Production and wear optimization of an MSSA-reinforced Al–Si–Mg composite: a Taguchi approach

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Abstract
In this study, Al–Si–Mg-MSSA composite is produced by reinforcing the aluminum alloy with mango seed shell ash (MSSA). For the development parameters of stirring time, processing temperature, MSSA content, and MSSA particle size, Taguchi design of experiment was employed for the optimization of the wear properties of the developed composites. The optimal wear rate of the MSSA-reinforced Al–Si–Mg composite was obtained to be 0.001517 mm³/N/m at stirring time, processing temperature, MSSA content, and particle size of 60 s, 720 °C, 20%, and 25 µm. Analysis of variance also proved the significance of MSSA particles in the reduction of the wear rate of Al–Si–Mg alloys. The wear behavior of the developed composite was successfully modeled using regression analysis with a prediction accuracy of 90.32%.

Keywords Composites · Aluminum matrix composites · Wear

1 Introduction
Aluminum metal matrix composites (AMCs) have found wide applications in our daily life because when in comparison with base aluminum alloy, they have exhibited good combinations of mechanical and tribological properties. With their unique properties, aluminum matrix composites have increasingly found application in industries like construction, automobile, and aviation. These unique properties such as low weight, high strength to weight ratio, low cost, and low wear rate make it a better choice for materials such as polymer composites. In respect to other metal matrix composites, AMCs have always been a better choice because of their corrosion resistance property, low density, good thermal conductivity, and low cost of production [1–4].

Using the Acheson process, some of these synthetic materials like silicon carbide (SiC) are prepared where silica sand and carbon are charged in the electric furnace-based reactors as source materials [5]. These processing techniques and the availability of these raw materials are a challenge in developing countries even though other authors have reported alternate processing methods like powder mixing, chemical vapor deposition, carbothermal reduction of silica, sol–gel processes, and also laser pyrolysis [1, 6, 7]. But all these methods also entail the use of advanced processing equipment which are not readily available in developing countries. The most cost-effective technique which poses no advanced equipment challenge is the carbothermal synthesis process which can be used in the production of silicon-based refractory compounds [6].

Agro-waste is continuously being explored as a reinforcement material for these AMCs, although some studies have used these agro-wastes with other synthetic materials (usually ceramics), forming a hybrid reinforcement for these metal matrix composites [8–10]. High silica agro-waste materials such as rice husk, mango seed shells, and donum palm seed shells are abundantly found in Nigeria [11]. These agro-wastes have been a source of environmental concern as their disposal poses risk to humans but they can also be harnessed and converted to silicon-based materials [12]. The

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availability, low weight, and low cost are a drive for its use as reinforcement for these AMCs. It has been reported that for metal matrix composites, these agro-wastes can either serve as direct precursors or can be ashed before further synthesis [13]. The produced ash serves as reinforcements in the development of the aluminum matrix composites. Some of the processes used in the development of these agro-waste ash–reinforced AMCs are liquid filtration, double stir casting, rheo-casting, and compocasting. The simplicity, flexibility, and viability of the double stir cast method have made it the most used in this development process.

Accessing synthetic reinforcement materials in some societies like developing countries is a challenge. Therefore, agro-waste materials, which have proven to be a viable alternative, must be developed. Different studies have been carried out on the use of agro-waste materials in the reinforcement of metal matrix composites. Adediran et al. [6] have successfully synthesized Si-based refractory compounds (SRC) from rice husk (RHs) via a carbothermal processing route. These SRCs comprise a good percentage of silicon carbide and other refractory compounds in their structures, thus making them a potential reinforcement material in the design of AMCs [14]. Also, RHs possess a good percentage of silica, hematite, and alumina as major refractory oxides in their structure [6].

Taguchi’s robust design of experiment method has successfully been applied to processes and systems in order to obtain the optimum performance with spending minimal resources [15]. The Taguchi optimization method involves the study of the effect of varied parameters with the S/N ratio. These S/N ratios are expressed in the form of either the “higher the better,” “the lower the better,” or “the nominal the best.” In the case of wear rate where the lower wear rate is expected to be achieved, “the lower the better” S/N ratio is applied. S/N ratio for the lower the better is expressed in Eq. 1.

\[ S/N_{LB} = -10 \times \log_{10} \left( \frac{1}{n} \sum_{i=1}^{n} y_i^2 \right) \] (1)

where \( S/N_{LB} \) represents the signal-to-noise ratio for the lower the better characteristics, \( y \) represents the responses, and \( n \) is the number of runs.

