



## ORIGINAL ARTICLES

# Microstructure, mechanical and fracture properties of groundnut shell ash and silicon carbide dispersion strengthened aluminium matrix composites

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**Abstract** The mechanical properties of aluminium hybrid composites reinforced with groundnut shell ash (GSA) and silicon carbide was investigated. GSA and silicon carbide with different mix ratios (10:0, 7.5:2.5, 5.0:5.0, 2.5:7.5 and 0:10) constituted 6 and 10 wt.% of the reinforcing phase, while the matrix material was Al–Mg–Si alloy. The hybrid composites were produced via a two-step stir casting technique. Microstructural examination, hardness, tensile and fracture toughness testing were carried out to appraise the mechanical properties of the composites. The results show that with increasing GSA in the reinforcing phase, the hardness, ultimate tensile strength (UTS) and specific strength of the composites decreased slightly for both 6 and 10 wt.% reinforced Al–Mg–Si based composites owing to the amount of the oxides of Al, Si, Ca, K<sub>2</sub> and Mg present in the composition of GSA. However, the percentage elongation improved marginally and was generally invariant to increasing GSA content while the fracture toughness increased with increasing GSA content. GSA offered a favourable influence on the mechanical properties of Al–Mg–Si hybrid composites comparable to that of rice husk ash and bamboo leaf ash.

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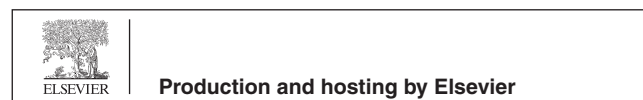
**1. Introduction**

Materials with good strength to weight ratio are becoming very essential in modern engineering designs especially for automotive and aerospace applications where improved machine efficiency and reduced fuel consumption are critical requirements to be satisfied. Also, modern infrastructures, equipment and machineries that are currently developed require materials that have a good combination of properties to match service demands. Aluminium matrix composites

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(AMCs) represent a class of materials that offer a wide range of properties that can measure up with the design requirements of some of the aforementioned applications (Surappa, 2003). AMCs are primarily reinforced with fibres or particulates which are usually ceramic materials (SiC, Al<sub>2</sub>O<sub>3</sub>, WC, B<sub>4</sub>C, TiO<sub>2</sub>, BN). They can be produced via solid route processing (such as powder metallurgy) and liquid metallurgy processing routes (rheocasting, compocasting, liquid infiltration, stir casting are a few examples) (Shabani et al., 2012; Kala et al., 2014). Without disregard to the technical competence of other processing routes available, stir-casting remains the most utilised technique due to its simplicity, flexibility, low cost acquiescence and commercial viability (Kala et al., 2014). Over the years, single reinforced composites have been mostly developed for use in several applications but have been observed to have some material property and cost related limitations (Dharmalingam et al., 2010). Efforts to optimise the performance of single reinforced MMCs and also to reduce the processing cost have paved way for the development of hybrid reinforced AMCs (Dharmalingam et al., 2010; Alaneme et al., 2014a).

In recent times, hybrid reinforced AMCs have attracted the interest of researchers and different design concepts have been adopted to select the appropriate combination of reinforcing materials (Alaneme et al., 2014b; Pandi and Muthusamy, 2012; Alaneme and Ajayi, 2015; Bodunrin et al., 2015). The combination of reinforcing materials has been noted to have an influence on the properties and processing cost of the composites. For example Iqbal et al. (2014), in their studies on the fatigue crack growth mechanism in cast aluminium hybrid composites reinforced with silicon carbide and alumina observed superior crack growth resistance in the lower stress intensity factor range for the composites containing both silicon carbide and alumina in comparison with aluminium matrix composite containing alumina alone. Siddesh Kumar et al. (2014), obtained superior wear resistance in MoS<sub>2</sub> and B<sub>4</sub>C hybrid reinforced aluminium composites as compared to the single reinforced aluminium/B<sub>4</sub>C composite. Montalba et al. (2015) reported that an increase in piezoelectric lead lanthanum zirconate titanate (PLZT) led to an improvement in the damping properties of aluminium based hybrid composites containing silicon carbide (SiC) and PZLT. Several other reports on property optimisation in metal matrix composites (MMC) using hybrid reinforcements are available in the literature (Dolata et al., 2012; Lei et al., 2014; Poovazhagan et al., 2013; Prasad et al., 2014). It can be noted from the above cited literature that synthetic reinforcements which are known to be relatively expensive and not readily available in most developing countries were used. Notwithstanding the attainment of improved properties in the respective cases, the problem of high processing cost was not addressed.

