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Article

Optimization of the Rheological Properties and Tribological Performance of SAE 5w-30 Base Oil with Added MWCNTs

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Abstract: The augmentation of lubricant oil properties is key to protecting engines, bearings, and machine parts from damage due to friction and wear and minimizing energy lost in countering friction. The tribological and rheological properties of the lubricants are of utmost importance to prevent wear under unembellished conditions. The marginal addition of particulate and filamentous nanofillers enhances these properties, making the lubricant oil stable under severe operating conditions. This research explores the improvement in SAE 5w-30 base oil performance after the addition of multiwalled carbon nanotubes (MWCNTs) in six marginal compositions, namely, Base, 0.02, 0.04, 0.06, 0.08, and 0.10 weight percentage. The effect of the addition of MWCNTs on flash and pour points, thermal conductivity, kinematic viscosity, friction coefficients, and wear are investigated and reported. X-ray diffraction and transmission electron microscopy are used to characterize the MWCNTs. The purity, crystallinity, size, shape, and orientation of the MWCNTs are confirmed by XRD and TEM characterization. Pour points and flash points increase by adding MWCNTs but inconsistency is observed after the 0.06 wt.% composition. The thermal conductivity and kinematic viscosity increase significantly and consistently. The friction coefficient and wear scar diameter reduce to 0.06 wt.% MWCNTs and then the trend is reversed due to agglomeration and inhomogeneity. A composition of 0.06 wt.% is identified as the optimum considering all the investigated properties. This composition ensures the stability of the tribo-film and hydrodynamic lubrication.

Keywords: nanolubricants; four-ball tribo-tester; thermal conductivity; flash and pour points; friction coefficient; wear scar diameter

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Highlights

- SAE 5w-30 oil-based nanolubricants were prepared by the addition of MWCNTs in six compositions.
- The tribological and rheological properties of the nanolubricants were measured and compared.
- The flash point, pour point, thermal conductivity, and kinematic viscosity increased whereas the friction and wear decreased with the addition of nanofillers.
- The 0.06 wt.% MWCNT/SAE 5w-30 nanolubricant emerged as the optimum best-performing composition.

1. Introduction

Currently, various lubricants are used to curb friction and wear in the rubbing parts inside countless mechanical systems including internal combustion engines, gears, bearings, turbines, and pumps [1,2]. By contrast, the quite prevalent reason for the deterioration of systems comprises the failure of the lubricants to protect wear. To improve lubricant properties, different nanoparticles have played a significant role in promoting performance [3]. Previous research findings suggest that the presence of nanomaterials in lubricant oil enhances its thermophysical and tribological properties [4–8]. Additionally, there are now many problems with engine oil such as extreme pressure, wear resistance, corrosive metals, and a polluted environment. In order to solve these problems, it has become popular to add carbon nanoadditives to lubricating oils to improve the quality of the lubricating oils. Most of this solid lubricant forms a lubricating film on the surface of the metal, thereby preventing the direct contact of the metal to exacerbate wear [9]. Engine oils are thixotropic fluids; their properties change with time. Under the impact of shear stresses, lubricant failure under severe conditions affects the microstructure and properties of the friction pair, resulting in their deterioration and failure. Lubricants are commonly used in automotive, rolling bearing, pumping, and other industries. Despite this, wear and friction behaviors can be predicted but under a high load, increased temperature, and sliding speeds the material still fails. Lubricants are exposes to degradation mechanisms; for example, physical and chemical mechanisms such as shear stress, lubricant evaporation, and impurity addition by pollutants and debris. Chemical degradation includes oxidation in addition to the depletion of improvers at a higher temperature. Furthermore, another form of degradation is the mixing of different types of lubricants vulnerable to reacting to each other under operating conditions [10–12]. Within the last few years, oils contained nanomaterials have exhibited breathtaking thermal and tribological properties compared to conventional lubricants. Engine oil-based nanolubricants utterly reduce friction and wear, conforming to the industrial standards and often exceeding them. Graphene and multiwalled carbon nanotubes (MWCNT) dispersed in conventional lubricants under different loads and conditions demonstrated improved tribological properties due to lower friction coefficients and load sharing [13]. Zvereva et al. [14] studied the influence of nanostructured additives, diproxamine, carbon nanotubes, and dehydrated carbonate sludge on lubricant performance and observed a reduction in viscosity by 10–20% in addition to an improvement in the viscosity index. Furthermore, nanodiproxamine improved the fuel viscosity at high temperatures (75 °C) and dehydrated carbonate sludge decreased the boiler fuel viscosity. Ahmadi et al. [15] studied the effect of MWCNTs in different concentrations on the flash point, viscosity, pour point, and thermal conductivity to improve the properties of lubricants. They employed a planetary ball mill for dispersing the MWCNTs in the base oil and recorded an improvement in the flash point and thermal conductivity of the nanolubricants concerning the base oil. Dinesh et al. [16] evaluated the impact of the addition of MWCNTs and zinc oxide nanoparticles on the performance of engine oil and found remarkable results. Saxena et al. [17] investigated the effects of nanolubricants on the thermophysical, rheological, and tribological properties of commercial lubricants. Eventually, an increasing nanoparticle volume fraction led to an increased performance of the lubricant [18,19]. CNTs have attracted much interest due to their remarkable mechanical, thermal, electrical, chemical, and optical properties [20,21]. CNTs can mitigate friction and wear in loaded contacts due to their shape, high aspect ratio, and high flexibility. The effect of carbon nanotubes (CNTs) and the addition of paraffinic oil on the flash point, kinematic viscosity, pour point, tribological properties, and thermal conductivity coefficient of gear oil was studied [18]. Due to the unrivaled properties of nanolubricants compared to standard engine oils, the lubricant performance and operational characteristics improved remarkably after the dispersion of nanoparticles into the base oil. Bahaa et al. [22] studied the rheological and tribological properties of a commercial engine oil of 15W50 grade dispersed with CNTs and graphene nanosheets (GNs). At a concentration of 1.5 wt.% CNTs and 0.5 wt.% GNs, the kinematic viscosity increased by about 76.8%, the thermal conductivity increased by 77%, the pour point and the flash point were enhanced by 20 and 15%, respectively, and the wear and friction coefficients were reduced by 78 and 48%, respectively. The literature review highlights the problems associated with conventional lubricants and their failure to perform in severe operating conditions. The research gaps in the present context include sparse information about the effect of nanofillers in the lubricant, lacking an immediate solution to the poor performance of conventional lubricants by the addition of the lubricants. The research gaps also demand an optimum composition of a nanofiller-based lubricant for a better overall performance. The present study investigates the optimum weight percentage (wt.%) of MWCNTs, which may be utilized as an additive in commercial lubricant oil to improve its performance. The rheological properties, such as pour and flash points and kinematic viscosity, and the tribological properties, such as the wear and friction coefficients, have been measured to determine the performance of the base oil and after the dispersion of the MWCNT additives.

