Nigerian Research Journal of Engineering and Environmental Sciences 8(1) 2023 pp. 162-171
p ISSN: 2635-3342; e ISSN: 2635-3350

Nigerian Research Journal of Engineering and Environmental Sciences
Journal homepage: www.rjees.com

Original Research Article

Age-Hardening Behaviour of Cow Bone Particulates-Reinforced Aluminium Metal Matrix Composites in Different Quenching Media

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http://doi.org/10.5281/zenodo.8097445

ARTICLE INFORMATION

Article history:
Received 23 Aug. 2022
Revised 26 Oct. 2022
Accepted 07 Nov. 2022
Available online 30 Jun. 2023

Keywords:
Cow bone particulates
Mechanical characteristics
Aluminium composites
Stir casting
Age-hardening

ABSTRACT

The influence of cow bone particulates on the mechanical characteristics of aluminium metal matrix composites was studied in this work. Cow bone particulates in varied weight percentages were added to molten aluminium alloy by stir casting method to produce the composites. The composites were subjected to microstructural examination and mechanical characterisation at room temperature. The results revealed serial dislocation lines in the microstructure of the composites, which are the basis for the improvement in strength of the specimens. There were minimal micro porosities in the microstructure of the casts, which indicated a fair uniform distribution of the hard cow bone particulates in the aluminium matrix. The 15 wt. % cow bone particulates-reinforced composite quenched in salt water exhibited the highest hardness value of 184.65 HV, which is 23.1% higher than hardness of the 15 wt. % cow bone particulates-reinforced composite that was not age-hardened. This is also 50% higher than hardness of unreinforced cast aluminium alloy. The age-hardened composites demonstrated an inverse response to impact as the addition of particulates increased in the composites. The subjection of the specimens to heat treatment in different quenching media reduced their impact energies.

1. INTRODUCTION

The rate at which wastes are being generated in the rural and urban areas is high and alarming (Ogundele *et al.*, 2018). Among these wastes are metallic and agricultural wastes. Aluminium alloy scraps and cow bones are examples of metallic and agricultural wastes respectively. Their generation has tremendously increased, and their composition and magnitude have much impact on mankind (De-Sousa and Zanchet, 2018). The indiscriminate disposal of such wastes may cause harmful environmental impacts and increase the great global problem of their final disposal (Mandal *et al*., 2012). However, the recycling of such materials can reduce these problems because recycling protects the environment and saves cost. Furthermore, the conversion of these wastes into valuable engineering materials (composites) via age-hardening process and stir casting method is value addition.

Age-hardening, which is known as precipitation hardening is a heat treatment process that is used to impart strength to metals and their alloys. The term "heat treatment" indicates a sequence of controlled heating and cooling, which lead to microstructural modifications, and thus to changes in the properties of a material. In many cases, heat treatment is necessary to achieve optimal properties, which allow the use of a material for industrial purposes (Fracasso, 2010). Heat treatment is done to increase strength, hardness, ductility, and reduce the residual stresses within a part. Residual stresses are caused by forming processes (e.g. stretching, forging, extrusion) or by machining processes (e.g. cutting, grinding) (Roney *et al*., 2017).

Age-hardening is termed precipitation hardening as it makes use of solid impurities or precipitates for the strengthening process. The metal or alloy is aged by either heating or keeping it stored at lower temperatures so that precipitates are formed. Although, age hardening process increases the tensile and yield strength, the precipitates that are formed inhibit the movement of dislocations or defects in the metal's crystal lattice. Any obstacle that makes dislocation movement difficult hardens materials (Polat *et al*., 2015). Age hardening increases the hardness of metal alloys by a relatively low-temperature heat treatment that causes precipitation of components or phases of the alloys from the supersaturated solid solution (Hossain and Kurny, 2013). The process increases tensile and yield strength, elasticity (stiffness/rigidity), ductility, hardness, wear resistance, reduces toughness (impact energy), aids machinability, and allows for little or no distortion of the part or component. The hardness of the quenched alloy increases as a function of ageing time**.** Solution heat treatment of aluminium is done to obtain the maximum concentration of hardening solute in solution by heating the alloy to a temperature at which a single phase will be created. This encourages the solute atoms that are originally part of a two-phase solid solution to dissolve into the solution and create one single phase in equilibrium (Demir and Gunduz, 2009; Temmar *et al*., 2011).

