STUDY OF RHEOLOGICAL AND TRIBOLOGICAL PROPERTIES OF METAL OXIDE NANOPARTICLES BLENDED LUBRICATING OIL

A thesis submitted to the
University of Petroleum and Energy Studies

For the Award of
Doctor of Philosophy
in
Mechanical Engineering

By
Harsh Gupta

June 2021

Supervisors
Dr. Gagan Anand
Dr. Santosh Kumar Rai

UPES
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Department of Mechanical Engineering
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Dehradun-248007, Uttarakhand

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DECLARATION

I hereby declare that my research work entitled **"Study of Rheological and Tribological Properties of Metal Oxide Nanoparticles Blended Lubricating Oil",** under the guidance of **Dr. Gagan Anand,** Sr. Associate Professor of Applied Sciences Deptt., University of Petroleum & Energy Studies and **Dr. Santosh Kumar Rai,** Scientist-E, Wadia Institute of Himalayan Geology, Dehradun, for the award of Doctor of Philosophy from University of Petroleum and Energy Studies, Dehradun is my own original work and has not been submitted for any assessment or degree/diploma or award at the University of Petroleum and Energy Studies or any other University/Institutions.

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Harsh Gupta

SAP ID: 500048422

Date: 14.06.21

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THESIS COMPLETION CERTIFICATE

This is to certify that the thesis entitled, "**Study of Rheological and Tribological Properties of metal oxide nanoparticles blended lubricating oil**" has been submitted by **Harsh Gupta** (SAP ID: 500048422) in partial completion of the requirements for the award of the degree of Doctor of Philosophy (Engineering) of the University of Petroleum & Energy Studies and is carried out by him under my supervision and guidance. He has carried out the work at the Mechanical Engineering Department, University of Petroleum & Energy Studies.

It is certified that this thesis has not been submitted to any other university for the award of any other diploma or degree.

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ABSTRACT

Nanoparticles or nanomaterials are the bridge between macroparticles and atoms. Nanoparticle diameter particle size ranges between 1 and 100 nm. Nanolubricants are composed by the dispersion of nanoparticles in a base oil or lubricant. Nanolubricants are nano fluids, i.e. colloidal nanoparticle suspension, meant specifically for machine lubrication of the engine. Nanolubricants endorse a significant reduction of friction and wear, which exhibit remarkable tribological properties. [16, 90]

Characterization and rheological studies of five nanoparticles are addressed in this study. Five different nanolubricant samples were identified using XRD and Photoluminescence Spectroscopy, and the rheological behaviour of these nanolubricants was investigated. The nanolubricants Newtonian behaviour was validated by the shear stress and shear rate data. The Newtonian behaviour of these five nanolubricants was further validated by the shear rate's independence from viscosity. Viscosity was unaffected by shear rate or the type of nanolubricant utilised, as was the case in the majority of previous investigations in this field. Shear viscosity was also found to be temperature-dependent. The results revealed a nonlinear decrease in shear viscosity as the temperature rose. This nanolubricant's nonlinear behaviour opens up a plethora of high-temperature uses in the automobile industry.

Particle size analyser, photoluminescence, and UV visible spectroscopy were used to characterise CuO nanoparticles. Using photoluminescence, the maximum emission of CuO nanoparticles was found to be between 455 and 475 nm. UV-Vis Spectroscopy, on the other hand, shows maximal absorbance at 430 nm with a band gap energy of 2.88 eV. Two of the four surfactants tested exhibited little to no increase in nanoparticle dispersion in oil after the testing. Gum arabic coating of CuO nanoparticles resulted in a considerable increase in dispersion. The samples were sonicated for 90 minutes, then vibrated for 30 minutes before being set aside for two days. The sedimentation method was used to determine the stability. In comparison to Engine Oil with a rise in temperature, all CuO - Engine Oil blends demonstrated a larger decrease in shear stress and viscosity. With the biggest reduction in viscosity, shear rate, and torque, the 1.0 wt percent CuO in Engine Oil mixture appears to exhibit the most promising outcomes. CuO nanoparticles in semi-synthetic engine oil shown better capability in reducing friction and wear, with the coefficient of friction lowered upto 75-90 percent. On the pin-on-disc machine, this reduction ranged from 20N to 100N loads. With the drop of the friction coefficient, the wear would also decline. An XRD study conducted using Debye Scherrer's equation assisted in determining the sizes of coated and non-coated CuO nanoparticles. The crystalline particles had an average size of 15.3 nm and a non-coated nanoparticle had a size of 18.21 nm. The dispersion in the lubricant is indicated by these data. The FTIR data demonstrates how coated and non-coated CuO nanoparticles function. Gum arabic was found in plenty in the coated combination, according to the report. The presence of enough gum arabic resulted in higher degrees of dispersion, as evidenced by the 6:1 ratio in this case. The wear on the object's surface grows when higher loads are added. By incorporating a 1% CuO nanosized particle addition into the base lubricant, the wear has been significantly decreased. CuO particles have a high level of solubility and stability in a given lubricant. CuO nanoparticles are therefore fully qualified for use as a multipurpose additive.

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Last but not the least I would like to thank each one who has been instrumental in the successful completion of this thesis in one way or another. My apology for those if I missed to mention the names of anyone.

Harsh Gupta

Dedicated to my parents

Prof. R. L. Gupta and Mrs. Chandrakanta Gupta

And the loving memories of my Jijaji

Late Mr. V.K. Singhal

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LIST OF SYMBOLS

Symbol	**Name**	**Unit**
^{o}	Degree	-
θ	Theta	-
π	Pie	-
C	Celsius	-
cSt	Centistokes	-
F_n	Normal Applied load	N
g	Gram	-
nm	Nanometre	-
mm	Millimetre	-
mL	Millilitre	-
cm	Centimetre	-
m	metre	-
km	Kilometre	-
s	Second	-
h	Hour	-
L	Litre	-
L	Sliding distance	m
r	Radius	mm
V	Volume loss	mm^3
W_s	Specific wear rate	mm^3/Nm

LIST OF ABBREVIATIONS

Al_2O_3	Aluminium oxide
Ag	Silver
Au	Gold
AFM	Atomic force microscopy
ASTM	American Society for Testing and Materials
COF	Coefficient of friction
Cu	Copper
CuO	Copper oxide
Fe	Iron
Fe_3O_4	Iron oxide
FTIR	Fourier Transform Infrared Spectroscopy
PP	Pour point
MO	Mineral oil
NP	New process
POD	Pin on disc
PM	Polarized microscopy
PL	Photoluminescence
PPM	Parts per million
RPM	Revolution per minute
SAE	Society of automotive engineers
SEM	Scanning electron microscope
SnO_2	Tin oxide
TiC	Titanium carbide
TiO_2	Titanium dioxide
TP	Traditional process
UV-VIS	Ultraviolet Visible Spectroscopy
VM	Viscosity modifiers
Wt	Weight
XPS	X-ray photoelectron spectroscopy

XRD	X-Ray Diffraction
WSD	Wear Scar Diameter
ZDDP	Zinc dialkyldithiophosphate
ZnO	Zinc oxide

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CHAPTER 1

INTRODUCTION

1.1. Study Background

Engine oil is considered to be the soul of an engine. In the absence of it, the whole machinery will turn out to be scrap. In today’s modern world the engines are generally made to operate at higher RPM to produce more power and torque as compared to engines of old technology in order to get better fuel efficiency. Engines are now developed to meet the demands of the modern world. Because contemporary design principles generally meet their viability for engine oils to adhere to the needs of sophisticated engine design, today's vehicle engines cannot run on just any oil [1, 2].

Petroleum products are largely refined and mixed with various additives to obtain the selected engine oil with optimum properties for specific service conditions. Motor oil is fundamentally delegated diesel motor oil which is, for the most part, set apart as "C" refers compression ignition and petroleum motor oil "S" refers spark ignition. The significant distinction between the two classes is the additives and the diffusion property of the motor oil. To the diesel motor, the additive property is of paramount significance and keeping in mind that in the gasoline engine the dispersive property is of considerable significance. The initial function of engine oil is to lubricate the engine parts such as lubricating body fluids such as the human ankle, knee, and the most complex part of the body, such as the spine. The fluids in the human body lubricate the bones, allowing them to fit and perform properly while preventing deterioration. As a result, lubrication was there at the start of production and hence is not a product of the industrial revolution [3, 4].

Due to certain limitations of petroleum products, recently, synthetic base oil captured the automotive market owing to their improved properties such as stability at higher temperature, suitable film strength and viscosity index.

These features are beneficial for existing engines that operate at high rpm and temperature [5-8].

Motor oil performs four fundamental capacities that help the engine run easily, work proficiently, and offer a long service life. A portion of these capacities are properties of the actual oil and keeping in mind that different capacities are principally identified with added substance bundles [9, 10].

1.2. Basics of lubrication

Lubrication – The most important property of engine oil is its viscosity; its resistance to rapid squeezing from between two surfaces is pressed simultaneously with respect to each other. Examples would be the piston-rod bearing on the piston ring surface or crankshaft against the cylinder. Engine oil provides a thin but crucial oil coating that facilitates the action of surfaces in close contact with minimum wear by resisting fast squeezing. It is called "hydrodynamic lubrication" when the viscosity is high enough to keep the two surfaces apart while preventing the maximum wear. It is illustratively dubbed "border lubrication" when the viscosity of the oil is insufficient to keep the two surfaces sufficiently distinct to prevent wear [11-13].

Cleaning – Within a motor, pollution from soot, ignition gases, and flash airborne particles that make it past the air filter creates an unforgiving environment. These materials will induce storage and sludge formation without proper preventative measures, making a motor run hotter and restricting oil flow, resulting in increased wear or possibly engine breakage. As a result, motor oils aid in the capture of pollutants, the prevention of storage, and the suspension of particles until they are removed by the channel or an oil change [14, 15].

