



Synthesis and characterization of ball-milled eggshell and Al₂O₃ reinforced hybrid green composite material

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Abstract

Eggshell is a by-product (a secondary product derived from a production process) which produces lots of environment pollution. However, some environment pollution can be reduced by utilizing this waste. In the present investigation, an attempt was made to utilize eggshell as the reinforcement material with alumina (Al₂O₃) in the development of composite material. Carbonized eggshell particles and Al₂O₃ ceramic particles were ball-milled up to 100 hours to obtain a uniform particle size of reinforcement particles. Results showed that by using Al₂O₃ with eggshell in the development of aluminium based composite material after the ball-milling, mechanical properties were significantly improved. However, toughness and ductility were reduced. Microstructure results showed a uniform distribution of eggshell and Al₂O₃ in aluminium alloy. Mechanical properties of the composite material were further improved after the heat treatment process. Thermal expansion, corrosion behaviour were also investigated to observe the effect of eggshell and Al₂O₃ addition in the development of aluminium based composite material.

1. Introduction

Aluminium metal matrix composites (AMCs) are gaining worldwide attention at present in the various field such as automobiles, marine, aerospace etc., because of their superiority over monolithic alloys. AMCs used in these industries due to the remarkable mechanical and physical properties including toughness, ductility, high specific strength, high specific modulus, corrosion loss, low density etc. However, the cost is also considered in the selection of materials. The best ways to reduce the cost is to use low-cost reinforcement which includes waste materials. Further, the cost of the composite material can be reduced by utilizing waste reinforcement particles [1-2]. Chicken eggshell (ES) is a by-product (a secondary product derived from a production process) which produce environmental problems. Eggshell dust can carry into the air (and your lungs, eyes...) whatever bacteria or microbes that have been associated with the egg or shell and may also carry airborne microbes that can therefore enter your lungs or eyes, etc. as you breathe. It is just as dangerous to breathe the dust after it has settled on clothing, furniture, hands, etc. Increased incidence of eye/eyelid infections, chronic sinusitis, lung problems, repeated, persistent or chronic congestion, coughs or colds, aggravation of asthmatic symptoms or emphysema-like symptoms, etc. are only some of the problems reported by eggers around the world [3-4]. Eggshell consists of around 95% calcium carbonates in the form of calcite and 5% organic materials such as Al₂O₃, SiO₂, S, P, Cl, and Cr₂O₃, MnO sulfated polysaccharides, X collagen, and other proteins. Chicken eggshell is widely used because of availability in bulk quantity with lightweight, economical and

environmentally friendly. The density of ES is lower than the mineral calcium carbonate. However, eggshell with other ceramic particles such as SiC showed various defects in the composite material after the solidification process due to mismatch density [5-6]. Utilization of eggshell in different processing is shown in Table 1.

Alumina (Al₂O₃) is the hardest ceramic particle (lower than diamond, silicon carbide and boron carbide and higher than other ceramic particles) which is widely used in the development of aluminium based composite material. Presence of alumina in the composite material always enhances the hardness and tensile strength of the composite. Further, alumina always showed better wettability and interfacial reaction layer with aluminium alloy. However, sometimes low particles size and high weight percent of alumina produced porosity inside the aluminium based composite material after solidification process.

Development of aluminium based composite material by using different ceramic reinforcement material was one of the most common trends in materials science and engineering [7]. Generally, aluminium is used in various applications where high strength and hardness were required. Some big challenges were faced due to the mismatch densities of reinforcement particles during the development of composite material after solidification process. Moreover, various researchers are developing aluminium based metal matrix composite materials with different reinforcement material and improved mechanical properties [8-10]. Table 2 shows the summary of aluminium based composite with outcome results.

From the literature review, it has been observed that very few researchers developed aluminium based composite material using eggshell and alumina as

reinforcement material. However, during the development of composite material, some difficulties were observed such as agglomeration of reinforcement particles due to mismatch densities. In this study,

reinforcement particles were ball-milled to form uniform particle size of reinforcement particles. Squeeze pressure was also applied to eliminate the porosity and agglomeration of reinforcement particles.