Quenching is the process that involves the rapid cooling of metals or their alloys by immersion in oil (commonly used in industry), water, brine and air from a high temperature, usually after heat treatment. This is usually done to maintain mechanical properties associated with the crystalline structure or phase distribution that would be lost upon slow cooling. In metallurgy, quenching is one of the critical steps in the heat treatment of metals and their alloys and is used to harden the final product. By adjusting the rate and intensity of the heating and cooling processes, the mechanical and metallurgical properties of metals/alloys are altered in order to achieve specific design parameters that are required by the part for a specific purpose (Fracasso, 2010; Roney *et al*., 2017).

Aluminium alloys have proven to be important materials in automotive, aerospace, and diverse engineering applications over time. However, aluminium alloys need to experience advancement with the ever-going technological evolution. Hence, the advent of composites. Agricultural wastes are getting more popular as reinforcers in composites because of their low cost, ease of processing environmental sustainability, and availability. Many studies (Purohit *et al*., 2012; Das *et al*., 2014; Ankesh *et al*., 2016; Arun *et al*., 2016; Umunakwe *et al*., 2017) have been carried out on the influence of particulates on the properties of different kinds of aluminium alloys. The results indicated that the particulates reinforced aluminium matrix composites exhibited improved tensile strength, flexural strength, modulus of elasticity, ductility, hardness, and wear resistance among other properties. Thus, the aim of this study is to investigate the effect of age hardening on the mechanical characteristics of cow bone particulates reinforced aluminium metal matrix composites using different quenching media.

2. MATERIALS AND METHODS

2.1. Material and their Processing

The materials used in this work are aluminium scraps, cow bones, and quench media (salt water, palm oil, and water). X-ray Fluorescence (XRF) was done to check the elemental composition of the aluminium scrap and the cow bone as shown in Table 1. The aluminium scraps were cleaned to remove dirt and were resized to smaller sizes by cutting before charging into the furnace for melting and de-slagging. The cow bones were soaked in water containing detergent for 5 days to remove fat and oil residue. The bones were then sundried for 2 days to remove moisture followed by a comminution process (crushing using a mortar, pestle, and grinding with a grinding machine). The cow bone particulates were sieved using British standardised sieves (BSS) to varied size range (150-300 µm). A mini pal compact energy dispersive X-ray fluorescence spectrometer was used to determine the chemical composition of the aluminium scrap and cow bone particulates and the results are presented in Tables 1 and 2. The various stages of materials preparation and processing are shown in Figure 1.

Table 1: Chemical composition of the aluminium scrap

Element	Al	Si	Fe	Cu	Mn	Mg	Zn	Cr
Weight (%)	87.47	10.53	0.6674	0.5781	0.2255	0.1341	0.1584	0.0269

Table 2: Chemical composition of cow bone particulates (Abdulkareem *et al.*, 2019)

Element	Ca	P	CO	K	Mg
Weight (%)	36.05	16.43	4.58	0.85	0.74

Figure 1: (a) As received cow bones (b) soaking of cow bones in detergent water (c) sun dried cow bones (d) crushed cow bones (e) ground cow bone particulates (f) sieved (150 to 300 µm) cow bone particulates (g) aluminium scrap (h) re-sizing (cutting) of the scrap

2.2. Composites Production

As presented in Table 3, weighed quantities of the aluminium alloy scrap were charged into a crucible furnace, which was heated to 720 ºC in order to melt the alloy. Thereafter, appropriate quantities of the cow bone particulates were separately added to the molten aluminium alloy. The mixture was stirred using a stainless steel stirrer for 10 minutes to avoid clustering and to achieve good dispersion of the particles in the molten matrix. Thereafter, the slurry was steadily poured into a sand mould, which was embedded with a cylindrical pattern of diameter 20 mm and length 150 mm and allowed to cure for one hour. Thereafter, they were removed from the moulds. The casts were prepared and machined to suitable ASTM standard dimensions for the various tests. Prior to the addition of the particulates to the molten alloy, they were preheated to improve wettability and reduce porosity in the cast.