Alaneme et al. (2013a), Prasad (2006) and Escalera-Lozano et al. (2007) are among researchers that have considered the development of less expensive AMCs by using industrial and agro waste derivatives as reinforcing materials in hybrid reinforced AMCs. This class of AMCs is referred to as low-cost high performance composites since the incorporation of these reinforcing materials did not only reduce the cost of the composites but in most cases had a positive effect on the performance of the composites provided the mix ratio is monitored (Alaneme et al., 2014a). Among the most investigated industrial and agro waste derivatives that have been used as rein-

forcing materials in AMCs include coal fly ash (FA), red-mud, rice husk ash (RHA), bamboo leaf ash (BLA) and bagasse ash (Bahrami et al., 2015; Lancaster et al., 2013; Loh et al., 2013; David Raja Selvam et al., 2013; Soltani et al., 2015). These reinforcing materials (BLA-0.36 g/cm<sup>3</sup> and RHA-0.31 g/cm<sup>3</sup>) usually have lower densities than the synthetic reinforcing materials (silicon carbide-3.18 g/cm<sup>3</sup> and alumina-3.96 g/cm<sup>3</sup>). They are also readily available as wastes and from chemical analysis observed to contain refractory oxides such as aluminium oxide, iron oxides and silicon oxides that make them attractive as reinforcing materials. GSA obtained from combustion of groundnut shell is another agro waste derivative that should be considered as potential reinforcing material in composite development. Although, articles published on the use of GSA as reinforcement in aluminium matrix composites are sparse, there are strong reasons for its advocacy. Firstly, nominal chemical compositions of GSA from the literature show a high alumina and silica content which are known to function as reinforcing materials (Alaneme et al., 2015). Unlike the other agro waste derivatives (RHA and BLA) that have been investigated in the past, the silica content in GSA is slightly lower while the alumina content is higher when compared to RHA and BLA. Secondly, Nigeria is one of the largest producers of groundnut in the world producing, 2,962,760 tons after China (16,114,231 tons) and India (6,933,000 tons) in 2011 (Ibrahim et al., 2013). There is current effort to increase production capacity by additional 120,000 metric tons in the next few years. This implies that groundnut shells will contribute significantly to the solid waste in the country. Ground nut shell can potentially be processed for use as reinforcing material in AMCs, thus contributing to reduction in current environmental waste management challenges.

In this research work, we considered the use of groundnut shell ash (GSA) and silicon carbide as hybrid reinforcements in the development of Al-Mg-Si based composites. The microstructural features, density measurements and mechanical properties were investigated to ascertain the viability of using GSA as a reinforcing material in the development of aluminium matrix composites.

## 2. Materials and methods

### 2.1. Materials

Al-Mg-Si alloy billets with chemical composition: Al (98.71 wt.%), Si (0.45 wt.%), Fe(0.22 wt.%), Cu(0.02 wt.%), Mn (0.03 wt.%), Mg (0.50 wt.%), Cr (0.03 wt.%), Zn (0.02 wt.%), and Ti (0.02 wt.%); determined using spark spectrometric analysis, was selected as aluminium matrix for this investigation. Silicon carbide (SiC) and groundnut shell ash (GSA) were selected as reinforcing materials for the development of the hybrid composites. The silicon carbide procured was of high chemical purity with average particle size of 28 µm while groundnut shell was obtained from the dump site of an open market within Akure metropolis. The groundnut shell was processed by burning to obtain groundnut shell ash following procedures explained in detail by Alaneme et al. (2015). Briefly, dried groundnut shell was placed in a metallic drum and burnt in open air. The ash was collected after 24 h and then subjected to conditioning in a muffle furnace at a

temperature of 600 °C for 3 h. Groundnut shell ash of particle size less than 50 µm was used in the research. The chemical composition of the groundnut shell ash indicates it consists of: SiO<sub>2</sub> (34.2 wt.%), Al<sub>2</sub>O<sub>3</sub> (12.42 wt.%), Fe<sub>2</sub>O<sub>3</sub> (14.0 wt.%), CaO (14.3 wt.%), MgO (2.0 wt.%), Na<sub>2</sub>O (0.048 wt.%), K<sub>2</sub>O (15.46 wt.%), P<sub>2</sub>O<sub>3</sub> (2.1 wt.%), MnO (0.36 wt.%), SO<sub>3</sub> (0.64 wt.%), and LOI (4.85 wt.%). Magnesium for improving wettability between the Al–Mg–Si alloy and the reinforcements was also procured.

