



Article Tribological Performance and Rheological Properties of Engine Oil with Graphene Nano-Additives

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Abstract: Nanoparticles dispersed in lubricants are being studied for their ability to reduce friction and wear. This paper examines SAE 5W-30 oil enhanced with dispersed graphene nanoplates for tribological and rheological properties. Graphene nanoplate (GNs) concentration effects on the rheological and tribological properties of 5W-30 base oil (0.03, 0.06, 0.09, 0.12, and 0.15 wt percent) were tested. Under various loads, a four-ball testing model was used to conduct a tribological analysis (200, 400, 600, and 800 N). Kinematic viscosity is calculated, and base oil and nanofluid-added 5W30 lubricant are compared for thermal conductivity and flashpoint. Wear scar and coefficient of friction improved by 15% and 33% with nano-additives. When related to the base oil, the flashpoint, thermal conductivity, kinematic viscosity, and pour point all increased, by 25.4%, 77.4%, 29.9%, and 35.4%, respectively. The addition of GNs improved the properties of 5W30 engine oil.

Keywords: engine tribology; GNs nanomaterials; lubricant additives; tribo-film mechanisms

1. Introduction

Today, nearly half of mechanical part failures can be attributed to insufficient lubrication in mechanical assemblies [1–3]. Reduced wear and friction can be achieved using lubricants. Low speed or heavy load can result in a lack of lubricating oil film formation, leading to severe wear on the contact surface when mechanical assemblies start up, gear pairs mesh, or the direction of the engine piston changes [4–7]. MoS2, WS2, and other nanomaterials have been developed to strengthen the lubricant film [8,9]. Despite recent advancements, tribological behavior is still a significant challenge under these conditions.

Graphene, a new lubricant material with outstanding tribological properties, thermal conductivity, and mechanical strength, has piqued interest [10–12]. A graphene-based solid lubricant is just one of many examples of graphene's exceptional wear resistance and friction-reducing properties. Generally, the tribological performance can be significantly enhanced by coatings such as graphene tribo-pairs on steel materials [13,14]. Graphene has also been shown to have significant macro-scale superlubricity (0.004), according to Berman et al. [15]. Most conventional methods of reducing wear and friction at sliding



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). interfaces use oil lubrication [16–20] for practical applications. However, without additives, the base oil cannot provide adequate lubrication. According to Nehme and colleagues, the tribological properties of base oil are affected by additives [18,19].

Compared with conventional lubricants, oils containing nanomaterials have excellent thermal and tribological properties. Nano-lubricants based on engine oil reduce friction and wear to the point where they meet or exceed industry standards. According to Zvereva et al. [21], nanostructured additives reduced viscosity by 10–20% while improving the viscosity index. Nanodiproxamine and dehydrated carbonate sludge improved fuel viscosity at high temperatures (75 °C). Dinesh et al. [22] found that engine oil with MWCNTs and zinc oxide nanoparticles added performed better. More nanoparticles with an additive in the lubricant improved the performance and efficiency of the products [18,19]. GN is the ideal material for reduced friction and wear because of its shape, high aspect ratio, and high flexibility. After dispersing nanoparticles into the base oil, the lubricant's performance and operational characteristics dramatically improved due to the superior properties of nano-lubricants. The base oil and GN additive performances have been evaluated by testing with the base oil rheological and tribological properties.

2. Experimental Procedures

2.1. Materials

GNs were combined with a fundamental fluid procured from the Nanotech Research Organization to create the nanofluids used in this study. In this case, the existing in-use commercial SHELL HELIX 5W-30 oil and its details are shown in Table 1. Table 2 lists the characteristics of the experiment nanoparticles. Because Dimethylformamide (DMF) is colorless, has low volatility, and has an extremely high dielectric constant, it was chosen as the solvent in this study. DMF was chosen as a solvent for mixing nanoparticles into the oil to prevent nanoparticle aggregation. As a result of friction, particle accumulation causes debris to appear on friction surfaces.

