Tribological performance of aluminium hybrid self-lubricating composites reinforced with green synthesized graphene

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Abstract

In this contemporary research work, graphene nanosheets were synthesized by the green method using agricultural waste and characterized by FESEM, XRD and Raman spectroscopy. Vortex liquid metallurgy technique is used to fabricate AA7075 hybrid composites reinforced with graphene and hard ceramic. The samples were evaluated for tensile strength, hardness and tribological behaviour. The influencing wear parameters percentage of graphene reinforcement, load, sliding speed and sliding distance were varied three levels each. An empirical relationship was formulated using face-centred central composite design considering wear weight loss and coefficient of friction as output responses. The influencing parameters on the output responses were determined by employing analysis of variance and the objective is to find the optimal process parameters. Graphene enriched the mechanical and wear behaviour. The composite having a 0.5 reinforcement percentage of graphene exhibited higher hardness and higher tensile strength. The optimal combination of tribological parameters for minimum wear loss and friction coefficient was found for 0.5% of graphene reinforcement, 30 N load, 2.5 m s⁻¹ sliding speed, 1000 m sliding distance. The worn surface was also analyzed using FESEM and inverted microscope images.

1. Introduction

Aluminium hybrid metal matrix composites (AHMMC) with ceramics, nanoparticles and other reinforcements have been inherited in various applications of defence, aircraft and marine structures, automobile and tribological components because of their distinct characteristics, mechanical stability and durability at elevated temperature, high strength and fatigue resistance, high toughness and stiffness, low density compared to that of monolithic aluminium alloys, controlled thermal expansion coefficient, excellent wear and corrosion resistance [1, 2].

Stir casting, also known as the vortex method, is a cost-effective and commonly used method for producing near-net-shaped AHMMC in which reinforcements are uniformly distributed into the matrix by stirring the molten liquid melt [3, 4]. Ceramic particulate reinforcements like Al₂O₃, TiC, SiC, TiO₂, ZrO₂ are generally used as reinforcements to improve the overall efficiency and strength of the composites. Among which, boron carbide (B₄C) is explored as kind of a substitute to SiC and Al₂O₃ as a result of its distinct traits, such as high hardness, high wear-resistant, specific property of bio isotope to neutron absorptions, low density, good thermal and chemical stability with that of aluminium matrix finding applications in storage tank material in the nuclear industry, tank armour, ceramic bearings, cutting tools, a replacement for Be alloys in the aerospace industry [5, 6]. Vettivel examined the mechanical characteristics of Aluminium 7075 reinforced with boron carbide and rice husk ash and it was discovered that B₄C is the influencing factor for increased tensile strength and hardness [7]. Baradeswaran evaluated the wear rate of Aluminium 7075/B₄C composites in which he concluded that B₄C particles transferred the load on the sliding surface, causing to decrease in wear rate [8].
Graphene, an advanced engineered material known for its exceptional properties of high intrinsic mobility, optical transparency of 97.7%, one TPa of young's modulus value, stiffer and thinnest material, extraordinary electrical and thermal conductivity values having wide applications in transistor and semiconductor industry, reinforcements in advanced composites, aircraft material, sensors, solar panels, batteries [9, 10]. Many researchers have synthesized graphene using conventional techniques. However, each approach has its own set of benefits and drawbacks. The simplest technique to produce graphene is by mechanical exfoliation, but the yield obtained is low and time-consuming. The thickness of the graphene was not uniform ordered after sonication and centrifugation in the liquid exfoliation process [11]. The usage of toxic reducing agents like KMnO4 and H2SO4 and hazardous gas emissions increases the complexity in using the hummers process [12]. The high purity graphene could be achieved using the chemical vapour deposition technique, but it has difficulty achieving uniform carbon deposition and thickness control complications [13]. The ordered and controlled graphene could be formed by epitalial growth on SiC but at the operating at a higher temperature.