There appears to be sparse literature on the characterization of AMCs reinforced with Si-based refractory compounds derived from mango seed shells upon its abundance in Nigeria. The current study attempts to explore the mechanical and microstructural characteristics of aluminum metal matrix composites developed from Si-based refractory compounds of the mango seed shell. Si-based refractory compounds have been established to be an excellent wear-resistant reinforcement for aluminum-based alloys [6]. This present work takes advantage of the availability of the mango seed shell to develop a mango seed ash–reinforced Al–Si–Mg-MSSA composite with varying compositions of the MSSA content of 5%, 10%, 15%, and 20%. The surface properties of the composite materials are studied. The Al–Si–Mg would be sourced from scraped motorcycle hubs. The data from these findings would add to the existing database of cost-effective and technically efficient secondary reinforcement phases to improve aluminum metal matrix composites.

2 Experimentation

2.1 Materials and equipment

Some of the equipment used include scrap motorcycle hubs, charcoal furnace and stirrer, digital weigh balance, cope and drag, a die cavity mold, a scanning electron microscope (SEM), a wear testing machine, and an optical microscope. The mango seeds (agro-waste) were obtained from consumed ripened mango fruits.

2.2 Methodology

2.2.1 MSSA preparation

The mango seed shells were collected, washed thoroughly, dried in the sun for 3 days, decorticated manually to remove the seed from the shell, and ground into powder. The powder of MSSA was packed in a steel box and ashed in an airtight condition in a furnace at 600 °C for 4 h to obtain the ash. The ash was sieved using a sieve size of 25 µm, 50 µm, 75 µm, and 100 µm. These ashes are used in the processing of the Al–Si–Mg-MSSA composite.

2.2.2 Production of the Al–Si–Mg alloy

Scrap motorcycle hubs were collected, melted in the furnace, poured into bar-shaped mold cavities, and allowed to cool. The aluminum matrix obtained from the typical hub of motorcycle was broken down into small sizes and weighed. This makes it easier to charge into the crucible. A sand mold was prepared with diameter 30 mm and the length 80 mm and used to produce the test bars. The scrap motorcycle hub to be melted was first charged into the crucible. The scrap motorcycle hub was melted and the crucible was heated to 750 °C in order to superheat the aluminum alloy melt.

2.2.3 Al–Si–Mg-MSSA production

The Al-7%Si-0.3%Mg bars already produced from the scrap motorcycle hubs were reheated to 660 °C in the crucible. The mango seed shell ash was heated to 120 °C to dry off possible moisture absorbed or oxides and other volatile materials.
The heated ash particles were activated by further heating the ash to 500 °C. The MSSA was added to the molten aluminum alloy in varying proportions of 5%, 10%, 15%, and 20% respectively according to the conditions of the Taguchi orthogonal design. The crucible in which the bars were melted again was removed from the furnace and there was continuous stirring for 30, 60, 90, and 120 s with a stirring speed of 100 rpm upon the addition of MSSA for dispersion of the ash in the aluminum alloy at a temperature of 690 °C, 720 °C, 750 °C, and 780 °C respectively. The composite was then poured into a mold and left to cool at room temperature.

2.2.4 Wear test

The test was carried out according to the ASTM D 6079–97/EN 590 standards using the Anton Paar Strasse 208,054 Wear testing machine. The surfaces of the samples which were cut to dimensions 10 mm × 10 mm × 3 mm were prepared metallographically and polished. Acetone was used in cleaning the surface of the polished samples and the steel balls which were then allowed to dry naturally. During the test, the depth of wear and the coefficient of friction were observed for 90 s. The sliding against the test samples was achieved in a reciprocating motion mode at a frequency of 5 Hz and a reciprocating speed of 1.5 mm/s. Other parameters employed include an 8 N nominal force delivered at 0.1 m/s² acceleration, 1.5 m/s velocity, and 3 mm stroke. The experiment was conducted at 28 °C ambient temperature. The motion was reciprocating sliding, with a steel ball diameter of 3.967 mm and grade 24, 440, with a preload of 10 s. The experiment was repeated thrice and the average results calculated. Also, using the parameter obtained, the wear rate was derived in line with the method of Adegbenjo et al. (2018) using single-trace analysis.

