Experimental study of Carbon Nanotubes to enhance Tribological Characteristics of Lubricating Engine Oil SAE10W40

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Abstract. Wear and friction had always been key factors in the functioning of advanced machines. Machine components containing high-performance oil cannot withstand increasing temperatures and intense pressures; therefore, additives improve different lubricants' parameters and performances. Lubricating oil is the only element that can minimize wear and friction while simultaneously protecting materials from wear. The relative movement between two surfaces is impossible without a lubricant. As a result, for a more complex machine, stern lubrication is necessary. Nanomaterials play a vital role and have tendency to improve tribological properties. Carbon nanotubes (CNTs) are most popular due to numerous applications in different fields on account of their excellent chemical, electrical, thermal, mechanical, and optical properties. This study aims to research the tribological activity of carbon nanotubes (CNTs) with a four-ball tribotester. Furthermore, SWCNTs and MWCNTs as an additives in SAE10W40 motor oil were also tested to evaluate its tribological characteristics. In compared to industrial engine oil without SWCNT and MWCNT, using MWCNTs as additives in the engine oil reduced the worn scar diameter by 67 percent, while utilising SWCNTs as an addition reduced the worn scar diameter by 38 percent. The average friction coefficient with MWCNTs is decreased by 56%, and with SWCNTs is decreased by 48%; this may be due to the effect of viscosity. The provided experimental result conveys that the lubricating oil with multiwall carbon nanotubes exhibit higher performance in anti-wear and decrease friction reduction compared with single-walled carbon nanotubes.

Keywords: Lubricant, Tribological properties, Carbon Nanotubes, Friction, Additive

1. Introduction

In the manufacturing process, machines play a vital role in different fields of the production industry. Wear and friction have limited the life of mechanical parts of devices, which may result in mechanical energy loss and, as a result, more fuel is needed to accomplish the same task. Also, it consumes more power and quickly overheats its working parts, resulting in equipment failure [1]. Therefore, critical challenges to be resolved effectively for an advanced production industry are friction and wear [2]. To avoid equipment failures, efficient lubrication is crucial. Oil is the most ancient method of reducing friction and wear. However, with the advancement in manufacturing technology, high pressure, and temperature occurred due to friction. Also, high speed makes the oil unfit, which needs the oil's lubrication characteristics to be enhanced. The high-quality lubricants are continuously formulated with improved additives to overcome these problems [3].

Lubrication additives are an essential feature of all lubricant systems because they add properties to the formulated lube - oil that the base oil lacks. Thus, additives are those chemical matter which are mixed with lube oil to transmit certain characteristics to the finished oil [4]. Because of the limitations of basic oils' characteristics, they are unable to match the needs of a high-performance lubricant. Additives improve the qualities of the existing base oil, reduce unfavourable properties, and introduce new features to the base oil in a number of ways [5]. By improving the properties of
lubricating oils, additives have contributed in the development of better prime movers and manufacturing equipment[6]. Anti-friction, anti-wear, extreme pressure, anti-corrosion, and anti-oxidant properties of lubricants can all be improved by adding additives to them. Few examples of these additives are organo-zinc phosphate compounds and organo-molybdenum compounds [7]. Traditional additives such as sulphides [8], chlorides [9], and phosphates [10] are used to protect materials from producing excessive wear and tear. However, these may have at least one of the following disadvantages like copper and/or lead corrosion, less soluble, finished lubricant grow dark, and increased levels of sulphur and/or phosphorus in the finished lubricant. Hence, there is a demand for additives in lubricant which will alter the overall qualities, performance of lubricants and also act as more eco - friendly and dealing with pollution control systems used in automotive and diesel engines[11].

Automobile engine manufacturers and machines are increasingly focusing on features such as low fuel consumption, high speed, and maximum kinetic energy [2]. They are constantly experimenting with the fabrication of automotive engine designs that contribute to higher friction and quick component wear. A high-performance lubricant [12] is one of the most important options for meeting all of these criteria. Engine oil is the most widely used lubricant. As a result, one of the most critical problems in advanced manufacturing processes is to boost properties like fuel consumption and friction. The search for improved lubricant additives to enhance tribological qualities including lubrication, friction, and wear continues [13].

