Journal of Materials and Manufacturing 4(1): 35-47 (2025)

Journal of Materials and Manufacturing

Tribological and microstructural enhancement of engine oil using reduced graphene oxide and nano-graphite additives

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Abstract

This study investigated the tribological and microstructural properties of fully synthetic engine oil (SEO) Shell 5W-30, enhanced with 0.5% concentrations of reduced graphene oxide (RGO) and nano-graphite (NG). The tribological performance of the prepared samples was assessed using a pin-on-disc tribometer under the test conditions of 150 RPM speed and a 981N load. Microstructural analysis was conducted using scanning electron microscopy (SEM). Additionally, Fourier transform infrared spectroscopy (FTIR), thermo gravimetric analysis (TGA) and UV-Vis spectroscopy were used to examine the functional group interactions, thermal stability and infrared absorption properties, respectively. The results demonstrated that adding 0.5 weight % RGO to Shell 5W-30 oil significantly improved the tribological performance compared to NG-based lubricants by effectively reducing both frictional forces and the coefficient of friction (approximately 50%). SEM analysis revealed that RGO produced a noticeably smoother worn surface with smaller pits, resulting in the lowest coefficient of friction and frictional forces among all tested samples including NG-based lubricants and unmodified shell 5W-30 oil. FTIR analysis revealed a significant reduction in hydroxyl and alkoxy groups following the addition of RGO to shell 5W-30 oil compared to nano-graphite-based lubricants. TGA analysis further indicated more substantial thermal degradation in RGO, attributed to the presence of oxygen-containing functional groups. These findings suggest that incorporating RGO to Shell 5W-30 oil could optimize the lubricant performance across a wider range of applications, particularly in automotive sector, where friction reduction is critical for improving engine efficiency.

Keywords: Reduced graphene oxide; Nano graphite; sythesized engine oil Shell 5W-30; Coefficient of friction.

1. Introduction

In mechanical assembly systems, nearly half of the mechanical parts failures are attributed to inadequate lubrication. The use of lubricants is essential for Reducing friction and wear. During start-up, especially under heavy loads or at low speeds, insufficient formation of lubrication oil film can lead to sever wear and increased friction at contact surfaces. To address this, Nano-materials such as WS2 and MoS2 have been developed to reinforce the lubricating film. In metal-to-metal surface contact, the inclusion of such additives in these lubricants promotes the formation of a highly protective, low-shear boundary layer, thereby enhancing surface protection and significantly improving tribological performance [1, 2]. Reduced friction contributes to improved fuel efficiency, while decreased wear enhances the durability and longevity of

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DOI: 10.5281/zenodo.15766837
Received: 7 June 2025, Revised: 23 June 2025, Accepted: 29 June 2025
ISSN: 2822-6054

mechanical components [3]. Both solid and liquid lubricants play a crucial role in ensuring the efficient and reliable operation of modern engines and mechanical systems over prolonged periods. In this context , graphene and its derivatives have attracted considerable attention as advanced lubricant additives, either in solid form or as colloidal dispersions, due to their exceptional lubricity, high mechanical strength, excellent thermal stability, and strong resistance to oxidation [4-7]. When used as additives, graphene-based compounds can significantly enhance the performance of conventional lubricants by providing superior protection against abrasive wear and friction. This capability is primarily attributed to two key factors. Firstly, the weak van der Waals forces between graphene flakes, along with their temperature-dependent translational and rotational motion, facilitate smooth interlayer sliding, thereby reducing shear resistance [8, 9]. Conversely, graphene-based materials interact synergistically with other lubricant additives to improve tribological performance. Under sever wear conditions, they promote the formation of island-shaped protective coatings, while during uniform wear, they continuously develop a stable protective layer on sliding surfaces—enhancing lubrication efficiency and surface durability [10].

Both mechanisms contribute to a significant reduction in the coefficient of friction (COF). Moreover, exceptional mechanical strength of graphene derivatives offers enhanced protection against corrosion and abrasion [11]. The lubricating capability of graphene in macro scale steel-on-steel sliding contacts under boundary lubrication conditions has been widely reported in recent tribological studies [12, 13]. While the incorporation of nano-additives into the lubricants represents a significant advancement in enhancing lubrication efficiency, the uncontrolled addition of nano-materials canlead to particle aggregation. Gravitational forces may cause these agglomerates to precipitate, potentially compromising the stability and performance of the lubricant [14]. Aggregation occurs when the van der waals forces between nano-materials exceed the repulsive forces, and the attractive interactions are not counteracted by Brownian motion [15]. Agglomeration in nanomaterial dispersions accelerates sedimentation, thereby reducing the lubricant's friction-reducing efficiency [16, 17]. Therefore, formulating highly effective and reliable nano-lubricants with long-term stability remains challenging, as they tend to settle over extended storage periods, potentially leading to increased mechanical wear between the contact surfaces [18]. . Recent studies indicate that the optimal concentration of graphene-based additives in lubricating oils typically lies within the range of 0.01% to 1%, effectively achieving a balance between long-term dispersion stability and substantial reductions in the coefficient of friction (COF) [19-22]. This optimal concentration varies depending on the specific properties of graphene-based additive, including functional groups modifications, number of layers, flake size, and the nature of the sliding contact [23].

