure 1: Photographs of (A) eggshells, (B) eggshell powder, and (C) carbonized eggshell powder.

to the aluminum matrix significantly enhances strength, modulus, abrasive wear resistance, and thermal stability. The density of SiC (3.2 g/cm^3) is nearer to that of aluminum alloy AA2014 (2.8 g/cm^3). The resistance of SiC to acids, alkalis, or molten salts up to 800°C makes it a good reinforcement candidate as a secondary reinforcement for aluminum based Metal matrix composites (MMCs). In addition, SiC is simply available and has good wettability with aluminum alloys [1].

The size of each reinforcement particles is selected at $25 \mu\text{m}$. However, it is not easy to obtain the exact particle size ($25 \mu\text{m}$) of all reinforcement particles (SiC and carbonized eggshell powder). Hence, the particle size with a standard deviation of $2 \mu\text{m}$ (mean, $25 \mu\text{m}$) was selected. SiC particles were directly purchase from the market, with a mean particle size of $25 \mu\text{m}$ (SD, $2 \mu\text{m}$). Carbonized eggshell powder was ball milled to obtain uniform particle size (mean, $25 \mu\text{m}$; SD, $2 \mu\text{m}$).

2.4 Composition selection

On the basis of the results based on pilot investigations, the compositions of reinforcements (SiC and carbonized eggshell) were selected and are shown in Table 4. The total percentage of both reinforcements varies from 2.5 to 12.5 wt.% fraction in AA2014 matrix material. If the weight percentage of reinforcement increases more than 12.5%, then no more effects on the physical and mechanical properties of hybrid MMC were observed.

2.5 Processing of hybrid MMCs

AA2014/carbonized eggshells/SiC hybrid green MMC used in this study was fabricated by electromagnetic stir casting technique at

Table 4: Sample designation and reinforcement weight ratio.

Sample no.	Sample designation	SiC (wt.%)	Eggshell (wt.%)
1	G1-AA2014/2.5% SiC/2.5% eggshell	2.5	2.5
2	G2-AA2014/2.5% SiC/5% eggshell	2.5	5
3	G3-AA2014/2.5% SiC/7.5% eggshell	2.5	7.5
4	G4-AA2014/2.5% SiC/10% eggshell	2.5	10
5	G5-AA2014/2.5% SiC/12.5% eggshell	2.5	12.5
6	G6-AA2014/5% SiC/2.4% eggshell	5	2.5
7	G7-AA2014/5% SiC/5% eggshell	5	5
8	G8-AA2014/5% SiC/7.5% eggshell	5	7.5
9	G9-AA2014/5% SiC/10% eggshell	5	10
10	G10-AA2014/5% SiC/12.5% eggshell	5	12.5
11	G11-AA2014/7.5% SiC/2.5% eggshell	7.5	2.5
12	G12-AA2014/7.5% SiC/5% eggshell	7.5	5
13	G13-AA2014/7.5% SiC/7.5% eggshell	7.5	7.5
14	G14-AA2014/7.5% SiC/10% eggshell	7.5	10
15	G15-AA2014/7.5% SiC/12.5% eggshell	7.5	12.5
16	G16-AA2014/10% SiC/2.5% eggshell	10	2.5
17	G17-AA2014/10% SiC/5% eggshell	10	5
18	G18-AA2014/10% SiC/7.5% eggshell	10	7.5
19	G19-AA2014/10% SiC/10% eggshell	10	10
20	G20-AA2014/10% SiC/12.5% eggshell	10	12.5
21	G21-AA2014/12.5% SiC/2.5% eggshell	12.5	2.5
22	G22-AA2014/12.5% SiC/5% eggshell	12.5	5
23	G23-AA2014/12.5% SiC/7.5% eggshell	12.5	7.5
24	G24-AA2014/12.5% SiC/10% eggshell	12.5	10
25	G25-AA2014/12.5% SiC/12.5% eggshell	12.5	12.5

parameters of 12 A (current), 180 s (time), and 700°C (matrix pouring temperature) and immediately extruded on UTM machine at 60 MPa using cylindrical H13 tool steel die coated with graphite [20]. It has been reported by various researchers that with sufficient time, any type of ash has the ability to absorb moisture from the surrounding air. Basically, when any type of reinforcement was mixed with matrix alloy, then with reinforcement some atmospheric air was also entered in matrix alloy. Carbonized eggshell particles have the ability to absorb the moisture, resulting minimum porosity obtained. Because carbonized eggshell particles have very good reactivity (ability to absorb the air moisture) to air at elevated temperature, the air trapped with carbonized eggshell particles reacts with carbonized eggshell inside the AA2014 melt, resulting in very low porosity formed inside the hybrid MMC. In the present investigation, a precipitation hardening process (heat treatment technique) was used to improve further mechanical properties. The heat treatment process was conducted at a solution time of 4.5 h, an aging temperature of 250°C, and an aging time of 13.5 h [3].