Table 3: Specimens' formulation

Specimens	Aluminium scrap		Cow bone particulates	
	wt. %	kg	wt. %	kg
1st	85	4.25	15	0.75
2nd	95	4.75	5	0.25
3rd	100	5.00	0	0.00

2.3. Age-hardening Treatment

The age-hardening treatment for the composite specimens produced was performed. The selected cast specimens were solution-treated by heating to a temperature of about 550 ºC in a furnace to obtain a single-phase solid solution α. The specimens were held at this temperature for 1 hour before quenching. Quenching was done in prepared media (water, palm oil, and saltwater), and ageing was done by holding the quenched composites at 180 ºC for 2 hours.

2.4. Characterisation of the Specimens

Standard procedures especially the American society for testing and materials (ASTM) methods of analysis were adopted in the characterisation of the specimens.

2.4.1. Microstructural examination

The microstructure of the selected as-cast aluminium alloy and composites was examined using a digital metallurgical microscope at x100 magnification. Prior to viewing of specimens with optical microscope, emery papers of grit sizes ranging from 150 to 2500 µm were used to polish the surfaces of the specimens. Thereafter, fine polishing was done using a suspension of polycrystalline diamond of particle sizes ranging from 0.5 to 10 µm with ethanol solvent. Each specimen was etched with 1 HNO_3:1 HCl solution (Alaneme and Bodunrin, 2013) for 1 minute prior to viewing with the optical microscope.

2.4.2. Mechanical characterisation

The tensile strength of the specimens was determined using a universal tensile testing machine of maximum test load of 5,000 kg and crosshead speed of 1.5 mm/min in accordance with the ASTM E8 (2022) standard. Each of the specimens was machined appropriately and was tightly locked between the upper and lower cross beams of the machine. The load was applied to set the specimen in the grips. The applied load was increased gradually until necking and subsequently fracture occurred. The same procedure was applied for all the specimens. The maximum load was determined for each of the specimen and the tensile strength was calculated using Equation (1).

$$P_{UTS} = \frac{P_{max}}{A_0} \quad (1)$$

Where, P_{UTS} = ultimate tensile stress, P_{max} = maximum load, A_0 = cross sectional area

The specimens were prepared according to the ASTM E384-16 (2017) standard test method for microhardness of materials. The specimens of the hardness test were successively ground using 110, 220, 320, and 400-micron grades of emery papers. The measure of indentation was recorded from the hardness tester. The hardness test was conducted on each specimen using a diamond indenter, and the diameter of the impression made by the indenter was recorded as displayed by the Vickers microscope. The corresponding values of hardness were recorded. The impact energy of the specimens of dimension 55 x 10 x 10 mm with a V-notch of 2 mm depth at the middle was determined in accordance with ASTM E23 (2018) standard using a Charpy impact tester. Each of the specimens was placed vertically in-between the grips of the testing machine and clamped into position. The pendulum's angle, weight, impact energy and striking velocity of 140°, 22 kg, 300 J and 5 m/s respectively were used to break the specimens.