Table 1. Helix 5W-30 base oil properties.

Property	Value	
Lubricant Grade	SAE 5W-30	
Kinematic Viscosity @ 40 °C mm ² /s, ASTM D445	73.95	
Kinematic Viscosity @ 100 °C, mm ² /s, ASTM D445	12.02	
Pour Point, °C, ASTM D97	-48	
Density @ 15 °C, kg/m ³ , ASTM D4052	848	
Flash Point, Cleveland Open-Cup, _C, ASTM D92	220	

Table 2. Specifications of nanoparticles.

Nanoparticles	GNs	
True density	$\leq 2.20 \text{ g/cm}^{3}$	
Average lateral dimension (x and y) length	\leq 5 μ m	
Average through-plane dimension (z) thickness	~10–20 nm	

2.2. Characterization of the GNs

The pure phase and crystal structure of the GNs were determined using a PANalytical X'Pert Pro X-ray diffraction (XRD) machine with X'Pert data collector software 2.2. This was accomplished through the use of a peak-finding algorithm and analysis software. Transmission electron microscopy (TEM), Jeol JEM1200, was used to examine the GNs' structure and dimensions.

2.3. Nanolubricant Processing and Testing Equipment

The physical property tests are depicted in Figure 1, which shows the equipment used in these processes. It began by dissolving the N, N-dimethylformamide (DNF) in

the GNs (DMF). For 45 min, the nanoparticles were dispersed in a new way to avoid accumulation, as suggested in previous studies [22–25], with a DMF concentration of 3% by weight. A SCILOGEX D500 homogenizer was used to homogenize the mixture without agglomeration. After that, the GNs were stirred with the base oil. For testing and analysis, six specimens were prepared. As a control, we prepared five samples of nano-oil with varying amounts of nanoparticles (GNs), ranging from 0.03% to 0.15% of the weight of the oil. Thermal and tribological properties were measured by a K2D thermal property analyzer, a HZNQ 1101, an open-cup flash point apparatus, and a four-ball tester, as depicted in Figure 1, respectively.

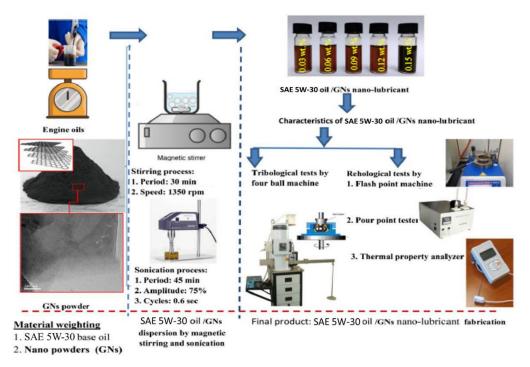


Figure 1. The sequence of preparation and characteristics of SAE 5W-30 oil/GNs nano-lubricant.

2.4. Rheological and Tribological Examinations

The four-ball tribo-tester is shown graphically in Figure 1. The oil temperature is measured at the highest pressure and temperatures during the test. This experiment was run for 60 min at room temperature with a top ball loaded to 800 N and rotating at 10,050 revolutions per minute around the perpendicular. An ASTM D-5183 load cell was used to measure the friction coefficient automatically. After that, it would be possible to compute an average value. The ASTM D-2596 method was used to measure the nanofluid's extreme pressure (EP) after it had been cranked up to 1,550 revolutions per minute. The wear of three stationary lower balls was estimated using the wear scar diameter (WSD). Five separate tests were carried out on each of the nanofluid mixtures. The average wear scar diameter (WSD) and friction coefficient were determined by measuring five lubricants and hybrid nano-lubricants tested samples. Each specimen conducted ten friction coefficient readings, and an average value was calculated.