2. Experimental Work

2.1. Materials

MWCNTs, purity 97%, were purchased from Nano Tech Co., Ltd, 6th October, Giza, Egypt. The length and diameter of the MWCNTs were 2–20 μm and 10–12 nm, respectively. Lubricant oil shell Helix 5W-30, which is a popular grade and widely used for lubrication in engines and pumps, was used as the base oil [23,24]. The main properties of base oil are presented in Table 1.

Table 1. Properties and specifications of base oil shell Helix 5W-30.

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

2.2. Characterization of the MWCNTs

A PANalytical X'Pert Pro X-ray diffraction (XRD) machine loaded with X'Pert data collector software detected the purity, phases, and crystal structure of the MWCNTs. A high score and analysis software were employed to identify the peaks. The structure and dimensions of the MWCNTs were examined by transmission electron microscopy (TEM), Jeol JEM1200.

2.3. Fabrication of the Nanolubricant and Test Equipment

Figure 1 shows the equipment used for the preparation, testing, and analysis of the nano-oil. First, MWCNTs were stirred in N,N-dimethylformamide (DMF).The percentage of DMF was 3% by the weight of the nanoparticles, which dispersed innovatively to avoid the agglomeration for 30 min as suggested in earlier studies [22,25]. The stirred MWCNTs were then mixed with the base oil and shaken up using a SCILOGEX D500 homogenizer to ensure the homogeneity of the composition without agglomeration. The specimens were prepared in six compositions for testing and analysis. The base oil without MWCNTs was taken as a reference sample without nanofillers (0.00) and five specimens of nano-oil with the composition of MWCNTs of 0.02, 0.04, 0.06, 0.08, and 0.1% by weight were the other five compositions. The tribological properties of the samples were tested by using a four-ball tester (shown in Figures 1 and 2), the pour point was measured on an HZNQ 1101, the flash point was determined by using an open-cup flash point apparatus, and the thermal properties were measured on a K2D thermal property analyzer, as shown in Figure 1.