3. RESULTS AND DISCUSSION

3.1. Microstructure

The micrographs (Figure 2 – 5) reveal serial dislocation lines, which are the basis for the improvement in strength of the specimens. Further, observation shows minimal micro porosities in the microstructure of the cast specimens indicates that the distribution of particulates in the respective matrix is fairly uniform. The micrographs also show increased particulates content and grain boundaries in the composites, which enhanced strength as demonstrated by the 15 wt. % particulates reinforced composite. This implies that increased particulates content promoted strength enhancement compared to the unreinforced and 5 wt. % particulates reinforced specimens, which demonstrated lower tensile strength and hardness. The age-hardened specimens revealed the presence of small Mg_2Si precipitates within the aluminium matrix that kept precipitating out from solid solution due to aging as shown in Figure 3 – 5 which is similar to the report by Kareem *et al.* (2017).

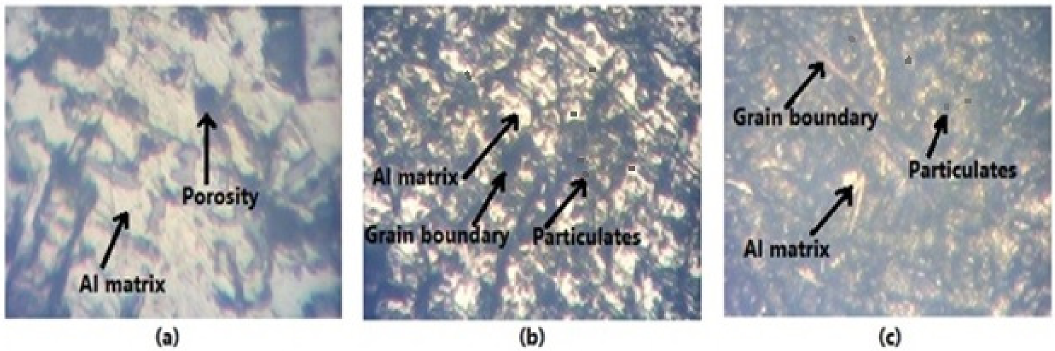

Figure 2: Optical micrographs of non-heat-treated specimens (x100) (a) unreinforced (b) 5 wt. % particulates-reinforced (c) 15 wt. % particulates-reinforced

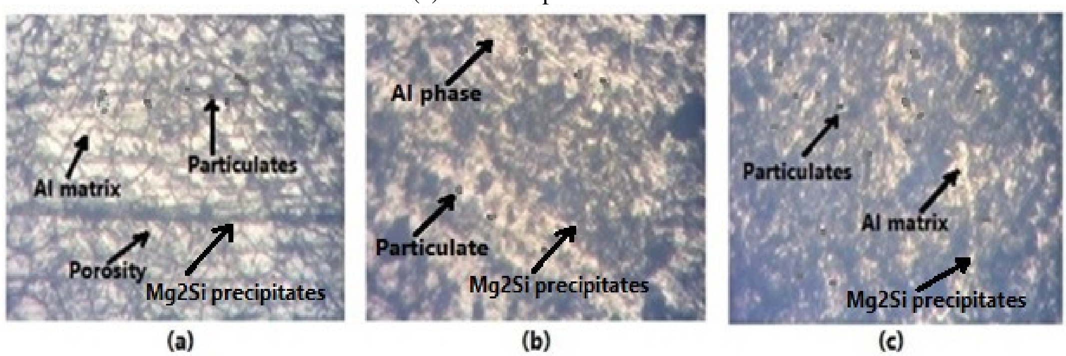

Figure 3: Optical micrographs of the specimens heat-treated and quenched in palm oil (x100) (a) unreinforced (b) 5 wt. % particulates-reinforced (c) 15 wt. % particulates-reinforced