Cooling – During operation, several elements of an internal combustion engine overheat. Piston rings, cylinder heads near exhaust valves, and turbocharger bearings, to mention a few, may reach temperatures of above 600 °C. The surplus heat is absorbed by the motor oil, which then distributes it to various parts of the motor, where it is transported via the cooling system. External air, which bypasses the oil pan, and, on rare occasions, an oil cooler, help to cool the oil [16, 17].

Protection – Engine oil protects engine components from corrosion by neutralizing acids formed in the engine as a result of combustion pollutants and water condensation. Condensation is particularly important on short trips and, unexpectedly, in cold weather when the motor does not run long enough at maximum speed to disperse all of the moisture. Consequently, the oil should likewise be designed, in order to anything burn up in the ignition chamber does not deliver pollutants. As the toxins could harm the vehicle's exhaust system or other expensive outflow control parts [18, 19].

Hydraulics – Motor oil is used as a pressure-driven liquid in contemporary engines to operate with features such as variable cam timing, uprooting, compression ratio, and lifter-controlled chamber deactivation. To achieve these capabilities, motor oil is used to drive a pin to a lock/open position [20, 21].

1.3. Introduction to Conventional Lubricant

SAE Viscosity Grade

The viscosity of oil determines how well it flows at a particular temperature. Engine oils are classified by viscosity grade, often known as oil weight, according to the Society of Automotive Engineers (SAE International) classification system. The viscosity grades are provided based on the oil flow rate at standard temperature through the specified orifice. The more it takes, the higher is the viscosity based on SAE grade. These grades are defined by numbers, the lower the number the lower is viscosity that flows more easily which indicates thinner oil. 0, 5, 10, 15, 20, 30, 40, and 50 are some of the common ratings having applications to automobile engines. Low viscosity grades are added to their numbers to make it simpler to start the engine in cold winter weather: 0W, 5W, 10W, 15W, and 20W. It's worth mentioning that 20-weight oil is available in two different formulas: "W" and "high-temperature."

Two digits make up the viscosity rating for multi-grade motor oils. After the first number, there is a "W" that defines how the oil performs at cold temperatures; accurate temperature changes with viscosity grade 2. The second figure indicates how the oil performs at 100 degrees Celsius (212 degrees Fahrenheit). In cold temperatures, for example, the SAE 10W-30 oil flows as freely as the 10W oil directly, but at warmer temperatures, it retains the same viscosity as the 30-weight oil directly.

Multi-viscosity oils are created by combining base stock with pore point depressants, which let the oil flow more freely in cold temperatures, and viscosity index correctors, which prevent the oil from thinning as the temperature rises [22-24].

Conventional Mineral Base Oil

Mineral oils (MO) are hydrocarbons and inorganic substances made from crude oil. These hydrocarbons range in size from C5 to C200, and the basis stocks are made from raw petroleum fractions collected during vacuum refining. Without refining, vacuum distilled fractions are not appropriate as foundation stock. Solvent extraction, dewaxing, and hydrocracking are some of the other purifying methods. The end product is made up of additional hydrocarbons based on the API group of the source stock [25, 26].

1.4. Role of additives

Engine oil contains chemical additive packages in addition to the basic stock to improve performance in a variety of areas. Over time, these additional compounds get depleted as they fulfill their functions. Additive material depletion, in addition to thermal and oxidative degradation of the base oil, is a major element in determining oil replacement intervals. The following descriptions summarize the most common types of additives:

1.4.1. Antioxidants

In the combustion chamber of engine oil, oxygen and heat are the main sources of oxidation of hydrocarbons. Oxidation reaction rate can be increased if needed with the help of transition metals which can be like iron, nickel, copper, and many others. Conditions inside an internal combustion engine establish an ideal climate for oxidation with metal motor parts going about as oxidation catalysts.

Oil deterioration is accelerated by oxidation, which reduces the oil's usefulness, shortens its service life, and damages the machinery that the oil is supposed to lubricate. Oxidation prevention agents are important engine oil additives that help to enhance the base oil's oxidative blockage [27-29].

Common antioxidants include:

- Compounds containing Sulphur and nitrogen
- Sulphur compounds
- Compounds of Phosphorus
- Amino Acids Aromatic
- Compounds of copper
- Boron Compounds are a kind of boron that may be found in

1.4.2. Anti-Wear/Extreme Pressure Components

Zinc dialkyldithiophosphates (ZDDPs) have been utilized as a multi-purpose addition in motor oil for more than 50 years. ZDDPs are generally employed as an anti-wear addition, but they also protect the base oil from oxidation and erosion. ZDDPs have a light EP feel to them as well.

ZDDPs react with surface asperities (roughness) to minimize metal-on-metal contact as an anti-wear agent. ZDDPs react with the entire metal surface when applied pressures are high enough to compress the thin layer on the surface, reducing wear. In a broad sense, the anti-wear film is made up of ZDDP degradation products, with temperature having a direct impact on the thickness and composition of the products. The ZDDP film's nature has been widely examined, but no research gives a succinct description of the film's behavior in automotive lubrication regimes [30-32].

1.4.3. Detergents

One of the most common side effects of oxidation is the creation of sludge and other products that, owing to polarity differences, tend to separate from the bulk lubricant. These detergent additives work by suspending polar oxidation products and neutralizing acids produced by oxidation and combustion.

Detergents reduce corrosion, rust, and sludge accumulation in engines due to their dual purpose. Detergents feature a metal-containing polar (hydrophilic) head and a hydrocarbon (hydrophobic) tail to provide high solubility within the bulk lubricant [4, 33, 34].

1.4.4. Dispersants

Dispersants function in tandem with detergents to keep polar oxidation products suspended. One of the most significant distinctions between detergents and dispersants is their relative basicity. Metal salts of organic acids with excessive basicity, such as metal hydroxides and metal carbonates, are detergents. Because dispersants lack fundamental properties, they have little or no acid-neutralizing capacity. Dispersants, on the other hand, have a larger molecular weight than the detergent's hydrophobic part. As a result, dispersants are more effective in cleaning the engine's interior environment.

The oxidative breakdown of the components that make up the bulk lubricant produces a slew of unwanted compounds. Fuel combustion, in addition to the oxidation mechanism already outlined, is a significant source of lubricant oxidation. Fuel breakdown products, which are potent oxidizers, travel through the piston rings and into the lubricant, causing blowby. These oxidizers attack the hydrocarbon component of the lubricant. Resin, varnish, and sludge are formed as a result of the process. The particular formation is determined by the deposit's composition as well as the temperature of the engine component where the deposit is forming. Dispersants prevent deposits from forming by suspending them in a micelle-like structure. The structure of the dispersant, which is characterized by oxygen- or nitrogen-based polar head and a large non-polar tail, allows for this. While the polar group links with the deposit, the non-polar component keeps the micelle floating in the lubricant [35, 36].

1.4.5. Viscosity Modifiers

To increase the viscosity index, viscosity modifiers (VMs) are added to the basic oil. In the 1960s, this sort of addition permitted the creation of multi-grade engine oils. VMs allow the engine to start at low temperatures while also providing enough viscosity to protect the engine from wear at higher temperatures. The viscosity of most fluids decreases as the temperature rises. VMs function by interacting with the base oil in a temperature-dependent manner. At low temperatures, there is less interaction with the oil; at higher degrees, there is more contact. The contact increases the VM's effective volume fraction, which increases the lubricant's viscosity. The VM is added to a low-viscosity base stock to create a multi-grade oil [37, 38].

1.4.6. Pour Point Depressants

At lower temperatures, paraffinic components in a base oil are prone to wax production. The waxes may clump together and prevent the lubrication from passing through. The pour point temperature is used to assess a lubricant's capacity to flow at low temperatures. Pour point (PP) depressants function by preventing the lubricant's waxes from adhering to one another [39, 40].

1.4.7. Foam Inhibitors

The oil that has been whipped into a froth or foam by rotating engine components has fewer lubricating and cooling properties. Foams in engine oil can cause oil pressure loss and cavitation. When additive packages are added to base oil, foam stability increases. Foam inhibitors function by lowering surface tension at the air-lubricant contact, making it easier for foam bubbles to break. Foam inhibitors are typically used in extremely little dosages (below 20 ppm) [10, 41].

1.5. Role of Nanoparticles

Heat removal and control is a crucial difficulty for every technology that works with high power and small compact. Many scientists from all around the world are interested in using nanofluids to resolve these problems. A nanofluid can be manufactured to order for a given application and utilised as a flexible cooling solution that adapts to the needs of a system in a number of scenarios. Nanofluids could be the world's first intelligent and adaptable coolant.

Nanofluids are the nanoparticles having better stability in a base fluid with a variety of characteristics. Their particular characteristics provide unrivalled potential for an extensive variety of applications. These nanofluids have several present and future uses in sectors such as nuclear reactors, transportation, electrical energy, mechanical, magnetic, solar absorption, and biological domains. CuO, Al_2O_3, SiC, TiO_2, TiC, Ag, Au, Cu, and Fe nanoparticles are among the many additional particle materials utilized in nanofluid production. In nanofluid research, they are often employed [31, 34, 42,43].

Nanoparticles (particles with a diameter of a few nanometers) may now be produced with remarkable simplicity thanks to recent advances in nanotechnology. As a result, it has recently been proposed to suspend these nanoparticles in a base liquid to improve heat conductivity. A nanofluid is defined as a suspension of nanoparticles in a base fluid. Nanoparticles fluidize quickly inside the base fluid due to their tiny size, and as a result, blockage of channels and erosion of channel walls are no longer a concern. When it comes to suspension stability, it's been proven that using the right dispersants helps avoid particle sedimentation [44, 45].