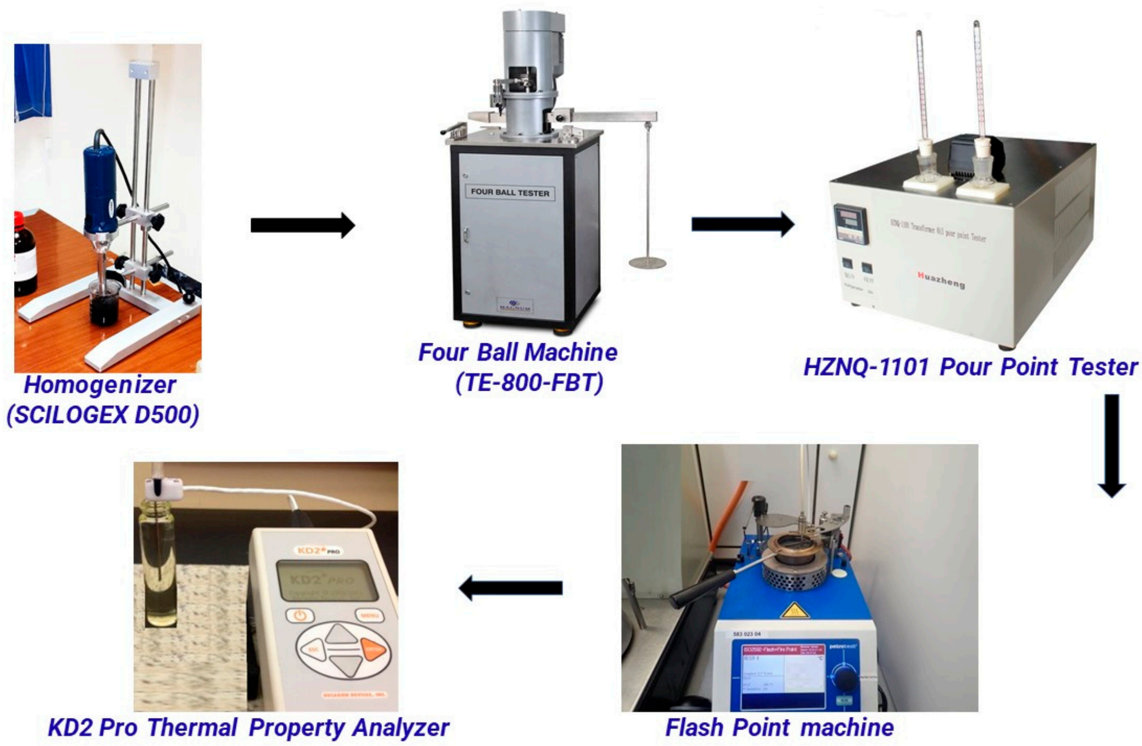

Figure 1. A snapshot of the equipment used for the preparation, testing, and analysis of the nano-oil.

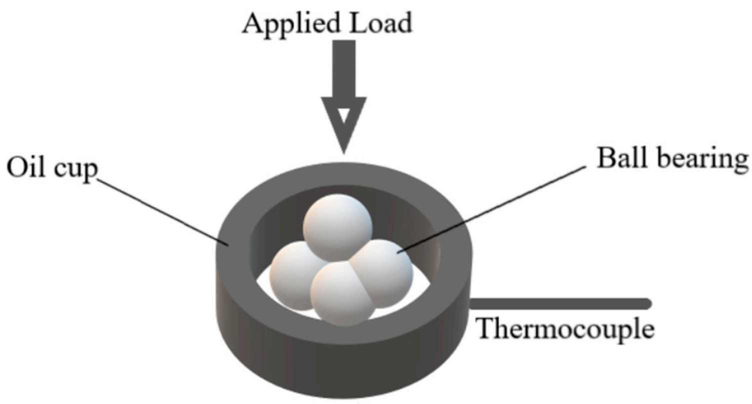

Figure 2. Schematic diagram of the four-ball tribo-tester.

2.4. Tribological Tests

The tribological properties of commercial oil SAE 5w-30 at different filler fractions were measured using a four-ball tribo-tester [19,26], depicted in Figure 2. The tests were performed under severe conditions to evaluate the wear and friction torque. An upper ball (made from GCr15 steel, Ra = 0.016 µm and 61 HR) rotated along a perpendicular axis with a rubbing speed of 1200 ± 60 against three stationary balls under a varied load up to 800 N for 60 min at room temperature. This related to a contact pressure of approximately 1 GPa with a mean contact pressure of approximately 0.7 GPa [27]. A load cell directly gave the values of the coefficient of friction per ASTM D-5183. Thereafter, the average values were calculated. The extreme pressure (EP) of the nanofluid was measured according to ASTM D-2596 after the speed increased to 1770 rpm. The wear scar diameter (WSD) method was used to calculate the wear from the three stationary lower balls to generate statistically reliable results. Five tests for each nanofluid composition were performed.

2.5. Pour Point, Flash Point, and Thermal Conductivity Measurements

The pour point is the minimum temperature at which a liquid forfeits its flow properties. It is the pivotal behavior of lubricant oil to flow at a particular temperature. The virgin oil and the oil with added nanoparticles were poured inside a container and held in a test tube holder. After solidification, the test tubes were extracted and a thermometer measured the temperatures at which the solidified samples began to melt and flow [28]. Using the ASTM D-97 standard, the temperature was noted and the pour point of the oil samples was recorded. The flash point temperature is used to measure the propensity of the test samples to brew a flammable mixture with air under specified states. It is considered to be one of the most valuable properties to estimate the overall flammability of a material. Using the ASTM D-93 standard, the flash point was measured to examine the response to heat and an ignition source. The flash points were experimentally fixed by heating the liquid in a container and then introducing a modest flame on top of the liquid surface. The flash point corresponds with the temperature at which the oil sample ignites [29]. Different techniques have been used to estimate the thermal conductivity of nanofluids by using steady-state [30], temperature oscillation [31], transient hot-wires [32], and 3-omega [33] methods. A KD2-Pro device was used to measure the thermal conductivity of the nano-oil samples through the transient hot-wire method.

2.6. Measurement of the Kinematic Viscosity

The kinematic viscosity of the base and nano-oil were measured by a kinematic viscometer, which indicates the resistance of the lubricant to shear and drift under gravity and gradient. Two types of oil viscosities were studied: the kinematic and dynamic viscosity. The dynamic viscosity represents the forces required to make the fluid flow whereas the kinematic viscosity gives the flow rate under an applied force. Dynamic viscosity is also known as absolute viscosity. A kinematic viscometer governs the flow rate of a lubricant. A capillary tube automatic viscometer measured the kinematic viscosity conforming to the D445 (ISO 3104) standard. Typically, kinematic viscosity changes with the temperature. It is measured in centistokes (CST) or mm^2/s. The ISO 3448 grading system for the viscosity of most lubricant oils reports it to be 40 °C. An average of five readings ensured the reproducibility of the results at two test temperatures, namely, 40 °C and 100 °C. The minimum, maximum, and midpoint viscosity of the oil at 40 °C corresponding with the SAE 5w-30 grade should be 90, 110, and 100 CST, respectively. Most engine oils are exposed to high temperatures. The kinematic viscosity for engine oils is referenced at 100 °C as per the SAE J300 standard. At 100 °C, the interference of engine soot contamination reduces [34].