Graphene have been utilised as reinforcement to enhance strength in advanced composites. Marc Leparoux investigated the mechanical characteristics of aluminium matrix composites (AMC) reinforced with graphene oxide made through powder metallurgy, concluding that the inclusion of graphene oxide greatly increased the hardness and ultimate tensile strength (UTS) when compared to pure aluminium alloy [14]. Haiyan Gao investigated the properties of graphene nanoplatelets reinforced AMC fabricated by continuous casting. The load transfer on GNP increases the UTS [15]. The compressive yield strength and microhardness of graphene nanoplatelets (GNPs) reinforced with AZ31 magnesium alloy were found to be significantly improved utilising the stir casting process, indicating good wettabiliy between matrix and GNP [16].

In this present work, a novel, green and efficient graphene was synthesized using a natural precursor. The synthesized graphene was incorporated into the aluminium matrix employing the liquid metallurgy technique. Mechanical properties like microhardness and tensile strength were studied. Additionally, the tribological performance was also studied by varying the tribological parameters and were optimized.

2. Experimentation

2.1. Materials
Aluminium 7075, having zinc as the predominant alloying element, is selected as the matrix because of its high strength and exceptional mechanical properties. AA7075 is extensively used in highly stressed parts such as gears, sprockets, aircraft fittings, and defence applications [17]. B4C with 325 mesh size is selected as the ceramic reinforcement.

2.2. Green synthesis of graphene
The agricultural wastes creating an environmental impact has made researchers focus on their utilization of it. Rice Husk Ash (RHA), one among them having significant elements of 91% of SiO2, 4% of carbon, 3% of Al2O3 and other minor elements, have widely used in applications to produce all forms of silica, porous and activated carbons, binder material in the construction industry, anti-cleansing agents, water purification, solar panels and many others [18].

Rice husk (RH) received from agricultural lands were washed with distilled water and dried. Brown RH was heated for one hour at 200 °C to obtain black RHA. 20.0 g of potassium hydroxide (KOH) was mixed with 5.0 g of RHA in a mortar. Ceramic wool was surrounded around the crucible compacted with the KOH:RHA mixture. Few grams of RHA are filled at the top layer to prevent the oxidation reaction. The crucible was placed in a larger crucible and it was annealed at 800 °C for two hours. After activating, the sample was subjected to continuous stirring for about seven hours in deionized water to eradicate the entrapped elements. Further, it was filtered and the sample was exposed to the drying process.

2.3. Characterization
The crystal structure was studied using an X-Ray diffractometer using Cu-Kα radiation source at a scan range from 10 to 80 degrees. The morphology of the synthesized graphene was examined with the field emission scanning electron microscope (FESEM). The vibrational modes of molecules present in the sample were examined using Raman spectroscopy (LabRAM HR800) at 532 excitation wavelength.

2.4. Preparation of the composite
AHMMC were produced by the controlled bottom pouring stir casting technique. AA7075 was introduced into the graphite coated furnace and heated at 850 °C till the metal to be melted. B4C and graphene (nGr) were preheated at 300 °C for 30 min to increase the wettabiliy of the matrix and the reinforcement. The graphene reinforcement is varied as 0.2%, 0.5% and 0.8%, while the percentage of B4C is kept constant as 5%. The mould
was preheated at 300 °C to achieve uniform solidification. 1% of magnesium is added into the melt, which acts as a degassing agent and improves the bonding. The impeller was lowered into the metal matrix and stirred at 500 rpm. The preheated reinforcements were introduced into the vortex of the liquid melt developed and the stirring was continued for 5 min to ensure uniform reinforcement distribution in the matrix. The molten liquid melt was poured into the preheated die and subjected to solidification. Figure 1 depicts the process of green synthesis of graphene and tribological testing of hybrid composite fabricated by stir casting technique.

### 2.5. Mechanical and wear testing

The casted samples were machined and evaluated for their tensile strength as per the ASTM E8 (Longitudinal direction) standard using an automated servo-hydraulic tensile test machine (FSA M100). The Vickers hardness of the composite was examined using Mitutoyo HM 200 automatic microhardness tester. Before testing, the samples were polished using abrasive grits of grade from 200 to 1200. The ASTM E384 was used to perform the

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**Figure 1.** Process flow chart of synthesis of graphene and fabrication of composites.

**Figure 2.** FESEM surface morphology of hexagonal fashioned rice husk derived graphene.
hardness test, with each sample being subjected to a load of 100g at three separate locations and the average results were taken.