For the past decade, nanoparticles as lubricant additives have been a major focus of research. In thin film lubrication, a variety of metals and metal oxide nanoparticles have been investigated as lubricant additives. Nanoparticle lubricant additives were shown to minimize friction and wear in tribo-surfaces in these investigations. Nano lubricants, like any other colloidal solution, have a suspension stability difficulty. Several scientific researchers have incorporated nanoparticles to lubricating oils to rise extreme pressure, wear, and friction-reducing characteristics, thus increasing and extending the efficiency and service life of the equipment. Furthermore, nanoparticles added to lubricating oil may greatly reduce friction and improve their capacity to bear the load of the components, indicating that they have a lot of promise as lubricant additives. Traditional lubrication technology has been improved and reformatted, thanks to the use of sophisticated nanomaterials. Of course, the concentration of nanoparticles in a lubricant is a critical factor. To enhance friction and wear behaviour, a low concentration of nanoparticles is sufficient:

For a variety of systems, a concentration of less than 2 wt percent was determined to be optimal [28, 46].

The addition of copper oxide (CuO) nanoparticles to engine oil and their friction and wear characteristics were studied. CuO nanoparticles in engine oil were used to make samples with various percentages of CuO nanoparticles (0.2, 0.5, 0.75, and 1 wt. percent). When associated to ordinary engine oil deprived of CuO nanoparticles, CuO nanoparticles blended to engine oil demonstrated better friction wear declining capabilities, as well as reducing the COF by 24 percent and 53 percent at 0.5wt percent concentrations, respectively. They reported that adding CuO nanoparticles to base oil at a low concentration increases the lubricant's friction-reduction properties. Base oil containing CuO nanoparticles has better tribological qualities than base oil without nanoparticles concerning load bearing ability, wear, and friction reduction possibilities. The results revealed that a concentration of 0.5wt percent was the best [47, 48].

It is reported that CuO nanoparticles used as additives in lubricating oils demonstrate superior friction reducing as well as an anti wear action [87]. CuO nanoparticles mixed in base of synthetic engine oil, offered better results in reduction of friction coefficient and suggest that it is a great low friction additive [88]. It has been also shown that CuO nanoparticles offer better efficiency during heat transfer [89].

1.6. Outline of the thesis

The present study includes six chapters which are stated in this section for the purpose to outline the work performed during this investigation.

Chapter 1 describes the main concept prior to undertaking this investigation. It also includes the research gap that has been found in order to address concerns that have not previously been investigated. The goal of the study, as well as the novelty identified throughout the research, are included in the next part. The significance and novelty of this study are also highlighted at the end of this section.

Chapter 2 mentioned the literature survey related to the previous work stated so far. The introduction of nanolubricants is explained in depth throughout the initial phase. Following that, the resources, benefits, and manufacturing procedure for nanolubricants are presented. The advantages of employing nanoparticles in engine oil are described in detail, taking into account their benefits, applications, and global availability. The tribological study that was conducted using metal oxide nanoparticles as an option, as well as the characteristics that were considered throughout the investigation, are also described. Lubricant market demand is also stated at the end of this chapter.

Chapter 3 discussed the objective of this study and what are the shortcomings that are remaining to be addressed are mentioned.

The materials and methods explored before the rheological and friction and wear investigation of metal oxide nanoparticles were discussed in Chapter 4. This chapter went over the specifics of the chemical process that is used to make nanolubricants. The equipment utilised in the characterisation procedure and their rheological investigations are explained in depth, as well as the tribological characterisation of nano lubricants and the procedure of surface analysis.

The result and discussion sections are found in Chapter 5. The tribological analysis was performed in this chapter while taking into account a variety of situations, including variations in sliding speed and weight. The comparison of various metal oxide nanoparticles is also presented with additional rationale, taking into account prior research. For reason, the rheological study of several nano lubricants is also discussed in length. In order to acquire a complete analysis of the surfaces addressed during this study, the measuring methods for the wear scar diameter analysis are also revealed.

The findings of this investigation were presented in Chapter 6. The findings obtained are correctly correlated with the investigation's objectives. This section also includes recommendations for future research that can be used to improve the properties of metal oxide nanoparticles. The articles published based on this study are also involved at the end of the sub-sections.

Chapter 7 includes the references taken during the present study.

1.7. Significance and novelties of the study

The further sections described the details about the significance and novelty involved in this study.

1.7.1. Significance

- Only a few research on nano lubricants' tribological behavior have been published, and no discussion on the effect of loads and concentration has been mentioned in the literature.
- There have been very few studies comparing the lubricity of nano lubricants to that of traditional lubricants. The majority of studies have compared nanoparticles to mineral oil while focusing on a single nanoparticle.
- There is no detailed discussion of the effect of coating CuO based nanoparticles under various conditions in the literature.
- The rheological features of several nano lubricants were not previously compared.

1.7.2. Novelty

In light of the aforementioned concerns, the current study focused on the use of nanoparticles in tribological applications. Nano lubricants were developed, and tribological research was conducted with various loads and concentrations. Rheological experiments of several nanoparticles combined with engine oil are discussed, as well as their comparison.

The Novelty elements in this research work are: (i) Utilization of the different nanoparticles blends for the tribological analysis and their comparison with the engine oil, (ii) Rheological characterization of different metal oxide nanoparticles blended with engine oil, (iii) Selection of the best suitable nano lubricant and its comparison with the engine oil with coating and without coating.

CHAPTER 2

LITERATURE REVIEW

2.1. Introduction

Sadeghinezhad et al. [49] conducted a scientific study on graphene nanofluids. It provides a comprehensive summary of recent graphene nanofluid advancements, with a focus on the various constraints that determine the nanofluids thermal characteristics in various fields. According to the previous studies, it was reported that the graphene-based nanofluids have received more attention than the many other critical aspects of nanofluid stability. More theoretical and experimental work is necessary to advance nanofluid stability. The nanofluids stability for a longer period has only been studied in a few cases.

Stability at high temperatures hasn't been tested as thoroughly. In addition, the outcome of several factors on the rheological and tribological characteristics was analyzed in this study. The results reveal that the concentration of nanoparticles, the effect of their addition, acidity have a major effect on the graphene nanofluid properties.

- Normally, the physicochemical properties of a nanofluid increase linearly with the concentration of nanoparticles.
- The temperature doesn’t have a major outcome on the thermal conductivity of nanofluids.

According to Emad et al., [50] graphene is a capable material for the exchange of heat during the process. However, there are minimum studies were conducted by taking graphene as nanofluids and are needed to be studied further. Many sectors have a pressing demand for better fluids that can efficiently transport heat. Nanofluids are more efficient at transferring heat than traditional fluids. As a result, when this nanofluid is employed to improve the design and the fluid. The various studies were performed and they have different effects according to the conditions considered during the analysis. They also discovered a paucity of knowledge in the areas of nanofluids thermal characteristics and their capability of heat exchange. The long-duration stability study of nanofluids is also critical for both research and industrial usage. The long-term stability of the most investigated nanofluids, as well as the efficacy of thermal management systems, have yet to be validated. The nanofluids have several benefits, including reduced cooling system size, enhanced reliability, lower pumping-power requirements, reduced pollutants, and increased energy and fuel efficiency. As a result, the focus of this study has been on the thermochemical characteristics and graphene nanofluids applications. For a deeper understanding of graphene nanofluids, more research is needed. Nanofluids containing a distinct nanoparticle have received a lot of interest from the authors, and hybrid graphene nanofluids have also been studied. As a result, future research can focus on the application of hybrid nanoparticles and defining the key factors that influence the thermophysical characteristics of graphene nanofluids.

As per Berman, et al., [51] graphene is a new emerging lubricant. A summary of current breakthroughs in the application of graphene from nano-scale to macro-scales is presented. The study was focused to evaluate the tribological properties that presented better results compared to graphene oxide and graphite. In addition, the current investigation on graphene as an additive and for the construction of self-lubricating nano-composite materials is described. The following important facts have been confirmed by these devoted studies:

- Theoretical calculations suggested that graphene's low friction is strongly dependent on the surfaces in contact and the affinity of the graphene film sustainability on the sliding surface.
- Theoretical predictions of reduced friction with the quantity of graphene layers were confirmed by AFM tests. It was demonstrated that when sliding happens without stick-slip, friction can reach the ultra-low friction regime at specified weights.
- The tribological studies performed on a micro-scale analyzed that friction increases with the introduction of graphene having defects in their structure and also the chemical modification of graphene leads to

further increase in the value of friction.

- Micro- and macro-scale tribological studies clearly demonstrated how graphene differs from graphite in relation to minimizing friction and wear of the surface without having any influence on the environment i.e dry or wet.

The monolayer graphene oxide (GO) sheet possibilities as water-based lubricant additives were performed by Kinoshita et al. [52]. The GO dispersion's friction coefficient was around 0.05, and after 60,000 friction cycles, the tungsten carbide (WC) ball and flat plate displayed no visible wear. The GO sheets are thought to act as defensive coverings by adsorbing the lubricated ball and flat plate surfaces. These findings point to the possibility of using GO sheets as a water lubricant to decrease friction and wear on surfaces.

Berman et al. [51] analyzed the tribological features of graphene in a dry nitrogen atmosphere. In a dry nitrogen atmosphere, the 440C steel was used. The findings show that modest concentrations of graphene on the sliding surface can achieve relatively low friction coefficients compared to the enormously great and unhinged friction of steel without graphene.

His findings show that graphene has considerable potential as a solid lubricant for mild stresses and that it may be easily coated on tribological surfaces using SPG-containing graphene particles.

According to Obasi, et al., [53] the outcome of additives on engine oil performance was studied. The additive has a significant impact on engine oil properties such as physical properties such as colour and viscosity; physiochemical properties such as dispersant power, friction and wear reduction, antirust and foaming resistance; and chemical properties such as oxidation and corrosion resistance. The viscosity of a fluid at a specific temperature is measured. The viscosity of the mix at 1000°C increases as the additive content increases, according to the results of the test. These viscosities cannot be overlooked; they must be considered to keep the engine oil mixture within the SAE range of 16.5–17.5.

The graph is practically a curve, illustrating that the rise in the concentration of the additive in the oil mix, the viscosity enhances, until the increase in viscosity dropped at a specific point on the graph, 15.58 cst. It indicates that after this point, no matter how much additive is added to the oil blend, there will be no discernible increase in viscosity.