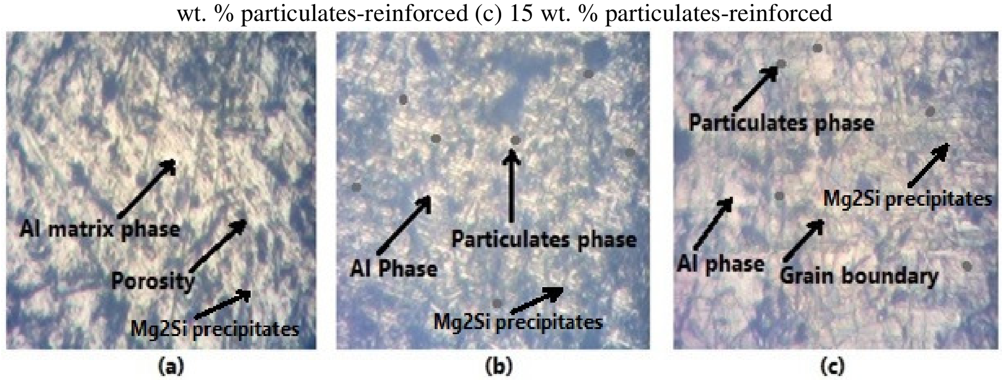

Figure 4: Optical micrographs of specimens heat-treated and quenched in salt water(x100) (a) unreinforced (b) 5 wt. % particulates-reinforced (c) 15 wt. % particulates-reinforced

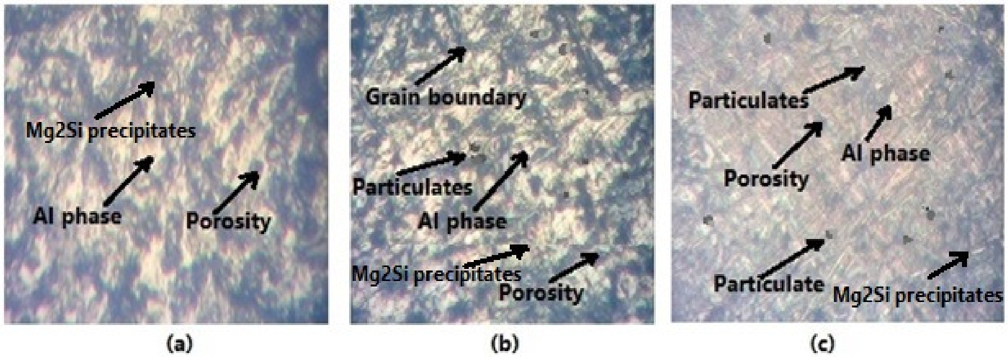

Figure 5: Optical micrographs of specimens heat-treated and quenched in water (x100) (a) unreinforced (b) 5 wt. % particulates-reinforced (c) 15 wt. % particulates-reinforced

3.2. Tensile Strength

The stress-strain curves of the specimens are shown in Figures 6, 7, 8 and 9. Among the non-heat-treated specimens, the 5 wt. % cow bone particulates-reinforced composite exhibited the highest tensile strength of 158.69 MPa as shown in Figure 6. However, when the specimens were age-hardened, some of them exhibited improved tensile strength. The 15 wt. % particulates-reinforced specimens exhibited higher tensile strength than other specimens in Figures 7 and 8. Furthermore, it exhibited the highest tensile strength of 179.96 MPa as shown in Figure 9. This shows that age-hardening enhanced the tensile strength with particulates addition. This could be due to precipitation hardening, consisting of fine, uniform precipitation of the solute particles obtained by aging from the supersaturated state of α-Al matrix (Kareem *et al.*, 2017). This agrees with the report by Agunsoye *et al.* (2013), Kareem *et al.* (2017) and Abdulkareem *et al.* (2019). The tensile strength of the composites is also enhanced because of the strong bonding of the particulates to the matrix, increased dislocation density near matrix-reinforcement interface, and the grain refining strengthening effect, which agrees with the report by Seshappa *et al.* (2018).