2.6 Corrosion test

The corrosion test of all fabricated composites was conducted in a bath tub with high alkalinity. All samples of composites were immersed in a bath tub with high alkalinity at room temperature 120 h in 3.5 wt.% NaCl aqueous solution. Corrosion rate was calculated at assuming uniform corrosion over the entire surface of the composites condition. The corrosion rate in millimeters per year (mmy) was calculated from the weight loss using the following equation [21, 22], where the constant (K) can be varied:

$$\text{Corrosion rate (CR)} = \frac{\text{Weight loss (g)} \times K}{\text{Alloy density (g/cm}^3) \times \text{Exposed area (A)} \times \text{Exposure time (h)}}$$

In the given equation, $K = 8.75 \times 10^4$, exposed area = 9 cm², and exposure time = 120 h.

2.7 Porosity analysis

The experimental densities of the composites were determined by means of the Archimedes principle. The theoretical densities of composites were calculated using a rule of mixtures, given as follows:

$$\begin{aligned} \text{The theoretical density of hybrid MMC} &= \text{wt. fraction of AA2014} \\ &\times \text{density of AA2014} + \text{wt. fraction of SiC} \times \text{density of SiC} \\ &+ \text{wt. fraction of carbonized eggshell} \\ &\times \text{density of carbonized eggshell.} \end{aligned} \quad (1)$$

Porosity (P) is the percentage of the pore volume to the total volume with the volume of a substance [3]. It is defined by

$$P = \left(1 - \frac{\rho_{\text{Experimental}}}{\rho_{\text{Theoretical}}} \right) \times 100 \% \quad (2)$$

3 Results and discussion

3.1 Mechanical behavior

The mechanical properties of AA2014/carbonized eggshell/SiC hybrid green MMCs produced are presented in Figures 2–5. It can be observed from Figure 2 that after the addition of carbonized eggshell particles up to the weight fraction of 7.5% in AA2014/2.5% SiC composites, the

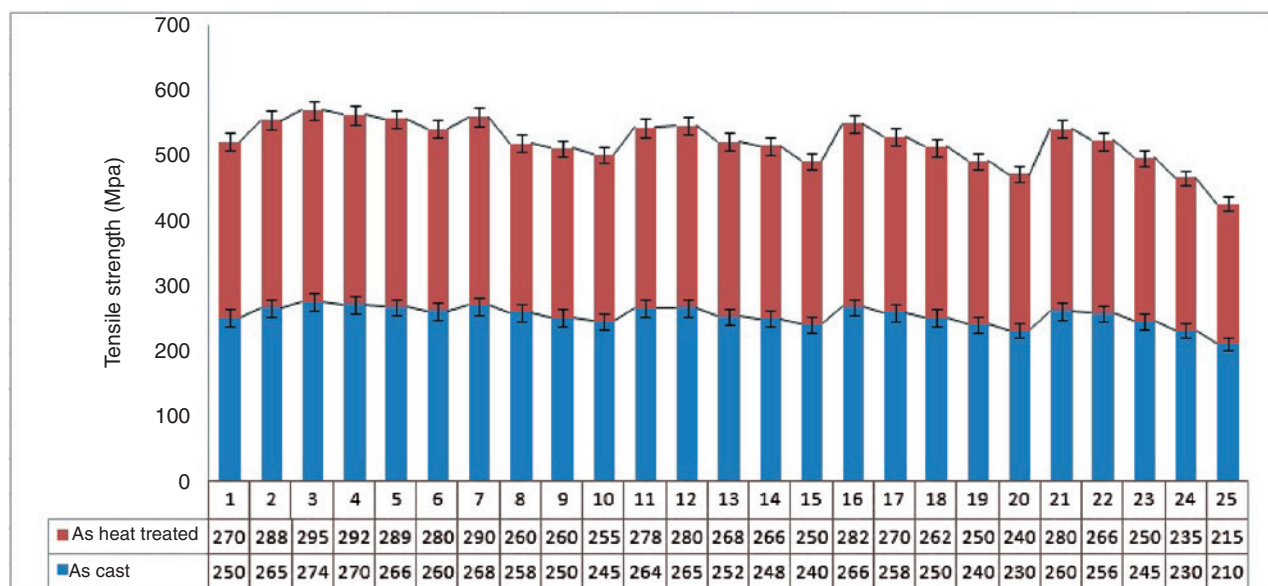


Figure 2: Tensile strength of hybrid green MMCs.