The name tribology comes from the Greek word tribos, which means "study of things that rub" or simply "rubbing" [14]. Friction, lubrication, and wear in all contacting pairs are dealt with in this branch of engineering. As a result, tribology is known as the science and technology of interacting surfaces moving relative to one another, as well as the problems that come with it. Tribological awareness increases the service life, protection, maintenance, repair, and wear of technical equipment, as well as the efficiency of interacting machine components ranging from spacecraft to household items, resulting in major cost savings and economical benefits [15]. Industrial lubricants are mainly used to increase component life and quality by reducing material wear and surface friction [16]. This quest for energy efficiency has led to the use of nanoparticles as lubricant additives in current research. Nanoparticles are the newest class of lubricant additives that gained popularity among other additives utilized in the past [17]. Nanotechnology, by the use of nano additives, has the potential to enhance lubricant efficiency. Nanomaterials can thus be used as lubricant oil modifiers to improve tribological properties and reduce wear and friction [18].

Nanotechnology is today one of the world's most rapidly expanding topics of research, with applications in many fields of technology and science [19]. Nanomaterials, due to its unique features, such as the quantum and small size effect, surface and interface effects, have emerged as a new additive alternative in recent years. The use of nanoparticles in Tribology has attained much attention recently [20]. The addition of nanoparticles to oil has also been confirmed to improve extreme-pressure properties and load-carrying capability, as well as lowering friction coefficients [18]. Small scale effects, temperature insensitivity, and strong tribological properties are all significant advantages of nanoparticles employed as lubricant additives to improve conventional lubricants [21]. They are excellent as lubricant additives because their size, which is generally less than 100 nm, permits them to enter the contact zone and is thermally stable at high temperatures [22].

Nanomaterials are frequently employed in a variety of human activity domains, such as cancer therapy, modified textiles, energy storage, rocket explosives and propellants, as illustrated in figure 1. Nano lubricants, according to several studies, are efficacious in reducing friction and wear. They used a range of nanoparticles obtained from polymer, metal, organic, polymer, and inorganic components to synthesize nanolubricants. Carbon Nanotubes, graphite [24], molybdenum disulfide [25], Silica Oxide [26], Aluminum Oxide [27], Copper Oxide [28], Titanium Oxide [29], and Zinc Oxide [30] are nanoparticles that have been studied as pure oil additives. Carbon nanotubes (CNTs) have aroused a lot of attention due to their exceptional mechanical, thermal, electrical, chemical, and optical capabilities. Superior mechanical strengths, CNTs in various forms have been investigated for
tribological applications[31]. This paper looked at the tribological characteristics of Single-Walled Carbon Nanotubes (SWCNTs) and Multi-Walled Carbon Nanotubes (MWCNTs) as an additive in Engine Oil to improve friction-reduction, load-carrying capacity of pure oil, and wear-reduction capabilities.

Figure 1. Examples of Nanomaterial with different structures [32]

1.1 Overview of lubrication and lubricants
The oldest method of reducing wear and friction in sliding contact bodies was liquid lubrication. Lubricant isolates adjoining parts by creating a fluid film between them. Metal-to-metal interaction is reduced by the fluid film [33]. Therefore, Lubricants are substances that are used to reduce the force of friction between the moving parts of a system in contact. The process of minimizing friction between the moving parts of a machine that comes into contact is known as lubrication.

Lubricants play a major role in machinery element safety, as they:

• Separate moving parts from one another
• Remove heat from contact by passing through.
• Ensure a clean surface.
• Transport functional additives toward the surface
• Power transfer in applications such as hydraulics, automatic transmission, and brakes [34].

