SLIDING WEAR CHARACTERISTICS OF BORON CARBIDE AND NOVEL SQUID QUILL ASH REINFORCED ALUMINIUM 6061 HYBRID COMPOSITES

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Reinforcement of metallic and non-metallic particulates will enhance or improve the tribological and mechanical properties of metal matrix composites. In the present study, Aluminium 6061 was reinforced with agro-waste based novel Squid Quill Ash (SQA) and Boron Carbide (B₄C) by employing a stir casting process. Sliding wear behavior of Al/B₄C/SQA hybrid composites investigated by varying SQA content (0.5%, 1%, and 1.5%), sliding speed (400, 500, and 600 RPM), and applied load (10, 20, and 30 N) using Pin on Disc apparatus. Experiments were performed based on Taguchi L27 orthogonal design. The role of control parameters on wear rate was estimated using the Analysis of Variance method. Statistical analysis showed that applied load has a significant effect on the wear rate of the hybrid composites followed by SQA content and Sliding Speed. Material removal during wear test mainly due to the combined effect of formation of cracks, abrasion, and grooves on the target surface.

Keywords: squid quill, wear rate, hybrid composites, reinforcement, sliding distance

INTRODUCTION

Composite materials are extensively used in engineering applications due to low weight to strength ratios, better mechanical properties, and wear resistance over conventional alloys. Aluminium (Al) is the most commonly used matrix material in the modern composite industries[1][2]. Al was first produced in a laboratory in 1825 from aluminium chloride[3]. Since the 1960s Al has been reinforced with fibers, particles, or whiskers, and adding particles into matrix material was known as dispersion strengthening; and these materials are commonly known as metal matrix composite (MMCs)[4][5]. Modern industries focused to reduce the weight of automobile structures, and the use of Al in automobiles was increased[6]. Improving the strength and toughness of Al will help in replacing the steel and ferrous metals in automotive structures[7][8]. The mechanical and tribological properties of the Al matrix composites were enhanced by reinforcing ceramic particles into the matrix during the casting process[9]. Boron Carbide, Silicon Carbide, Aluminium Oxide, Silicon Oxide, Graphite, Tungsten Carbide, Magnesium Oxide, etc. are the common ceramic particulates used in metal matrix industries[10][11]. Recently researchers were started to explore the feasibility of non-metallic particulates like rock dust, rice husk ash, fly ash, bone ash, etc. as reinforcements with metal matrix[12]. Reinforcement of non-metallic particulates with Al will enhance the mechanical properties and also reduce the manufacturing cost of the structural components[13]. Particulates derived from agro-industrial waste can be considered as the reinforcement material and combined with metallic reinforcements to enhance the properties of MMCSs[5]. Agro-waste produced by the meat industries specifically bones has a higher potential to be a part of the composite industry. Ochieze et al.[14] estimated the coefficient of friction and wear depth of Al356 alloy reinforced with cow horn particulates. Analysis showed that the wear rate decreased as the percentage of particulates increases (20%). Wear loss is mainly characterized by scars, pits, and grooves on the target surface. A similar kind of study was conducted by Babaremu et al.[15] by reinforcing cow horn and corn cob particulates in different weight percentages with Al 8011 alloy. Specimen containing 20% of cow horn and corn cob particulates showed improvement in tensile strength and yield stress by 54% over the control sample. The hardness of the specimen containing 15% cow horn and corn cob particulates improved by 52.6% over the control sample. As the percentage of cow horn increased the hardness value of the hybrid composites decreased. Zinc-Aluminium (ZA27) alloy reinforced with lamb bone ash (LBA) and Boron Carbide (B₄C) showed better tensile, hardness, and compressive strength as compared to base alloy[16]. The density of the composites reduced by incorporating LBA particles. As the weight percentage of LBA content increased porosity and cluster formation of reinforcements were increased. Hybridization of Al matrix with SiC and agro-waste based rice husk has improved the coefficient of friction of the material. Reinforcement of hybrid materials with Al alloy improved the corrosion resistance as compared to base alloy[17]. Studies conducted by Alaneme et al. showed the addition of 6 to 10% of ground nutshell ash and Silicon Carbide with Al enhanced the fracture toughness and failure strain of the material [18]. Hardness of the hybrid composites were decreased with the increase in the weight percentage content of ground nut shell. The available study shows that the incorporation of agro-waste into metal matrix composites will enhance the mechanical,
corrosion, and wear properties of the material. In this present study; an attempt has been made to improve the sliding wear characteristics of Al matrix material reinforced with B₄C (1%) and novel Squid Quill Ash (SQA), which was derived from agro-waste in different weight percentages (0.5%, 1%, and 1.5%). The Squid Quill is a transparent, tough, and flexible skeletal element of squid fish and is made up of chitin[19]. These bones are rich in Calcium Carbonate and are converted into SQA and reinforced with Al. The influence of process parameters on wear characteristics of Al/ B₄C/SQA was studied according to Taguchi L27 orthogonal array. An optical microscope was used to identify the mode of material removal from the target surface.