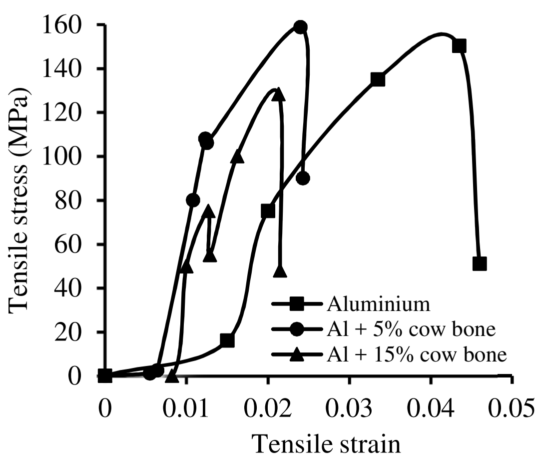

Figure 6: Stress-strain curves of the non-heat-treated specimens (Al alloy and composites)

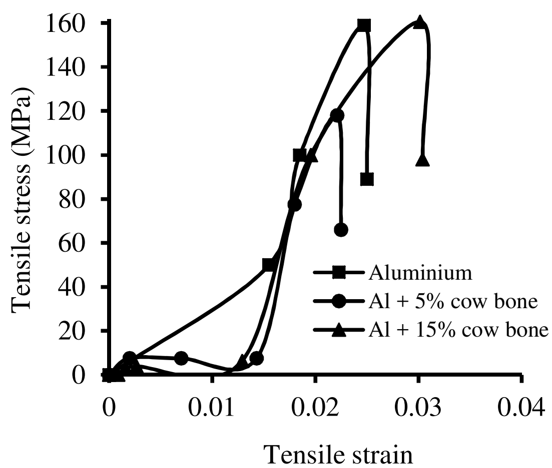

Figure 7: Stress-strain curves of the age-hardened specimens (water quenched)

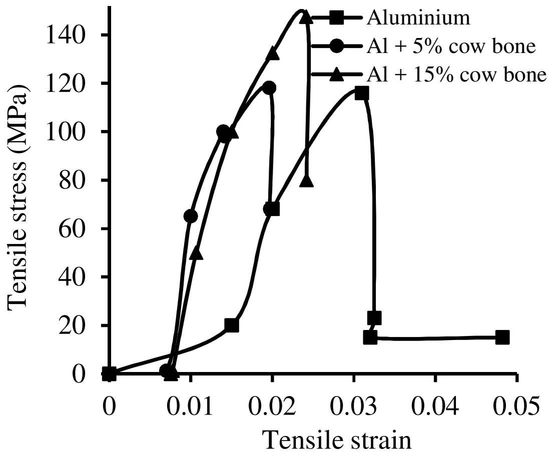

Figure 8: Stress-strain curves of the age-hardened specimens (palm oil quenched)

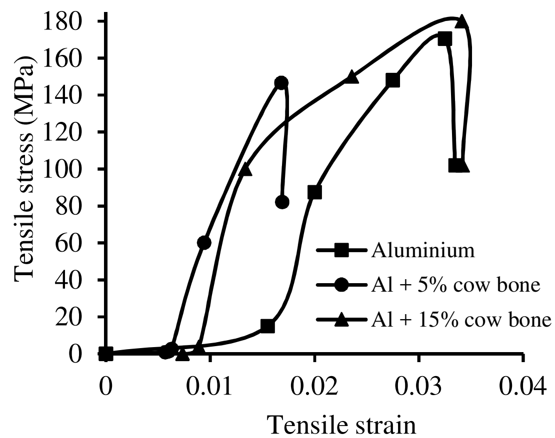

Figure 9: Stress-strain curves of the age-hardened specimens (salt-water quenched)

3.3. Hardness

The hardness values of the specimens increase in response to age-hardening compared to the untreated specimens as shown in Figure 10. Furthermore, there is an increase in the hardness as the weight fraction of cow bone particulates is increased in the specimens. The 15 wt. % cow bone particulates reinforced composite quenched in salt water exhibited the highest hardness value of 184.65 HV, which is 23.1% higher than hardness of the 15 wt. % cow bone particulates-reinforced composite that was not age-hardened. This is also 50% higher than hardness of unreinforced cast aluminium alloy. As shown in Table 2, cow bone contains calcium, which is hard, and the hardness of composite materials is directly proportional to the quantity of integrated hard particles (Milos *et al*., 2011). The presence of hard and well-bonded cow bone particulates in Al matrix, which impeded or restricted the movement of dislocations, increased hardness of the aluminium matrix composites. This agrees with the report by Seshappa *et al.* (2018) who investigated the effect of silicon carbide reinforcement on the mechanical properties of aluminum metal matrix composites.