## 2.2. Composite production

Two step stir casting process was utilised to produce the composites in accordance with Alaneme and Aluko (2012a). The process started with the determination of the quantities of groundnut shell ash (GSA) and silicon carbide (SiC) required to produce 6 and 10 wt.% reinforcement consisting of GSA and SiC in weight ratios 0:1, 1:3, 1:1, 3:1, and 1:0 respectively. The groundnut shell ash and silicon carbide particles were initially preheated separately at a temperature of 250 °C to eliminate dampness which helps reduce particle clotting and improves wettability and dispersion of the particles with the molten Al–Mg–Si alloy. The Al–Mg–Si alloy billets were charged into a temperature controlled gas-fired crucible furnace and heated to a temperature of 750 °C ± 30 °C (above the liquidus temperature of the alloy) to ensure the alloy melts completely. The liquid alloy was then cooled in the furnace to a semi solid state at a temperature of about 600 °C. The preheated GSA and SiC particles along with magnesium were then charged into the semi-solid melt at this temperature (600 °C) and stirring of the slurry was performed manually for 5–10 min. The composite slurry was then superheated to 800 °C ± 50 °C and a second stirring performed using a mechanical stirrer. The stirring operation was performed at a speed of 400 rpm for 10 min before casting into prepared sand moulds fitted with metallic chills.

## 2.3. Sample designation

The composites produced were given designations based on the weight percent of the reinforcing phase and the weight ratio of SiC and GSA in the reinforcement. B1, B2, B3, B4, and B5 were used to designate the 6 wt.% reinforcements containing 100% SiC (1:0), 75% SiC + 25% GSA (3:1), 50% SiC + 50% GSA (1:1), 25% SiC + 75% (GSA), and 100% GSA (0:1), respectively. The designations C1, C2, C3, C4, and C5 were used as representations for the 10 wt.% reinforced composites containing 100% SiC (1:0), 75% SiC + 25% GSA (3:1), 50% SiC + 50% GSA (1:1), 25% SiC + 75% (GSA), and 100% GSA (0:1), respectively. The unreinforced alloy was designated as A0 to differentiate it from the composite grades.

## 2.4. Microstructural examination

The microstructures of the composites produced were examined using a JSM Jeol ultra-high resolution field emission gun scanning electron microscope (FEG-SEM). Prior to the examination, the surface of the samples were prepared by grinding and polishing following standard procedures. There-

after, samples were etched using Keller's reagent (95 ml water, 2.5 ml HNO<sub>3</sub>, 1.5 ml HCl, 1.0 ml HF).

## 2.5. Mechanical testing

Samples were machined from the as-cast composites using lathe machine for hardness, tensile and fracture toughness testing following standard procedures. Hardness test was conducted on the prepared samples using EmcoTEST DURASCAN microhardness testing machine at an applied load of 100 g for a dwell time of 10 s. Prior to hardness testing, the test samples were machined and polished to obtain a smooth plane parallel surface. A Vickers hardness scale was utilised for the hardness measurement following recommendations of ASTM E 92-82 standard (ASTM E92, 2004). Multiple hardness tests (a minimum of five measurements) were performed on each sample and the average value was taken as a measure of the hardness of the specimen. Tensile tests were performed on the tensile samples prepared from the as-cast composites in accordance with the specifications of ASTM E8M-91 standard (ASTM E8M, 2013). The samples for the test were machined to round specimen configuration with 6 mm diameter and 30 mm gauge length. The test was carried out at room temperature using an Instron universal testing machine operated at a quasi-static strain rate of 10<sup>-3</sup>/s. The strength and deformability parameters determined from the tensile test are ultimate tensile strength, yield strength, percentage elongation and the specific strength.