Several studies have explored the tribological benefits of nanoparticles additives. For instance, Mirzaamiri et al. [24] added nano-diamonds to the water, and reported 88% reduction in wear and a 70% decrease in friction attributed to the ball-bearing effect of the nano-diamond particles. Wu et al. [25] introduced sulfonated graphene into the water, resulting in a 25.8% increase in viscosity, while the wear scar diameter (WSD) and coefficient of friction (COF) were reduced by 74% and 15.7%, respectively. Xu et al. [26] investigated the effects of graphene nano-sheets (GNS) and silver (Ag) hybrids in the phenolic composites and found that a 9 weight percent GNS/Ag hybrid led to a 72% reduction in wear rate and 40% decrease in COF. According to Wang et al.[27], thicker copper covered with molybdenum disulphide showed more severe wear but achieved a lower friction coefficient (COF). Similarly, Yu et al. [28] reported that the use of hydrated silica tribofilm lowered the friction coefficient of MoAlB ceramic as low as 0.12. . Pham et al. [29] demonstrated that incorporating SiO_2 nanoparticles into the engine oil improved the anti-oxidation properties of lubricants. Simonovic et al. [30] observed that abrasive wear happens at loads exceeding 8 N; , although WSC-coated ceramics exhibited reduced wear at low loads due to a higher presence of WS_2 monolayers. Chen et al. [31] determined that combining multi-layer graphene (MLG) with Si_3N_4 improved the wear resistance and lowered the coefficient of friction (COF) compared to Si_3N_4-based and carbon-rich MLG-based ceramics.

Previous studies have demonstrated that nano-particles such as reduced graphene oxide (RGO) performs effectively as lubricant additive in enhancing tribological and microstructural properties and long term stability of lubricants across various concentrations and loading conditions. Friction and mechanical failure between worn surfaces remain major contributors to energy loss in automobile engines, highlighting the importance of effective and advanced lubricants for enhancing the engine efficiency. While several studies have explored the application of nanoparticles in lubricants, there remains a notable lack of comprehensive analysis between lubricants with and without such additives. In particular, the direct impact of RGO on the frictional forces and the coefficient of friction (COF) has not been comprehensively investigated in widely used commercial lubricants such as Shell 5W-30 oil. This gap emphasizes the need for further research into the role of different nanoparticle additives to further improve the efficiency and reliability of lubricants, especially in the automotive applications, where bridging the divide between theoretical performance and real-world functionality is critical.

To address this issue, the present study conducts a comprehensive investigation of the tribological and microstructural properties of Shell engine oil (SEO) 5W-30 incorporating 0.5 weight % reduced graphene oxide (RGO) and 0.5 weight % nano-graphite (NG). In addition to evaluating frictional forces, and coefficient of friction, this study implies scanning electron microscope (SEM), Fourier-transform infrared spectroscopy (FTIR), UV-Vis spectrograph and thermo gravimetric (TGA) analysis to enable thorough comparative assessments of un-modified 5W-30 oil, RGO modified and NG modified lubricant samples.

2. Materials and Methods

2.1. Materials

In the present study, following materials were used: acetone, sodium bicarbonate, potassium permanganate, hydrogen peroxide (30%), hydro iodic acid (HI, 55%), graphite powder, and sodium nitrate all of which were purchased from Sigma-Aldrich. Glacial acetic acid and sulfuric acid (95–98%) were obtained from Merck (Darmstadt, Germany) and Honeywell Advanced Materials (NJ, USA), respectively. The fully synthetic SEO Shell 5W-30containing the trace elements such as calcium (Ca), zinc (Zn), phosphorus (P), sulfur (S), and magnesium (Mg), was supplied by Shell Company. The specifications of SEO Shell 5W-30 were verified against PSAB71 2296, Ferrari; Fiat 9.55535-Z2; Renault RN0700, RN0710; and Chrysler MS-10725. The properties of SEO Shell 5W-30 are summarized in Table 1.