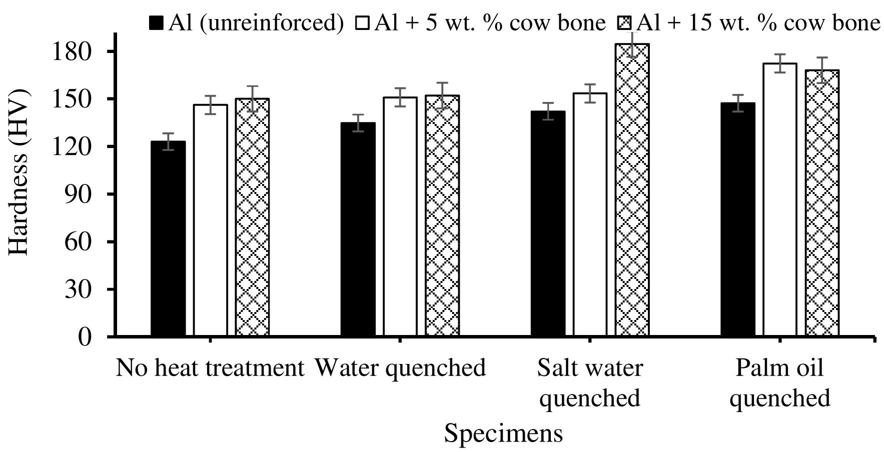

Figure 10: Hardness of as-cast aluminium alloy and aluminium matrix composites subjected to different ageing conditions

During quenching, the alloy quickly cooled from solution locking the strengthening elements within the aluminium matrix. The alloy after quenching, became meta-stable, where some silicon and magnesium crystals attempted to precipitate out as Mg_2Si but could not, since at room temperature, there was not enough energy for precipitation to occur (Kareem *et al*., 2017). During ageing, the specimens were raised to a high enough temperature (180 °C) to initiate precipitation of the Mg_2Si. During ageing, the precipitates diffused out as dispersed phases, which anchored the matrix and impeded deformation resulting in increased strength and hardness. Hence, the combined mechanisms of dispersion and precipitation hardening enhanced the mechanical properties of the specimens. Figures 6 – 11 show that there is an increase in strength and hardness due to ageing with corresponding decrease in impact energy values. This improvement is due to precipitation hardening, consisting of fine, uniform precipitation of Mg_2Si obtained by ageing from the supersaturated state of α-Al. These agree with the report by Kareem *et al.* (2017) who investigated the effects of precipitation hardening on mechanical properties of multistage stirred cast AA6063 composites.

3.4. Impact Energy

The untreated and unreinforced cast alloy exhibited an impact energy of 24.34 J as illustrated in Figure 11. The untreated aluminium matrix composite reinforced with 15 wt. % cow bone particulates exhibited the highest impact energy of 65.28 J. This was followed by the untreated aluminium matrix composite reinforced with 5 wt. % cow bone particulates, which exhibited an impact energy of 46.24 J. This shows an increase in the impact energy of the untreated composites with an increment in particulates addition. However, the age-hardened composites demonstrated an inverse response to impact as the addition of particulates increased in the composites. This implies that the subjection of the specimens to heat treatment in different quenching media reduces their impact energies (Kareem *et al.*, 2017). The reverse response of the specimens to the age-hardening process could be attributed to the presence of small precipitates within the aluminium matrix, which simultaneously precipitated out from the solid solution as the material is being treated (Kareem *et al.*, 2017). In addition, as the particulates increased in the matrix, they were no longer isolated in the ductile α-Al matrix. Hence, cracks were not arrested by the ductile matrix and gaps propagated easily between the particulates leading to reduced ability to absorb impact energy. The decrease in impact energy could be because higher percentage of hard reinforcing particulates may increase preference of grain segregation in the ductile matrix, which will consequently lead to snappy crack formation and ultimate rupture during impact (Palanivel and Koshy, 2011). This pattern of decrease in impact energy conforms to the report by Palanivel and Koshy, (2011) on increase in impact energy and drastic decrease at higher percentages of reinforcement.