The dry sliding wear test was done using a computer-controlled DUCOM pin-on-disc wear tester following ASTM G99. Prior to the testing, the end surface to be tested were polished using emery paper. The rotating disc was cleaned before and after using acetone for each test to remove the debris present on the counter surface. The sample was pressed against EN 31 steel of 64 HRC having the size of diameter 165 mm and 8 mm thickness. The sliding path on the sample surface is considered circular. The wear weight loss was computed and assessed by weighing the sample before and after the test.

2.6. Response surface methodology
The influence of prominent tribological parameters graphene reinforcement (%), load (N), sliding speed (m s\(^{-1}\)), and sliding distance (m) on wear loss and coefficient of friction (COF) was investigated using a face-centred central composite design (FCCCD). The tribological testing was carried out as per FCCCD of 30 experimental runs for four independent factors at three levels coded as \(-1\) (minimum), 0 (central), +1 (maximum). Table 1 shows the process parameters and their levels. The total 30 experimental design consists of six replicate central points, eight axial points and sixteen factorial points. For FCCCD, \(\pm \alpha = \pm 1\), indicating axial points at the centre of each face of factorial space [19]. The statistical and regression analysis of the quadratic model was carried out using Design Expert v.11. and each coefficient is tested at 95% confidence limits.

3. Results and discussion

3.1. Characterization and mechanical properties evaluation
Thin layered hexagonal structures of graphene flakes were formed with sharp edges eliminating the amorphous carbon through the insinuation of potassium atoms into rice husk ash [20], as shown in figure 2. The broad diffraction peak formed at 26.3° in the XRD spectra, as shown in figure 3(a), corresponds to the breakage of amorphous carbon and the development of graphene in the 002 plane [21]. Raman spectra (figure 3(b)) estimates the layer thickness of graphene as well as its defectiveness. Discarded and structural defects are represented by the D band at 1356 cm\(^{-1}\). The G band at 1588 cm\(^{-1}\) is responsible for the in-plane vibrations of C–C stretching. The development of sp\(^2\) hybridised carbon atoms is confirmed by the 2D band at 2731 cm\(^{-1}\).
Figure 4. Comparison of mechanical test results (a) Tensile test. (b) Vickers hardness test.

Table 2. Tensile and hardness test results.

| Sample | Alloy/Composite      | Tensile Strength (N mm\(^{-2}\)) | Vickers Hardness (VHN) |
|--------|----------------------|----------------------------------|------------------------|
| Sample 1 | AA 7073 base alloy   | 190.23                           | 109.4\(^a\)           |
| Sample 2 | AA 7073 + 5% B\(_4\)C | 235.07                           | 156.4                  |
| Sample 3 | AA7073 + 5% B\(_4\)C + 0.2% nGr | 271.11                           | 203.6                  |
| Sample 4 | AA 7073 + 5% B\(_4\)C + 0.5% nGr | 281.65                           | 219.5                  |
| Sample 5 | AA 7073 + 5% B\(_4\)C + 0.8% nGr | 245.56                           | 207.6                  |

Table 3. Dry sliding wear test results for 30 experimental runs.