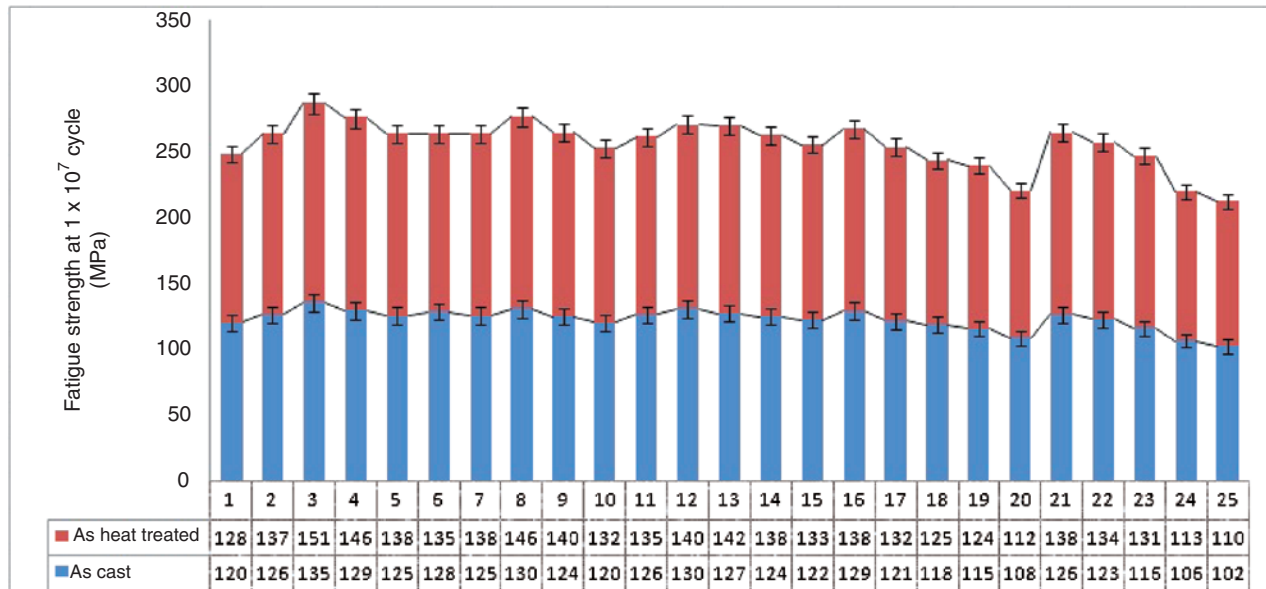


Figure 3: Fatigue strength of hybrid green MMCs.

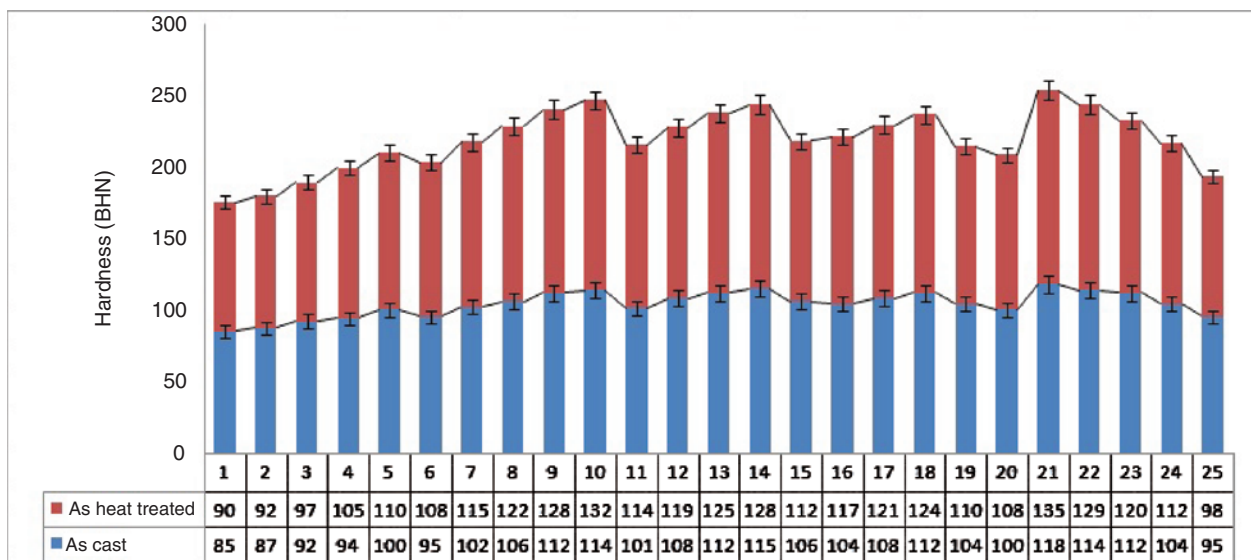


Figure 4: Hardness of hybrid green MMCs.

tensile strength of hybrid green metal matrix composite is maximum (274 MPa). Further, by increasing the weight fraction of carbonized eggshell particles in AA2014/2.5% SiC composite, tensile strength continues to decrease, and it is lowest at the 12.5% weight fraction of eggshell and 10% weight fraction of SiC. The presence of reinforcements (carbonized eggshell/SiC) produces a significant increase in the work hardening of the material during tensile testing. This increase in work hardening is more significant at a higher weight fraction of carbonized eggshell particles (presence of CaCO_3 , Cu, Al_4C_3 , SiC, CaSiO_3 ,