The crack propagation resistance (fracture toughness) of the composites was determined using a simplified fracture mechanics approach based on uniaxial tensile testing of circumferential notch tensile (CNT) specimens (Alaneme, 2011). The as-cast composites were machined for the CNT testing using gauge length, specimen diameter (*D*), notch diameter (*d*), and notch angle of 30 mm, 6 mm, 4.2 mm, and 60°, respectively. The specimens were then subjected to tensile loading to fracture using an instron universal testing machine. The fracture load (*P<sub>f</sub>*) was determined from the load-extension plots obtained and the fracture toughness (*K<sub>IC</sub>*) evaluated using the relation (Dieter, 1988):

$$K_{IC} = \frac{P_f}{(D)^{\frac{3}{2}} [1.72(\frac{d}{D}) - 1.27]} \quad (2.1)$$

The validity of the fracture toughness results assessed on the basis of the attainment of plain strain condition for the CNT fracture toughness evaluation was determined using relations specified by Nath and Dass (2006):

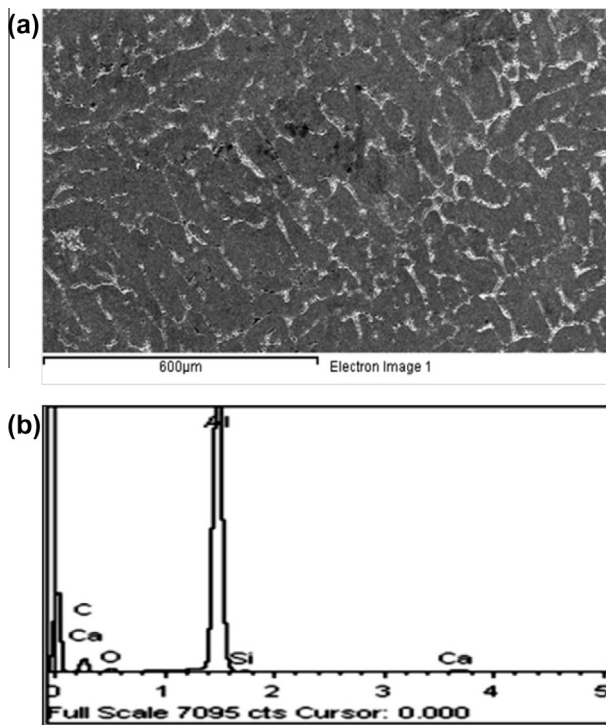
$$D \geq \left( \frac{K_{IC}}{\sigma_y} \right)^2 \quad (2.2)$$

where  $\sigma_y$ , represents the yield strength of the composite.

## 3. Results and discussion

### 3.1. Microstructure

The microstructure and EDS profile of a representative composition of the composite produced are presented in Fig. 1. The microstructure (Fig. 1a) shows the continuous phase which is aluminium with dispersed reinforcing particles. The



**Figure 1** Representative (a) microstructure and (b) EDS profile of Al-Mg-Si/10 wt% SiC-GSA composite produced (SiC:GSA mix ratio 1:1).

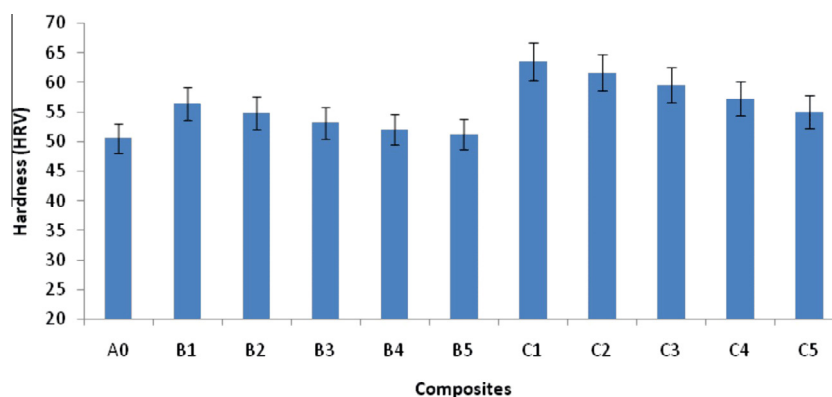
level of particle dispersion is easily discernible from the micrograph. The same microstructural attributes as those in Fig. 1 (a) were observed for the other composite grades produced for which reason their micrographs were not presented. The EDS profile (Fig. 1b) show the major elements present in the composites with Al, Si, Ca and O chiefly observable, confirming the presence of Al alloy, SiO<sub>2</sub>, CaO and Al<sub>2</sub>O<sub>3</sub>.