2.5. Measurements Properties

The temperature of the lubricating oil must be maintained at a precise level. 5W-30 oil and nanoparticle-enhanced 5W-30 oil were both tested in a container. Further, a thermometer was used to keep track of the temperatures at which each specimen started to melt and flow [25]. ASTM D 97 was used to determine the pour points and temperatures of the samples. The goal of determining a sample's flash point temperature is to determine whether or not it will catch fire when exposed to air. This property can significantly influence a substance's inherently flammable properties. It was possible to conduct an

in-depth investigation of heat and ignition sources thanks to an ASTM D 93 benchmark. On top of a cylinder filled with heated liquid, scientists used only a single, simple flame to determine flashpoints. Oil specimens begin to burn at a specific temperature known as the flashpoint [26]. The thermal conductivity of nanofluids has been determined using a variety of methods, including steady-state [27], temperature oscillation [28], transient hot wires [29], and 3-omega [30,31]. A KD2-Pro instrument was used to measure the thermal conductivity of nano-oil samples using a transient hot wire approach. The temperature in the test was controlled and maintained by a water bath.

2.6. Kinematic Viscosity Measurement

Kinematic oil viscosity was studied. When a force is applied, the fluid's dynamic viscosity is measured; the kinematic viscosity of the fluid is also measured. It is also known as absolute viscosity or dynamic viscosity. The researchers used an ISO 3104-compliant capillary tube automatic viscometer to measure kinematic viscosity. Most of the time, the temperature has an impact on kinematic viscosity. The kinematic viscosity is measure in centistokes. According to ISO 3448, the viscosity of most lubricating oils is 40 °C operating temperature. Taking five average readings from 40 °C to 100 °C ensured consistent results. Viscosity index (VI) was calculated by standard practice from kinematic viscosity at 40 and 100 °C according to ASTM D2270 and ISO 2909.

3. Results and Discussions

3.1. Characterization of GNs

A strong peak at $\theta 2 = 26.35^{\circ}$ in Figure 2b demonstrates that the nanoparticles are GNs. Nanoparticle dimensions can be measured by TEM images of GNs in Figure 2a by examining the structure of GNs [14]. GNs have a length of 2.31 µm and a thickness of 1.3 nanometers, as depicted in Figure 2a, confirming the data in Table 2.

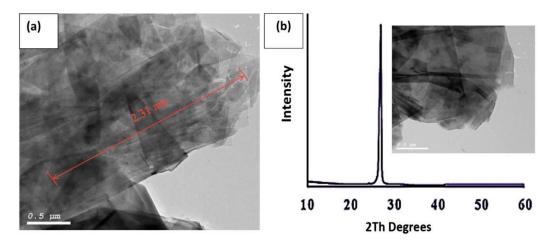
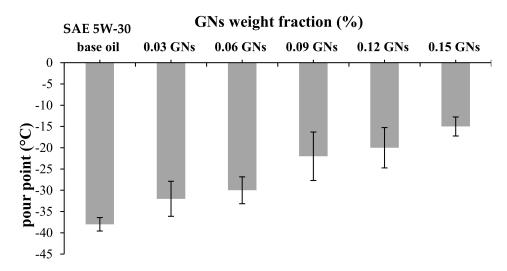


Figure 2. (a) HRTEM images of GNs; (b) XRD patterns of GNs [14].

3.2. Pour Point and Flashpoint

Overall productivity is determined by a lubricant's ability to reduce wear during the early stages of cold start, thereby improving an engine's efficiency. Pumping oil into the outlets is difficult due to a lack of oil circulation. When a lubricant becomes too thick and stops flowing, it reaches its pour point temperature. Reduced wear during the initial stages of cold starting improves a lubricant's overall performance and increases an engine's efficiency. According to standard test conditions, oil can flow freely without stirring if the pour point is below a specific temperature. GN addition significantly impacted these phenomena, as shown in Figures 3 and 4. The pour point of base oil increases by 37% when adding nano-additives (GNs) at a percentage concentration of 0.15 weight percent. However, the trend is reversed when the percentage concentration is increased to over 0.15 wt. percent, as shown in Figure 3. To illustrate, the flashpoint was raised by 25.4 percent



when 0.12 percent GN was added to the base oil 5W30, but when the GN concentration increased to 0.15 wt. percent, it decreased again, as shown in Figure 4.