3. Results and Discussions

3.1. Characterization of the MWCNTs

Figure 3a shows the TEM imagery, giving valuable intrinsic information, size, and dimensions of typical MWCNTs added to the SAE 5w-30 base oil. The diameter of the MWCNTs was ~8 nm and the length were ~10–20 µm. Figure 3b gives the XRD pattern of the MWCNTs, revealing their crystallographic structure. One prominent peak emerged against the background noise at $2\theta = 26.5°$, identified as MWCNTs by the Highscore plus data cards with an hkl value of (0 0 2). The purity of the MWCNTs and their size were therefore confirmed.

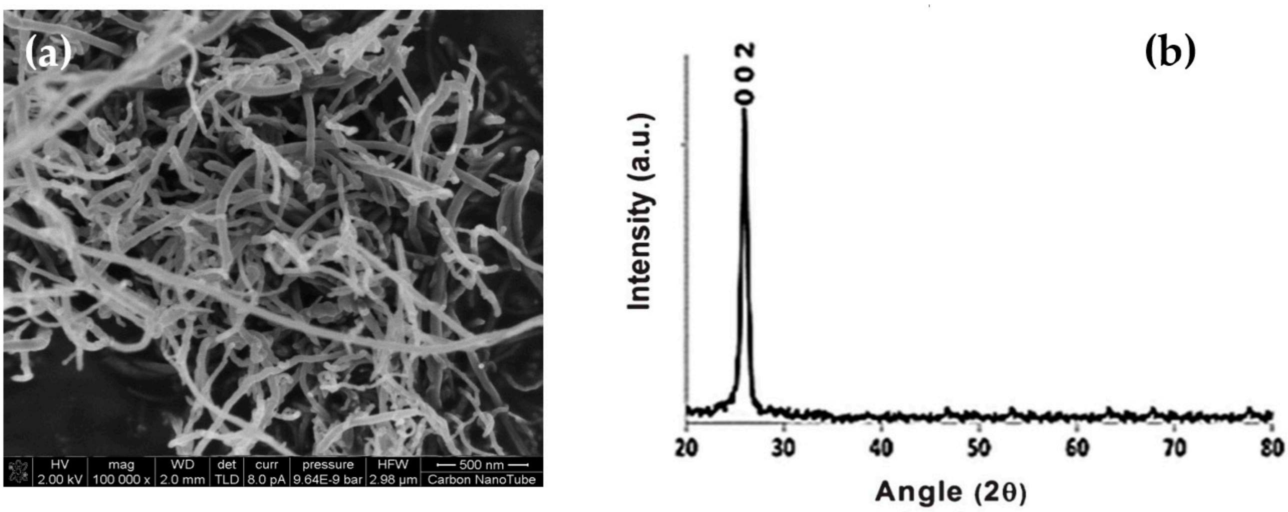

Figure 3. (**a**) The TEM image of the MWCNTs. (**b**) The XRD patterns of the MWCNTs.

3.2. Pour Point and Flash Point

The pour point temperature indicates the temperature at which a lubricant becomes too viscous and ceases to flow. It is an important property and gives the overall performance of a lubricant and boosts the efficiency of an engine by reducing the wear at the initial stage of cold starting. Due to the absence of oil circulation among the engine parts, the oil is difficult to pump into the channels. When engines and machines are cold and the viscosity of the oil is at its highest, the pour point of the lubricant determines the ease of starting. Several attempts have been made to increase the pumpability and flowability of oil so that it may reach the moving parts as quickly as possible. The published literature asserts that the pour point of the oil is the temperature below which the fluid loses its ability to flow normally. Figure 4 depicts the pour point temperature of the different compositions of the base and nano-oil. The pour point of the base oil is the lowest whereas the addition of the MWCNTs increased the pour point temperature until 0.08 wt.% by about 74% and it decreases again at 0.10 wt.%. The nanoadditive formed stronger non-polar covalent bonds with the molecules of the base oil. The obtained results indicated that beyond the concentration of 0.06 wt.%, the effect of MWCNTs tended to saturate. Beyond 0.08 wt.%, MWCNTs had no ratable impact on the pour point of the nanolubricants.

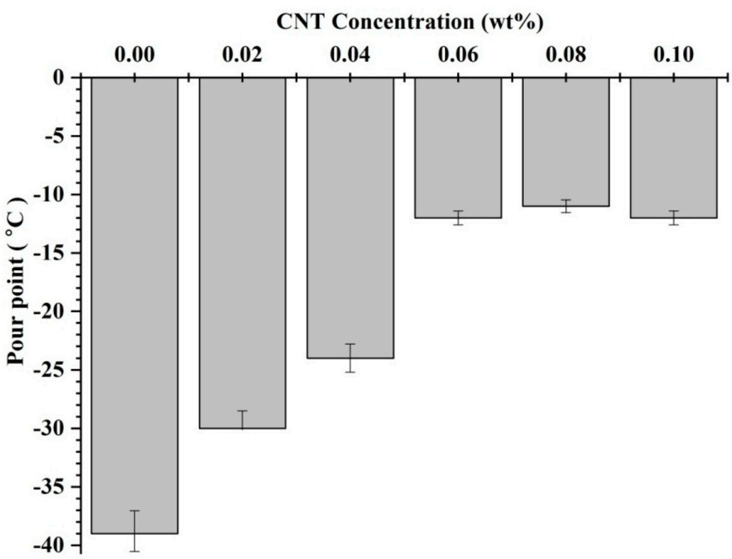

Figure 4. Pour point temperatures of the different compositions of the nano-oil.