2.2. Samples Preparation

In this study, reduced graphene oxide (RGO) and nano-graphite (NG) were used in powder form. Reduced graphene oxide (RGO) was synthesized in-house using a standard protocol involving the preparation of graphine oxide followed by chemical reduction with hydro iodic acid. Specifically, graphite oxide was reduced using a combination of hydro iodic acid and acetic acid via a modified chemical vapor deposition (CVD) process. For sample preparation, 15 mL (approximately 8g) of SEO Shell 5W-30 oil (/8 g) was placed to a 20 mL vial. Subsequently, 0.5 weight % of reduced graphene oxide (RGO) and 0.5 weight % of nano-graphite (NG) nanoparticles with the size of 5nm were introduced into the vial. To unsure a uniform dispersion of nano-particles within the lubricants, the mixture was subjected to ultrasonic agitation for 2 hours. Following sonication, samples were centrifuged using a Hettich Universal 320 R centrifuge at 4100 rpm for 40 minutes to eliminate aggregates and improve stability. The resulting samples were then used for tribology testing and microstructure analysis. Samples preparation is illustrated in Fig. 1.

Table 1. The Properties of (SEO) shell 5W-30

Characteristics	Values
Total Base Number (TBN)	9.2 mg KOH/g
Viscosity at 40ºC	62 cSt
Flash Point	168ºC

2.3. Tribological Testing

In this study, the tribological performance of lubricants, specifically the coefficient of friction (COF) and frictional forces was evaluated using a pin-on-disc tribo-tester (model TR-20LE), as shown in Fig. 2. The test configuration involved a stationary pin made of pure aluminum (A1100) in contact with rotating disc composed of SKD11 tool steel. The required force was applied via a loading pan and resulting frictional force was measured using a load cell. A small quantity of lubricant (0.02g) was applied to the disc surface, which was designed with a groove to the retain lubricant during rotation and prevent leakage. The tests were conducted under a constant normal load of 981 N, at a sliding speed of 0.2 m/s for duration of 60 minutes. After each test, the aluminium pin was thoroughly cleaned with acetone to remove any residual oil from the pin surface. The coefficient of friction (COF) was calculated by dividing the frictional force by the normal load, providing a measure of antifriction performance of the lubricants. To assess the anti-wear performance, the wear scar diameter (WSD) on the pin surface was measured using high-resolution optical microscope. Important key parameters related to the tribological testing setup are summarized in Table 2.

Table 2. Dimensions and parameters used

Pin and Disc Dimensions and Parameters	
Length of pin	300 mm
Diameter of pin	8 mm
Radius of curvature	3 mm
Steel disc dimensions	165x 8 mm
Roughness (Ra)	0.01-0.3 µm
Test speed	150 RPM
Normal load	981 N
sliding speed	0.2 m/s
Track radius	30 mm

2.4. Microstructural Analysis

The morphology characteristics of prepared samples were examined using scanning electron microscope (SEM, Camscan MX 200), operated at accelerating voltage range of 80–120 kV. To analyze the presence of organic functional groups on the surfaces of nano-graphite (NG) and reduced graphene oxide (RGO), Fourier transform infrared spectroscopy (FTIR, Perkin Elmer, USA) was employed. The FTIR spectra for both NG and RGO were recorded over the wavenumber range of 400 cm^{-1} to 4000 cm^{-1}. The optical absorption properties of both NG and RGO were further investigated using UV-visible spectroscopy. Absorption spectra were obtained using a double beam Lambda 35 UV-visible spectrophotometer (Perkin Elmer, USA), scanning solution samples within the wavelength range of 200–800 nm. Thermo gravimetric analysis (TGA) was performed to assess and compare the thermal stability of NG and RGO providing insights into their decomposition behavior and structural integrity under elevated temperatures.