1.2 Lubrication Types
When it comes to forms, there are several types to choose from. The three types of lubrication are represented in Figure 2: boundary, mixed, and complete film. Despite the fact that each one is different, they all rely on additives or lubricants contained in oils to preserve them from wear and tear. Aside from this, there are two types of full-film lubrication: hydrodynamic and elastohydrodynamic.

(a) Hydrodynamic lubrication occurs when a fluid layer effectively separates two sliding surfaces (relative to each other).

(b) In Elastohydrodynamic lubrication, the film is thinner, the friction is higher, and the effect is more noticeable when the surfaces are rolling. Elastohydrodynamic describes how the elasticity of the film deforms the rolling surface. [35].

(c) In conditions with frequent starts and stops, as well as shock loading, boundary lubrication occurs. If complete film cannot be created owing to speed, load, or other circumstances, certain lubricants contain extreme-pressure (EP) or anti-wear (AW) compounds to protect surfaces [36].
1.3 Role of Nanomaterials in lubricants
Since most existing lubricants have reached their maximum performance capacity, new ones must be formulated that are capable of achieving energy efficiency in a number of fields and under increasingly difficult conditions [42]. The most significant attribute of nanomaterial is their small size, which allows them to enter microscopic spaces between rough surfaces in contact and modify the contact's tribological performance. As a result, nanoparticles provide an alternative to lubrication by directly introducing third-body entities into the interface. To improve tribological properties, a minimal concentration of nanoparticles (up to 1%) is enough.

The following are some of the benefits of lubricant having nanoparticles as additives:

a) Insolubility in non-polar base oils.
b) Their reactivity with other lubricant additives is low.
c) A huge film formation on a variety of surfaces.
d) Longer lasting.
e) Non-volatility is high, allowing it to withstand high temperatures.

1.4 Nanomaterial parameters responsible for enhancing the performance of lubricant oils
Nanomaterial size and morphology: The nanoparticles' size has a great impact on the tribological behavior of nano lubricants in a number of different ways. The nanoparticles' small size allows them to reach the rubbing surface, reducing friction as well as wear mechanisms [44]. The size of nanoparticles can also affect lubricant homogeneity. It was noticed that decreasing the particle size increased the dispersion of nanoparticles [45]. The shape and structure of nanoparticles are extremely important. Nanoparticles come in five different shapes: spherical, onion-shaped, granular, tube-shaped, and sheet-shaped [46].

Dispersion stability of nanoparticles: Because of their large surface area to volume ratio, nanoparticles possess high surface energy. The particles in suspension can bind together to form agglomerates in order to achieve the equilibrium state. This phenomenon is known as Agglomeration/floculation [47]. The sedimentation rate is affected by the agglomeration of nanoparticles, as well as the ability to protect against wear and friction. As a result, the nanoparticles' dispersion stability is essential [48].

1.5 Carbon nanotubes (CNT) – lubricants
Carbon nanotubes (CNTs) are streamlined, tubular structures made by rolling c-c covalent bonds in a hexagonal plane. CNTs have the highest yield strength (~ 1 TPA) of any substance [49]. Nanometer-
sized carbon nanotubes can self-assemble at sliding asperity contacts, reducing the sliding contact area and shear stresses, leading to reduced friction and wear. Recent progress in nanoparticles have caught the eye of researchers for their critical number of applications. The nanoparticles have been identified to have superior mechanical and chemical properties. Carbon Nanotubes are an example of a breakthrough that has fascinated the attention of researchers due to their superior mechanical and chemical properties [50].

Carbon nanotubes are of two types: single-walled carbon nanotubes (SWCNT) and multiple-walled carbon nanotubes (MWCNT). Single-walled carbon nanotubes (SWCNT) are cylindrical in shape and can be formed by rolling a graphene sheet. As graphene is derived from honeycomb lattice, and sheets are seamless cylinders that reflect a single atomic layer of crystalline graphite. An MWCNT is a graphene sheet stack that has been folded up into concentric cylinders. Millions of atoms make up each of these nanotubes. This molecule is tens of micrometers long and has a diameter of 0.7 nanometers [51]. SWCNTs are typically just ten atoms around the circumference and have a one-atom thickness. Nanotubes can be thought of as a one-dimensional structure with a length-to-diameter ratio (aspect ratio) of around 1000 [52].