| Exp.Run | Reinforcement (%) | Load (N) | Sliding Speed (m s\(^{-1}\)) | Sliding Distance (m) | Wear weight loss (g) | COF   |
|---------|-------------------|----------|------------------------------|----------------------|----------------------|-------|
| 1       | 0.8 (+1)          | 40 (+1)  | 1.5 (+1)                     | 2000 (+1)            | 0.0404               | 0.4525|
| 2       | 0.5 (0)           | 20 (−1)  | 2.5 (0)                      | 1500 (0)             | 0.0307               | 0.4256|
| 3       | 0.8 (+1)          | 20 (−1)  | 3.5 (+1)                     | 2000 (−1)            | 0.0395               | 0.4417|
| 4       | 0.8 (+1)          | 40 (+1)  | 3.5 (+1)                     | 2000 (+1)            | 0.0418               | 0.4793|
| 5       | 0.5 (0)           | 30 (0)   | 1.5 (−1)                     | 1500 (0)             | 0.0328               | 0.4424|
| 6       | 0.5 (0)           | 30 (0)   | 2.5 (0)                      | 1000 (−1)            | 0.0278               | 0.4167|
| 7       | 0.5 (0)           | 30 (0)   | 2.5 (0)                      | 1500 (0)             | 0.0282               | 0.4261|
| 8       | 0.5 (0)           | 40 (+1)  | 2.5 (0)                      | 1500 (0)             | 0.0297               | 0.4386|
| 9       | 0.5 (0)           | 30 (0)   | 2.5 (0)                      | 1500 (0)             | 0.0285               | 0.4266|
| 10      | 0.2 (−1)          | 40 (+1)  | 3.5 (+1)                     | 1000 (−1)            | 0.0331               | 0.4528|
| 11      | 0.5 (0)           | 30 (0)   | 2.5 (0)                      | 2000 (−1)            | 0.0301               | 0.4323|
| 12      | 0.5 (0)           | 30 (0)   | 2.5 (0)                      | 1500 (0)             | 0.0290               | 0.4278|
| 13      | 0.2 (−1)          | 20 (−1)  | 1.5 (−1)                     | 2000 (−1)            | 0.0369               | 0.4737|
| 14      | 0.2 (−1)          | 30 (0)   | 2.5 (0)                      | 1500 (0)             | 0.0302               | 0.4474|
| 15      | 0.2 (−1)          | 20 (−1)  | 1.5 (−1)                     | 1000 (−1)            | 0.0366               | 0.447|
| 16      | 0.2 (−1)          | 20 (−1)  | 1.5 (−1)                     | 1000 (−1)            | 0.0373               | 0.4546|
| 17      | 0.8 (+1)          | 20 (−1)  | 3.5 (−1)                     | 1000 (0)             | 0.0364               | 0.4573|
| 18      | 0.2 (−1)          | 20 (−1)  | 1.5 (−1)                     | 1000 (−1)            | 0.0342               | 0.4593|
| 19      | 0.2 (−1)          | 20 (−1)  | 3.5 (−1)                     | 2000 (−1)            | 0.0368               | 0.4876|
| 20      | 0.2 (−1)          | 20 (−1)  | 1.5 (−1)                     | 2000 (−1)            | 0.0358               | 0.4987|
| 21      | 0.2 (−1)          | 20 (−1)  | 3.5 (−1)                     | 1000 (−1)            | 0.0351               | 0.4579|
| 22      | 0.8 (+1)          | 20 (−1)  | 1.5 (−1)                     | 1000 (−1)            | 0.0366               | 0.4593|
| 23      | 0.8 (+1)          | 20 (−1)  | 1.5 (−1)                     | 1000 (−1)            | 0.0376               | 0.4357|
| 24      | 0.8 (+1)          | 30 (0)   | 2.5 (0)                      | 1500 (0)             | 0.0334               | 0.4348|
| 25      | 0.2 (−1)          | 20 (−1)  | 3.5 (−1)                     | 2000 (−1)            | 0.0366               | 0.4701|
| 26      | 0.5 (0)           | 30 (0)   | 2.5 (0)                      | 1500 (0)             | 0.0291               | 0.4261|
| 27      | 0.8 (+1)          | 20 (−1)  | 1.5 (−1)                     | 2000 (−1)            | 0.0386               | 0.4387|
| 28      | 0.5 (0)           | 30 (0)   | 2.5 (0)                      | 1500 (0)             | 0.0288               | 0.4282|
| 29      | 0.5 (0)           | 30 (0)   | 3.5 (1)                      | 1500 (0)             | 0.0329               | 0.4395|
| 30      | 0.5 (0)           | 30 (0)   | 2.5 (0)                      | 1500 (0)             | 0.0279               | 0.4225|
The defect in the hexagonal layer of carbon is indicated by the intensity ratios of D and G peaks, \( I_D/I_G \) of 0.72 [21]. The ratio of peak 2D and G intensities \( I_{2D}/I_G \) of 0.84 suggests that just a few layers of graphene nanosheets were developed [22].

The tensile and hardness test results are tabulated in table 2. In comparison to base alloy and AA7075/B\(_4\)C composite, graphene nanoparticle reinforced composed exhibited improvement in properties as indicated in figure 4. The inclusion of graphene nanoparticles and hard ceramic particles significantly increased the hybrid composite’s hardness and tensile strength. The graphene particles act as a 2D impediment at the matrix grain boundaries, causing grain refinement corresponding to the Hall-Petch theory [23].