2.2.5 Microstructural characterization

The inverted-type metallurgical microscope (Nikon, RangeX50 to X1500) at 200 times magnification was used for the microstructural study. Scanning electron microscopy (SEM) was used for the study of the surface morphology of fractured surfaces of the developed composites. The scanning electron microscope was operated at 15 kV and a working distance of 15 mm, selected areas of interest were focused, and micrographs were taken as reported in Ramezani et al. [16] and Hoseinzadeh et al. [17]. Before the microscopic examination, the samples were metallographically prepared using a series of grinding and polishing steps. Samples were then etched using Keller’s reagent (1.0 ml HF, 1.5 ml HCl, 2.5 ml HNO₃, 95 ml water).

3 Results and discussion

3.1 Optimization of Al–Si–Mg/MSSA particulate composite for low wear rate

The wear rate of the developed composites is presented in Table 1.

| Runs | Parameter settings | Wear rate |
|------|---------------------|-----------|
|      | Stir. time (sec)   | Pro. temp. (°C) | MSSA (wt. %) | Part. size (µm) | Mean (mm³/N/m) | S/N (dB) |
| 1    | 30                  | 690        | 5           | 100           | 0.04499       | 26.9377  |
| 2    | 60                  | 720        | 5           | 75            | 0.02248       | 32.9641  |
| 3    | 90                  | 750        | 5           | 50            | 0.01538       | 36.2609  |
| 4    | 120                 | 780        | 5           | 25            | 0.02036       | 33.8244  |
| 5    | 60                  | 750        | 10          | 100           | 0.03131       | 30.0863  |
| 6    | 30                  | 780        | 10          | 75            | 0.01348       | 37.4062  |
| 7    | 120                 | 690        | 10          | 50            | 0.01783       | 34.9770  |
| 8    | 90                  | 720        | 10          | 25            | 0.02371       | 32.5014  |
| 9    | 90                  | 780        | 15          | 100           | 0.01788       | 34.9526  |
| 10   | 120                 | 750        | 15          | 75            | 0.01628       | 35.7669  |
| 11   | 30                  | 720        | 15          | 50            | 0.05062       | 25.9136  |
| 12   | 60                  | 690        | 15          | 25            | 0.03671       | 28.7043  |
| 13   | 120                 | 720        | 20          | 100           | 0.01318       | 37.6017  |
| 14   | 90                  | 690        | 20          | 75            | 0.01614       | 35.8419  |
| 15   | 60                  | 780        | 20          | 50            | 0.02452       | 32.2096  |
| 16   | 30                  | 750        | 20          | 25            | 0.04225       | 33.0632  |

Table 1 Wear rate of the Al–Si–Mg composites
Table 1 shows the wear rate of the developed Al–Si–Mg/MSSA particulate composite at different runs (combinations). It also depicts the wear rate properties of the materials through the experimental runs carried out during the study and the signal-to-noise ratio. From Table 1, the wear rate general mean and the S/N ratio mean of the developed Al–Si–Mg/MSSA composites are 0.02432 mm³/N/m and 33.0632 dB respectively. The S/N ratio was calculated from the lower the better performance characteristics for the wear rate.