Al, MnO_2 , Al_2O_3 , and Mg_2SiO_4 , as shown in Figure 6). However, it decreases at a higher weight fraction of carbonized eggshell and SiC (more than 7.5% of carbonized eggshell and more than 2.5% of SiC). This may be due to the clustering of carbonized eggshell particles in AA2014/SiC composite. It can be observed that after the addition of SiC particles in AA2014/eggshell composite, the heat-treatable property of green MMC significantly improved. After the heat treatment process, approximately 7.66% tensile strength improved as compared with cast AA2014/7.5% eggshell/2.5% SiC green metal composite. However, after

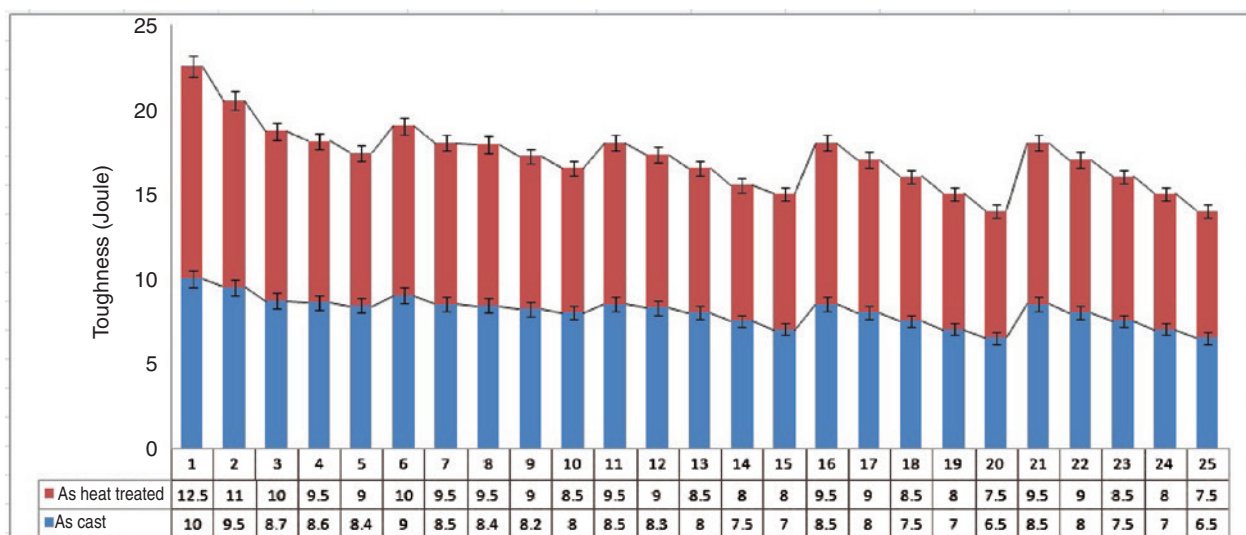


Figure 5: Toughness of hybrid green MMCs.