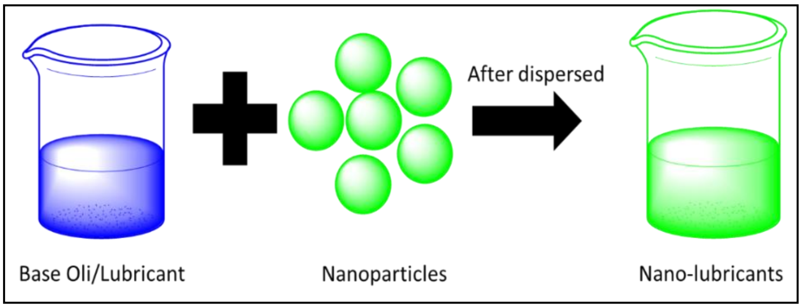


Fig. 1. Samples preparation

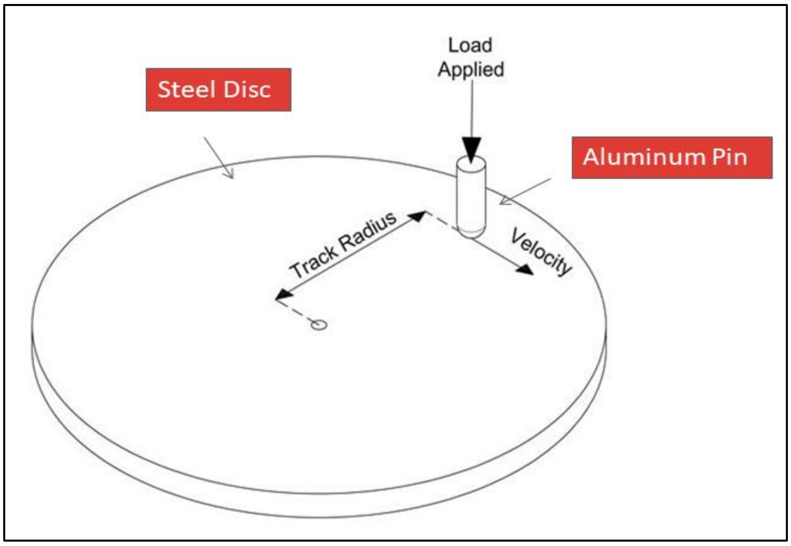


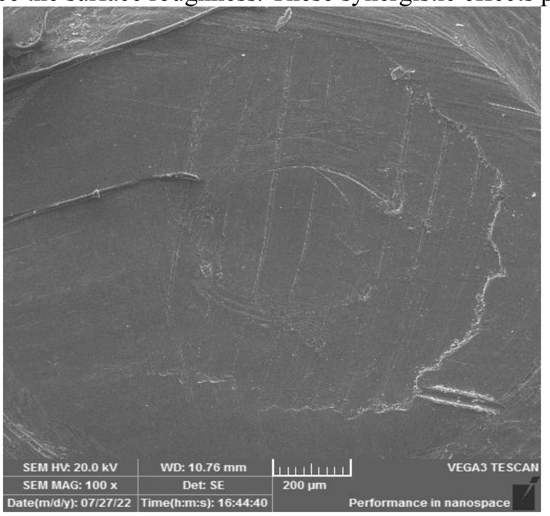
Fig. 2. Samples preparation and schematic diagram of pin-on-disc

3. Results and Discussion

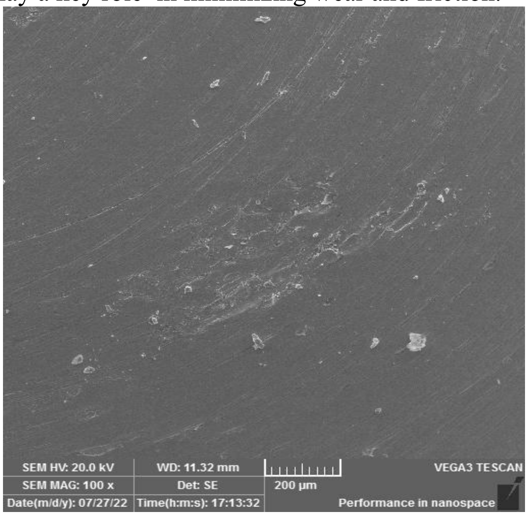
3.1. Microstructural

3.1.1. SEM Analysis

Scanning electron microscopy (SEM) was employed to examine the worn surfaces after tribological testing. SEM images of the worn surface of the tested aluminum pin-on-disc are presented in Fig. 3(a-c). Fig. 3(a) shows the worn surface of an aluminum pin lubricated with unmodified SEO Shell 5W-30 at 100 x magnifications. The micrograph reveals some pits and surface wears, likely attributed to adhesive wear mechanisms. Although some pits and grooves still remain in the unmodified SEO Shell 5W-30. Fig. 3(b) depicts a comparetively smoother worn surface with the addition of 0.5 weight % of nano-graphire (NG) particles. The reduction in pits size compared to the base SEO Shell 5W-30 lubricant led to further decrease in coefficient of friction (COF) and frictional forces, though minor defects and grooves were still visible. Fig. 3(c) illustrates a significantly smoother surface achieved with the addition of 0.5 weight % reduced graphene oxide (RGO). In compared to the both SEO Shell 5W-30 oil and NG-enhanced lubricants, the worn surface treated with RGO exhibited a marked improvement in surface integrity, showing minimal pits, lowest coefficient of friction (COF), and frictional forces among the samples. These observations align with the findings of Paul, G.et al. [32], who reported that graphene deposition on the worn surfaces, contributes to both a mending effect-filling in micro cracks and surface defects, and a polishing effect, which collectively reduce the surface roughness. These synergistic effects play a key role in minimizing wear and friction.