The flash point of a lubricant is important in determining its volatility and resistance to flammability, along with the fire point. The minimum temperature at which the lubricant generates substantial combustible vapors may generate a flash if mixed with air and brought into contact with a flame [35]. The flash point determines the safe temperature at which a lubricant may be stored and transported without the risk of self-ignition. Higher flash points are desirable for the thermochemical stability of a lubricant. Figure 5 shows the flash point results of the compositions of the nano-oils reinforced by MWCNTs. The addition of MWCNTs increased the flash point dramatically from the base oil to 0.06 wt.%, decreasing its volatility and making the handling of the nano-oils easy. The flash point saturated at 0.08 wt.% concentration rather tended to decline again, and further dropped significantly between 0.08–0.10 wt.%. Earlier studies reported the same trend of increasing flash points to a certain composition of the nanoadditives and then a decrease [35,36]. The reason for the decrease was not revealed. The increase of the flash point may be attributed to the increase in the thermal conductivity, imparting the resistance to the formation of combustible vapors. The high thermal conductivity of MWCNTs could inspire an increase in the flash point for nanolubricants compared to the base lubricating oil and thus let it remarkably improve [37]. The nanoadditive formed stronger non-polar covalent bonds with the base oil, thus requiring higher energy for depletion. Therefore, the thermal stability increased, raising the flash point. After 0.08 wt.%, the dispersion of the MWCNTs was inhomogeneous due to agglomerations, retarding the formation of non-polar covalent bonds. As a result, the vapor formation increased again at a lower temperature.

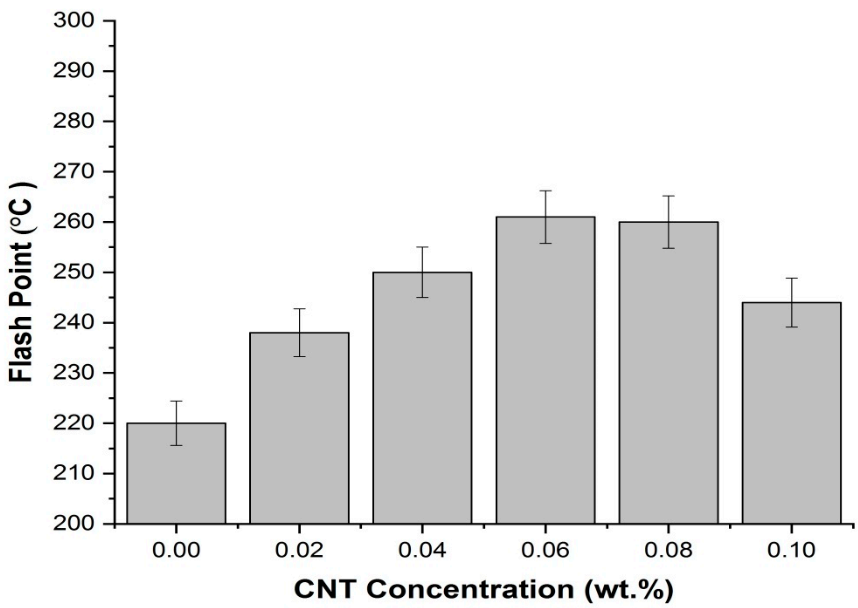

Figure 5. Flash point temperatures of the different compositions of the nano-oil.

3.3. Thermal Conductivity

The thermal conductivity of a lubricant plays a significant role in cooling the engine and machine parts and transferring heat generated due to friction or the combustion of fuel whilst maintaining itself at a reasonable temperature at which it is thermally stable and within the flash point threshold. Generally, the presence of nanoparticles inside a base oil ameliorates its thermal conductivity. The metallic oxides and nanoparticles have a high ratio of surface to volume. Various researchers have indicated impressive physical and mechanical properties for this innovative form of carbon including the thermal conductivity of CNTs, which has gained a great deal of attention. Recent measurements of the conductivity of CNTs confirmed conductivities of about 3000 W/m K for multiwalled carbon nanotubes (MWCNTs) [38]. Homogenous and stable nanofillers with diverse structures exhibited a better thermal conduction phenomenon [35,36,39]. Figure 6 shows the thermal conductivity of the virgin oil and the nanolubricants in different compositions. The fluid comprising MWCNTs showed an ultimate boost in the thermal conductivity, consistent with the increase in the concentration of the nanofillers, which was ascribed to the excellent thermal conductivity of the MWCNTs and their ability to dissipate the bulk heat quickly. The improvement in the thermal conductivity at compositions 0.02, 0.04, 0.06, 0.08, and 0.10 wt.% of MWCNTs was 1.6, 6.4, 10.4, 14.5, and 18.6%, respectively. The nano-oil with 0.06 wt.% MWCNT exhibited the highest slope and improvement in thermal conductivity due to the surface properties of the MWCNTs and their microscopic activity. Furthermore, the random movements of the nanofillers suspended in the oil resulting from the molecular impact and their transitions increased the thermal conductivity [18]. These molecular movements increased with an increase in the temperature therefore increasing the thermal conductivity. Due to the improvement in thermal conductivity and the movement of the nanofillers within the oil, oil sludge fashioning may have been prevented on the inner surface of the engine. The presence of the MWCNTs effectively reduced the thickness of the lubricant layer when the temperature increased, making the engine oil less prone to burning due to the restricted thermal storage and increasing its efficiency. Uniform dispersion favors the thermal conductivity of the nanofluids whereas the larger separation among them reduces it, so the higher the concentration of MWCNTs, the higher the thermal conductivity. A high thermal conductivity is desirable for stability and cooling. However, it does not directly interfere or influence other properties such as friction coefficients, wear, flash points, pour points, and kinematic viscosity.