Table 1. Waste eggshell and its utilization

Ref. No.	Author's Name	Material Used	Results Outcome and Conclusions
1	Patricio Toro <i>et al.</i> (2007)	Polypropylene (PP) composite/eggshell (ES) and Polypropylene (PP) composite/ calcium carbonate (CC)	The chicken eggshell contains 95% calcium carbonate.
2	Libor Severa <i>et al.</i> (2010)	Eggshells	E-modulus of eggshells ranged from 47.4 to 53 GPa.
3	Sneha Lunge <i>et al.</i> (2013)	Alumina/eggshell	Eggshell can enhance hardness due to the presence of hard phase such as CaCO ₃
4	S.B. Hassan <i>et al.</i> (2013)	Al-Cu-Mg/eggshell particulate	Eggshell can be used with aluminium alloy and enhanced mechanical properties.
5	Munlika Bootklad <i>et al.</i> (2013)	Thermoplastic starch/eggshell powder composites	Eggshell as reinforced compared with CaCO ₃ as reinforcement material
6	Manasa K.Rath <i>et al.</i> (2014)	Eggshells	Developed NiO-Ce0.8Gd0.2O1.9 (GDC) composite using eggshell.

Table 2. Aluminium based composite materials and their outcomes

Ref. No.	Author's Name	Material Used	Results Outcome and Conclusions
7	E.J. Zoqui <i>et al.</i> (2002)	A356 aluminium alloy	Refined rheocast structure obtained at 1200 watt using Electromagnetic stir casting technique
8	S. Nafisi <i>et al.</i> (2006)	Al-Si-Fe alloy	The grain structure is quite coarse with pouring at high temperature
9	T. W. Kim <i>et al.</i> (2009)	A6061 aluminium alloy	Due to excessive vertex, the air is interrupted into the melt matrix material
10	Shyam Lal <i>et al.</i> (2013)	Al7075/SiC/Al ₂ O ₃	Argon gas was used at the surface of melt composite material to eliminate the porosity
11	S. P. Dwivedi <i>et al.</i> (2014)	A356/SiC/Fly-ash	Hardness and tensile strength improved
12	Rong Chen <i>et al.</i> (2000)	A356alloy/SiC	Mechanical properties were improved after the heat treatment process
13	S.A. Sajjadi <i>et al.</i> (2012)	A356 aluminium alloy/Al ₂ O ₃	Tensile and compressive strength were significantly improved
14	Du Jun <i>et al.</i> (2007)	AlSi12CuMgNi/Al ₂ O ₃ / carbon short fibres hybrid composites	Heat treatment behaviour of the aluminium alloy was observed.
15	A. Arun Premnath <i>et al.</i> (2012)	Al6061/Al ₂ O ₃ /Graphite	Hardness and tensile strength can be enhanced by using Al ₂ O ₃ and Graphite simultaneously
16	T. S. Mahesh <i>et al.</i> (2012)	A356/SiC/B ₄ C	Ceramic particles SiC and B ₄ C can be used simultaneously with aluminium alloy
17	C.U. Atuanya <i>et al.</i> (2012)	Al-Si-Fe alloy	Tensile strength and the hardness increased after adding the breadfruit seed hull ash particulate
18	Poovazhagan L. <i>et al.</i> (2013)	Al 6061/nano SiC/nano B ₄ C	Ductility of the composites reduces marginally as the hybrid ratio increases
19	P. Ravindran <i>et al.</i> (2013)	Al 2024-5 wt% SiC- 10 wt% graphite	The wear loss increased with increasing sliding distance
20	Keneth Kanayo Alaneme <i>et al.</i> (2013)	Al-Mg-Si/RHA/Al ₂ O ₃ hybrid composites	The specific strength, percent elongation and fracture toughness is better when RHA and Al ₂ O ₃ were used together
21	K. Umanath <i>et al.</i> (2013)	Al6061/SiC/Al ₂ O ₃	Wear resistance significantly improved by using SiC and Al ₂ O ₃

2. Materials and methods

2.1 Matrix material

In the present investigation, aluminium alloy (major compositions are Al about 99.8%, Cu about 0.40%, Mg about 0.8%) was the matrix material. Aluminium is the most demanding material in automobile industries as well as aerospace applications due to its lightweight and good specific strength with less corrosion loss. Aluminium alloys are used in the fabrication of various parts such as internal parts of brake and rotors, I.C engines piston, and different structural parts [22,23].