(a)



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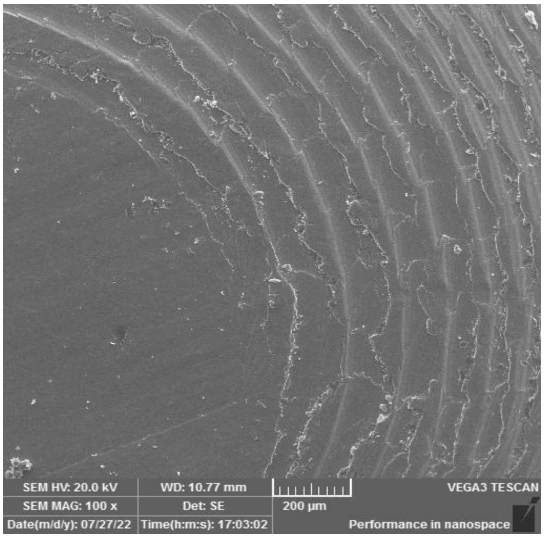


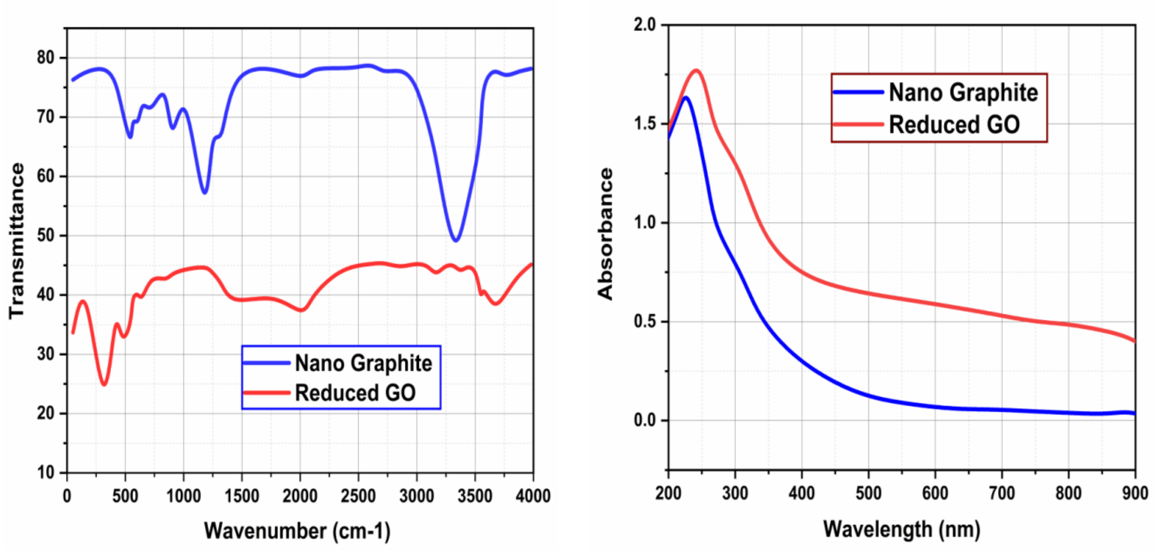
(c)

Fig. 3. SEM images of (a) Shell 5W-30 oil (b) 0.5wt.% NG (c) 0.5wt.% RGO

3.1.2 FTIR Analysis and UV Spectrograph

Figure 4(a-b) presents the fourier transform infrared (FTIR) spectra and UV-visible absorption spectra of nano-graphite (NG) and reduced graphene oxide (RGO) nanoparticles. In Fig. 4(a), the FTIR spectra are shown in the range of 500 to 4000 cm^{-1}. The hydroxyl group exhibits a stretching vibration at 3450 cm^{-1}, the carbonyl (C=O) group stretches at 1728 cm^{-1}, and the epoxide (C–O) group shows stretching vibrations at 1229 and 1061 cm^{-1}. The high intensity of the major peaks in RGO confirms the presence of numerous oxygen-containing functional groups. . The peak observed at 1627 cm^{-1} corresponds to the vibrations of adsorbed water molecules. After chemical reduction, the intensities of hydroxyl and alkoxy group peaks were significantly reduced, while a stretching vibration of phenolic (C=C) ring appears at 1585 cm^{-1}. However, achieving efficient removal of oxygen functional groups from the carbon planes may require a sufficiently acidic environment.

In the UV-visible spectra (Fig. 4(b)), the primary absorption peak of graphite appears at 230 nm, corresponding to the transition of the aromatic C–C ring, with a secondary absorption peak at 303 nm attributed to the n-π* transition of the C=O bond groups. Upon reduction, RGO peak shifts to 255 nm due to the removal of oxygen-containing groups and restoration of aromatic domains, which allows electrons excitation at lower energy levels.



(a) (b)

Fig. 4. (a) FTIR analysis **(b)** UV-visible spectra of NG and RGO

3.1.3 Thermo Gravimetric Analysis (TGA)

Thermo Gravimetric Analysis (TGA) was conducted to assess the thermal stability of nano graphite (NG) and reduced graphene oxide (RGO) nanoparticles as illustrated in Fig. 5. The analysis indicates that nano graphite (NG) undergoes a single-step weight loss, while RGO exhibits a three-step degradation process. The first weight loss in RGO occurs between 50°C and 120°C, and is attributed to the evaporation of absorbed water molecules. The second stage, observed from 120°C to 440°C, corresponds to the decomposition of oxygen-containing functional groups present in the RGO structure. The third phase, occurring above 440°C, is associated with the decomposition of unstable carbon in the structures and the pyrolysis of oxygen functional groups, leading to the release of gaseous products such as CO and CO2. These findings emphasize the distinct thermal behaviors of NG and RGO, with RGO demonstrating more substantial thermal degradation due to of its higher contents of oxygen-containing functional groups.