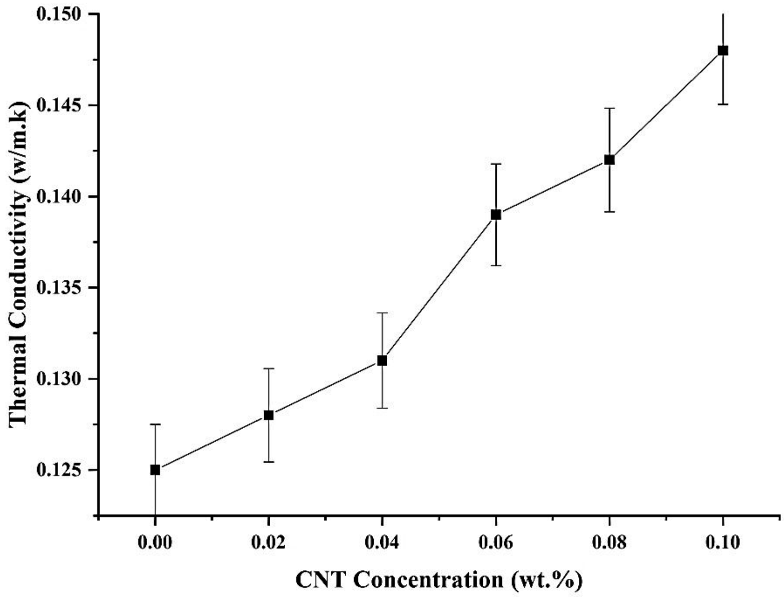

Figure 6. Thermal conductivity of the different compositions of the nano-oil.

3.4. Kinematic Viscosity

The kinematic viscosity of many lubricants is important for their proper use; for example, the flow of fuels through pipelines, injection nozzles, and orifices and the determination of the temperature range for the proper operation of fuel in burners [40]. The kinematic viscosities of the nanolubricants were measured at 40 °C and 100 °C at different concentrations 0.02, 0.04, 0.06, 0.08, and 0.10 wt.% in the context of the survey of the influence of carbon nanotube concentration and tempering on the viscosity of the lubricants. As illustrated in Figure 7, the viscosity of all samples decreased. The nanotube coalesced to form large asymmetric particles as the concentration increased, preventing the movement of oil layers on one another and thereby increasing the viscosity. In general, according to various studies, the way in which nanofluids change viscosity is influenced by different factors such as the fluid, nanoadditives of the type, concentration rate, and nanoadditive dispersal method within the basic fluid as well as the dispersion into the base fluid of nanoadditives. In this study, the viscosity of the base oil was reduced by about 75% at a concentration of 100 °C and 0.1% by the addition of carbon nanotubes. In the case of the nanolubricants, it was increased by approximately 31.7% at 40 °C and 0.1 wt.%, the greatest reduction and increase in viscosity, respectively. Lastly, the addition of MWCNTs to SAE 5w-30 oil had a significant effect on the low concentrations of viscosity of the base oil. Figure 8 shows the effect of the MWCNT concentration on the viscosity index of the lubricant film. The viscosity index is an important parameter indicating the temperature-related flow properties of an oil. Selecting an oil without considering its VI for a certain application can lead to premature wear and costly damage of machinery. A higher VI is more desirable because it enables the lubricant to provide a more stable lubricating film over a wider temperature range [41]. It was found that the maximum raise of the viscosity index of the nanofluids above the net lubricant oil was at the 0.10 CNT concentration.