2.2 Primary reinforcement material

In the current investigation, Al_2O_3 or Alumina, ceramic particles have been considered as a primary reinforcement material. Alumina is the fourth hardest ceramic particles. The hardness of diamond, boron carbide, silicon carbide is 7000, 2800 and 2500 Knoop hardness respectively. The hardness of alumina (Al_2O_3) is 2100 Knoop hardness. High hardness of alumina particles enhances the tensile strength and hardness significantly [20,21].

2.3 Secondary reinforcement material

Eggshell waste was collected from the food store. It was washed thoroughly in water and then dried for about 25 h. The dried eggshell was ball-milled. The ball-milled eggshell particle was carbonized for 24 h at the temperature of 500°C.

2.4 Ball-milling process of reinforcement particles

Figure 1 shows the ball-milling process (mechanical alloying). The mixture of two reinforcement particles in a single entity was obtained by using a ball milling process. In the present investigation, Al_2O_3 ceramic particles and carbonized eggshell particles with an average mean particle size of 50 μm , has been alloyed mechanically by the ball milling process. The ball-milling process was carried out up to 100 h to obtain the mixture of alumina powder and carbonized eggshell particles in a single entity. Figure 2 shows the formation of ball-milled reinforcement particles (a mixture of Al_2O_3 and carbonized eggshell powder) in a single entity from the waste eggshell.

2.5 Development of composite material

Figure 3 shows the line diagram for ball-milled reinforced composite development. Waste eggshell was collected from the food store. Collected eggshell was ball-milled to obtain in powder form. Powder eggshell has been carbonized for 24 h at the

temperature of 500°C. Carbonized eggshell powder and alumina particles (Al_2O_3) were further ball-milled up to 100 h to form uniform particle size of reinforcement particles. Ball-milled reinforcement particles were preheated (up to 400°C) to enhance wettability. Reinforcement particles with different composition as shown in Table 1 have been mixed with aluminium alloy. A prepared composite material in the mushy zone (Mushy zone is porous layers of dendritic crystals that commonly form during solidification of multi-component melts) was transferred to a UTM (universal testing machine) for squeeze pressure (it has the potential to eliminate the gas defects associated with high pressure die casting). Squeeze pressure was applied to reduce porosity and blowholes (bubble-shaped cavities) from the composite.

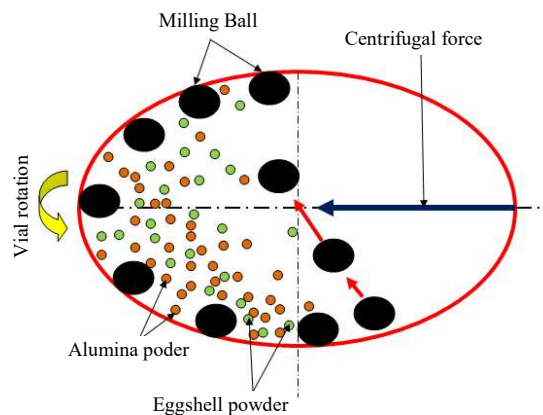


Figure 1. The ball-milling process (mechanical alloying).



Figure 2. Formation of ball-milled reinforcement particles in a single entity from the waste eggshell.