The dislocation pile-up occurs as the nanoparticles restrict the aluminium’s dislocation motions according to the Orowan mechanism [16]. These two are the primary contributors to the composite’s increased strength. In addition, according to the dislocation density mechanism, an increase in the shear module and dislocation density enhanced the composite’s hardness. The composite having 0.5% graphene exhibited higher tensile strength and hardness. At 0.8 weight percentage of graphene, the mechanical properties decreased, attributing to the agglomeration of particles. The results were in accordance with [24].

### 3.2. Evaluation of tribological performance

Table 3 shows the tribological results. The wear weight loss was calculated for each experimental run by subtracting initial weight and final weight after wear. Figure 5 depicts the influence of tribological factors on wear loss using surface plots. Weight loss dropped up to 0.5 percent of graphene inclusion, then increased and when the load and sliding distance were increased, the weight loss increased. The wear loss declined and then increased until the sliding speed of 2.5 m s\(^{-1}\). The wear loss was maximum for 0.8% of graphene reinforcement, and it may be attributed to the agglomeration of nanoparticles at higher nano reinforcement percentages leading to the porosity of composites resulting in a massive amount of mass loss [25]. In addition, there is high contact.
Figure 6. 3D plots interaction effects of (a) AV s B . (b) AV s C . (c) AV s D . (d) B Vs C on the coefficient of friction.

Table 4. ANOVA for wear weight loss.

| Source      | Sum of Squares | Df  | Mean Square | F-value | p-value |
|-------------|----------------|-----|-------------|---------|---------|
| Model       | 0.0005         | 14  | 0.0000      | 234.71  | < 0.0001 significant |
| A-Reinforcement | 0.0000       | 1   | 0.0000      | 254.43  | < 0.0001 |
| B-Load      | 9.225E-07      | 1   | 9.225E-07   | 6.12    | 0.0258  |
| C-Sliding speed | 5.304E-11     | 1   | 5.304E-11   | 0.0004  | 0.9853  |
| D-Sliding distance | 0.0000      | 1   | 0.0000      | 176.34  | < 0.0001 |
| AB          | 3.414E-06      | 1   | 3.414E-06   | 22.64   | 0.0003  |
| AC          | 8.032E-07      | 1   | 8.032E-07   | 5.33    | 0.0357  |
| AD          | 1.796E-06      | 1   | 1.796E-06   | 11.91   | 0.0036  |
| BC          | 2.833E-07      | 1   | 2.833E-07   | 1.88    | 0.1907  |
| BD          | 5.718E-06      | 1   | 5.718E-06   | 37.91   | < 0.0001 |
| CD          | 2.234E-06      | 1   | 2.234E-06   | 14.81   | 0.0016  |
| A²          | 0.0000         | 1   | 0.0000      | 150.38  | < 0.0001 |
| B²          | 4.445E-06      | 1   | 4.445E-06   | 29.47   | < 0.0001 |
| C²          | 0.0000         | 1   | 0.0000      | 272.07  | < 0.0001 |
| D²          | 2.848E-08      | 1   | 2.848E-08   | 0.1888  | 0.6701  |
| Residual    | 2.263E-06      | 15  | 1.508E-07   |         |         |
| Lack of Fit | 1.132E-06      | 10  | 1.132E-07   | 0.5007  | 0.8354  | not significant |
| Pure Error  | 1.131E-06      | 5   | 2.261E-07   |         |         |
| Cor Total   | 0.0005         | 29  |             |         |         |
pressure between the sample and the counter surface at higher loads, which tends to increase the friction between the mating surfaces causing high wear loss. Even though the weight loss was reduced from $1.5 \text{ m s}^{-1}$ to $2.5 \text{ m s}^{-1}$ sliding speed, higher weight loss was observed at higher sliding speed ($3.5 \text{ m s}^{-1}$) and the results were in accordance with $26, 27$.