EXPERIMENTAL METHODS

Materials

In the present study, Aluminium alloy 6061(Al-6061) containing 97.9% aluminium, 1% Magnesium, 0.6% Silicon, 0.28% Copper, and 0.2% Chromium was used as the matrix material. Al 6061 was chosen as a matrix material due to its high strength to weight ratio and heat treatability. The Al-6061 is reinforced with commercial grade B₄C having a grain size of 60 µm[9] and novel SQA. During the fabrication process, the weight percentage of B₄C maintained as constant (1%) and SQA was varied in three levels (0.5%, 1%, and 1.5%). A large number of Squid Quill were collected and were washed with tap water to remove the flesh and other wastes. Cleaned Squid Quill were dried in room temperature for 1 week and dried Squid Quill is shown in Figure 1(a).

![Figure 1: Structure of Squid Bone (a) Cleaned, (b) Burnt particles, (c) Morphology of the particles](image)

Dried Squid Quill were placed inside the muffle furnace at 400°C for 30 minutes. The white unpleasant smoke raised from the furnace indicated the complete burning of Squid Quill. These burnt black crystals of Squid Quill were taken out from the furnace and cooled to room temperature and is shown in Figure 1(b). After cooling the burnt Squid Quill were crushed into a fine powder and sieved into 100 µm to form dark black crystal powders known as Squid Quill or Cuttlefish ash (SQA). The prepared SQA was in the form of flakes as shown in Figure 1(c). The particle size of prepared SQA was heterogeneous and was varied between smaller to 100 µm.

Specimen preparation

The stir casting process was used to fabricate the specimens containing Al 6061 as matrix material and B₄C and SQA as reinforcements. B₄C mixed with SQA as a reinforcement to increase the intermolecular bonding between the reinforcements and matrix material. 800 grams of Al 6061 were placed inside the stir casting equipment and melted at a temperature of 750°C. The reinforcement particles were mixed with the molten Al using an electric stirrer and soon after mixing the particles the furnace closed to prevent energy losses. The molten Al-containing B₄C and SQA as reinforcements were poured into a preheated mold to prepare the required specimens. Three sets of stir casting trials were performed to get three different samples of Al reinforced with 1% B₄C and three different weight percentages of (0.5%, 1%, and 1.5%) SQA respectively and designated as Al/B₄C/SQA hybrid composites. After the post-fabrication process, all as-cast samples were subjected to homogenization process at 480°C to improve the structural grain distribution in the materials.

Sliding wear test

The tribological performance of the Al/B₄C/SQA hybrid composites was studied as per ASTM G 99-17 using Pin on Disk apparatus supplied by Magnum Engineers, Bangalore, India in ambient conditions. The equipment used in the present work can be used to study the friction and wear characteristics of the material in sliding by varying the normal load, rotational speed, and wear track diameter. The apparatus consists of a carbon steel rotating disc and stationary specimen holder as shown in the schematic representation Figure 2. Electronic sensors are used to record the tangential force and wear as a function of load and speed.

![Figure 2: Schematic representation of the pin on disc wear test](image)
Specimens for the sliding wear test were prepared as per ASTM G 99-17 with dimensions of 10 mm diameter and 25 mm in length[20]. The specimens made to rotate on Carbon steel disc having a diameter of 100 mm and thickness of 10 mm. The specimen sliding radius for all wear test were maintained as 40 mm. The contact surface area of the specimens was made flat to maintain the uniform contact over the rotating Carbon steel disc. Sliding wear tests were performed by varying rotational speed, applied load, and weight percentage of SQA for a sliding distance of 500 m. The wear rate of the specimens were estimated based on the weight loss method. The initial and final weight of the samples were measured using the digital weighing scale with an accuracy of 0.001 grams. The wear and wear rate of specimens were measured using Equation (1) and (2) respectively[21]:

\[ \text{Wear (W)} = W_i - W_f \]  
\[ \text{Wear Rate} = \frac{\text{Wear loss in grams (W)}}{\text{Sliding Distance in meters (SD)}}, \text{g/m} \]  

Where \( W_i \) and \( W_f \) are the initial and final weight of the specimens in grams respectively. The sliding distance (SD) of each experiment was maintained as 500 m.