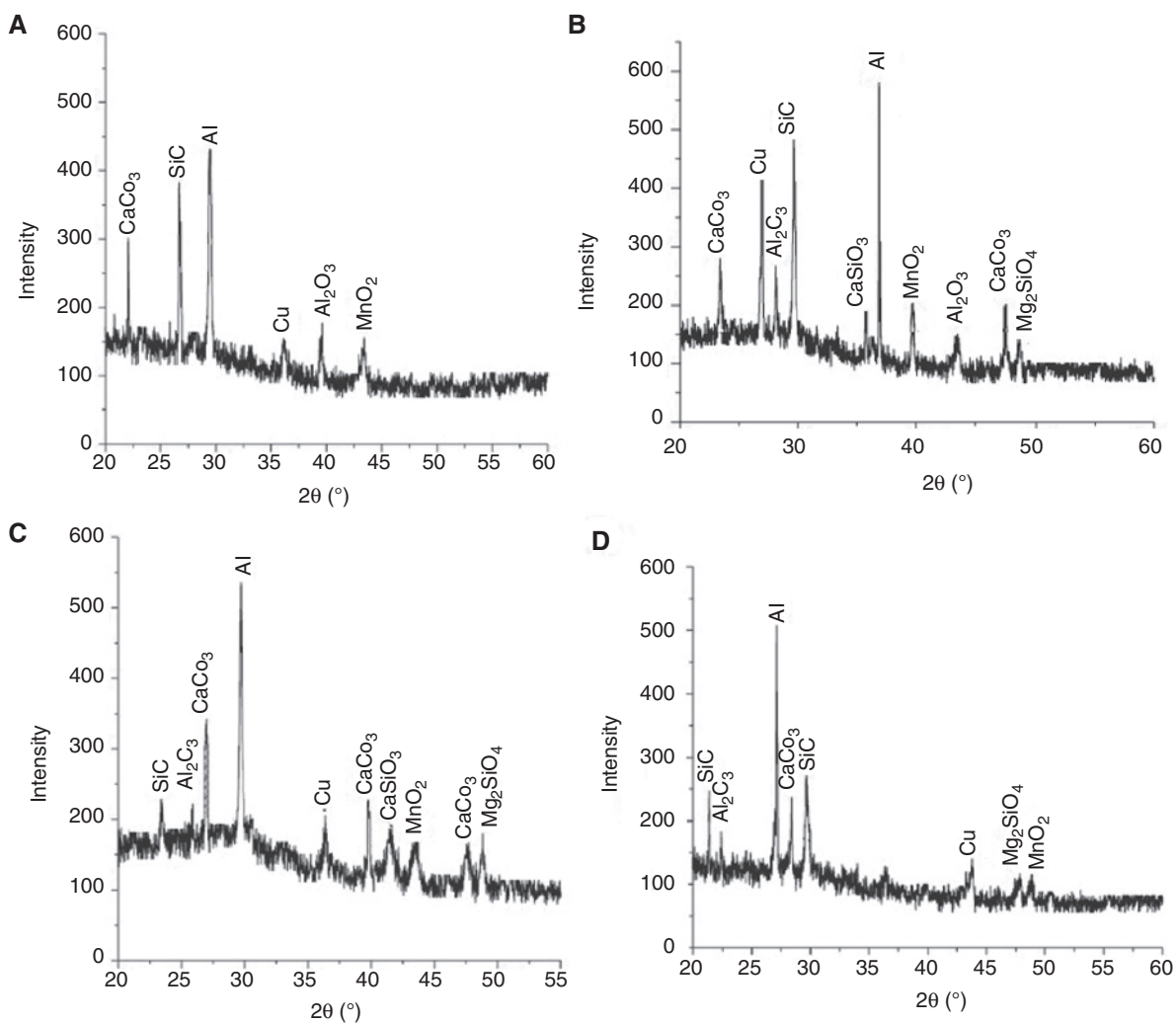


Figure 6: XRD results of (A) sample designation G1, (B) sample designation G3, (C) sample designation G5, and (D) sample designation G21.

the heat treatment of AA2014/7.5% eggshell/2.5% SiC green metal composite, approximately 62.22% tensile strength improved as compared with matrix alloy AA2014. Maximum fatigue strength was also observed for sample designation G3 (AA2014/7.5% eggshell/2.5% SiC green metal composite), as shown in Figure 3. After the heat treatment of AA2014/7.5% eggshell/2.5% SiC green metal composite, approximately 67.77% fatigue strength improved as compared with matrix alloy (90 MPa).

From Figure 4, it is observed that there is a general increase in hardness with the increase in the weight fraction of SiC particles in AA2014/2.5% carbonized eggshell composite. Maximum hardness was observed for sample designation G21 (AA2014/2.5% eggshell/12.5% SiC green metal composite). The X-ray powder diffraction (XRD) of AA2014/2.5% eggshell/12.5% SiC hybrid green MMC was conducted, as shown in Figure 6. XRD results showed the presence of SiC, Al_4C_3 , Al, CaCO_3 , Cu, Mg_2SiO_4 , and MnO_2 phases in hybrid green metal matrix composites. It was reported by various previous researcher that the presence of SiC, Al_4C_3 , Al, CaCO_3 , Cu, Mg_2SiO_4 , and MnO_2 phases significantly increases the hardness of composite. Approximately 125% hardness improved after the heat treatment as compared with matrix alloy. It is observed that the toughness (Figure 5) decreases with the addition of carbonized eggshell particles and SiC particles in AA2014 aluminum alloy. The reduction in toughness may be due to the presence of hard phases (Figure 6) in the hybrid MMC.

3.2 Microstructure analysis

Maximum tensile strength and fatigue strength were observed for AA2014/7.5% eggshell/2.5% SiC composite, whereas maximum hardness was observed for AA2014/2.5% eggshell/12.5% SiC composite. AA2014/12.5% eggshell/2.5% SiC composite shows minimum corrosion rate, whereas AA2014/2.5% eggshell/2.5% SiC composite shows maximum toughness. Hence, in view of the above facts, the microstructures of sample designations G1, G3, G5, and G21 were conducted. Figures 7 and 8A–D show a typical microstructure of AA2014 reinforced with SiC and carbonized eggshell (a, 2.5% SiC + 2.5% eggshell; b, 2.5% SiC + 7.5% eggshell; c, 2.5% SiC + 12.5% eggshell; d, 12.5% SiC + 2.5% eggshell) hybrid green metal matrix composites at lower and higher magnification. It shows a uniform distribution of carbonized eggshell and SiC particles in AA2014 matrix. This is an indication that the electromagnetic stir casting process adopted for the AA2014/SiC/carbonized eggshell hybrid green metal matrix composite production is reliable. Figure 9 shows a TEM image of AA2014/2.5% SiC/7.5% carbonized eggshell hybrid green metal matrix composite, which represents dendrites of aluminum and precipitates along the dendrite boundaries of SiC and carbonized eggshell particles. TEM image clearly shows the interface between the AA2014 matrix and the reinforcement materials (SiC particles and carbonized eggshell particles). It shows proper wettability and good interface bonding between AA 2014 matrix, SiC particles, and carbonized eggshell particles.