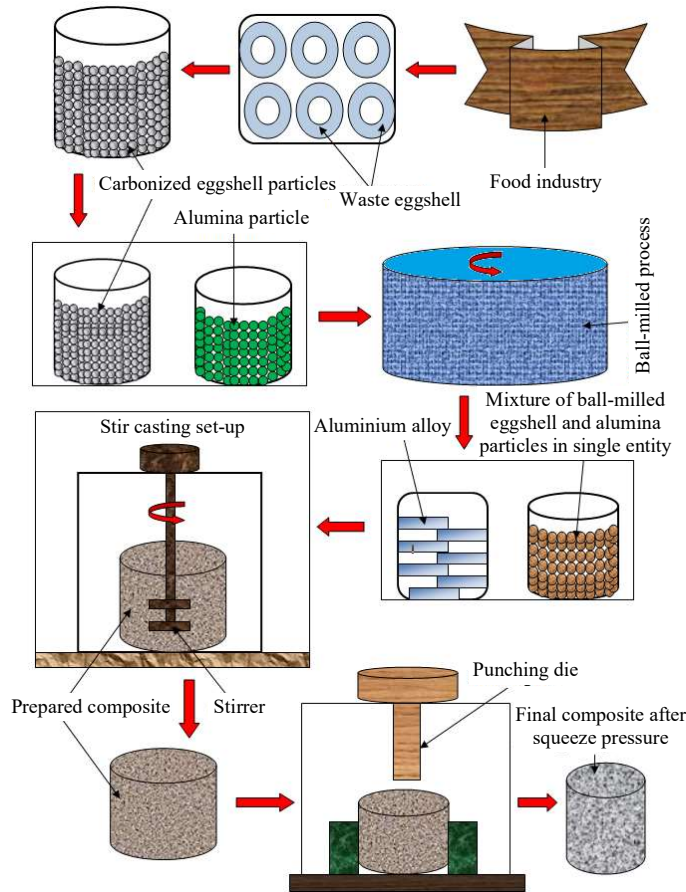


Figure 3. Processing of composite material.

Table 3. Composition of the composite.

Compositions					
Sample No.	Reinforcement (wt%)	Al/Carbonized eggshell	Al/Al ₂ O ₃	Al/Carbonized eggshell/Al ₂ O ₃ Composition with ball-milled	Heat-treated Al/ Carbonized eggshell/ Al ₂ O ₃ Composition with ball-milled
1	2.5	Al/2.5% carbonized eggshell	Al/2.5% Al ₂ O ₃	Al/1.25% carbonized eggshell /1.25% Al ₂ O ₃	Al/1.25% carbonized eggshell /1.25% Al ₂ O ₃
2	5	Al/5% carbonized eggshell	Al/5% Al ₂ O ₃	Al/2.5% carbonized eggshell /2.5% Al ₂ O ₃	Al/2.5% carbonized eggshell /2.5% Al ₂ O ₃
3	7.5	Al/7.5% carbonized eggshell	Al/7.5% Al ₂ O ₃	Al/3.75% carbonized eggshell /3.75% Al ₂ O ₃	Al/3.75% carbonized eggshell /3.75% Al ₂ O ₃
4	10	Al/10% carbonized eggshell	Al/10% Al ₂ O ₃	Al/5% carbonized eggshell /5% Al ₂ O ₃	Al/5% carbonized eggshell /5% Al ₂ O ₃
5	12.5	Al/12.5% carbonized eggshell	Al/12.5% Al ₂ O ₃	Al/6.25% carbonized eggshell /6.25% Al ₂ O ₃	Al/6.25% carbonized eggshell /6.25% Al ₂ O ₃

3. Results and discussion

3.1 Investigation of microstructure

Figure 4 shows the microstructure image of a composite material reinforced with carbonized eggshell and Al₂O₃ in a single entity. To prepare the microstructure sample, the grinding was done in such a way so that all the scratches are in the same direction and the grinded surface becomes flat. After this, the samples were polished properly using different grits of emery papers. Being sample, an aluminium alloy which is considered soft, it is rubbed over the emery paper for a small time. Then, it was rubbed over an emery paper of 400 grit and then over a very fine emery paper of 600 grit for a considerable time to get a smooth and clear surface of the samples. The sample was then polished on a fine polishing machine using alumina/diamond polishes. This was done to get a well polished and a smooth surface required for the further characterization of the samples. Similarly, all the samples were polished for a considerable time, over and over again until a very fine and smooth surface was obtained for further analysis. Polished unetched samples can show macroscopic cracks, pits, and so on, but no microstructural details because there is not yet any contrast producing feature on the surface. These will be revealed by the etching process. The etching process was carried out in Keller's solutions (Distilled water, Nitric acid, Hydrochloric acid, Hydrofluoric acid) [24-28]. A metallurgical microscope is used to observe the microstructure of composite. Microstructure image of ball-milled reinforcement particles shows the uniform distribution in the aluminium based matrix material after solidification. Microstructure image showed the good interfacial reaction layer and proper wettability between the reinforcement particles and matrix material. Proper interfacial reaction layer between the matrix material and reinforcement particles enhanced the mechanical properties of the composite. The black spot shows the presence of eggshell and Al₂O₃ particles in the aluminium based matrix material as shown in Figure 4.