### 3.2. Mechanical behaviour

The variation of hardness of the Al-Mg-Si alloy, single and hybrid reinforced Al-Mg-Si/SiC-GSA is presented in Fig. 2. The single and hybrid composites had superior hardness values when compared with the unreinforced Al-Mg-Si alloy (A0)

while the hardness of the GSA containing composites decreased with increase in weight percent of GSA in the composite. The decrease in hardness of the composite as GSA content increases is due to lower hardness value of the dominant refractory oxides (SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>) present in the GSA as compared with silicon carbide (Accuratus, 2013). The percent reduction or increase in the mechanical properties of the hybrid composites presented in Table 1, was calculated using the composite solely reinforced with SiC (B1 and C1) as a reference. From Table 1, it is however observed that the percent reduction in hardness when the GSA constituted 75% of the reinforcing phase was 7.65% and 9.8% for the 6 and 10 wt. % SiC-GSA reinforced Al-Mg-Si alloy matrix composites respectively. In Al-Mg-Si/10 wt.% SiC-BLA composites containing 40% BLA in its reinforcing phase, a hardness reduction of 10.94% compared to the single reinforced SiC was observed as compared to a reduction of 6.1% observed for the SiC-BLA containing 50% GSA in the reinforcing phase (the closest mix ratio for comparison purpose). This clearly indicates that GSA has a less depreciating effect on hardness in comparison with BLA in Al-Mg-Si based hybrid composites. This behaviour is most likely due to the composition of GSA which has relatively more alumina compared to BLA (bamboo leaf ash) (Alaneme et al., 2013b).

Symmetrical to the hardness trend observed in Fig. 2, the ultimate tensile strength (UTS), yield strength (Fig. 3) and specific strength (Fig. 4) of the single and hybrid composites were higher than that of the unreinforced alloy. Also, the strength parameters increased as the weight fraction of the reinforcing phase increases from 6% to 10%. However, the strength parameters (UTS, yield strength and specific strength) decreased with an increase in the weight percent of GSA in the reinforcing phase. Direct (load transfer from matrix to the reinforcement) and indirect strengthening (increased dislocation density at the matrix-particles interfaces due to thermal mismatch during processing) mechanisms have been reported to be responsible for enhanced strength in AMCs (Chawla and Shen, 2001). The low elastic modulus and hardness value of the dominant oxides in GSA (alumina and silica) in comparison with SiC is responsible for the slight reduction in strength of the GSA containing composites. This is because the load bearing capacity of the reinforcing phase is reduced due to the presence of GSA. During processing, thermal mismatch between the GSA reinforcements and Al-Mg-Si matrix would result in high dislocation density at the matrix-reinforcement

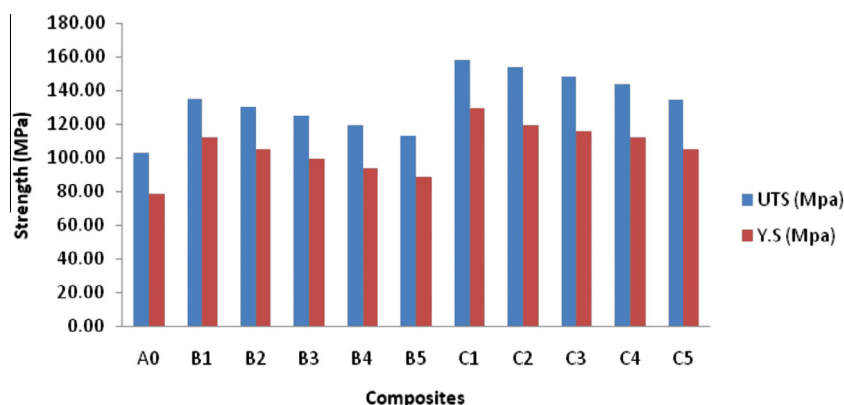
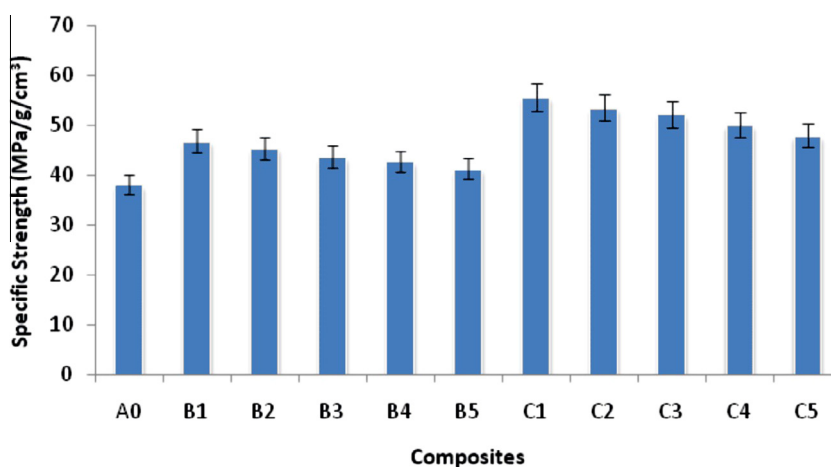