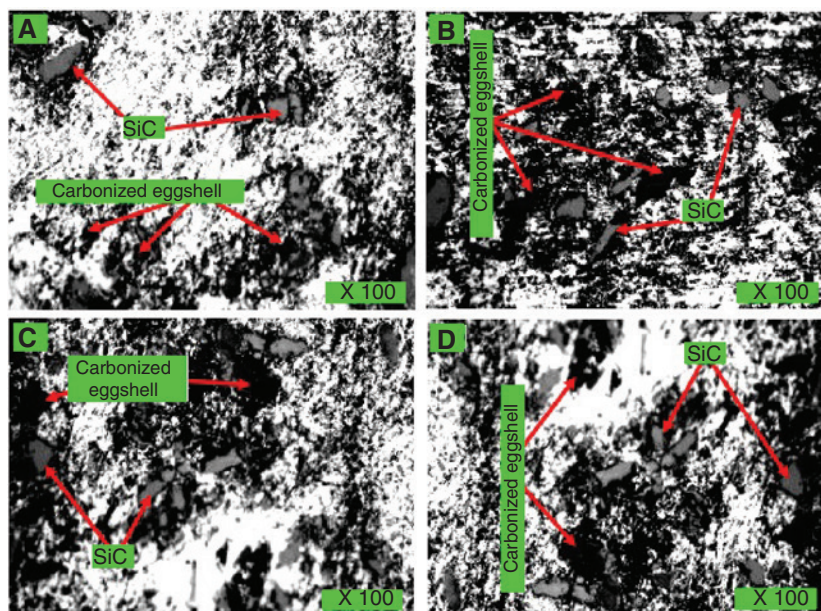


Figure 7: Microstructure of AA2014 reinforced ($\times 100$) with (A) 2.5% SiC + 2.5% eggshell, (B) 2.5% SiC + 7.5% eggshell, (C) 2.5% SiC + 12.5% eggshell, and (D) 12.5% SiC + 2.5% eggshell.

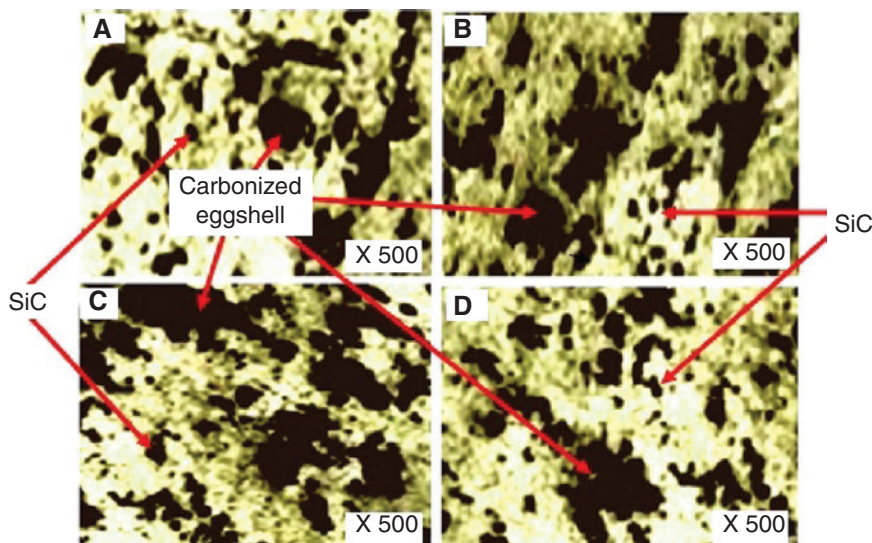


Figure 8: Microstructure of AA2014 reinforced ($\times 500$) with (A) 2.5% SiC + 2.5% eggshell, (B) 2.5% SiC + 7.5% eggshell, (C) 2.5% SiC + 12.5% eggshell, and (D) 12.5% SiC + 2.5% eggshell.

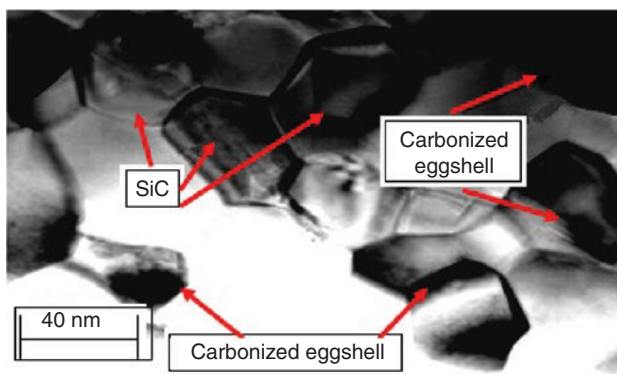


Figure 9: TEM image of sample designation G3-AA2014/2.5% SiC/7.5% eggshell composite.