**Design of Experiments**

The sliding wear properties of the material depended upon different process parameters such as applied load, sliding distance, sliding speed, the composition of the material, type of sliding disc, etc. To understand the effect of process parameters on the sliding wear of the material, experiments were performed according to the Taguchi statistical method. Taguchi analysis method is a systematic experimentation method that is mainly used to optimize the control parameters and to understand the contribution of control parameters on performance output. In the present study, pin-on-disc wear experiments were performed by varying three process parameters such as applied load, sliding speed, and weight percentage of SQA of the specimens. These three process parameters varied at three levels and the corresponding operating conditions at which the sliding wear tests are carried out are given in Table 1. Sliding wear experiments were performed as per L27 orthogonal design by varying process parameters at ambient conditions.

**RESULTS AND DISCUSSION**

**Elemental Analysis of SQA**

Scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDS) technique used to identify the structure and composition of the SQA. SEM images of SQA revealed the irregular and flaky shapes of particles due to the shear action during the bone-crushing process (Figure 1 (c)). Figure 3 represents the SEM micrograph of SQA and the corresponding EDS spectrum. EDS spectrum shows the composition of SQA and revealed the presence of a large amount of carbon content. The addition of carbon-rich filler material into the Al matrix will enhance the hardness and strength of the material.

**Statistical Analysis**

Sliding wear experiments were conducted as per Taguchi L27 orthogonal design by varying process parameters at different levels. Table 2 shows the experimental wear rate in microgram/meter (µg/m) obtained during the sliding wear test conducted at various combinations of process parameters. Sliding wear was estimated in each experiment according to Equation (2). Two sets of experiments were conducted in each combination of process parameters and average wear rate used as a process output response for the statistical analysis. The experimental sliding wear rate was converted into a signal-to-noise (S/N) ratio. In the present study, the main objective is to minimize the wear rate of the composites and lower the better characteristics approach of the Taguchi experimental design used in the analysis as per Equation (3)[22].

\[ S_N = -10 \log \left( \frac{1}{n} \sum y^2 \right) \]  

Where \( y \) is the observed data, \( n \) and \( n \) is the number of observations.
Table 2: Experimental wear rate for Taguchi L27 design

| Experiment Number | Process Parameters | Wear Rate (µg/m) |
|-------------------|-------------------|-----------------|
|                   | SQA content (%)   | Sliding Speed(RPM) | Load (N) |                  |
| 1                 | 0.5               | 400             | 10       | 1.9               |
| 2                 | 0.5               | 400             | 20       | 4.4               |
| 3                 | 0.5               | 400             | 30       | 4.6               |
| 4                 | 0.5               | 500             | 10       | 2.5               |
| 5                 | 0.5               | 500             | 20       | 3.3               |
| 6                 | 0.5               | 500             | 30       | 3.1               |
| 7                 | 0.5               | 600             | 10       | 4.4               |
| 8                 | 0.5               | 600             | 20       | 3.7               |
| 9                 | 0.5               | 600             | 30       | 3.3               |
| 10                | 1.0               | 400             | 10       | 3.9               |
| 11                | 1.0               | 400             | 20       | 4.6               |
| 12                | 1.0               | 400             | 30       | 5.1               |
| 13                | 1.0               | 500             | 10       | 3.8               |
| 14                | 1.0               | 500             | 20       | 4.2               |
| 15                | 1.0               | 500             | 30       | 5.5               |
| 16                | 1.0               | 600             | 10       | 2.6               |
| 17                | 1.0               | 600             | 20       | 4.3               |
| 18                | 1.0               | 600             | 30       | 4.9               |
| 19                | 1.5               | 400             | 10       | 3.7               |
| 20                | 1.5               | 400             | 20       | 3.6               |
| 21                | 1.5               | 400             | 30       | 4.8               |
| 22                | 1.5               | 500             | 10       | 1.9               |
| 23                | 1.5               | 500             | 20       | 2.9               |
| 24                | 1.5               | 500             | 30       | 4.6               |
| 25                | 1.5               | 600             | 10       | 2.3               |
| 26                | 1.5               | 600             | 20       | 3.2               |
| 27                | 1.5               | 600             | 30       | 4.8               |

ANOVA Analysis on wear rate

Analysis of variance (ANOVA) method is used to identify the influence of individual and interaction effect of process parameters on the wear rate of Al-containing B4C and SQA as reinforcements. ANOVA analysis carried out using Minitab software at a confidence level of 95%. To identify the influence of parameters on wear rate experimental values converted into S/N ratio[23][24][25][26]. The ranking of process parameters on wear rate based on the S/N ratio is shown in table 3. From the analysis, it can be observed that load applied during wear test has a significant effect (39.64%, p-value=0.022) on wear rate. Apart from the applied load, wear rate is influenced by the percentage of SQA content (14.31%) and by sliding speed (4.71%). In the process parameter interaction effect; wear rate is influenced by the interaction between SQA content and sliding speed (8.35%), followed by SQA content and applied load (7.09%). The effects of interaction between sliding speed and applied load negligible with a percentage contribution of 0.88%.