![Figure 3. Schematic diagram of Graphene rolled into A) Single-wall carbon nanotubes (SWCNT), B) Multiwall carbon nanotubes (MWCNT)[53].](image)

**2. Experimental**

In this study, the tribological effects of single-wall carbon nanotubes and multiwall carbon nanotubes are investigated by mixing it into the engine oil at definite proportions.

**2.1 Materials**

Commercial Engine oil SAE10W40 with a viscosity of 83.5 cSt at 40 degrees Celsius was used as the base lubricant. The lubricants' properties are described in Table 1. Single wall carbon nanotubes (SWCNTs) and multiwall carbon nanotubes (MWCNTs) bought from Nanoshel LLC were employed in varied ratios as solid additions to the base oil (0.1, 0.5, 1, and 2 wt%). SWCNTs had an average length and size of 8 um and 2 nm, respectively, while MWCNTs had an average length and size of 8 um and 20 nm, respectively.
Table 1. Specifications of Engine oil SAE10W40 used

| Property or Test                        | SAE Grade10W40 |
|-----------------------------------------|-----------------|
| Viscosity (at 40°C), cSt               | 83.5            |
| Viscosity (at 100°C), cSt              | 13.0            |
| Viscosity index                         | 155             |
| Density, g/ml at 15.6°C                 | 0.86            |
| Flash Point, °C                         | 212             |
| Pour Point, °C                          | 45              |

2.2 Synthesis

Oil blends were formulated by adding SWCNTs and MWCNTs at different concentrations of 0.1%, 0.5%, 1%, and 2%, respectively, which were then mechanically stirred for 30 minutes and ultrasonically agitated for 60 minutes. Ultrasonication was employed to achieve uniform dispersion of SWCNTs and MWCNTs in the lubricant to minimise aggregation. The nano lubricants were collected after ultrasonication for 2 hours. Figure 4 depicted the illustration of nano lubricants

![Synthesis of CNTs (SWCNT and MWCNT) added in Engine Oil](image)

Figure 4. Schematic illustration of the synthesis of CNTs (SWCNT and MWCNT) added in Engine Oil

3. Results and discussion

3.1 Fourier transforms infrared (FTIR) spectroscopy

FTIR analysis uses infrared light to scan samples in order to distinguish biological, inorganic, and polymeric components. Fourier transform infrared (FTIR) spectral tests of SWCNT and MWCNT were conducted at intelligent materials Pvt.Ltd. to confirm potential structural variations between the two series of samples. FTIR spectra of SWCNT and MWCNT ground in KBr pellets recorded using a spectrometer with 200 scans averaged at a resolution of 1 cm⁻¹. The FTIR spectra of the SWCNT and MWCNT are shown in Figure 5. The characteristic response of FTIR spectra for SWCNT and MWCNT shows peaks C=C, which are considered as a result of carbon groups. Carbon nanotubes' C=C stretching mode is more likely to be responsible for the absorption band at 1584 cm⁻¹, 1678cm⁻¹. Based on this result and the wave numbers it can be concluded that MWCNTs a strong and broad stretch peak at 3480 cm⁻¹ and a bending peak at 1584 cm⁻¹ are assigned the C=C bond[23].
Figure 5. FTIR spectra of SWCNT and MWCNT

3.2. Kinematic viscosity with the temperature at different concentrations

The lubricating mechanism relies heavily on kinematic viscosity. At temperatures of 40 °C and 100 °C, the effect of various concentrations on the viscosity of nano lubricant is calculated using SWCNT and MWCNT (shown in Tables 2 and 3). The result showed that enhancing the concentration of carbon nanotubes increased the kinematic viscosity of nano lubricants. At less concentration, though, the rate of viscosity shifts is much lower than at higher concentrations.