3.3 Density and porosity analysis

Figure 10 shows that both the theoretical and the experimental densities decreased with increasing percentage additions of carbonized eggshell particles in AA2014/SiC composites. Theoretical densities of SiC particles and carbonized eggshell particles were 3.21 and 2 g/cm³, respectively. Although minimum theoretical and experimental densities were observed for sample designation G5, sample designation G3 shows better mechanical properties, as discussed earlier. The overall theoretical density of AA2014/eggshell/SiC hybrid green metal matrix composite decreased with wt.% additions of carbonized eggshell particles. The theoretical density of the composites (AA2014) decreased from 2.8 g/cm³ at 0 wt.% to 2.75 g/cm³ at 7.5 wt.% for carbonized eggshell particles and 2.5 wt.%

SiC particles. The experimental density of the composites decreased from 2.78 g/cm³ at 0 wt.% to 2.73 g/cm³ at 7.5 wt.% for carbonized eggshell particles and 2.5 wt.% SiC particles. From the theoretical density analysis of AA2014/7.5% eggshell/2.5% SiC hybrid green metal matrix composite, it can be observed that there was an approximately 1.8% decrease in theoretical density.

In the present investigation, black deviation bars between theoretical density and experimental density are showing percent porosity, as shown in Figure 10. Shorter black deviation bar between theoretical and experimental densities shows low porosity, whereas longer black deviation bar shows higher porosity. A higher percentage of porosity leads to specimen failure. Minimum porosity was found to be 0.72% for AA2014/7.5% carbonized eggshell/2.5% SiC hybrid green metal matrix composites (sample designation G3).

3.4 Corrosion behavior

From Figure 11, it can be observed that corrosion rate continuously decreases after the addition of carbonized eggshell particles up to 12.5 wt.% in AA2014/SiC composite. The corrosion rate decreased from 5.79 mm/year at 0 wt.% to 3.74 mm/year at 12.5 wt.% for carbonized eggshell particles and 2.5 wt.% for SiC particle. Approximately 35.40% corrosion rate decreases after the addition of 12.5 wt.% carbonized eggshell and 2.5 wt.% SiC particles in AA2014 aluminum alloy. However, after the heat treatment process, the corrosion rate of hybrid green metal matrix composites further increased. A minimum corrosion rate

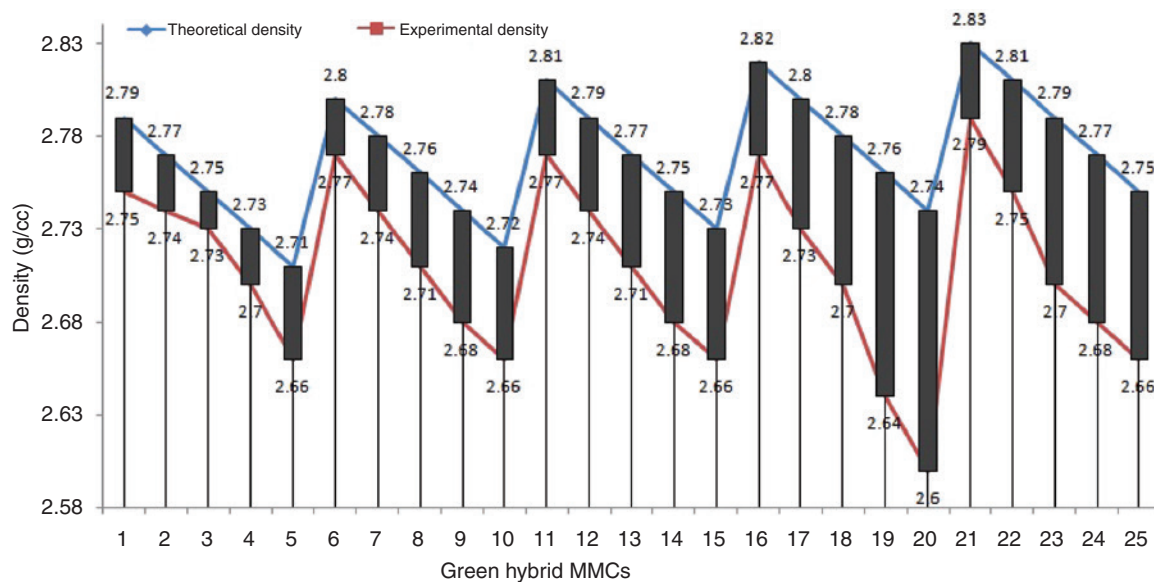


Figure 10: Density and porosity analysis of hybrid green MMCs.