Table 3: Response table for S/N ratio (smaller is better)

| Level  | SQA content | Sliding Speed | Load |
|--------|-------------|---------------|------|
| Low    | -10.495     | -11.894       | -9.144 |
| Medium | -12.541     | -10.567       | -11.497 |
| High   | -10.573     | -11.149       | -12.969 |
| Delta  | 2.046       | 1.327         | 3.825 |
| Rank   | 2           | 3             | 1     |
Table 4: Analysis of variance for wear rate with percentage of contribution

| Source          | DF | Seq SS  | Adj SS  | Adj MS  | F-value | P-value | Percentage of Contribution |
|-----------------|----|---------|---------|---------|---------|---------|---------------------------|
| SQA %           | 2  | 24.191  | 24.191  | 12.095  | 2.29    | 0.164   | 14.31                     |
| Sliding Speed   | 2  | 7.96    | 7.96    | 3.979   | 0.75    | 0.502   | 4.71                      |
| Load            | 2  | 67.009  | 67.009  | 33.504  | 6.33    | 0.022   | 39.64                     |
| SQA %*RPM       | 4  | 14.124  | 14.124  | 3.531   | 0.67    | 0.632   | 8.35                      |
| SQA %*Load      | 4  | 11.984  | 11.984  | 2.995   | 0.57    | 0.694   | 7.09                      |
| Sliding Speed *Load | 4  | 1.481   | 1.481   | 0.371   | 0.07    | 0.989   | 0.88                      |
| Residual Error  | 8  | 42.316  | 42.316  | 5.2895  |         |         | 25.03                     |
| Total           | 26 | 169.064 |         |         |         |         | 100.00                    |

Influence of process parameters on the wear rate

The influence of individual process parameters on the wear rate of Al/B₄C/SQA hybrid composites is shown in Figure 4.

![Figure 4: Main effects of process parameters on wear rate for Al/B₄C/SQA](image1)

Effect of process parameter on erosion process can be identified based on the slope of the line with respect to the central horizontal line of the plot. Figure 4 indicates that, applied load has the highest slope compared to the slope of the other parameters. The wear rate of Al/B₄C/SQA composites increases with the applied load and decreases with the addition of a higher percentage of SQA and sliding speed. The lowest wear rate was observed at lower applied load and the highest percentage of SQA and medium sliding speed. The interaction effect of process parameters on wear rate is shown in Figure 5. The wear rate of Al/B₄C/SQA is influenced by the interaction between the percentage content of SQA and sliding speed. Interaction between the percentage content of SQA and applied load is also shown a slight influence on the wear rate of the specimens.

![Figure 5: Interaction effect of process parameter on the wear rate](image2)

Surface analysis

An optical microscope (Olympus, BX53M) is used to capture the images of worn surfaces, and wear patterns were identified. For the analysis purpose, two specimens having low (1.5% SQA, 500 RPM, 10 N) and high (1% SQA, 500 RPM, 30N) wear were considered and images were captured at 100X and are shown in Figure 6 (a) and (b) respectively.

![Figure 6: Surface morphology of worn surface (a) Low wear, (b) High wear](image3)
The specimens having lower wear rates (Figure 6(a)) shown three types of failure patterns such as abrasion, cracks, and grooves. Material removal in this case mainly due to the formation of grooves on the target surface. Whereas abrasion and formation of multiple cracks in the target surface resulted in a high wear rate (Figure 6(b)). The lower content of SQA in the Al matrix resulted in the formation of cracks on the target surface. Formation of cracks on target surface due to abrasion between Al and sliding disc resulted in higher wear rate.

**CONCLUSIONS**

The present study is an attempt to utilize agro waste in the fabrication of hybrid metal matrix composites. Squid Quill Ash and Boron Carbide successfully reinforced with Aluminium matrix material. Sliding wear characteristics of the hybrid composites studied as per Taguchi L27 orthogonal array and the following conclusions were drawn.

- The wear performance of the hybrid composites depended upon the applied load, SQA content, and sliding speed. The applied load has a significant contribution (39.64%, p-value=0.022) on the wear rate of the composites.
- The wear rate of the hybrid composites decreased by increasing the SQA content (1.5%). EDS analysis revealed the presence of carbon content in SQA and helped in improving the wear characteristics of the composites.
- The formation of grooves, abrasion process, and cracks on the target surface dominated the material removal rate.

**Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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