Figure 3. Pour point of base oil and oil with various percentages of GNs.

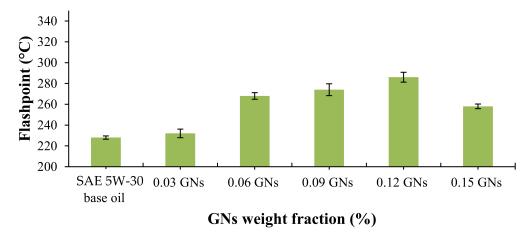


Figure 4. Flashpoint of oil alone and oil with various percentages of GNs.

3.3. Thermal Conductivity

In the cooling of engines and machines, heat flows are caused by friction or combustion and keep the lubricant stable. Within the flashpoint limit, thermal conductivity plays a critical role. Nanoparticles boost 5W-30 oil thermal conductivity. These materials have a high surface-to-volume ratio due to metallic oxide and nanoparticles; they improved thermal conductivity of nanofillers with a wide range of homogeneous and stable structures: Figure 5, SAE 5W-30 base oil and different GNs concentrations. An increase in the volume of nanofillers in the GN-containing fluid led to an increase in thermal conductivity, all thanks to the excellent thermal conductivity and effective heat dispersion of GN. At GN proportions of 0.03, 0.06, 0.09, 0.12, and 0.15 wt. percent, thermal conductivity increases by 7, 12, 6, 16.5, 19.7, and 29.9 percent, respectively. Thanks to their surface properties and microscopic action, the most effective way to increase thermal conductivity is to use nano-oil with 0.15 wt. percent GNs.

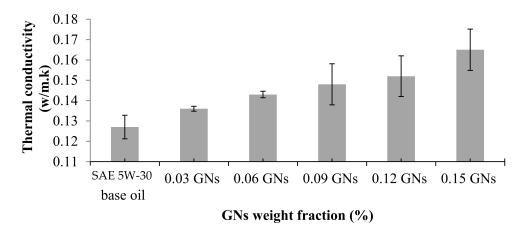


Figure 5. Thermal conductivity of different compositions of the nano-oil.

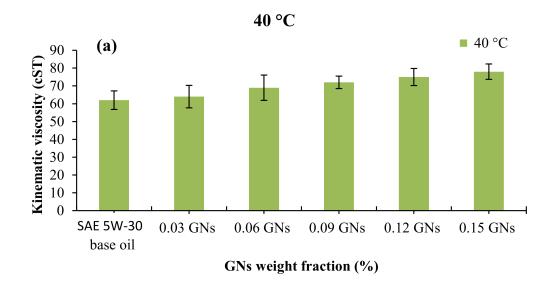
3.4. *Kinematic Viscosity*

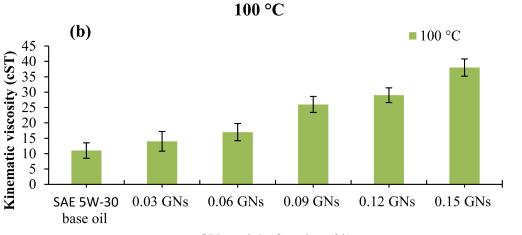
In selecting a suitable oil for any machine, kinematic viscosity is critical. Shearing of lubricating layers or planes over each other is possible because of this property. The sealing effect, internal friction, and heat development in machine components are all impacted. A lower temperature causes an increase in kinematic viscosity, while a higher temperature causes it to decrease. Kinematic viscosity decreases with age, exposure to other oils, water, particles, and other environmental factors. Kinematic viscosity at 40 and 100 °C are shown in Figure 6a,b. Because of their ability to form non-polar covalent connections, GNs were found to increase nano-oils' kinematic viscosity and shear resistance [31]. The viscosity index (VI) against the concentration of GNs is depicted in Figure 6c. Higher viscosity index (VI) promotes a more stable viscosity across a range of temperatures (independent of temperature). The values of VI are observed to increase with the addition of 0.09% and 0.15% of GNs by 8.5% and 10.5% for SAE 5W-30 oil samples, respectively. Lubricant samples with 0.03 wt.% and 0.06 wt.% GN nanoparticles seem not to influence the VI for all lubricant samples.