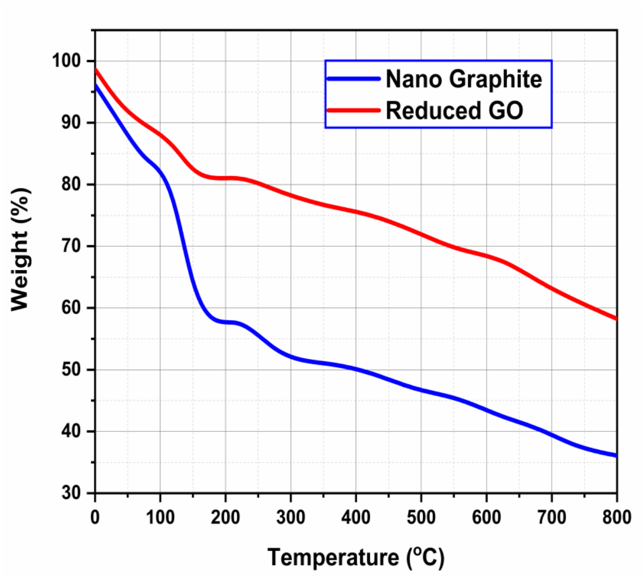


Fig. 5. TGA Analysis of NG and RGO

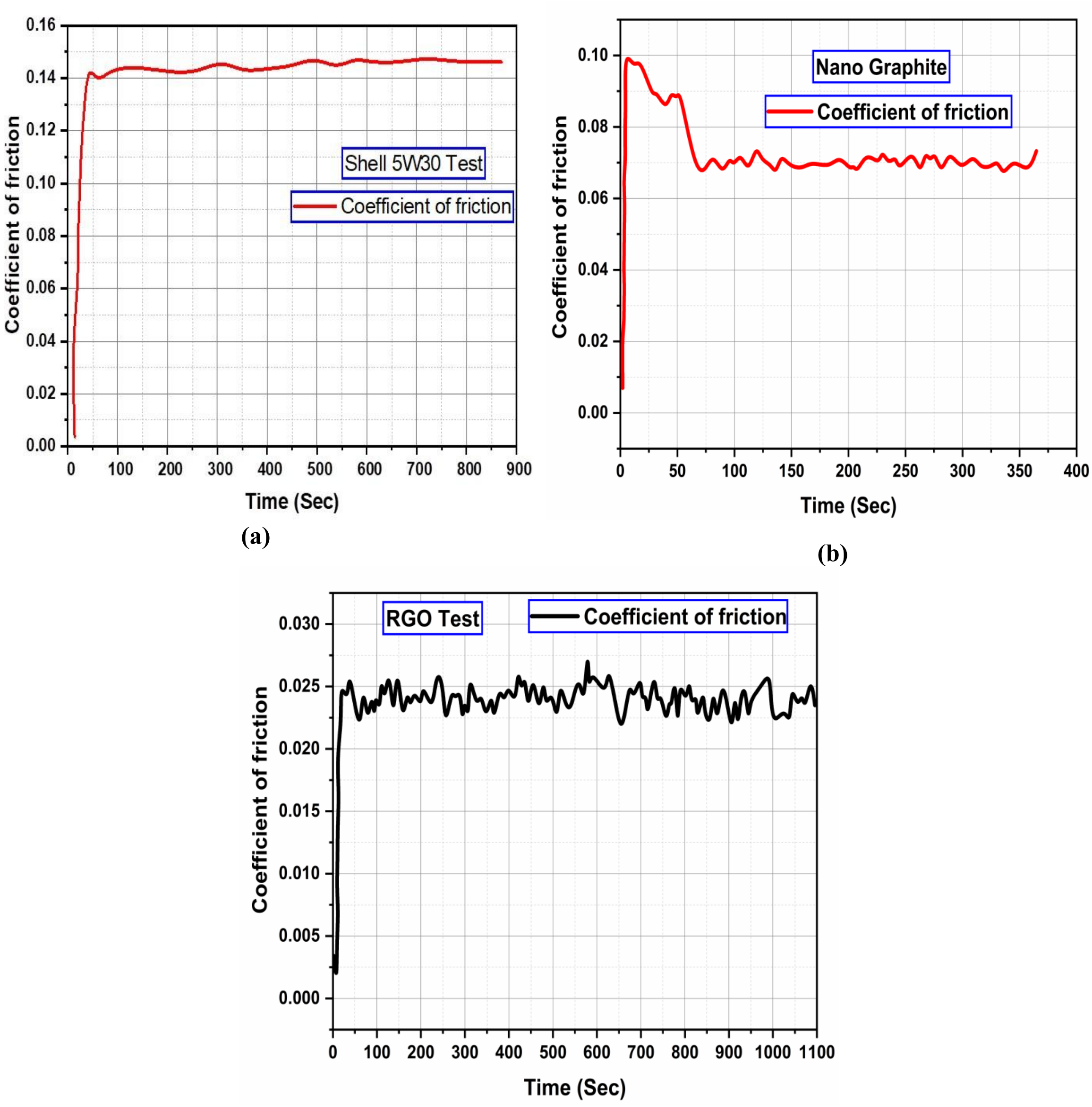
3.2 Tribology Testing

3.2.1. Coefficient of Friction (COF)

Fig. 6(a-c) illustrates the typical fluctuations in the coefficient of friction (COF) for SEO Shell 5W-30 with and without addition of 0.5 weight % reduced graphene oxide (RGO) and nano-graphite (NG) over time. The comparative analysis indicates that RGO-based nano lubricants exhibited superior tribological performance compared to those containing NG. Fig. 6(a) displays the coefficient of friction (COF) graph for SEO Shell 5W-30 without any nanoparticle additives, revealing the highest friction among all tested samples. The COF stabilizes after approximately 50 seconds, fluctuating between the upper limit of 0.16 and lower limit slightly above 0.14.

Fig. 6(b) presents the COF graph for the NG-based lubricant. Initially, the curve reaches a COF of 0.10 followed by a zigzag trend that stabilizes around 0.5 after 60 seconds. A peak of COF approximately 0.10 is observed in the beginning of the test (100 and 400 seconds), with subsequently fluctuations