A substance's density can be defined as its mass per unit volume. Density is critical in engine oil to enable its effective and efficient operation. According to the test, the higher the additive concentration on the oil mixture, the higher the oil density. The density is measured at 150 degrees Celsius, and the ASTM specification for multigrade engine oil density is between 0.886 and 0.906. The density vs. additive concentration figure is linear, indicating that the higher the additive concentration in the oil, the higher the density. It can be seen from the figure that at a density of 0.8870, increasing the additive concentration does not end in a rise in density. The lowest temperature at which a test flame allows the oil vapour to ignite under a specific test circumstance is called the flash point of the engine oil. The safe guard's flash point is simply a numerical value. Increases in additive content raise the oil's flash point, according to the tests. The increment is confirmed by plotting flash point against additive concentration. The graph is a linear graph that shows that the additive content enhancement in the blended oil increases the flash point temperature value. When blending oil with addition, a lot of additives should be avoided to keep the flash point safe.

2.2. Nanoparticles size effect

The foaming phenomenon causes lubrication issues in automotive and aviation engines because the presence of gas (air bubbles) in the oil makes it impossible to detect the true quantity and quality of the oil in the engine, which can lead to engine inefficiencies. The test illustrates the influence of the additive on engine oil foaming properties. As the additive concentration increases, the oil's ability to froth decreases. This is also supported by a graph showing foaming ability vs. additive concentration. The graph is negative, indicating that an increase in additive concentration will progressively kill the foam, regardless of its volume. On the stability of the foam created, the engine oil mix had no foam after 10 minutes of removing the air supply to the diffuser since the sample had returned to its original value. The foam present in the engine oil would have caused an increase in the volume of the test sample.

As a result, it is recommended that additives be added to produce the ideal engine oil qualities for improved engine performance.

A review was done by Rao et al., [54] for nanofluid synthesis, and a variety of particle materials are employed, including Al_2O_3, TiO_2, CuO, TiC, SiC, TiC, Au, Ag, Fe, and Cu nanoparticles. Carbon nanotubes are also used because of their extraordinarily high longitudinal (axial) heat conductivity. Some additives are mixed into the mixture in modest amounts to improve the nanoparticles stability in the fluids.

Nanoparticles employed in nanofluid preparation typically have a diameter of fewer than 100 nanometers. Nanofluid research has utilized particles as tiny as 10 nm. It's worth noting that, as a result of the clustering process, particles can form micrometer-sized clusters that affect the friction of the surface due to the abrasiveness created on the surface.

Spherical particles are most commonly employed in nanofluids. Nanoparticles that are rod-shaped, disk-shaped, and tube-shaped are also employed. The gatherings created by nanoparticles, on the other hand, may exhibit fractal-like shapes.

2.3. Nanoparticles mechanical applications

Nanoparticles have the capability to build a defensive layer with small hardness and elastic modulus on the worn surface. This is one of the primary reasons why some nanoparticles in fluids have good lubricant characteristics. Magnetic fluids are a type of nanofluid that is unique. Magnetic liquid rotary seals use the magnetic properties of nanoparticles in liquid to function in a wide range of applications with not at all preservation and enormously little leakage. Progressive lubricants can boost efficiency by reducing energy consumption and increasing the consistency of engineered systems. The goal of tribological study is to reduce friction and wear. Nanoparticles have sparked a lot of attention in the current years because they have tendency to resist the higher loads and also extreme pressure occurred on the surfaces in contact. On a four-ball machine, Zhou et al. [55] tested the friction and wear characteristics of Copper (Cu) nanoparticles. Cu nanoparticles demonstrated higher friction-reduction and antiwear capabilities as an oil additive than zinc dithiophosphate, particularly at higher loads, according to the findings. Meanwhile, the nanoparticles could significantly boost the base oil's load-carrying ability.

Eswaraiah et al. [56] investigated tribological properties of graphene-based nanofluids added to the engine oil. Probe sonication was used to homogeneously disperse different amounts of graphene in the base oil. The tribological characteristics of nanolubricants vastly enhanced at lower concentrations of around 81 % reduction in friction and 32 % drop in wear scar diameter were attained with the introduction of graphene. This clearly illustrates that graphene-based engine oil creates a full decline in friction without changing the surface. The rise in graphene concentration also causes an increase in COF and wear scar diameter, which can be attributed to particle coalescence and segregation. Nano bearings form between the balls, which helps to reduce wear and friction. The decisive mechanical strength and topological structure of graphene are thus linked to its extraordinary performance on tribological properties of the nanofluids.

Llie and Covaliu [57] performed the tribological characteristics of the lubricant with the introduction of titanium dioxide (TiO_2) as an additive. Following are the conclusions obtained from the study:

- The new approach produced nanoparticles that were introduced to oil and had good dispersion stability. In association to the base oil, the nanoparticle suspensions examined showed reduced friction and wear. Concerning nanoparticles concentration, TiO_2 suspensions made using the New Process (NP) and the Traditional Process (TP) had identical friction and wear behaviour. Such research could be useful in overcoming the challenges of using TiO_2 nanoparticles in lubrication during the cutting process in industries. The obtained results show that the manufactured nanoparticles have 50–100 nm average size and that the nanoparticles surface has been changed from oleophilic to oleophobic. Furthermore, the nanoparticles can be thoroughly disseminated in the engine oil completely beneath the NP, with no discernible influence on the property reference to oxidation.

- In a comparison of TiO_2 nanoparticles application under the conventional process and new process, the findings of tribological investigations demonstrate that TiO_2 nanoparticles beneath the new process have better anti-friction and wear-reducing properties in the conventional oil.
- Polarized microscopy (PM) and X-ray photoelectron spectroscopy (XPS) results show that an uninterrupted opposition film covering layers and tribo-chemical reactions like Ti_2O_3 and Fe_2O_3 forms, resulting in outstanding nanoparticles possibilities in engine oil.
- The primary component of this research's originality is that it addresses the oil-solubility issue by combining the effects of surface modification with lubricating oil's specific mix procedure. This approach was first utilised to make TiO_2 nanoparticle-based lubricants. TiO_2 nanoparticles used as additives in different lubrication sectors like engine oil, and gear oil applications should benefit from it.

CHAPTER 3

OBJECTIVES

There are some inherent drawbacks of engine oils such as short lifetime and non-uniform degradation patterns. Mixing of various nanoparticles has been proposed by the researchers to overcome these limitations.

1. Barriers and challenges have to be examined carefully due to size, shape and temperature of metal oxide nanoparticles (e.g. CuO, ZnO or TiO_2) before this nanofluid (mixture of engine oil - 10W30, 15W30 etc. and metal oxide nanoparticles) can be fully implemented in the industrial applications.
2. Study of particle characterization of metal oxide nanoparticles and stability of nano-lubricant (mixture of engine oil and metal oxide nano-particles) through UV-Vis spectroscopy.
3. Study of the engine oil performance mixed with metal oxide nanoparticles with reference to quantification of various rheological and tribological properties.
4. Study of the physio-chemical properties of the engine oil mixed with metal oxide nanoparticles.

CHAPTER 4

MATERIALS AND METHODS

The brief details of the methodology on the credibility of the different nanolubricants with their rheological and tribological characterization are mentioned in Fig. 4.1. The entire study includes two phases: Phase 1: Rheological characterization of different nanolubricants i.e. TiO_2, SnO_2, CuO, Fe_3O_4, and ZnO. The current study is aimed at the utilization of nanolubricants for the performance improvement of engine oil. The detailed discussions are mentioned in the upcoming sections.

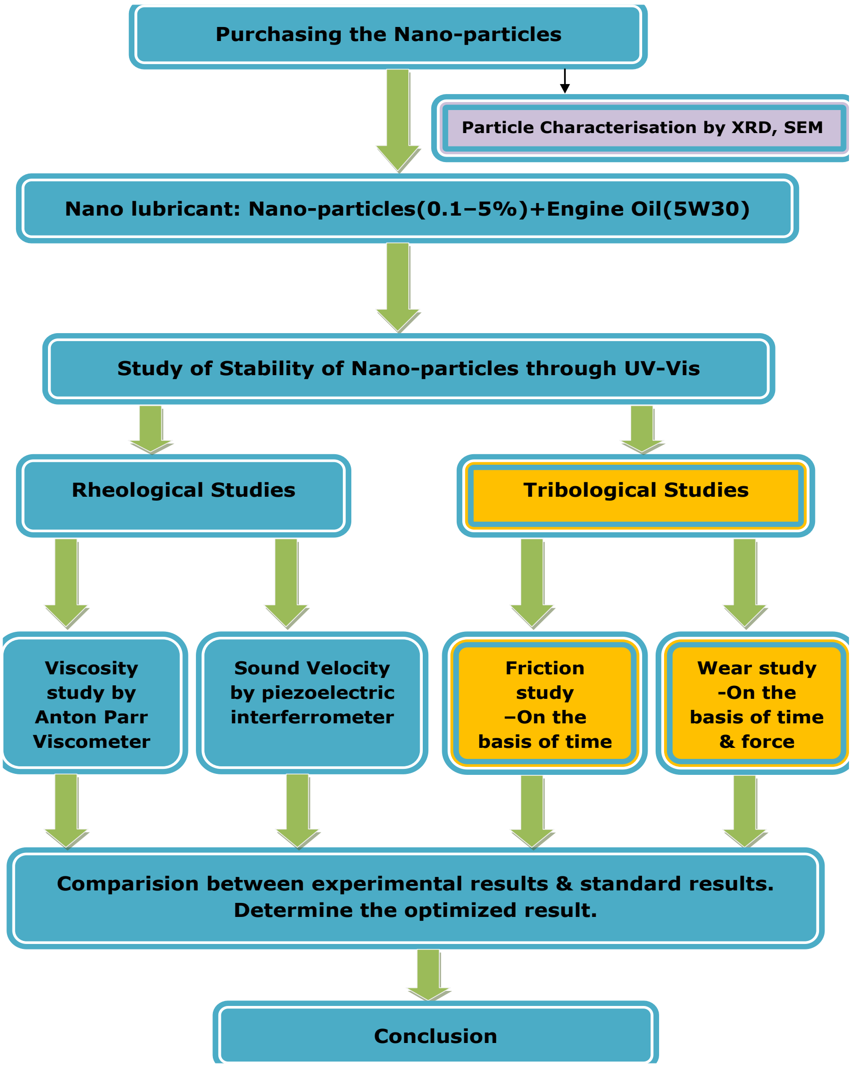


Figure 4.1. Schematic image of the processes included in the analysis.