Generally, at a higher sliding speed, the temperature increases between the sliding surfaces. The addition of graphene formed a thin 2D lubricant layer and decreased the wear loss. However, at $3.5 \text{ m s}^{-1}$, the temperature exceeds the lubricant film and causing the formation of material deformation and wear debris. The wear debris interlocks between the sliding surfaces and increased the weight loss of the composite. As the contact duration between the material and the disc increased, the wear loss increased with increasing the sliding distance. The composite’s hardness has a substantial impact on weight loss $28$. In our research, higher hardness was obtained at 0.5% of graphene reinforcement. Similarly, the wear loss was minimum at 0.5 graphene reinforcement percentage, which was in agreement with Archard’s law. The impact of wear parameters on COF was inferred from figure 6 3D plots. The COF drastically decreased from 0.2 to 0.5 weight percentage of graphene as the reinforcement forms a thin self-lubricant layer at the matrix boundary. The COF increased as the load and sliding distance increased $29$. As sliding speed increased, there is a gradual decrease in friction coefficient up to $2.5 \text{ m s}^{-1}$. The incorporation of graphene, a self-lubricant material, decreased the temperature causing the reduction in friction between mating surfaces.

### 3.3. ANOVA and regression analysis

The statistical approach analysis of variance (ANOVA) was carried out to define the importance of tribological characteristics on output responses. The ANOVA for the developed quadratic model for output response wear loss is tabulated in table 4.

| Source       | Sum of Squares | df | Mean Square | F-value | p-value |
|--------------|----------------|----|-------------|---------|---------|
| Model        | 0.0110         | 14 | 0.0008      | 395.80  | < 0.0001 significant |
| A-Reinforcement | 0.0010       | 1  | 0.0010      | 484.85  | < 0.0001 |
| B-Load       | 0.0010         | 1  | 0.0010      | 480.96  | < 0.0001 |
| C-Sliding speed | 0.0000       | 1  | 0.0000      | 6.82    | 0.0196  |
| D-Sliding distance | 0.0009   | 1  | 0.0009      | 436.65  | < 0.0001 |
| AB           | 0.0001         | 1  | 0.0001      | 42.95   | < 0.0001 |
| AC           | 0.0000         | 1  | 0.0000      | 21.27   | 0.0003  |
| AD           | 0.0006         | 1  | 0.0006      | 305.89  | < 0.0001 |
| BC           | 0.0003         | 1  | 0.0003      | 157.55  | < 0.0001 |
| BD           | 0.0002         | 1  | 0.0002      | 82.67   | < 0.0001 |
| CD           | 0.0002         | 1  | 0.0002      | 95.47   | < 0.0001 |
| A²           | 0.0006         | 1  | 0.0006      | 286.43  | < 0.0001 |
| B²           | 0.0001         | 1  | 0.0001      | 44.78   | < 0.0001 |
| C²           | 0.0006         | 1  | 0.0006      | 281.07  | < 0.0001 |
| D²           | 8.283E-06      | 1  | 8.283E-06   | 4.17    | 0.0593  |
| Residual     | 0.0000         | 15 | 1.988E-06   |         |         |
| Lack of Fit  | 9.737E-06      | 10 | 9.737E-07   | 0.2423  | 0.9729 not significant |
| Pure Error   | 0.0000         | 5  | 4.018E-06   |         |         |
| Cor Total    | 0.0110         | 29 |             |         |         |

**Table 5. ANOVA for the coefficient of friction.**

| Fit statistics | Wear weight loss | Coefficient of friction |
|----------------|------------------|-------------------------|
| $R^2$          | 0.9955           | 0.9973                  |
| Adjusted $R^2$ | 0.9912           | 0.9948                  |
| Predicted $R^2$| 0.9835           | 0.9926                  |
| Adeq. Precision| 51.1275          | 81.5338                 |

The fit statistics for the responses were tabulated in table 6. From table 6, The adjusted $R^2$ value and the predicted $R^2$ value are in fair agreement, indicating the model’s significance and feasibility. Equations (1) and (2)
Figure 7. Normal probability plot for (a) Wear loss. (b) Coefficient of friction.