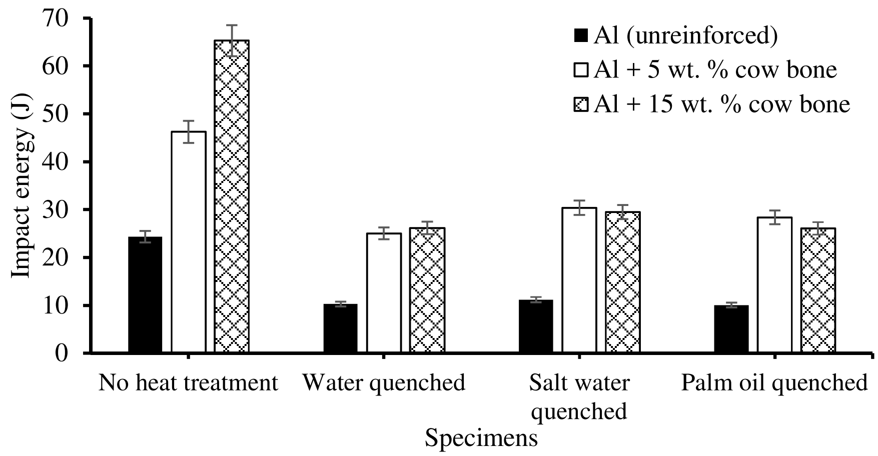

Figure 11: Impact energy of as-cast aluminium alloy and aluminium matrix composites subjected to different aging conditions

4. CONCLUSION

In this experimental study, aluminium matrix composites reinforced by 5 and 15 wt. % of cow bone particulates were developed by stir casting method and subjected to heat treatment processes. The effect of age-hardening on the mechanical properties of the composites was studied and compared with that of non-heat treated specimens. From the results of investigation and discussion of this research work, the following inferences can be drawn:

1. The microstructure of the composites revealed serial dislocation lines, which are the basis for the improvement in strength of the specimens.
2. There were minimal micro porosities in the microstructure of the casts, which indicates a fair uniform distribution of the hard cow bone particulates in the aluminium matrix.
3. The 15 wt. % cow bone particulates-reinforced composite quenched in salt water exhibited the highest tensile strength of 179.96 MPa. This shows that age-hardening enhanced the tensile strength with particulates addition.
4. The 15 wt. % cow bone particulates reinforced composite quenched in salt water exhibited the highest hardness value of 184.65 HV, which is 23.1% higher than hardness of the 15 wt. % cow bone particulates reinforced composite that was not age hardened. This is also 50% higher than hardness of unreinforced cast aluminium alloy.
5. The age-hardened composites demonstrated an inverse response to age hardening as the addition of particulates increased in the composites. The subjection of the specimens to heat treatment in different quenching media reduced their impact energies.
6. Generally, specimens that went through the salt-water quenching medium seemed to birth desirable properties with good cooling rate. Thus, choice of quenching medium and heat treatment are important parameters in developing better mechanical properties.
7. The specimens are suitable materials for engineering purposes due to increased strength. Thus, they can be used in areas where lightweight and high strength are required within the aerospace, automotive and electronic industries such as cylinder liners in engines, aluminium calipers and power electronic modules.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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