4.1. Rheological study of various nanolubricants

4.1.1. Sample preparation

In this study, five distinct oxide nanolubricants: TiO_2, SnO_2, CuO, Fe_3O_4, and ZnO were analyzed. The experiment employed nanoparticles that were purchased from Nano Labs, Jamshedpur, and blended with engine oil. These prepared samples have a labeled volume concentration by 1% of weight. The samples utilised in the studies are all labeled as being 99 percent pure. The nanoparticles were magnetically agitated for 30–45 minutes at 200–300 rpm or sonicated for 15–30 minutes in the engine oil of grade 5W30 before any of the characterization or rheology tests. This was done to achieve consistent nanoparticle dispersion in the engine oil.

4.1.2. Viscosity measurement

Rheology is the use of rheometers to study the flow and deformation of materials under applied forces [58]. Rheological qualities can be measured with a rheometer upto nanometer. Rheological qualities can be determined at the bulk scale using a mechanical rheometer, and at the nanoscale with a capillary viscometer. The viscosity of five distinct materials was determined in this experiment using an ANTON PAAR Rheolab QC Rheometer and Rheolab software. The Rheolab QC is a rotational rheometer with a Peltier temperature device for temperature ranges of 0–180^{o}C. The ANTON PAAR rheometer's software was used to record the corresponding shear stress, strain, and dynamic viscosity. Furthermore, the temperature of the measuring cup and the sample was precisely controlled by connecting a Peltier temperature device (C-PTD 180/AIR/QC) or a liquid temperature device (C-LTD 80/QC) to a water bath thermostat. Multiple tests were carried out at various temperatures, including room temperature, 30, 40, 50, 60, 70, and 80^{o}C. For each test, the shear rate was changed from 1 to 1000 (1/s).

4.2. Characterization of the nanoparticles

4.2.1. X-Ray Diffraction

XRD spectroscopy was used to characterise and identify the nanoparticles in all of the samples. X-ray powder diffraction (XRD) is a quick analytical technique that provides information on unit cell dimensions and is commonly used for crystalline material phase identification. The substance under scrutiny has been finely powdered and homogenised. The bulk composition of the samples was determined using XRD. The notion of productive interference of monochromatic X-rays with a crystalline sample underprops XRD. The Bruker EW D8 Advance was utilized to conduct the XRD test for this investigation, and it came with its own software for data analysis. The XRD spectrum for all five nanolubricants was conducted using this device.

4.2.2. FTIR Test

An infrared light source is used to measure the wavelength of an infrared region of a substance in a Fourier Transform Infrared Spectrophotometer (FTIR). A radiation source, a beam splitter, a moveable mirror, a stationary mirror, and a detector make up the FTIR machine. The movable mirror rotates back and forth throughout the experiment, emitting different wavelength patterns.

4.2.3. Particle Size Analysis

The Zetasizer (Malvern Zetasizer Nano) was used to extent the nanoparticle particle size. Using dynamic light scattering, the size of the nanoparticles is assessed (DLS). In a disposable polystyrene cuvette, the nanoparticles were dissolved in water and the system temperature was maintained at 25°C.

4.2.4. Photoluminescence

The study of light emission from particles after photon absorption causes electron excitation to a higher energy state is known as photoluminescence

(PL). When the electron returns to its lower energy state, this results in the emission of a photon.

4.2.5. UV-Vis Spectroscopy

UV- Vis spectroscopy (ultraviolet-visible spectroscopy) is a method for determining the wavelength at which maximal light absorbance occurs in both the ultraviolet and visible spectrum. The PerkinElmer UV-Vis Spectrometer was utilised for this study. Two disposable polystyrene cuvettes were employed, one containing the sample dissolved in distilled water and the other containing only distilled water as a control.

4.2.6. Coating process of nanoparticles

In a test tube with 30 millilitres (ml) of Toluene, 12 gm CuO nanoparticles were coated with 2 gm Gum Arabic (a ratio of 6:1). For 10 minutes, the mixture was swirled until it was completely dissolved. The coated particle was then placed in a sonication machine and sonicated for 15–20 minutes after it was fully dissolved and agitated. This was done to ensure that there were no leftovers of Toluene in the mixture. After the sonication was finished, the mixture was placed in a hot oven to evaporate the remaining Toluene. The mixture was baked for 19 hours at 60°C in a hot oven. After that, the coated nanoparticle was processed for 30 minutes until it was powdered. The coated nanoparticle was then mixed with one litre (L) of 5W30 mineral engine oil and swirled for around 30 minutes on a magnetic stirrer machine. After that, the mixed substance was ready to be put to the test.

4.2.7. Addition of surfactant to CuO nanoparticles

CuO nanoparticles do not disperse properly in engine oil and therefore need a surfactant to increase the degree of dispersion. Surfactants are known to decrease the surface tension between two liquids, a liquid, and a gas or between a liquid and a solid, and therefore coating the CuO nanoparticles with the surfactants will promote dispersion of the CuO nanoparticles in engine oil. Surfactants are competent enough to enhance the stability of the nanofluids as they trim down interfacial tension amid the molecules of engine oil and floated nanosized particles. Surfactants must be capable solubilizers, having lower toxicity, and cost-effective. All these attributes are exhibited by the surfactants namely; SDS, CTAB, SDBS, GA, and ARB [59]. However, most surfactants that are shown to improve dispersion are in powdered form and therefore require a solvent. The surfactant and solvent used in this report are as follows:

- Sodium dodecyl sulfate dissolved in Ethanol [SDS_e]
- Sodium dodecyl sulfate dissolved in Toluene [SDS_t]
- Gum Arabic dissolved in Ethanol [GA_e]
- Gum Arabic dissolved in Toluene [GA_t]

The surfactants were mixed with the solvent in centrifuge tubes and then sonicated for ten minutes. This was to make sure that it is completely dissolved. A small number of nanoparticles of CuO were then mixed into the surfactant-solvent mixture and then sonicated twice for 10 min each to allow for the coating to take place. The centrifuge tubes were then centrifuged for 5 minutes to allow the CuO nanoparticles to settle down to be collected. The liquid was poured out of the test tube carefully keeping the particles inside and then the test tube was put in the oven for 3 hours to dry the CuO nanoparticles. This also made sure that no solvent was left in the CuO nanoparticles which could contribute to the weight of the particles. To find which surfactant allows for maximum dispersion of nanoparticles in base 5W30 engine oil, small amounts of four differently coated CuO particles were then mixed to the 5W30 base oil in separate test tubes. All test tubes were then shaken to mix and then sonicated to cause dispersion. The test tubes were then monitored for a couple of hours to see which combination caused maximum dispersion and to check if the dispersed state was stable enough for experimentation. Figure 4.2 shows the samples of CuO nanolubricants with different surfactants.

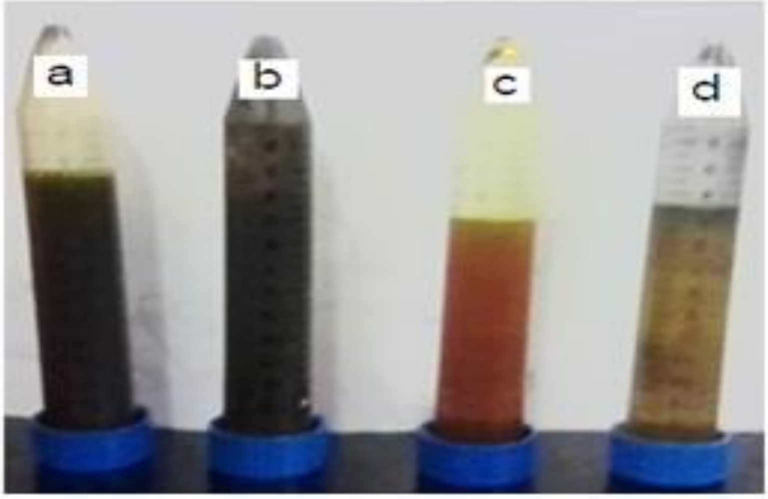


Figure 4.2. Dispersion of (a) GA_t, (b) SDS_e (c) GA_e, (d) SDS_t coated CuO Nanolubricants.

It is evident that CuO nanoparticles coated with Gum Arabic dissolved in Toluene causes the maximum dispersion of Oil followed Sodium dodecyl sulfate (SDS) dissolved in Ethanol. The other two combinations cause very little or no dispersion at all in oil. As Gum Arabic dissolved in Toluene produces the best results, it was chosen as the coating material for the rest of the experiment, i.e. the rheological tests. Another reason was that the Gum Arabic dissolved in toluene is more stable when observed in the long run compared to the other combinations.

4.2.8. CuO nanoparticles based samples development

For the experimentation, samples with precisely 0.5%, 1.0%, 1.5%, and 2.0% of CuO nanoparticles by weight percentage was taken. This required a straightforward calculation to find what the weight of the nanoparticles would be in a 20 ml sample of 5W30 engine oil. Four samples were produced from the nanoparticles and engine oil blend to be associated with the data with standard engine oil. 20 ml of 5W30 Engine Oil was then added to the test tubes using a measuring cylinder. To promote the dispersal of all nanoparticles in engine oil and increase the degree of dispersion, the test tubes were sonicated multiple times for 10 minutes [60]**.** The test tubes were monitored for a couple of hours to see if the dispersion was stable enough for testing and if not, it was sonicated again. Figure 4.3 shows all the prepared samples, the test tubes correspond to 0.5 wt %, 1.0 wt %, 1.5 wt %, and 2.0 wt % CuO nanoparticles by weight in 20 ml of 5W30 Engine Oil.