Table 2 is the response table for the wear rate of the developed composites where the wear rate of the composite was 0.02580 mm³/N/m, 0.02158 mm³/N/m, 0.03037 mm³/N/m, and 0.02402 mm³/N/m with S/N ratios of 32.50 dB, 33.74 dB, 31.33 dB, and 33.28 dB at stirring time of 30 s, 60 s, 90 s, and 120 s respectively. At processing temperatures of 690 °C, 720 °C, 750 °C, and 780 °C, the mean wear rate was 0.02684 mm³/N/m, 0.01709 mm³/N/m, 0.02709 mm³/N/m, and 0.03076 mm³/N/m with S/N ratios of 32.39 dB, 35.49 dB, 32.34 dB, and 35.54 dB respectively. The mean wear rates under the influence of MSSA content 5%, 10%, 15%, and 20% were 0.03784 mm³/N/m, 0.02875 mm³/N/m, 0.01828 mm³/N/m, and 0.01691 mm³/N/m with S/N ratios of 29.44 dB, 30.99 dB, 34.89 dB, and 35.54 dB respectively. The wear rates in respect to the particle size of MSSA at 100 µm, 75 µm, 50 µm, and 25 µm were 0.02892 mm³/N/m, 0.02750 mm³/N/m, 0.02631 mm³/N/m, and 0.01906 mm³/N/m at means and 31.62 dB, 32.25 dB, 32.40 dB, and 34.60 dB at S/N ratios. The stirring time had the highest rank with a difference between the highest and lowest mean wear rate as 0.00879 mm³/N/m and the MSSA reinforcement had a difference between the highest and lowest mean wear rate as 0.00986 mm³/N/m.

Figure 1 shows the effect of stirring time on the wear rate of the Al–Si–Mg/MSSA composite. There was a wear rate reduction with an increase in stirring time from 30 to 60 s. Figure 1 shows the lowest mean wear rate of 0.02158 mm³/N/m at means and 33.74 dB at S/N ratio on a stirring time of 60 s and highest wear rate at 90 s. Generally, it is observed that the wear rate increased with the increase in stirring time beyond 60 s. But at a much higher stirring time, i.e., greater than 90 s, the wear rate reduced. This is not disconnected from the fact that with more stirring time, there will be increased dispersion of the secondary phases in the alloy as reported in Ayar et al. [18].

The effect of processing temperature on the wear rate of Al–Si–Mg/MSSA composite is shown in Fig. 2. It shows that the wear rate of the composite decreases with the increased processing temperature from 690 to 720 °C where the lowest wear rate was observed. This is due to the ease of solidification within these temperatures. Beyond 720 °C, the wear rate increased with an increase in pouring temperature due to the formation of voids by bubbling at these higher temperatures. Above 720 °C, wear rate increases, probably due to formation of complex compound SiAl at higher temperature affecting the composite nature of the microstructure.

### 3.1.1 Effect of MSSA content on the wear rate of Al–Si–Mg/MSSA composite

Figure 3 shows the variation of wear rate with MSSA content. It shows that the wear rate reduces with an increase in MSSA content. The ash particles form a lubricating point at surfaces and an increase in these particle content increases the wear inhibition mechanism. The rate of reduction of wear is linear until 15% MSSA content where the rate of reduction in wear rate is reduced and this is due to the increase in more crystalline materials within the phases as observed by Stalin et al. [19].

### 3.1.2 Effect of particle size on the wear rate of Al–Si–Mg/MSSA composite

Figure 4 shows the variation of wear rate with particle size from 25 to 100 µm. It shows that the wear rate decreases with a decrease in particle size in agreement with Vishal et al. [20] who stated that particle size governs the mechanical and tribological characteristics of such composite materials. At 100 µm particle size, the peak of wear rate was observed to be 0.02892 mm³/N/m. The best (lowest) wear rate general mean and the S/N ratio mean of the developed Al–Si–Mg/MSSA composites are 0.02432 mm³/N/m and 33.0632 dB respectively. The S/N ratio was calculated from the lower the better performance characteristics for the wear rate.
rate (0.01906 mm³/N/m) was observed to be at the smallest particle size of 25 µm attributable to enhanced compatibility or setting over the grain boundary.

Figures 5, 6, 7, and 8 show the interaction effect of MSSA content (in weight percent), stirring time, processing temperature, and particle size. It could be observed that the highest
wear rate was observed at MSSA content between 12 and 18% and at a low stirring time of less than 60 s. So also, the wear rate was observed to be the highest at MSSA content between the range of 12 and 18% and pouring temperature of 700–740 °C. The highest wear rate was also observed at particle size within 30–70 µm. Figure 8 shows the interaction between stirring time and particle size.