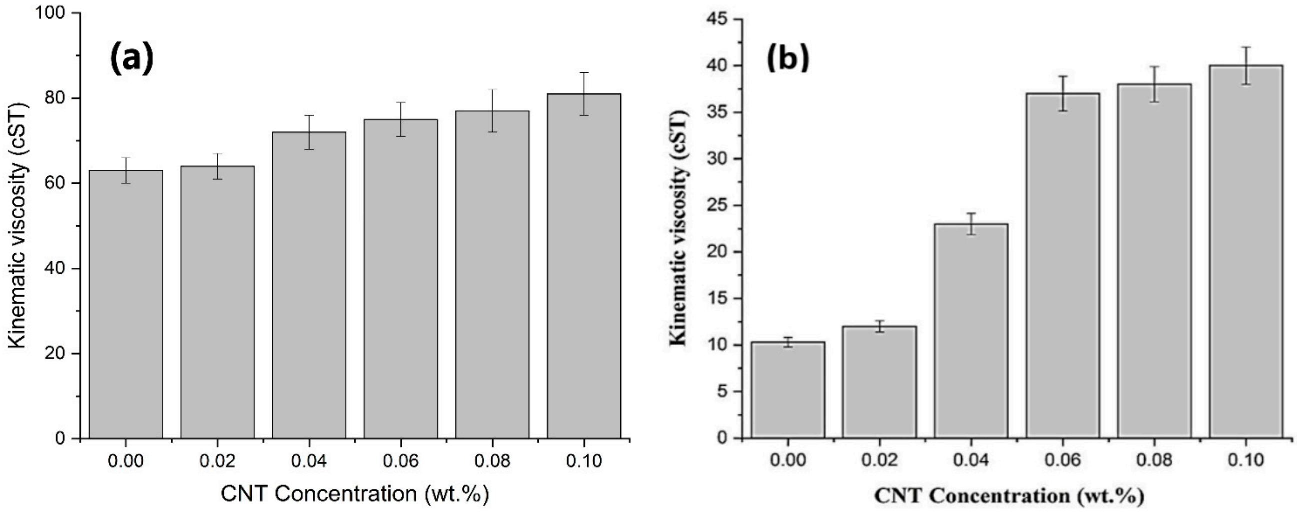

Figure 7. Kinematic viscosity of the different compositions of the nano-oil: (**a**) at 40 °C; (**b**) at 100 °C.

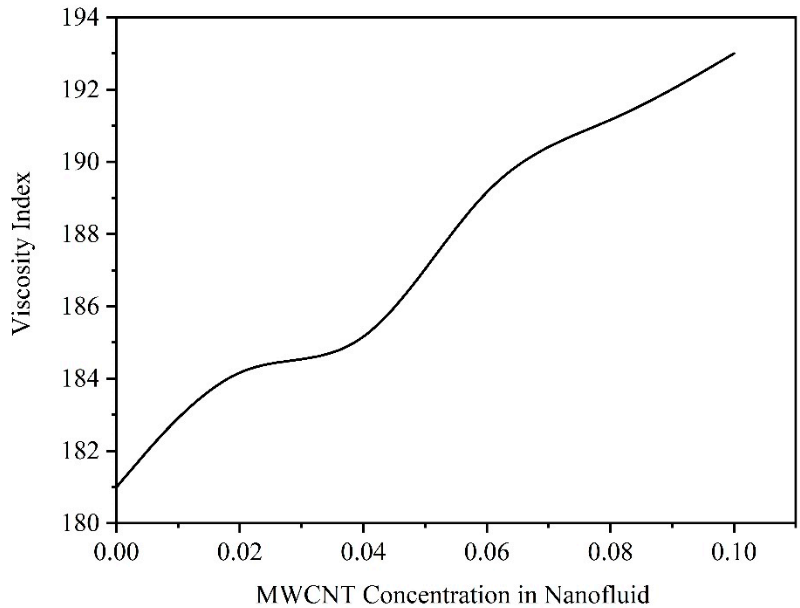

Figure 8. Viscosity index of the nanofluid vs. the CNT concentration.

3.5. Tribological Properties

The four-ball tribo-tester determines the wear and friction coefficients of a lubricant under extreme pressure. The lubricant exhibits three regimes, depending on the applied pressure: the hydrodynamic (desirable), the transitional or mixed, and the boundary layer (undesirable) [42]. Friction and wear create scars on the surface of the lower balls under the boundary layer lubrication and the average diameter of the wear scars (WSD) on the balls indicate their wear after measurement with a digital microscope. Figure 9a shows the worn surface of the ball under base oil lubrication and Figure 9b shows the scar created with 0.06 wt.% nano-oil. It conclusively emerged that the wear scar and the surface deformation was higher under the base oil lubrication. The wear scar under the base oil lubrication comprised noticeable subterranean furrows and channels whereas the scar under the nano-oil lubrication was smooth with shallow grooves.