**Figure 2** Variation of hardness of the Al-Mg-Si based composites produced.



**Table 1** Percent reduction in mechanical properties with an increase in GSA content in the hybrid reinforced Al–Mg–Si/SiC–GSA composites.

Mix ratio of GSA and SiC in wt.%	% Reduction in hardness		% Reduction in UTS		% Reduction in specific strength		% Increase in elongation		% Increase in fracture toughness	
	6 wt.%	10 wt.%	6 wt.%	10 wt.%	6 wt.%	10 wt.%	6 wt.%	10 wt.%	6 wt.%	10 wt.%
	25:75	2.8	3	3.8	3	3.1	3.9	3.13	7.32	14.5
50:50	5.7	6.1	7.1	6.3	6.8	6.2	1.18	9.46	15.7	7.90
75:25	7.6	9.8	11.7	9	8.8	10	0.42	7.67	16.9	10.59
100:0	9.2	13	16.5	14.8	11.9	13.9	1.67	9.03	18.32	14.12

**Figure 3** Variation of ultimate tensile strength and yield strength of the Al–Mg–Si based composites produced.**Figure 4** Variation of specific strength and yield strength of the Al–Mg–Si based composites produced.

interfaces. This must have enhanced the strength in the single reinforced Al–Mg–Si/GSA composites (sample B5 and C5) despite having lower load bearing capacity as compared with SiC.

An analysis of the sensitivity of the composite strength parameters to GSA content is presented in Table 1. It is noteworthy that for both 6 and 10 wt.% reinforced Al–Mg–Si hybrid composites the reduction in UTS was less than 12% even when 75% of GSA constituted the reinforcing phase. Also, the reduction in specific strength did not exceed 10% when 75% of the reinforcing phase contained GSA in both 6 and 10 wt.% reinforced Al–Mg–Si hybrid composites. This

suggests that the presence of GSA did not have a significant effect on the strength of composites considering the low cost benefits being offered by GSA. Alaneme and Adewale (2013) studied the influence of SiC–RHA weight ratios on the mechanical SiC–GSA behaviour of Al–Mg–Si matrix hybrid composites. Three grades of the hybrid composites containing 5, 7.5 and 10 wt.% of the reinforcing phase were produced. They reported that increasing the weight percent of the SiC–RHA hybrid reinforcement increased the strength of the composites while the strength of the hybrid composites for all grades dropped with increasing RHA in the reinforcing phase. This is consistent with our observation on the strength of Al–

Mg–Si/SiC–GSA hybrid composites. The percentage reduction in UTS obtained for 5 wt.% reinforced Al–Mg–Si/SiC–RHA composites was given as 4.9%, 8.9% and 12.5% when the reinforcing phase constituted 25%, 50% and 75% of RHA respectively. Comparing these values to the percentage reduction in UTS obtained in 6 wt.% reinforced Al–Mg–Si matrix hybrid composite (Table 1), it is observed that GSA has less effect on the percentage reduction in UTS. This trend was also confirmed when comparing the percentage reduction obtained in 10 wt.% SiC–RHA reinforced Al–Mg–Si hybrid composites to 10 wt.% GSA–SiC reinforced Al–Mg–Si hybrid composites. The percentage reduction in UTS for 10 wt.% SiC–RHA reinforced Al–Mg–Si hybrid composites was 4%, 8.2% and 13.2% when RHA constituted 25%, 50% and 75% of the reinforcing phase respectively.