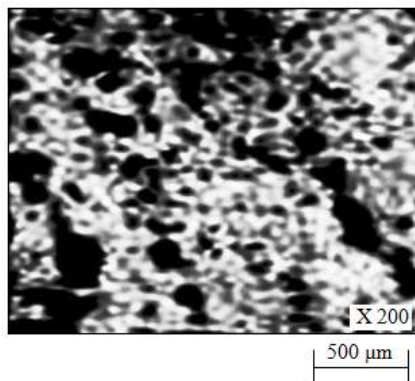


Figure 4. Microstructure image of carbonized eggshell and Al₂O₃ reinforced (visible in microstructure with black spots) aluminium based composite material.

3.2 Tensile strength analysis

Tensile strength of composite materials is shown in Figure 5. Tensile strength of base material (major compositions are Al about 99.8%, Cu about 0.40%, Mg about 0.8%) was also observed. It was found to be 132 MPa. Maximum tensile strength for ball-milled reinforced composite material (a mixture of eggshell particles and alumina) without heat treatment was found to be 182.25 MPa for composition Al/5% carbonized eggshell/5% Al₂O₃ composite material. Figure 4 (a) shows the microstructure of Al/5% carbonized eggshell/5% Al₂O₃ composite material with ball-milled reinforcement particles. Uniform distribution of reinforcement particles in the aluminium alloy enhanced the tensile strength of the composite. Further, Incorporation of hard Al₂O₃ ceramic particles with carbonized eggshell particles was responsible for enhancing the tensile strength of the composite. Presence of phases such as CaCO₃ (about 96%), S (about 2%), Mg (about 0.4%) and P (0.5%) in eggshell was also responsible for enhancing the tensile strength of composite material. However, uniform distribution of single entity reinforcement particles (a mixture of Al₂O₃ and eggshell) in matrix material was also accountable to enhance the tensile strength of composite material. Tensile strength of composite material was further improved after the heat treatment process due to grain refinement of microstructure. Hassan *et al.* [27] identified the effect of eggshell weight percent in Al-Cu-Mg alloy. Stir casting technique was used to develop the eggshell reinforced Al-Cu-Mg alloy based composite. Their results showed that about 14.28% tensile strength improved after adding 12 wt% carbonized eggshell in aluminium alloy. However, present study results showed that about 38.06% tensile strength improved by adding the ball-milled Al₂O₃ and carbonized eggshell particles in aluminium alloy. Tensile strength of heat-treated Al/5% carbonized eggshell/5% Al₂O₃ composite material was improved by about 46.55% concerning base material (major compositions are Al about 99.8%, Cu about 0.40%, Mg about 0.8%).

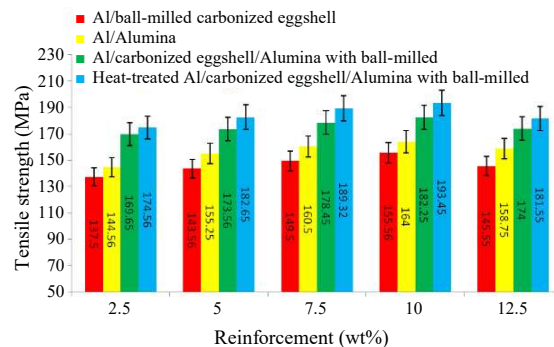


Figure 5. Tensile strength of composites.