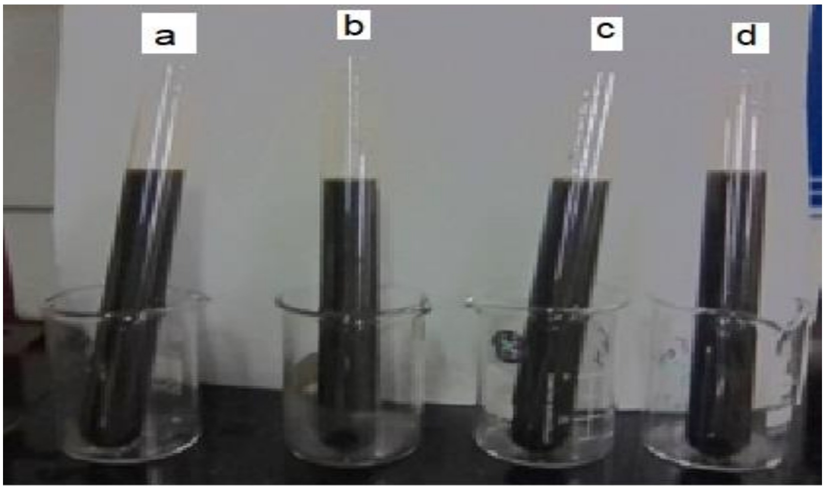


Figure 4.3. (a) 0.5 (b) 1.0 (c) 1.5 and (d) 2 wt% CuO Blended Engine Oil Samples.

4.3. Friction and wear analysis

4.3.1. Pin on disc tribometer

For the Friction and wear characterization of the selected lubricants, DUCOM make Pin-on-Disc Wear was used. The detailed description of the experimental setup is shown in Figure 4.4. The POD machine was connected to the computer equipped with a data acquisition system. It was inbuilt with software for the assessment of the frictional force, coefficient of friction, and wear at different operating conditions. An oil tank with a capacity of 3 litre was connected consisting of a hydraulic pump having a supply mode for the constant supply to the testing surface. With the help of a DC servomotor, the disc was spun around its centre, and the sliding speed was regulated with the controller attached by feeding the desired value into the system. The detailed specifications of the Pin-on-Disc wear tester are mentioned in Table 4.1.

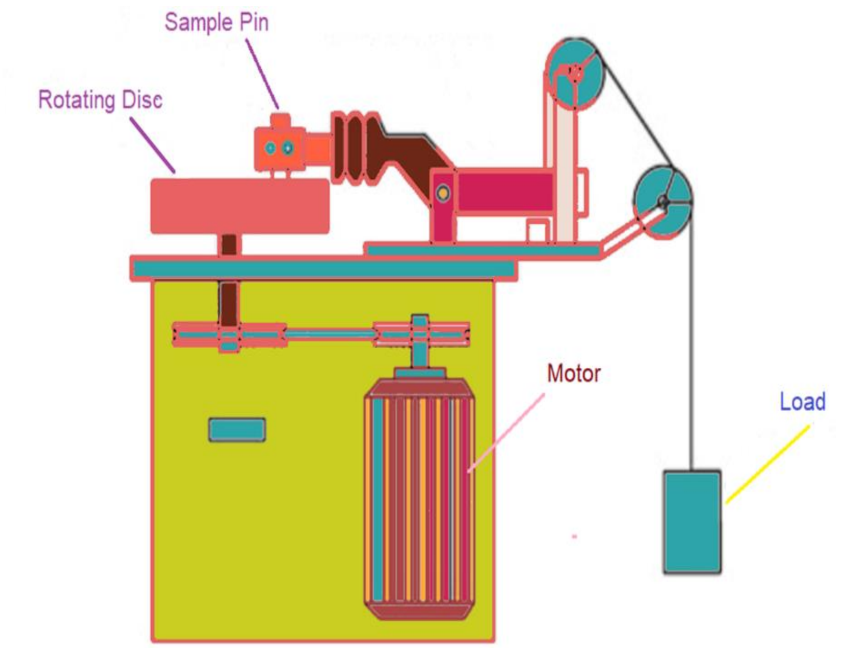


Figure. 4.4. Schematic image of the pin-on-disc experimental setup.

Table 4.1.

Specifications of the POD machine.

Parameters	**Min**	**Max**
Diameter of the pin, mm	3	12
Disc Diameter, mm		165
Rotating speed, rpm	200	2000
Applied load, N	1	200
Frictional force, N	1	200
Wear measurement range, µm	1	1200
Range of temperature, ^{o}C	1	300
Wear track diameter, mm	50	145

4.3.2. Conditions used on POD tribometer

The details are: applied load = 20 to 100 N; temperature = Ambient temperature; sliding speed= 900 rpm; sliding distance= 3000 m. The pin was held stationary during the process of the analysis by rotating the disc at the desired speed by loading the arm with selected loads. The friction track was equipped with sensors for obtaining the friction values. For the proper cleaning and making disc suitable for the test, emery paper with a range from 500 to 1200 µm was used for polishing them before and after conducting every experiment. Before the examination of the surface of the pin, it was cleaned in an ultrasonic bath in the presence of acetone maintaining a temperature of 65°C. Before worn surface characterization, the pin samples after cleaning were placed in an oven to prevent any oxidation on the samples ready for further examination. The pin was weighted using a weighing balance machine having an accuracy of around ±0.001 gm. The temperature was applied with help of an isolated chamber after keeping it on the pin on disc contact surface and the value was fed to the controller equipped with the setup for the possible temperature range.

4.4. Oiling concept

A single fused silica capillary tube consisting of an outer diameter of 370 µm and an inner diameter of 210 µm was used for the supply of the oil to the surfaces in contact. The capillary tube was capable to be bent as per the requirement of the surfaces in contact for proper flow during the process implemented. The bending of the capillaries was maintained at 3 mm from the surfaces in contact so that the proper formation of the lubricant layer can be obtained. The oil flow rate can be varied with a high precision range from 0.001 ml/min to 10 ml/min. Around 5 ml/min flow rate was conserved while conducting each test for determining tribological analysis.

4.5. Experimental procedure

The experiment was conducted on POD tribometer to assess the influence of the various lubricants used for the analysis. The material of the pin was obtained from the piston ring of the engine and it was fixed in the particularly considered pin holder to maintain a sliding contact to the surface of the disc. The dimensions of the pin were 30 mm in length and 8 mm in diameter with a spherical shape from the front side. The disc was obtained from the M/s Agrawal metal works, Roorkee City, Uttarakhand by using the melting and machining process applied for obtaining the desired shape of the disc. Because the pin's component was small, it was inserted into the grooves of another plate with the necessary proportions. On an electric weighing machine (10^{-4} g sensitive scale), the weight of the pin was measured, and wear was plotted based on the difference achieved under working conditions. Specific wear rate was obtained according to the Archard's equation:

$$W_s = \frac{V}{L*F_n} \qquad (4.1)$$

Where, W_s is the specific wear rate (mm^3/Nm), V is the volume loss (mm^3), L is the sliding distance (m) and $\mathrm{F_n}$ is the normal applied load (N).

Volume loss was obtained as per the ASTM standards. The lower part of the pin was round in shape taking a curvature radius of 4 mm.

$$V = \frac{\pi(WSD)^4}{64*r} \qquad (4.2)$$

Where, V is the volume loss in mm^3, WSD is the wear scar diameter in mm and r is the radius of the spherical pin in mm.

After conducting each test at desired operating conditions, optical microscope having 0.01 mm (ASTM D4172) resolution equipped with view 7 software for wear scar diameter characterization. The lower part of the pin was held on the platform of the microscope in a position of facing its scar upward towards the microscope lens. The microscope lens was adjusted and focused to get a clear image of the scar obtained on the surface of the pin. By hitting the capture button after detecting the scar, the image was collected and saved in the system. With the assistance of the software, wear scar diameter was measured in mm range through scale inbuilt to the view 7 software.

CHAPTER 5

RESULTS AND DISCUSSION

5.1. Rheological analysis of various metal oxide nanolubricants

5.1.1. XRD Analysis

XRD was used to validate the nanoparticles characterization using Bruker EW D8 Advance. XRD diffraction pattern demonstrates the single-phase monoclinic structure of the nanoparticles. In the region of 2 theta 20–80^o, X-ray diffraction was detected. With the use of an XRD spectrum, the crystalline nature, crystal phase, and crystallite size of the nanoparticles were investigated. The resulting XRD spectrum of nanoparticles is shown in Figure 5.1 and it was linked to the regular diffraction spectrum for each of the five nanoparticles (JCPDS No.: 88-1175 and 84-1286). The peaks found at (100), (002), (101), (102), (110), (103), (200), (112), and (201) allocated to various crystal planes of ZnO’s hexagonal structure, aligned well with the typical diffraction pattern for the peaks recorded at (100), (002), (101), (102), (110), (103), (200), (112) and (201). Minor peaks were identified, showing the sample's excellent crystallinity. Figure 5.1 shows the Fe_3O_4 nanoparticles diffraction pattern generated under usual conditions, which shows diffraction peaks at (311), (400), (511), (440) and other locations, which are the characteristic peaks of Fe_3O_4 crystal inside a cubic structure. The diffraction pattern for TiO_2 with significant peaks at (101), (004), (200), (105), and (204) were observed, which corresponded to the conventional TiO_2 spectrum as reported by Thamaphat et al. [61], the principal peaks in the diffraction patterns of SnO_2 and CuO also aligned and agreed with the conventional X-ray spectra of these nanoparticles.