Fig. 5 shows the variation of percentage elongation of the unreinforced alloy and the Al–Mg–Si/GSA–SiC composites produced. It is observed that for both 6 and 10 wt.% compos-

ite grades, the elongation of the Al–Mg–Si hybrid composites containing GSA and SiC improved marginally in comparison to single reinforced Al–Mg–Si/SiC composites (B1 and C1). As shown in Table 1, the percentage increase in the elongation for Al–Mg–Si/6 wt.% GSA–SiC was less than 4% despite having the SiC replaced by 75% GSA while for Al–Mg–Si/10 wt.% GSA–SiC the percent increment, though greater than 4% was less than 10%. The slight improvement in ductility did not follow a consistent trend with increasing weight ratios of GSA and SiC. Samples B2 and C3 exhibited the highest strain to fracture among the hybrid composites. Similar trend of invariance of percentage elongation to increase in weight ratio of other agro waste derivative hybrid reinforced aluminium composites has been reported (Alaneme et al., 2013a; Alaneme and Adewale, 2013).

Representative stress–strain curves of the composites from which the tensile properties discussed above were derived are presented in Fig. 6.

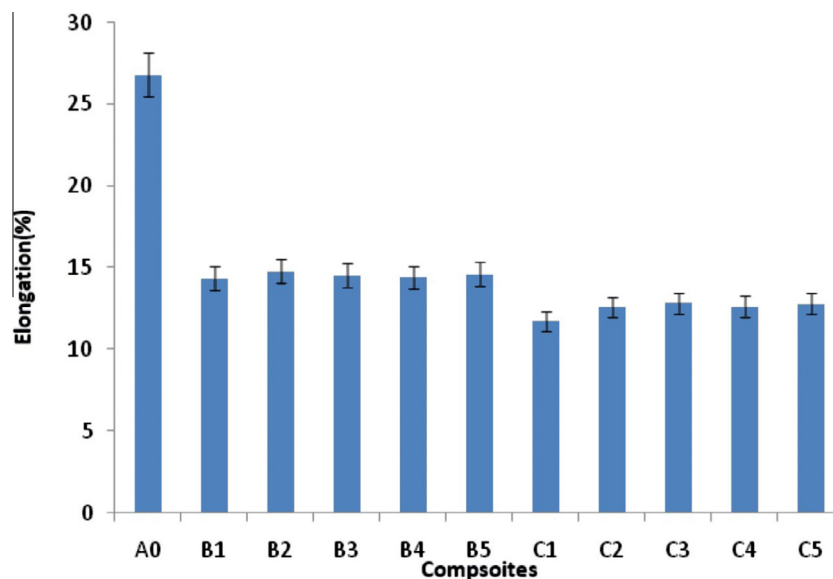


Figure 5 Variation of percentage elongation of the Al–Mg–Si based composites produced.

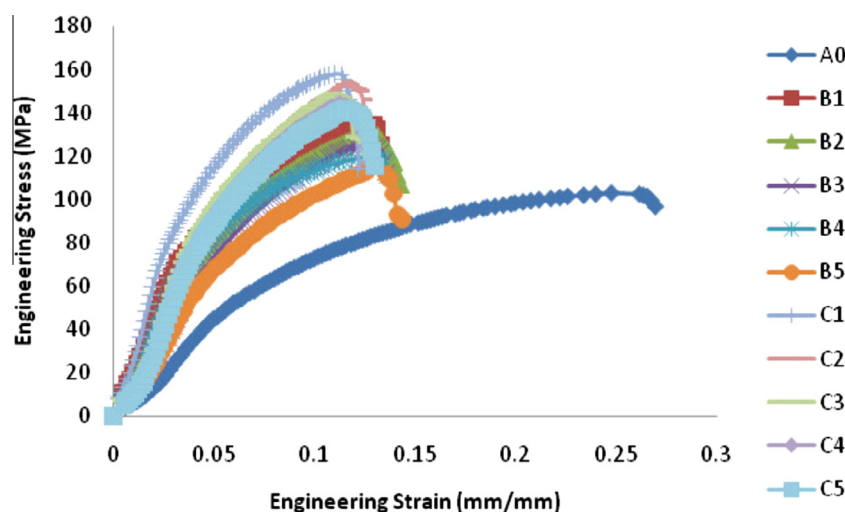


Figure 6 Representative stress–strain diagrams of the composites produced.