![Graph showing variation of wear rate mean and S/N ratio with MSSA content](image1)

![Graph showing variation of wear rate mean and S/N ratio with particle size](image2)
3.1.3 Analysis of variance for the wear rate of Al–Si–Mg/MSSA composite

Table 3 shows the analysis of variance of the wear rate of Al–Si–Mg/MSSA composite for the various factors that were considered. The MSSA reinforcement has the highest percentage contribution to the wear rate of the composite. Implying that for the factors considered, the MSSA content contributes 38.09% to the wear rate. In order to reduce the wear rate, the MSSA content must be taken into consideration. The particle size contributed 13.21% to the wear rate of the composite material. With a $P$ value less than 0.05 at a 95% confidence level, the MSSA content was observed to be a significant factor in the wear rate of the developed material.

3.1.4 Optimal wear rate of Al–Si–Mg/MSSA composite

The optimal development combination of the factors for the development of Al–Si–Mg/MSSA composite for low wear is obtained from the response table. From Figs. 1, 2, 3, and 4, it could be observed that the best (optimum) development parameters are stirring time of 60 s, processing temperature at 720 °C, MSSA content of 20%, and particle size of 25 µm, which is A2B2C4D4. The optimal wear rate
of the composite was predicted using Eq. 2 to be 0.00168 mm$^3$/N/m at means and 40.1804 dB at S/N ration.

$$W_{opt} = W_{St} + W_{Pr} + W_{Ma} + W_{Ps} - 3W_m$$  \hspace{1cm} (2)

where $W_{opt}$ is the optimal wear rate, $W_{Pr}$ is the lowest wear rate at the processing temperature, $W_{Ma}$ is the lowest wear rate at varied mango seed shell ash contents, and $W_{Ps}$ is the lowest wear rate at different varied particle sizes.

**Fig. 7** Interaction of MSSA content with particle size

**Fig. 8** Interaction of stirring time with particle size
To confirm the predicted optimum wear rate, a confirmatory experiment was carried out with the optimum set of parameters of A2B2C4D4. The confidence interval calculated from Eq. 3 showed that the interval lies on ±0.00021 mm³/N/m of the predicted optimal wear rate.

$$f_{\text{tab}(1,\text{DoF}_e)} \times V_e \times \left( \frac{1}{E} + \frac{1}{R} \right)$$  \hspace{1cm} (3)

Confidence interval =

Regression equation : Wear rate = 0.538 – 0.00689 A + 0.000134 B – 0.00332 C – 0.00073 D  \hspace{1cm} (4)

Table 3 Analysis of variance of means for wear rate of Al–Si–Mg/MSSA composite

| Source                     | DF | Adj SS   | Adj MS   | F      | P      | Percentage contribution |
|----------------------------|----|----------|----------|--------|--------|------------------------|
| Stirring time (sec.) (A)   | 1  | 0.00002  | 0.00002  | 0.25   | 0.627  | 1.07                   |
| Processing temperature (°C) (B) | 1  | 0.000014| 0.000014 | 0.17   | 0.686  | 0.75                   |
| MSSA reinforcement (wt. %) (C) | 1  | 0.000715| 0.000715 | 8.94   | 0.012  | 38.09                  |
| Particle size (µm) (D)     | 1  | 0.00248  | 0.00248  | 3.11   | 0.106  | 13.21                  |
| Error                      | 11 | 0.00088  | 0.00088  |        |        |                        |
| Total                      | 15 | 0.001877 |          |        |        |                        |

Table 4 Observation of wear rate confirmation test

| S/N | Trial number | Average wear rate (mm³/N/m) | S/N ratio (dB) |
|-----|--------------|----------------------------|----------------|
| 1   | 1            | 0.00151                    | 56.38          |
| 2   | 0.00151      |                            |                |
| 3   | 0.00153      |                            |                |
| 4   | 0.00157      |                            |                |

3.1.5 Wear modeling

A mathematical model for the combination of stirring time (A), processing temperature (B), MSSA content (C), and particle size (D) was derived from regression analysis carried out using the Minitab® 19 statistical software which was used for the prediction of the wear rate properties of the developed composite. The regression analysis model is presented in Table 6.