Kinematic viscosity was found to stabilize over the 40–100 °C temperature range when looking at Figure 6a,b. This is evident when comparing the kinematic viscosity data in Figure 6a,b. Many variables affect the kinematic viscosity of lubricating oils supplemented with nanofillers [32]. Furthermore, the lubricating film thins out under extreme temperatures, pressures, or speeds, allowing the surfaces to come into contact. GNs may have avoided border lubrication by increasing oil viscosity and index of viscosity. The best viscosity is found in nano-oil containing GNs at a concentration of 0.09 wt.%.

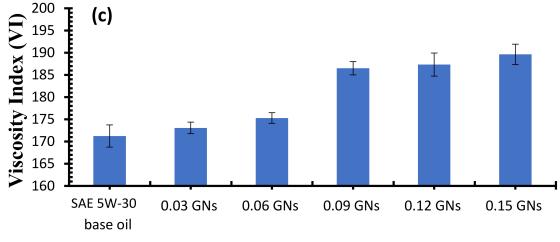
3.5. Tribological Properties

A four-ball tribo-tester measures the lubricant's wear and friction coefficients under extreme pressure. According to the pressure at which it is applied, transitional, hydrodynamic, and boundary layer regimes of lubrication can be distinguished. A layer of friction, lubrication, and wear causes scarring on the lower balls. The average diameter of the so-called wear scars can be used to gauge the amount of wear on the balls (WSD). While Figure 7a,b depict 5W-30 scars, the worn surface of the ball in both cases has been lubricated with nano-oil, which is used in concentrations as low as 0.12 weight percent. There is no denying that 5W-30 lubrication causes more wear scars and surface deformation. EDX of the worn surfaces exposed that a tribo-film collected of GNs, iron, and chromium reduced the friction between the surfaces in contact.









GNs weight fraction (%)

Figure 6. Nano-oil kinematic viscosity at (a) 40 $^{\circ}$ C, (b) 100 $^{\circ}$ C, and (c) viscosity index for different compositions.

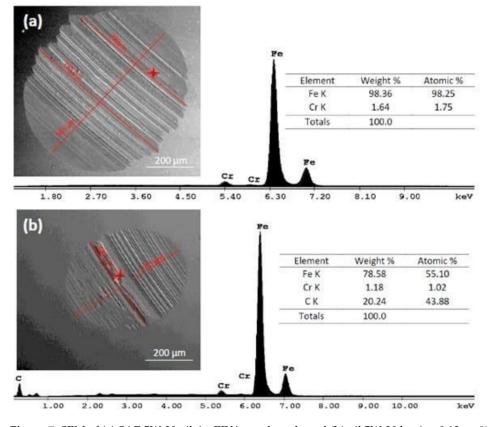


Figure 7. SEM of (**a**) SAE 5W-30 oil, its EDX at red mark, and (**b**) oil 5W-30 having 0.12 wt.% GNs, its EDX at red mark.

The mean WSD and friction coefficient are shown in Figure 8. As GN content increases from 0.00 to 0.12 wt. percent, a resilient hydrodynamic lubricant film forms, reducing WSD and friction coefficient. Low concentrations of GN tend to have higher surface-to-volume ratios [33,34]. Due to this volume, amalgamation, uniformity, friction, and WSD increased. Another possibility is that as the concentration of GNs increases, a protective layer of GN particles forms on the pin surface, which increases friction by trapping oil, nanoparticles, and surfaces together. This confirms the presence of carbon elements on the worn surface. We can conclude that the oil lubrication process was carried out by forming a tribo-film of GNs, iron, chromium, and a small portion of other elements on the ball surface (See Figure 7).