3.3 Hardness test analysis

Figure 6 shows the hardness of ball-milled reinforced composite material. Hardness has been measured on three random points for each sample. Averages of three hardness values were considered for each sample. The hardness of the base material found to be 59 BHN. Maximum hardness was found to be 89 BHN before heat treatment of composition Al/5% Al₂O₃/5% carbonized eggshell composite material. Microstructure image (Figure 4) shows the proper interfacial reaction layer between the matrix material and reinforcement material. Proper interfacial reaction layer between the matrix and reinforcement material reduced the porosity and blow holes develop inside the composite after the solidification. Microstructure image of Al/5% Al₂O₃/5% carbonized eggshell composite material with ball-milled reinforcement particles shows no porosity inside the composite. Resulting, the hardness of this composite was better than other selected composition. Further, the Presence of hard phases such as CaCO₃ [29] in carbonized eggshell and Al₂O₃ in alumina was responsible for enhancing the hardness of the composite [27,29]. The hardness of the composite was further improved after the heat treatment process. Refined grain structure after the heat treatment process enhanced the hardness significantly. Hassan *et al.* [27] showed that hardness of aluminium based composite material can be improved by using carbonized eggshell particles as reinforcement material. Their results revealed that hardness of Al-Cu-Mg/12 wt% eggshell was improved by about 25.4% with respect to the base material. Present study results showed that the hardness of heat-treated and without heat-treated composite material reinforced with ball-milled 5 wt% eggshell and 5 wt% Al₂O₃ in single entity improved about 50.84% and 60.16% respectively concerning base material.

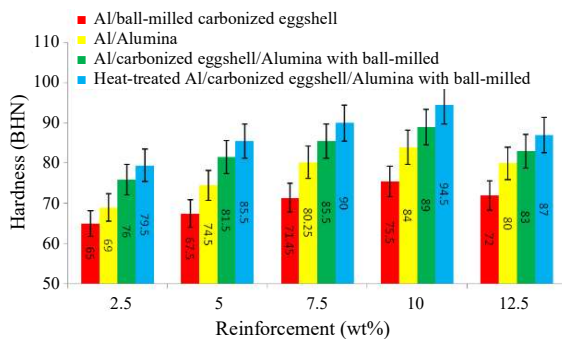


Figure 6. Hardness of composites.

3.4 Toughness (Impact strength)

Toughness (Sample dimension was 10 mm × 10 mm × 55 mm with 45°V notch at the centre of 2 mm depth according to ASTM A370 standard) of composite measured on impact testing machine. Impact strength

analysis of composite material is shown in Figure 7. The base material impact strength was tested. It was 18 J·m⁻³. The toughness of the composite material reduced after the addition of ball-milled eggshell particles and Al₂O₃ ceramic particles in a single entity. The microstructure of composite material (Figure 4) shows the fair distribution of reinforcement particles. However, the fair distribution of reinforcement particles enhanced the tensile strength and hardness of the composite. But, reduced the impact strength of composite material. Presence of hard phases in eggshell particles (CaCO₃) and Al₂O₃ ceramic particles are the main reason in reducing the impact strength of the composite. The toughness of the composite material improved after the heat treatment process.

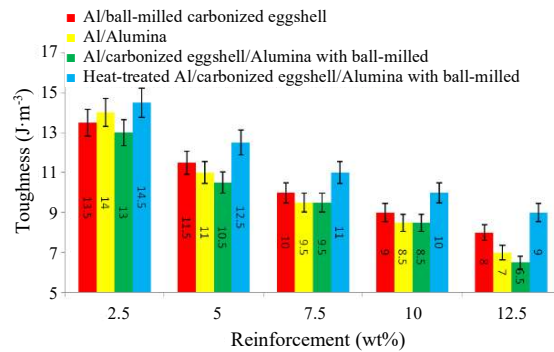


Figure 7. Impact strength of composites.

3.5 Ductility

Ductility of the composite material was observed to identify the percent elongation (% EL). Ductility of the base material was calculated. It was 16.5% EL. Figure 4 shows that the addition of ball-milled reinforcement particles reduced the plasticity deformation behaviour of composite material. Incorporation of hard particles such as carbonized eggshell and Al₂O₃ increased the tensile strength but decreased the ductility of the composite. Ductility of the composite is continuously decreased by adding the reinforcement particles in the aluminium alloy as shown in Figure 8. However, ductility of hybrid metal matrix composite was increased after using the heat treatment process.

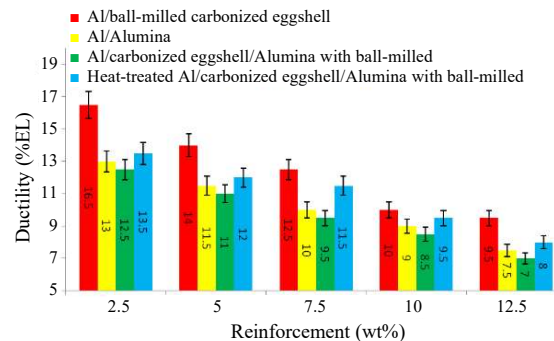


Figure 8. Ductility of composites.