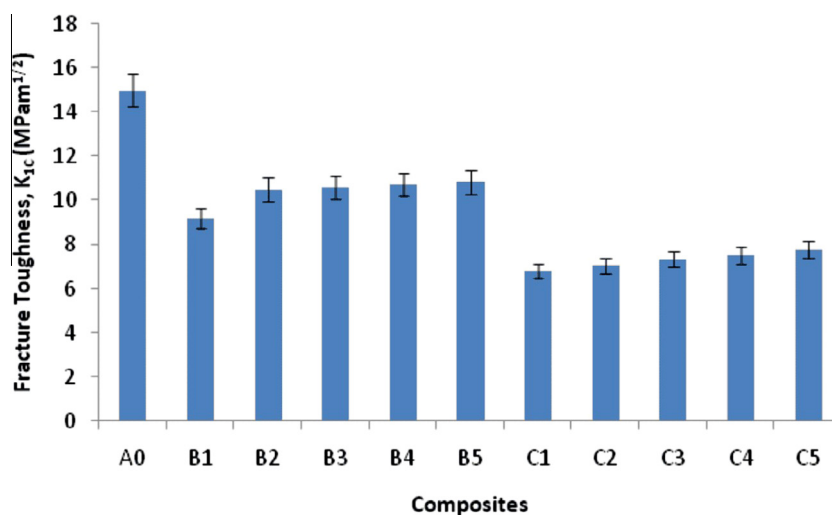


Figure 7 Variation of fraction toughness of the Al-Mg-Si based composites produced.

Fig. 7 shows the variation of fracture toughness of the unreinforced alloy, single and hybrid and Al-Mg-Si/SiC-GSA composites produced. The fracture toughness values were reported as valid plane strain fracture toughness since it met the conditions stated by Nath and Dass (2006). It is observed that fracture toughness of the composites is lower than the unreinforced alloy. Also, the hybrid counterparts have slightly superior fracture toughness when compared to single reinforced Al-Mg-Si/SiC composites (B1 and C1). Equally of note is the increase in fracture toughness with increase in GSA content for both 6 and 10 wt.% Al-Mg-Si/GSA-SiC composites. This indicates that the composites containing GSA are relatively less susceptible to crack propagation. This can be ascribed to the softness of GSA in comparison with SiC. The fracture micro-mechanism of particulate reinforced MMCs have been attributed to particle cracking, interfacial cracking and particle debonding (Alaneme and Aluko, 2012b). Hard and brittle ceramic particulates which SiC epitomizes are more susceptible to rapid crack propagation. The improvement in fracture toughness of the 6 wt.% Al-Mg-Si hybrid composites was much more significant than the 10 wt.% reinforced Al-Mg-Si hybrid composites. An average of 15.7% improvement in fracture toughness was achieved in 6 wt.% reinforced Al-Mg-Si/SiC-GSA composites while an average of 7.33% was achieved for other 10 wt.% reinforced composite grades.

Based on the aforementioned observations, it suffices to say that the incorporation of GSA as a complementing reinforcement in the development of Al-Mg-Si/SiC-GSA composites did not have an adverse effect on the mechanical properties of the composites. MMCs are mostly reported to exhibit enhanced strength at the expense of ductility and fracture toughness (Alaneme and Aluko, 2012b). However, the use of GSA as a complementing reinforcing material in the development of aluminium based hybrid composites showed a very slight reduction in strength levels while a modest improvement was achieved in ductility and fracture toughness of the composites. GSA can thus serve as an alternative to other agro-waste ash used as reinforcing materials despite the lower percentage of silica in its composition when compared with RHA and BLA that have been mostly investigated.

#### 4. Conclusion

- Hardness and tensile strength increased with increasing weight percent of the reinforcing phase but the strength and hardness dropped slightly with an increase in GSA content in the reinforcing phase. The percentage reduction in hardness and strength achieved in GSA containing composites were lower than both RHA and BLA reinforced aluminium hybrid composites reported in previous work.
- Percentage elongation improved marginally with increasing GSA content. The improvement did not follow a consistent trend with increasing SiC-GSA weight ratios.
- Fracture toughness ( $K_{1c}$ ) improved with an increase in GSA content. For the 6 wt.% Al-Mg-Si/SiC-GSA grades, an average improvement of 15.7% was achieved as compared to 7.33% achieved in the 10 wt.% composite grades.
- The use of GSA as complementing reinforcement is viable for the production of low-cost high performance aluminium matrix composites. GSA can be utilised as reinforcement based on its overall positive effect on the mechanical properties of the composites even at higher weight fractions in comparison to RHA and BLA.

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