Table 2. Viscosity change of engine oil with additive SWCNT at different concentration with temperature

| Property | Engine Oil (SAE10W40) | SAE10W40 + SWCNT (0.1%) | SAE10W40 + SWCNT (0.5%) | SAE10W40 + SWCNT (1%) | SAE10W40 + SWCNT (2%) |
|----------|-----------------------|-------------------------|-------------------------|------------------------|------------------------|
| Viscosity at 40°C, cSt | 83.5 | 98.2 | 105.9 | 139.2 | 180.6 |
| Viscosity at 100°C, cSt | 13.0 | 18.4 | 23.6 | 67.3 | 73.0 |
| Viscosity index | 155 | 164 | 179 | 186 | 198 |

Table 3. Viscosity changes of engine oil with additive MWCNT at different concentration with temperature

| Property | Engine Oil (SAE10W40) | SAE10W40 + MWCNT (0.1%) | SAE10W40 + MWCNT (0.5%) | SAE10W40 + MWCNT (1%) | SAE10W40 + MWCNT (2%) |
|----------|-----------------------|-------------------------|-------------------------|------------------------|------------------------|
| Viscosity at 40°C, cSt | 83.5 | 101.1 | 112.9 | 147.2 | 195.6 |
| Viscosity at 100°C, cSt | 13.0 | 22.4 | 34.1 | 73.3 | 87.0 |
| Viscosity index | 155 | 168 | 182 | 190 | 112 |

3.3. Scanning electron microscope (SEM)

SEM (scanning electron microscope) is used to examine surfaces of materials by scanning a beam of directed low-energy electrons over the material on a regular basis. The behaviour of the electron
beam allows high-energy backscattered electrons to be released, and low-energy secondary electrons from the specimen's surface are captured with a detector. Figure 6 shows images of SWCNT and MWCNT captured with a SEM at a magnification scale of 500 nm. It shows that the SWCNT has a smooth surface, whereas MWCNT has bundles of tangled tubes due to the multiwall structure.

Figure 6. SEM image of (a) SWCNT and (b) MWCNT

3.4. Four ball tribotester

Figure 7 shows the four ball tribotester that were utilised in this study to analyse the tribological characteristics of nano lubricants. There are four balls in this device: one on top and three on the bottom. The lubricant sample to be analysed is held in a ball pot with the three bottom balls. The one ball on top, which is fastened on a collet, rubs against the below balls. Each ball's surface is cleansed with acetone. The upper ball spins at appropriate rpm. As the three lower balls touched the fourth upper ball, the load was divided over three spots. The temperature of the lubricant-filled pot (oil cup) rises to 75 degrees Celsius. The drive motor was then switched on and set at 1200 rpm to drive the top ball. The rotating spindle creates frictional torque, resulting in a scar on the bottom pair of balls. The heater was then turned off for an hour, and the oil cup assembly was taken out from the cup. The test balls had a diameter of 12.8 mm and a roughness of Ra = 0.017 mm and were constructed of AISI E 52100 steel. The variation in temperature of a liquid sample and the friction torque between the balls were both measured. The wear scar diameter was used to measure the wear on the steel ball surfaces (WSD). An optical microscope is used to measure the sizes of the wear scars on the lower stationary balls.

Figure 7. Fourballtribotester
3.5 Testing of tribological properties and surface analysis

The diameters of worn scars are used to evaluate the anti-wear capabilities. Recalculating the worn scar diameter of the three spheres yielded the average WSD for each lubrication test. After adding SWCT and MWCNTs to SAE10W40 engine oil, Figure 8 illustrates the worn surfaces. The worn scar diameters of SWCNT and MWCNT engine oil-lubricated specimens are smaller than those of engine oil without CNTs. With the addition of SWCNTs and MWCNTs in the engine oil as additives, the worn scar diameter is decreased by 38 percent and 67 percent, respectively, when compared to commercial engine oil without SWCNT and MWCNT. The inability of the tribochemical reaction film to form on the rubbing surfaces may be due to the higher shear strength of the adsorbed oil on the balls’ surface. As a result, the rolling effect of the SWCNT and MWCNT nanoparticles may be to contribute the smaller worn scar diameter.