Table 7. Confirmatory test for A0B0C0D-1 optimal process parameter.

| Responses                  | Predicted | Experimental | Error (%) |
|----------------------------|-----------|--------------|-----------|
| Wear weight loss (g)       | 0.0276    | 0.0270       | 2.17      |
| Coefficient of friction    | 0.4175    | 0.4124       | 1.22      |

Figure 8. Inverted microscope images of worn surface.
describes the second-order equation in terms of coded factors for wear loss and the COF.

\[
\text{Wear weight loss} = +0.0287 + 0.0014 A - 0.0002 B + 1.7166 \times 10^6 C + 0.0012 D + 0.0002 AB + 0.0002 AC + 0.0003 AD + 0.0001 BC + 0.0005 BD + 0.0003 CD + 0.0029 A^2 + 0.0013 B^2 + 0.0039 C^2 + 0.0001 D^2
\]

\[
\text{Coefficient of friction} = +0.4262 - 0.0073 A + 0.0073 B - 0.0009 C + 0.0069 D + 0.0023 AB + 0.0016 AC - 0.0062 AD - 0.0044 BC + 0.0032 BD - 0.0034 CD + 0.0148A^2 + 0.0059 B^2 + 0.0147 C^2 - 0.0018 D^2
\]

(1)

(2)

Where A is the graphene reinforcement percentage, B is load, C is sliding speed and D is sliding distance. The normal probability plot indicates the points scattered on the plot are along the straight line with no justifiable outliers, indicating that the dispersion of regression residuals is within standard ranges, as shown in figure 7.

The optimal process parameter for both wear weight loss and COF occurred at A0B0C0D-1 coded factor conditions. The confirmatory test was carried for the same process parameters and the responses were measured. Table 7 shows the values obtained in the confirmatory test are closest to the predicted value. The percentage of error between predicted and experimental value for weight loss is 2.17% and COF is 1.22%, which is very low, indicating an excellent correlation.

3.4. Worn surface morphology

The worn surface was analyzed using an inverted optical microscope. The wear grooves were narrow observed for a sample having minimum wear loss A0B0C0D-1 as shown in figure 8(a). The plastic deformation and plowing were seen in figures 8(b) and (c) for the sample tested at all maximum input factors. Plastic deformation may have occurred as a result of the increase in temperature at higher sliding speed [30]. The surface morphology of the worn sample was studied using FESEM. The graphene formed a lubricant layer on the pin surface, as shown in figures 9(a) and (b), resulting in reduced friction between the sliding surfaces and the wear.
loss was minimum for the A0B0C0D-1 test condition. Fractured surface, micro pits and plowing of metals were occurred due to abrasive wear, as shown in figure 9(c) for A + 1B + 1C + 1D + 1 coded factor condition.

4. Conclusions

In this research work, the graphene was synthesized by a novel method and incorporated into an aluminium matrix and the hybrid composite was fabricated using liquid metallurgy technique. Then, the sample was investigated for mechanical properties, tribological behaviour and summarized conclusions were as follows.

- FESEM results reveal the graphene synthesized by the eco-friendly method of using natural precursor exhibited sharp edge graphene nano flakes.
- XRD peak at 26.3° confirmation the formation of graphinic structure. The intensity ratios of Raman spectra revealed the presence of layered graphene with fewer defects.
- The hybrid composite’s tensile strength and hardness were greatly enhanced compared to the base alloy, when graphene and hard ceramic reinforcements were added to the aluminium liquid melt.
- The occurrence of a dislocation mechanism caused by graphene at matrix grain boundaries improved the composite’s strength. At 0.5 weight percent of graphene, high hardness and higher tensile strength were observed.
- The inclusion of graphene enhanced the composite’s tribological performance as graphene forms the thin lubricant layer between the sample and rotating disc, which decreases the temperature and reduces wear loss and COF.
- The worn surface analyzed using an inverted microscope and FESEM revealed that only narrow grooves were formed as graphene reduced the friction. Thus, graphene reinforced aluminium composites can be used as self-lubricating composites.
- The statistical results conclude that the terms are significant as the p-values are <0.05 and the predicted $R^2$ value corresponds with the adjusted $R^2$ value for both responses.
- The optimal tribological parameters based on the study was found to be A0B0C0D-1, i.e., graphene reinforcement 0.5 percentage, load 30 N, sliding speed 2.5 m s$^{-1}$ and sliding distance 1000 m for which the wear weight loss and coefficient of friction were minimum.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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