staying below that threshold. Fig. 6(c) illustrates the COF graph for RGO-based nano-lubricant in a pin-on-disc tribology test. A noticeable zigzag trend is observed throughout the entire test duration. The addition of 0.5 weight % RGO to the SEO Shell 5W-30 oil significant reduces the coefficient of friction (COF) with the maximum peak of COF occurring just above 0.025 between 500 and 600 seconds. Over the entire duration, the COF remained between just below 0.030 and slightly above 0.020.The results clearly indicate that the nano-lubricants containing 0.5 weight % RGO exhibited approximately 50 % reduction in COF compared to those samples with 0.5 weight % NG added to SEO Shell 5W-30.

These findings are consistent with the study by Baigi, V. et al [33], who developed nano-lubricants using varying weight percentages of reduced graphene oxide (RGO) and zirconia (ZrO_2) nanoparticles in the paraffin oil. Their results also demonstrated reduced coefficient of friction (COF) and also exhibited less fluctuations in friction over time at different weight concentrations.



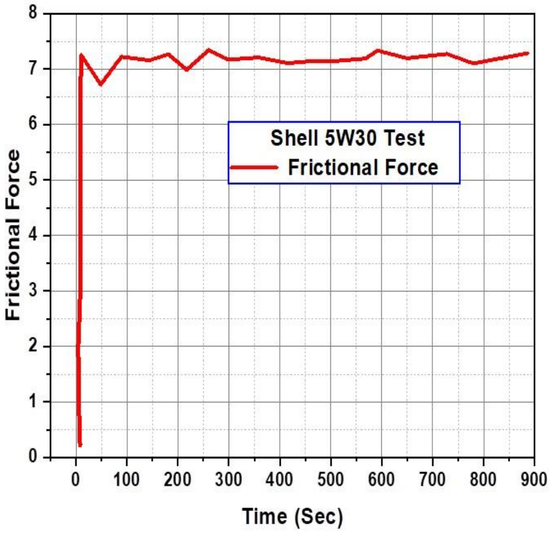
(a) (b) (c)