Figure 8. SWCNT and MWCNT in Engine oil SAE10W40 at various volume concentrations as a function of wear scar diameter.

During tribological experiments, the friction coefficient was calculated based on friction torque measurements. The effect of SWCNT and MWCNT concentrations on the friction coefficient of SAE10W40 engine oil is shown in Figure 6. As can be seen, adding SWCNT and MWCNT to engine oil SAE10W40 changes the friction coefficient when applied to commercial engine oil SAE10W40. The friction coefficient of CNTs is smaller than the friction coefficient of engine oil without additive. Furthermore, for MWCNTs, the average friction coefficient is reduced by 56 %, while with SWCNTs, it is reduced by 48 %; this may be due to the effect of viscosity. Higher viscosity fuel oils, such as SAE10W40, have a lower friction coefficient.

Figure 9. The Coefficient of Friction variations with SWCNT and MWCNT in Engine oil SAE10W40 at different volume concentrations.
Figures 10 and 11 illustrates the coefficient of friction and wear scar diameter for nano lubricants and engine oil at optimal concentrations as a result of applied loads. With the addition of SWCNT and MWCNT to the engine oil, the friction coefficients and wear scar diameter were significantly decreased. It has been observed that adding MWCNT to engine oil produces better results than using SWCNT at the same concentration.

**Figure 10.** The wear scar diameter variations with load applied containing Engine Oil SAE10W40, 1 wt. % SWCNT and 1 wt. %MWCNT.

**Figure 11.** The Coefficient of Friction variations with the load applied containing Engine Oil SAE10W40, 1 wt. % SWCNT and MWCNT.
Figure 12. SEM analysis of the wear scar of steel balls lubricated by (a) Engine Oil SAE10W40 (b) 1wt. % SWCNTs Engine Oil SAE10W40 (c) 1wt. % MWCNTs Engine Oil SAE10W40.

The specimens' worn surfaces were lubricated with base oils comprising SWCNTs and MWCNTs nanoparticles were analyzed using SEM analysis, as seen in Fig 9. The MWCNT Engine oil SAE10W40-lubricated wear scar track on the ball has a larger, smoother surface than the SWCNT Engine oil SAE10W40-lubricated wear scar track. Engine oil SAE10W40 without any additive, on the other hand, indicates roughening on the wear surface of the ball, with narrow and deeper grooves, as shown in Fig 9. A lubricating film may have formed due to the existence of carbon nanotubes, avoiding or reducing direct steel-to-steel contact that results in a smoother surface.

4. Conclusion

The four-ball tribotester was used to find the nano lubricant properties of Engine oil SAE10W40 containing SWCNTs and MWCNTs. The tests show that the addition of MWCNTs and SWCNTs to the Engine oil SAE10W40 improved the lubrication properties of engine oil without additives.

- The kinematic viscosity of nano lubricants had increased by increasing the concentration of CNTs.
- These results showed that using MWCNTs as additives in the engine oil decreased the worn scar diameter by 67%. With SWCNTs as an additive, the worn scar diameter is reduced by 38% in comparison to the industrial engine oil without SWCNT and MWCNT.
- The average friction coefficient with MWCNTs is decreased by 56%, and with SWCNTs is decreased by 48%; this may be due to the effect of viscosity.
- The observations prove that by adding MWCNTs has enhanced the anti-wear friction coefficient of Engine oil SAE10W40 compared to SWCNTs when both are taken at the same concentration, i.e., 1 %w/v. These experimental results revealed that on adding MWCNT nanoparticles, wear has been reduced and resulted in a smoother surface with less scars, implying that the MWCNTs greatly decreased metal contact.
- The anti-wear mechanism is because of accumulation of MWCNTs and SWCNTs on the surface, that reduces shearing stress and improves tribological properties.

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