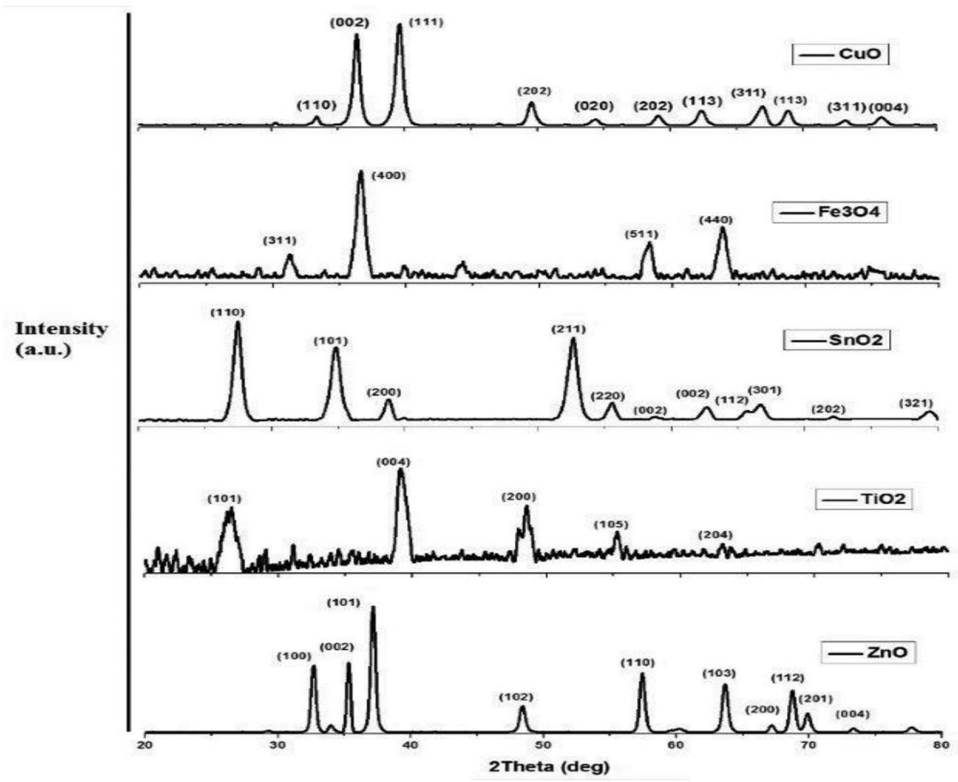


Figure 5.1. XRD spectrum of various nanoparticles.

5.1.2. Stability analysis of various nanolubricants

Figure 5.2 shows the photoluminescence of five oxide nanoparticles at room temperature, exhibiting their emission band. UV and visible emission bands are the two types of emission bands. The visible band spans 400 to 800 nm, while the UV band spans 100 to 400 nm. Figure 5.2 shows the absorption, which is centered on the 490–510 nm emission band. The oxygen vacancies are primarily responsible for the UV peak in polycrystalline oxide nanoparticles [86]. By comparing the excitation peak of the absorption graph, the nanoparticle may be identified. Only the excitation peak from 400 nm is considered in the graph below. The excitation peak of each of the five nanoparticles studied in this work matches the standard spectrum perfectly. The clumping of nanoparticles could be the cause of the slight difference in excitation wavelength.

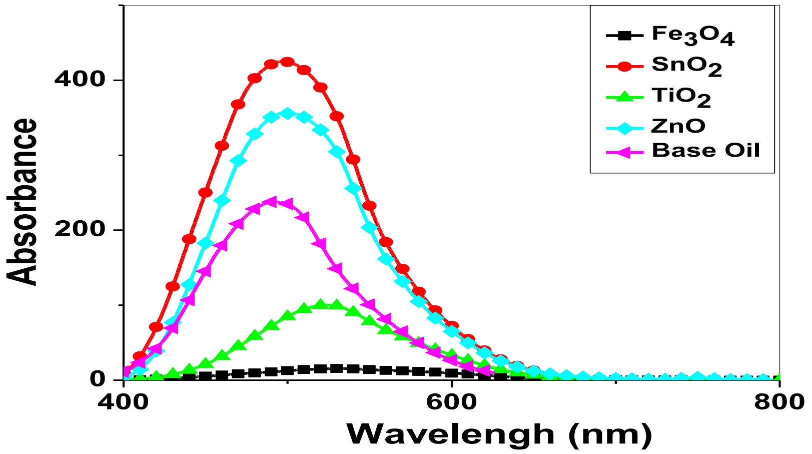


Figure 5.2. PL absorbance spectrum of five different nanolubricants.

5.1.3. Particle size analysis

For all of the samples, Figure 5.3 illustrates the intensity versus size (nm). All of the particles have a size distribution of 20 to 80 nm, as can be seen. As demonstrated in Table 5.1, further data analysis allows us to track the average particle size of each sample. The obtained outcomes are steady with those reported in the previous study [62]. As a result, it verifies that the particle size achieved is on the nano scale.

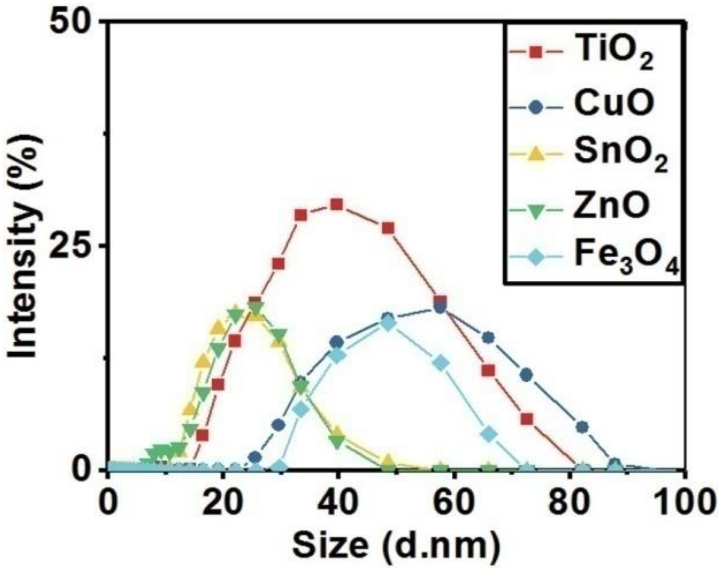


Figure 5.3. Various nanoparticles Size characterization.

Table 5.1.

Average particle size of various nanoparticles.

Composition	Average particle size (nm)
CuO	35
TiO_2	45
SnO_2	46
ZnO	47
Fe_3O_4	61

5.1.4. Rheological behavior of lubricants

The rheological data in Figure 5.4 indicate the effect of shear rate (at fixed temperature) and temperature (at fixed shear rate) on the viscosity of nano-lubricants. The analysis was done to confirm the rheological properties of five distinct nanolubricants, namely SnO_2, TiO_2, Fe_3O_4, CuO, and ZnO. The common linear equation of shear stress and shear strain rate was detected in the data acquired from the rheological tests of these nanolubricants, indicating the Newtonian character of these nanolubricants. The viscosity of these nanolubricants is shown in Figure 5.4 with effect of shear rate. The Newtonian character of these nanolubricants is further confirmed by this relationship. This relationship found in this study fits with Murshed, Santos, and Castro's findings. The usage of silicon oil and a high shear rate could explain why the viscosity appears to be a little greater than the plot above. However, for these nanolubricants, both figures accord with the shear independent viscosity relation. The plot also shows that as the temperature increases, the shear viscosity declines. Hu et al. [16] discovered similar results.

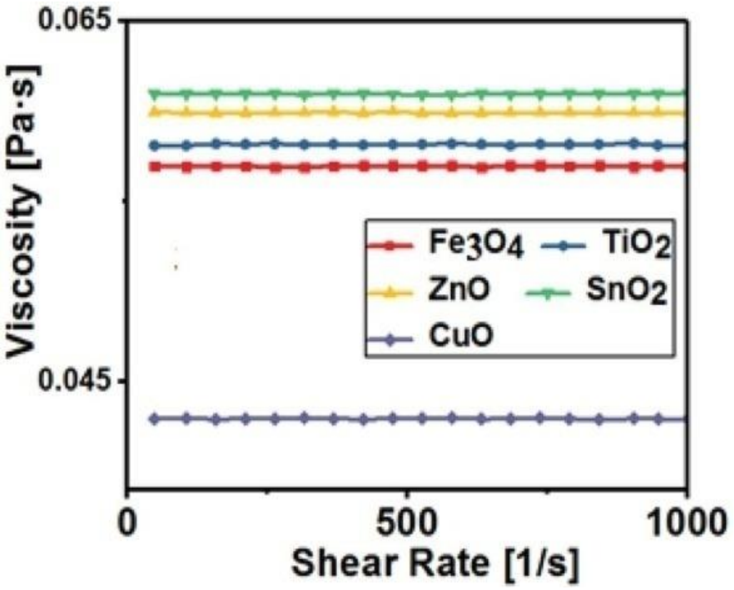


Figure 5.4. Dynamic viscosity versus shear rate of different nanolubricants at constant temperature of 40°C.

Figure 5.5 shows the influence of shear rate on shear viscosity for all five nanolubricants when the temperature was adjusted from 30° to 80°C. The shear viscosity (for all nanolubricants) is free from the shear rate however not of the temperature, as shown in the figure. Temperatures ranged from 30 to 80°C. For each nanoparticle, the experiment was carried out independently. The results for all five nanoparticles support the pattern of a significant drop in shear viscosity with increasing temperature (with 30°C indicating the highest viscosity and 80°C indicating the lowest viscosity). This result is in line with the findings of Sundar et al. [63]. The explanation for the drop in viscosity is that the molecules behaviour has changed. The interatomic/ intermolecular distance increases as the temperature rises, resulting in a decrease in viscosity [64].