3.6 Corrosion behavior

Corrosion behaviour of the composite was observed in 3.5 wt% NaCl for 120 h. Weight of each sample was kept 9 g. Figure 9 shows the mechanism of corrosion developed in the aluminium based composite material. Corrosion in the material takes place due to the chemical reaction of metal. Deterioration occurred in the surface properties of composite material due to the solution of H₂O and NaCl. The corrosion resistance of the composite material was increased by using the mixture of carbonized eggshell and alumina particles reinforcement particles after ball-milling in the development of composite material [24, 25]. Minimum weight loss was observed for the heat-treated Al/1.25% carbonized eggshell /1.25% Al₂O₃ composite material. It can be observed that by increasing the weight percentage of reinforcement particles (a mixture of eggshell and Al₂O₃), corrosion resistance of the composite material began to decrease. This decrement may be observed due to the formation of Al (OH)₃ at the surface of composite in the presence of O₂ and OH⁻. Results showed that corrosion resistance of the composite material has been significantly improved after the heat treatment process. The corrosion resistance of the composite material after the heat treatment improved due to the refined grain structure. Refined grain structure prevents the formation of oxide, hydroxide, or sulfide at the surface of composite material. Resulting, weight loss of the composite decreased after the heat treatment process. The corrosion weight loss of the investigated material is shown in Figure 9.

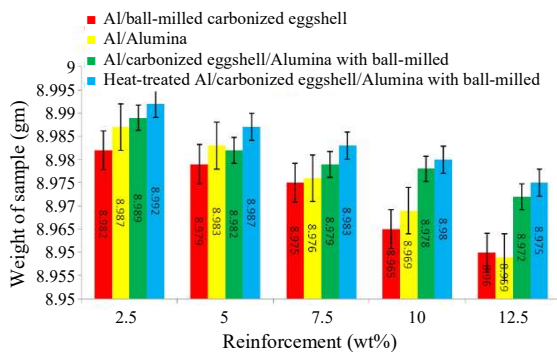


Figure 9. Corrosion weight loss of composites.

3.7 Thermal Expansion Behavior

Thermal expansion behaviour of composite material has been performed to observe the material sustainability in a high-temperature environment. In the thermal expansion test, the specimen size has been taken as 25 mm × 10 mm × 10 mm (2500 mm³ volume) throughout the experiment before thermal expansion test. Thermal expansion test was performed in a muffle furnace at 450°C for 24 h. After thermal expansion test, again volume of the samples has been measured. Minimum volume difference has been observed for

sample Al/5 wt% carbonized eggshell/5 wt% Al₂O₃ composite material as shown in Figure 10. However, material sustainability was further improved after the heat treatment process.

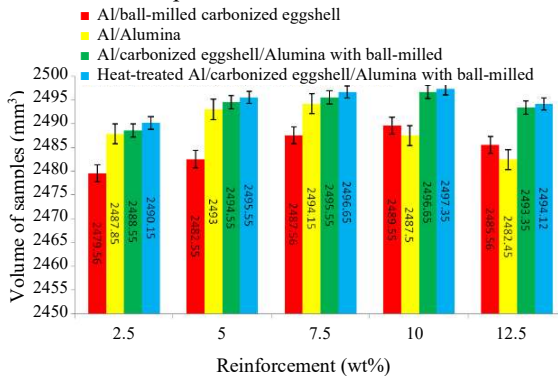


Figure 10. Thermal expansion behaviour of composites.

4. Conclusions

Conclusive points from the present study are given below.

1. The carbonized eggshell powder can ball milled with Al₂O₃ ceramic particles to obtain in single entity particles.
2. Ball-milled reinforcement particles in single entity enhanced the tensile strength and hardness about 38.06% and 50.84% respectively. However, toughness and ductility were reduced.
3. Corrosion resistance and thermal sustainability of composite material by using ball-milled reinforcement particles were significantly improved.
4. Mechanical behaviour, corrosion resistance and thermal expansion of the composite further improved after the heat treatment process.

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