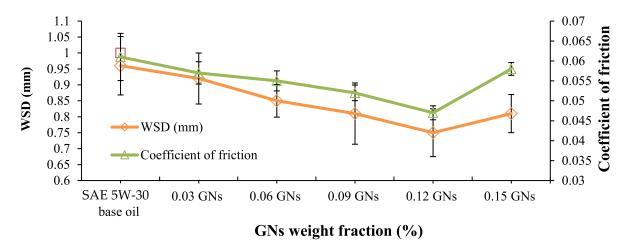


Figure 8. A wide range of GN concentrations were studied in terms of friction coefficient and WSD.

When the GN particles were elevated above the ideal limit, friction was increased on the surfaces (i.e., 0.12). An additional 0.12 concentration level of wear rate was observed; as the GN particle concentration increased, when GN particles reached their maximum effective range, particle wear and surface abrasion increased. The more nanoparticles per volume, the better the nanoparticles deposited. The lubricant's tribological properties were studied by Choi et al. [35] and Yu et al. [36] using nanoparticles, and the results were the same. Hydrodynamic lubrication is an excellent method for reducing friction and wear. On the other hand, there has been progress in the kinematic and thermal properties of GNs. The 0.09 weight percent dispersion of GNs allowed for the development of a tribo-film, which reduced friction and wear.

3.6. Comparison between Present Work and Previous Work

This section compares tribological properties in the present results and previous studies, as shown in Figure 9. However, the differences between the kinds of additive particles used and types of oils used and the comparison between the obtained tribological properties are considered. According to the published manuscripts, different nanoparticles have been used, such as CNTs, mixing individually with biodiesel oil [37,38]; on the other hand, dual CNTs and GNs have been added to SAE [23]. The coefficient of friction and WSD with individual CNTs with biodiesel and engine oil was the highest at a content of 0.06 wt.% and 0.09 wt.%, respectively. At the same time, average values of coefficient of friction and WSD were obtained in the samples of dual CNTs and GNs. Specifically, in the current study, the coefficient of friction and WSD presented the lowest value in the synthesized GNs with SEA oil. Overall, this is due to the excellent mechanical properties and two-dimensional physical appearance of GNs, whereas for nanoscale, as the number of graphene layers increases, the local stiffness of the contact area increases, leading to a decrease in the deformation that emerges from point-to-point contact and, hence, a decrease in friction [14].

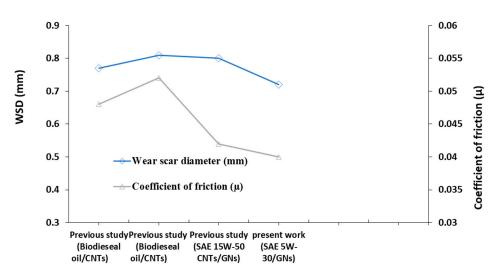


Figure 9. Comparison between present work and previous work for tribological properties [23,37,38].

4. Conclusions

We successfully synthesized oil with nano-additives of GNs in order to improve the performance of the engine. Different properties were focused on in this work, such as tribological and rheological, to study the effect of adding nano-additives of GNs on the tribological and rheolo5gical properties of oil engine performance.

According to the above observations, we can identify the main and new points:

- 1. increase in flashpoint by 25.4% when the percentage of GNs was increased;
- increase in thermal conductivity by increasing the content of additive nanoparticles of GNs;

- 3. reduced wear rate at the concentration of 0.12 wt.% GNs;
- 4. reduced friction by increasing the concentration of GNs below 0.12 wt.%;
- 5. the viscosity index of SAE 5W-30 oil lubricant was increased by 8.5% using 0.09 wt.% GNs as an additive.

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