Table 6 shows the regression analysis model for the wear rate. Also, Eq. 4 shows the regression model for the wear rate for the factors considered. R-square value of 90.32% shows the accuracy of the regression model developed. It explains the suitability of the model in predicting the wear rate under the factors considered. A comparison of the predicted wear rate using the regression equation and the experimental values of the Al–Si–Mg/MSSA composite is shown in Fig. 9.

3.2 Microstructural analysis

3.2.1 Microstructural analysis using SEM

Figure 10 shows an almost uniform distribution of Al–Si–Mg alloy consisting of primary grains of α-Al solid solution (white) surrounded by interdendritic regions of coarse plates of Al-Si eutectic (deep black) in which various intermetallic

Table 5 Confirmatory test result for the optimal wear rate of Al–Si–Mg/MSSA composite

| S/N ratio (dB) | Optimal process parameter settings | Predictive values | Experimental values |
|----------------|-----------------------------------|-------------------|---------------------|
| 40.1804        | A2B2C4D4                          | 56.38             |                     |
| Mean wear rate | A2B2C4D4                          | 0.00168           | 0.001517            |
phases are present including the precipitates of Mg$_2$Si inter-metallic compound, etched with Keller’s solution.

At 5%wt content, the mango shell ash (MSSA) particle reinforcement in the grain boundaries of the Al–Si–Mg matrix was observed. The structure reveals the precipitates of Mg$_2$Si and platelet of eutectic Si particles in α-Al matrix with MSSA particles.

Also, at 15% wt MSSA content, there was more presence of the reinforcement within the grain boundaries of the Al–Si–Mg matrix. The structure revealed the precipitates of Mg$_2$Si and networks of eutectic Si particles in α-Al matrix with MSSA particles. This showed that there was good interfacial bonding between the 15% MSSA particles and the Al–Si–Mg matrix. The presence of magnesium in the matrix helps in enhancing the wettability of the MSSA particles in the metal matrix.

A higher percentage of the presence of reinforcement within the grain boundaries of Al–Si–Mg matrix was observed at the 20% wt content of MSSA reinforcement. The structure revealed the precipitates of Mg$_2$Si and networks of eutectic Si particles in α-Al matrix with non-uniform distribution of mango seed shell ash particles (MSSAp). The excess presence of reinforcement (MSSAp) beyond 15% resulted in a poor distribution of MSSAp in the aluminum matrix. This made the composite slurry, too thick, and reduce the fluidity of the molten metal which adversely affected the mechanical and physical properties of this sample, etched with Keller’s solution.

Table 6 Regression analysis model for wear rate

| Term                       | Coef   | SE coef | T value | P value | VIF |
|---------------------------|--------|---------|---------|---------|-----|
| Constant                  | 0.04207| 0.0091  | 4.62    | 0.001   |     |
| Stirring time (s) (A)     | 0.000173| 0.000965| 0.18    | 0.861   | 1   |
| Processing temp. (℃) (B)  | 0.000435| 0.000386| 1.13    | 0.284   | 1   |
| MSSA content (wt.%) (C)   | −0.000146| 0.000039| −3.8    | 0.003   | 1   |
| Particle size (µm) (D)    | −0.000615| 0.000386| −1.59   | 0.139   | 1   |

R-square = 0.9032

Fig. 9 Predicted and experimental wear rate of Al–Si–Mg/ MSSA composites
4 Conclusion

1. Al–Mg–Si /MSSAp composite was successfully developed using the stir casting technique and the samples were used for the wear test.

2. The addition of mango seed shell ash particulate to Al–Si–Mg has resulted in microstructural changes which confirm that it can be used as a reinforcement for AMCs.

3. The optimal wear rate of the MSSA reinforced Al–Si–Mg composite is 0.001517 mm$^3$/N/m at stirring time, processing temperature, MSSA content, and particle size of 60 s, 720 °C, 20%, and 25 µm.

4. The wear behavior of the developed composite has been successfully modeled with a prediction accuracy of 90.32%.
Availability of data and material Not applicable.

Code availability Not applicable.

Declarations

Ethics approval Not applicable.

Consent to participate Not applicable.

Consent for publication Not applicable.

Competing interests The authors declare no competing interests.

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