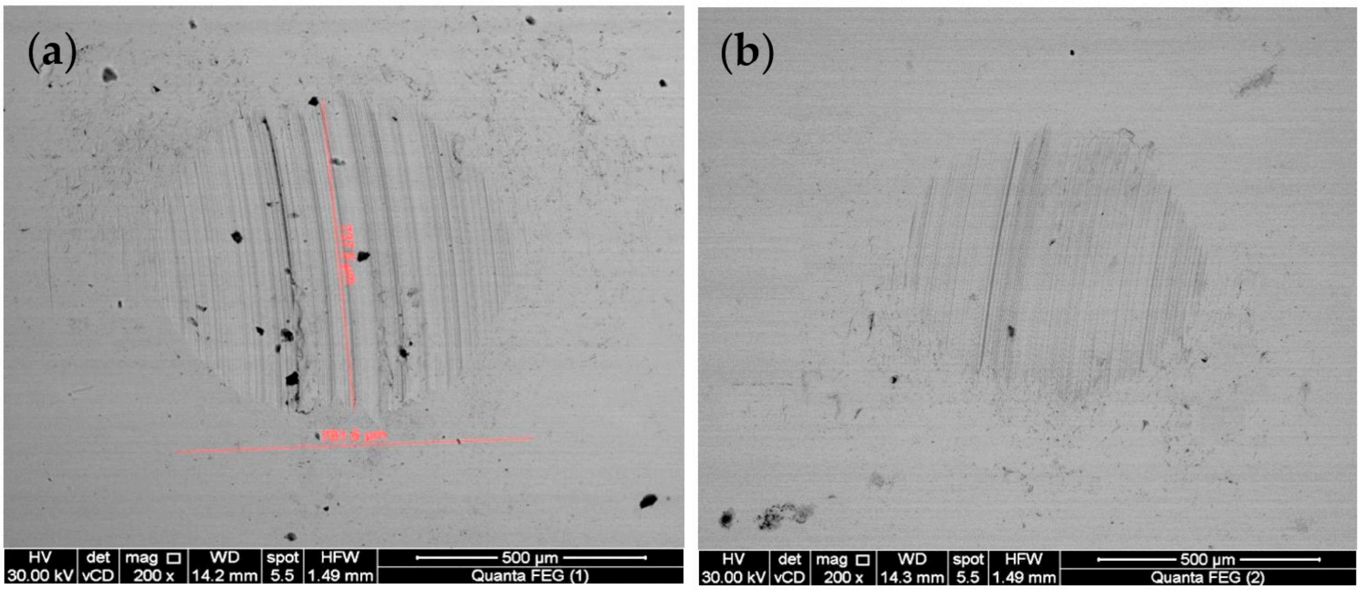

Figure 9. SEM of the wear scar of the steel balls lubricated by (**a**) SAE 5w-30 base oil; and (**b**) 0.06 wt.% MWCNT nano-oil.

Figure 10 depicts a plot of the average WSD and the friction coefficient for all the nano-oil compositions. The WSD and the friction coefficient reduced with an increase in the concentration of the MWCNTs from 0.00 to 0.06 wt.%. Beyond this concentration, the agglomeration and inhomogeneity led to an increase in both the friction and WSD. Typically, a low concentration of MWCNTs exhibits a non-polar nature with a higher surface to volume ratio [43]. In the 0.06 wt.% concentration, the dispersion of the MWCNTs was sound enough to impart the optimum conditions for the formation of the tribo-film, reducing friction and wear.

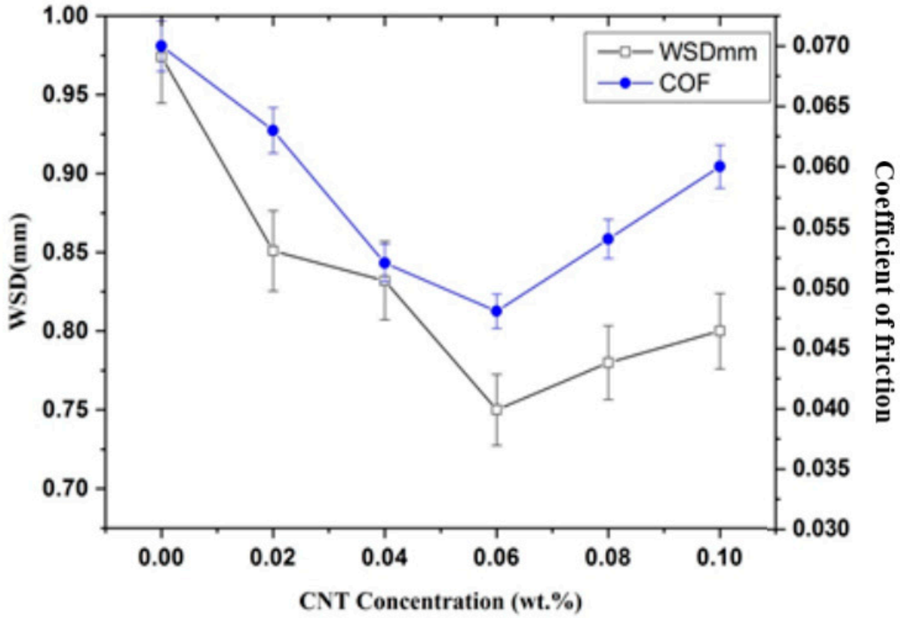

Figure 10. Friction coefficient and wear scar diameter measured for different CNTs concentrations

4. Conclusions

This article explores the development of SAE 5w-30 oil-based nanolubricants. The addition of MWCNTs increased the pour point temperature to 0.08 wt.% by about 74% and it decreased at 0.10 wt.%. The flash point increased from the base oil to 0.06 wt.%, decreasing its volatility and making the handling of the nano-oils easy. The flash point saturated at the 0.08 wt.% concentration and dropped significantly between 0.08 and 0.10 wt.% due to inhomogeneity and agglomerations, retarding the formation of non-polar covalent bonds. The 0.06 wt.% nano-oil exhibited the highest slope and improvement in thermal conductivity due to the surface properties of the MWCNTs and their microscopic activity. The WSD and the friction coefficient reduced with an increase in the concentration of the MWCNTs from 0.00 to 0.06 wt.% due to the formation of a stable hydrodynamic lubricant film. Beyond this concentration, the agglomeration and inhomogeneity led to an increase in both. The improved performance and stability of nanolubricants offer great opportunities for reductions in friction and wear, increasing the service life of lubricants and machine components and considerable fuel-energy savings.

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