Fig. 6. Variation in COF with changing time for shell 5W-30 oil with RGO and NG

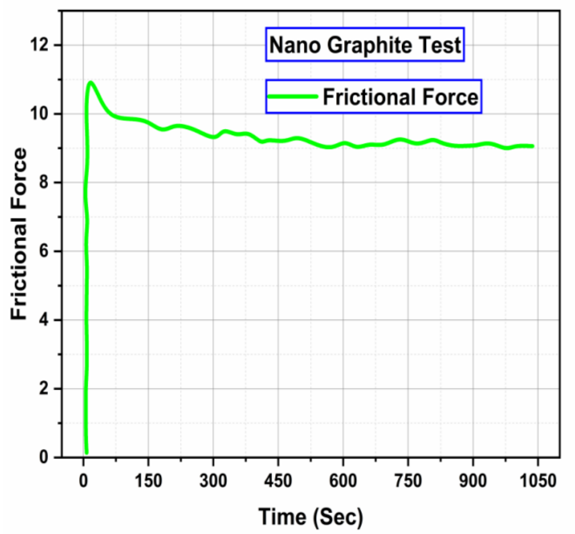
3.2.2. Frictional Forces

Fig. 7(a-c) illustrates the variation in frictional forces over time for SEO Shell 5W-30 oil containing 0.5 weight % nano-graphite (NG) and reduced graphene oxide (RGO). The comparative analysis indicates that RGO-based nano lubricants exhibited superior tribological performance compared to those containing NG. The frictional force in NG-based nano-lubricants displayed significant fluctuations and higher peak values throughout the test duration. In contrast, RGO-based nano-lubricants demonstrated a more stable frictional force trend with minimal variations, effectively reducing the friction and wear. This improvement is attributed to enhanced surface interaction provided by RGO, which facilitates both the polishing and mending effects, leading to smoother contact surfaces and reduced metal-to-metal interactions.

Moreover, the layered structure and improved dispersion of RGO enhance its load bearing capability while maintaining low friction. These findings confirm that the addition of 0.5 weight % RGO markedly enhances the antifriction properties of the lubricant, making it a more effective additive than NG for reducing frictional forces.



(a)



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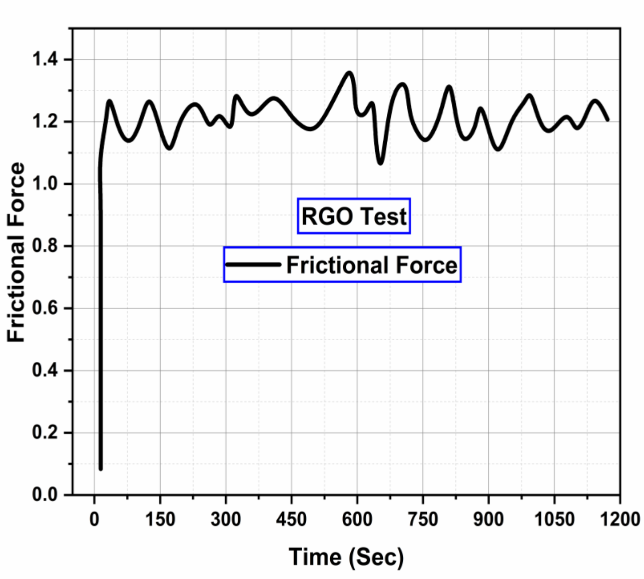

(c)

Fig. 7. Variation in COF with changing time for shell 5W-30 oil with RGO and NG

4. Conclusion

In this study, the tribological and microstructural properties of synthesized engine oil Shell 5W-30 enhanced with 0.5 weight % NG and 0.5 weight% RGO were successfully investigated. The primary focus was to analyze the effects of these nanoparticles on the performance of engine oil, with particular emphasis on tribological and microstructural properties. A pin-on-disc tribo-meter was employed to evaluate the tribological properties of the lubricants, and their characteristics were further examined using scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), and UV spectroscopy and thermo gravimetric analysis (TGA). The key conclusions drawn from the results are as follows:

- In terms of tribological performance, the incorporation of 0.5 weight % RGO into the Shell 5W-30 significantly reduced both the coefficient of friction (COF) and frictional forces compared to 0.5 weight % nano-graphite (NG) and unmodified shell 5W-30 oil.
- The SEM analysis revealed the smoother worn surface and minimized surface defects in 0.5 weight % RGO-based lubricated samples indicating a substantial improvement in wear resistance and lubrication efficiency compared to both 0.5 weight % NG and base shell 5W-30 oil samples.
- FTIR analysis confirms a significant reduction in hydroxyl and alkoxy groups after the addition of 0.5 weight % RGO into shell 5W-30 oil as compared to NG, suggesting successful chemical reduction and enhanced thermal stability.
- UV-visible spectroscopy revealed a red shift in the absorption peak for RGO from (230-250 nm) attributed to the restoration of π-conjugated system indicating fewer oxygen functional groups and more graphitic character compared to NG-lubricants.
- TGA analysis indicated that RGO exhibits multi stage thermal degradation due to oxygen-containing functional groups while NG showed a single step decomposition highlighting their different thermal stabilities.

These findings suggest reduced that graphene oxide (RGO) offers superior tribological, chemical, and thermal characteristics over nano-graphite when used as additives in synthetic engine oil, making it a highly suitable candidate for advanced lubrication formulations.

Author Contribution Statement

Conceptualization, Hafiz Muhammad Umer, Muhammad Shoaib Naseem, Barkat Ullah, Hassan Raza Channar; methodology, Muhammad Shoaib Naseem, Barkat Ullah, Hassan Raza Channar; validation, Barkat Ullah, Hassan Raza Channar; investigation, Hassan Raza Channar, Hammad Khalid, writing—original draft preparation, Hassan Raza Channar; writing—review and editing, Hassan Raza Channar, Hammad Khalid. All authors have read and agreed to the published version of the manuscript.

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