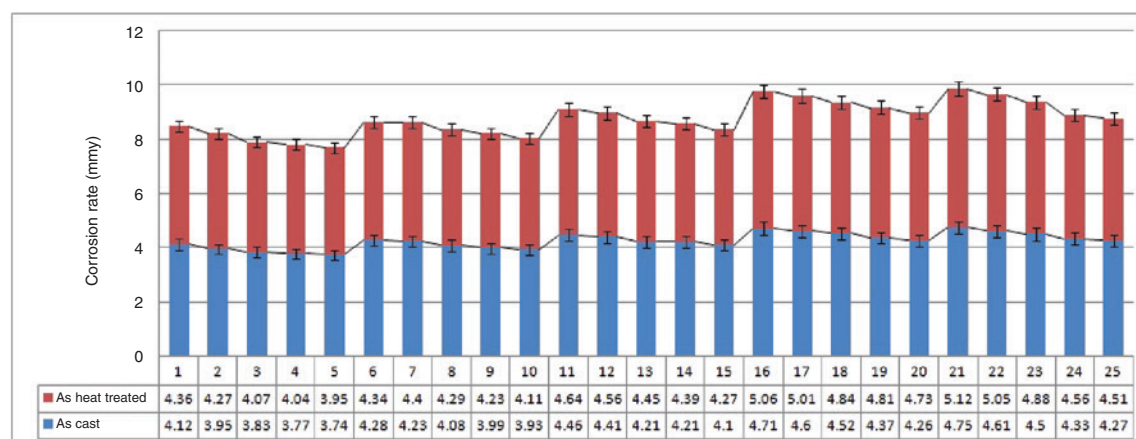


Figure 11: Corrosion behavior of hybrid green MMCs.

was observed for sample designation G5, but sample designation G3 shows better mechanical properties, as discussed earlier. The corrosion rate of sample designation G3 was found to be 3.83 mm/year. Sample designation G3 (AA2014/7.5% eggshell/2.5% SiC composite) shows approximately 33.85% lower corrosion rate as compared with matrix alloy AA2014, which is acceptable.

3.5 Cost estimation

The cost of AA2014 is approximately 300 Indian rupees (INR) per kilogram, whereas the cost of SiC powder is 700 INR per kilogram [3, 20]. Eggshell particles are available as waste, so these are available free of cost. From

Figure 12, it can be observed that the cost of hybrid green metal matrix composites continuously decreases after the addition of carbonized eggshell particles in AA2014/SiC composite. Sample designation G3 (7.5% eggshell and 2.5% SiC) shows better tensile and fatigue strength, whereas sample designations G5 (12.5% eggshell and 2.5% SiC) and G21 (2.5% eggshell and 12.5% SiC) show low corrosion rate and maximum hardness, respectively, as discussed earlier. The costs of sample designations G3, G5, and G21 are estimated at 287.5, 272.5, and 342.5 INR. The costs of sample designations G3 and G5 were found to be approximately 5.83% and 9.16% lower than AA2014 alloy. The cost of sample designation G21 was found to be 14.16% higher than AA2014 alloy, which is not acceptable from the cost point of view of green hybrid metal matrix

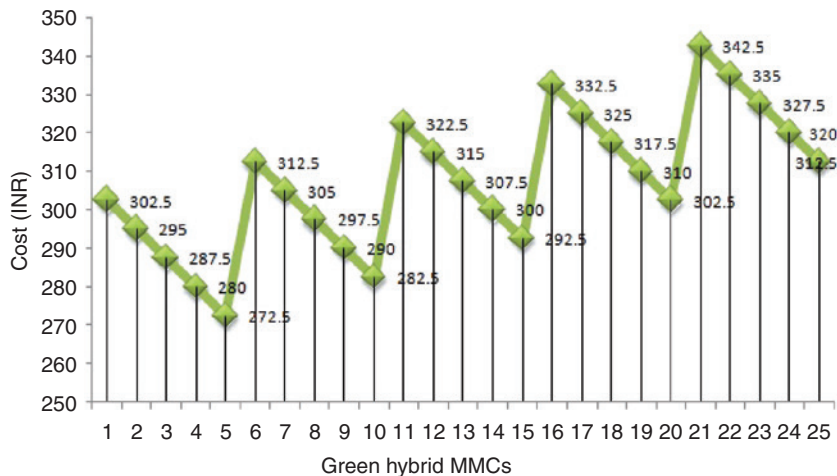


Figure 12: Cost estimation of hybrid green MMCs.

composite. Minimum cost (272.5 INR) and corrosion rate were observed for sample designation G5. The hardness of sample designation G5 after heat treatment was found to be 110 BHN, which is acceptable from the mechanical property point of view as well as the cost of composite point of view.

4 Conclusions

The microstructural characteristics, mechanical behavior, physical behavior, corrosion behavior, and cost estimation of AA2014/waste eggshell/SiC hybrid green metal matrix composites were investigated in this research. The results show the following:

1. Microstructure images show that carbonized eggshell particles and SiC particles are uniformly distributed in AA2014 aluminum alloy. TEM image shows proper wettability between reinforcement particles and aluminum alloy AA2014.
2. Maximum tensile strength and fatigue strength were found to be for sample designation G3 (reinforced with 7.5% eggshell and 2.5% SiC). The cost and the density of AA2014/7.5% eggshell/2.5% SiC composite were found to be 5.83% and 1.8% lower than matrix alloy, respectively, which is acceptable.
3. Minimum cost and corrosion rate were observed for sample designation G5 (reinforced with 12.5% eggshell and 2.5% SiC).
4. Maximum hardness was observed for sample designation G21, but composite overall cost is 14.16% higher than AA2014 aluminum alloy. Minimum cost and corrosion rate were observed for sample designation G5. The hardness of sample designation G5 after

heat treatment was found to be 110 BHN, which is acceptable.

5. Minimum porosity was found to be 0.72% for AA2014/7.5% carbonized eggshell/2.5% SiC hybrid green metal matrix composites.

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