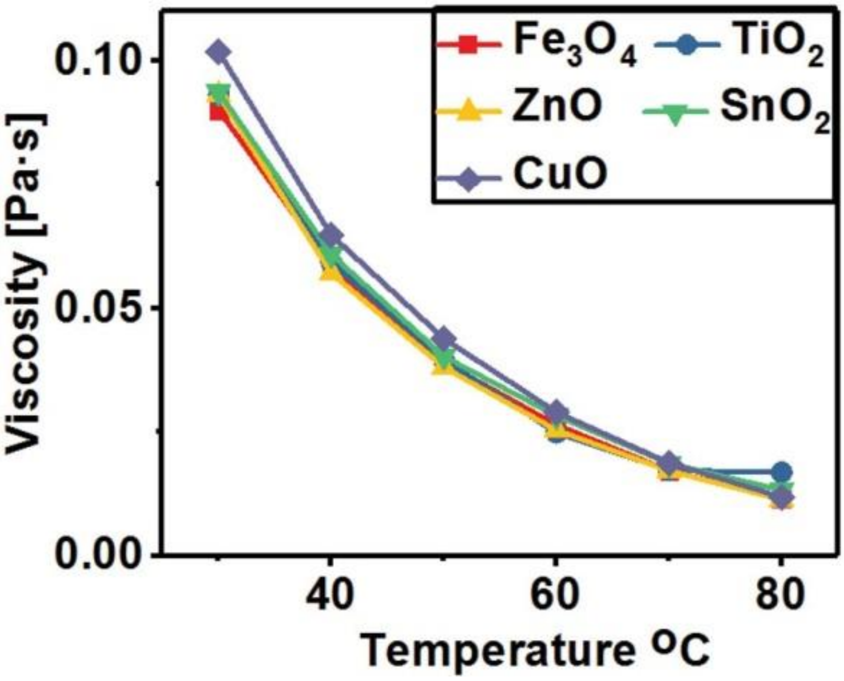


Figure 5.5. Dynamic viscosity versus temperature of different nanolubricants at constant shear rate of 1000(1/s).

Figure 5.6 shows how the shear viscosity of nanolubricants varies with temperature. The graph clearly establishes that when the temperature rises, the viscosity of all five nanolubricants declines significantly. The nanolubricants were used at a concentration of 1% by weight in this experiment. The concentration dependence of viscosity has been studied in a number of ways. According to these investigations, as the concentration of nanoparticles rises, so does the viscosity. The results in Figure 5.6 are consistent with the viscosity data for several nanolubricants as discussed by Tshehla [64] . A similar pattern was also reported by Sundar et al. [63] and investigated the influence of Nano fluids on viscosity using a variety of base fluids (ethylene glycol, transformer oil and water). Different base fluids showed similar nonlinear temperature dependence with viscosity [63, 65]**.** Several studies have found that the effect is the same regardless of which base fluid is used.

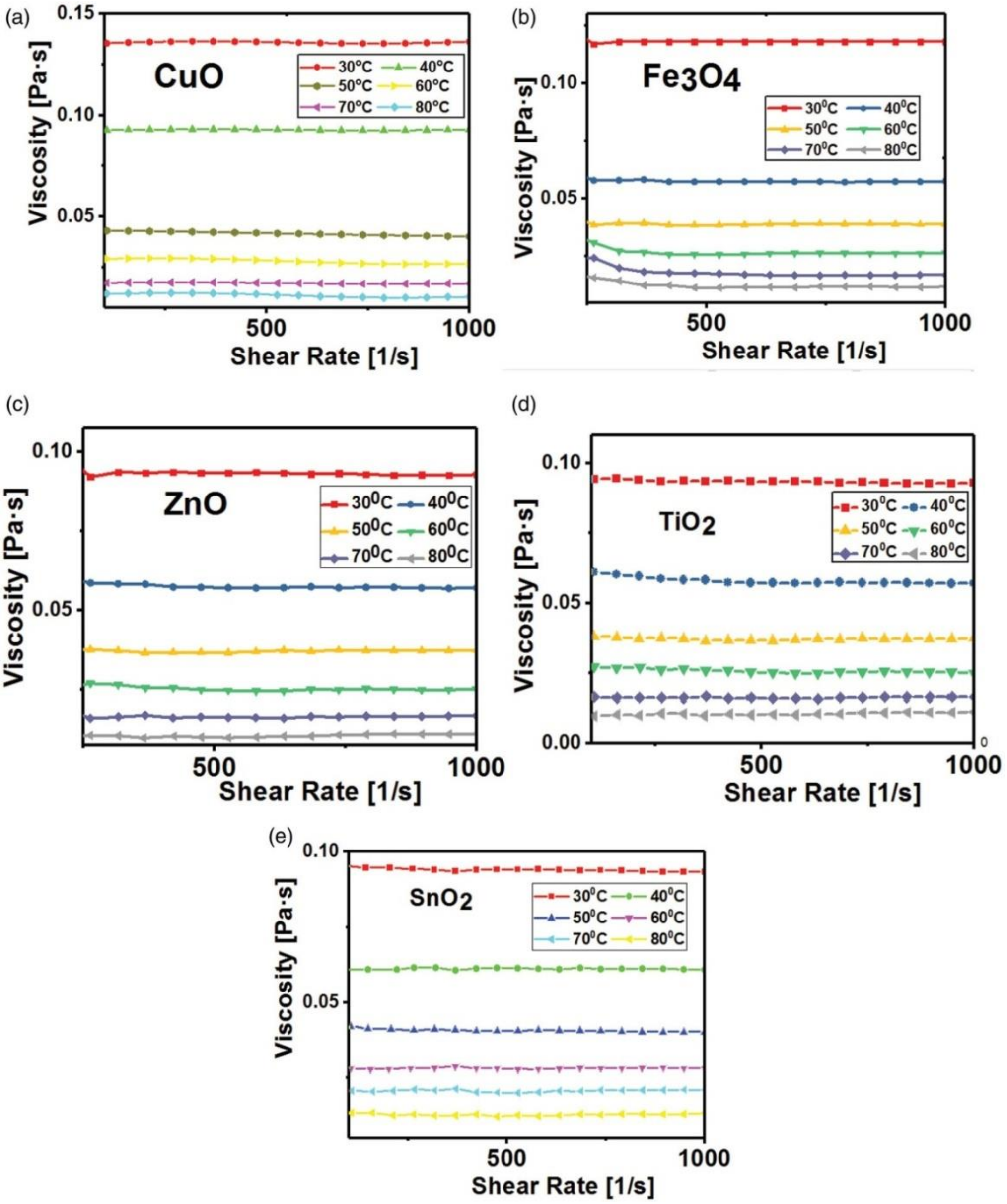


Figure 5.6. Viscosity vs Shear rate of nanoparticles at different temperatures (a) CuO, (b) Fe_3O_4, (c) ZnO, (d) TiO_2, and (e) SnO_2.

5.2. Characterization of CuO based nanolubricants

5.2.1. X-Ray Diffraction (XRD) characterization

X-Ray diffraction (XRD) machine is a potent tool for characterizing different materials such as crystal structures and atomic spacing. Figure 5.7 shows the XRD spectrum of coated, gum arabic CuO and uncoated CuO nanoparticles. Two main peaks on the XRD graph are located at 2θ = 46° and 2θ = 49° as seen in Figure 5.7 (JCPDS no.: 88-1175 and 84-1286).

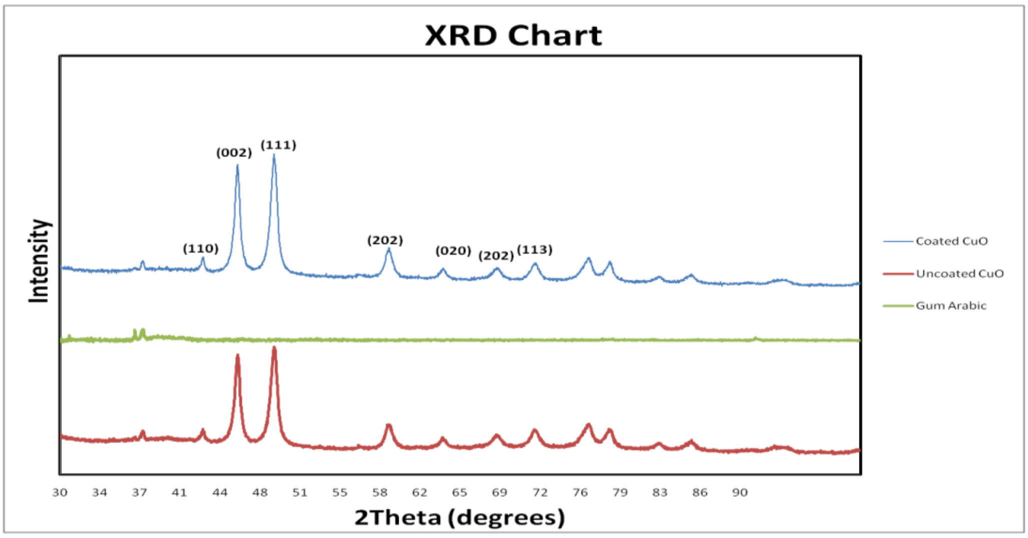


Figure 5.7. XRD spectrum of coated, Gum Arabic and uncoated CuO nanoparticles.

The multiple peaks in both coated and non-coated particles ascribe the formation of CuO monoclinic crystal phase [66]. Debye Scherrer equation was applied to analyze the crystallite size of coated CuO and non-coated CuO nanoparticles were assessed to be 15.3 nm and 18.21 nm. The crystallization size is bigger in non-coated because it’s less dispersant. These calculations agree with the literature [67, 68].

Debye Scherrer Equation:

$$D = K \lambda / (\beta \cos \theta)$$

where, D = Crystallite size

k = Scherrer constant (Correction or Shape factor) = 0.9

λ = Wavelength of Cu target = 1.5406 Å

β = Full width at half maximum (FWHM) of the most intense diffraction peak plane in radians

θ = Bragg’s angle in degrees

5.2.2. FTIR Test

Fourier Transform Infrared Spectrophotometer (FTIR) uses an infrared light source to measures the wavelength of an infrared region of a material. FTIR machine comprises of the source of radiation, beam splitter, movable mirror, stationary mirror, and the detector. The movable mirror moves back and forth throughout the experimental process to emit different wavelength patterns. The FTIR spectrum analysis is shown in Figure 5.8, the CuO nanoparticles major peak at 3437.66 cm^{-1} shifted to 3434.45 cm^{-1} for coated nanoparticle making it wider.

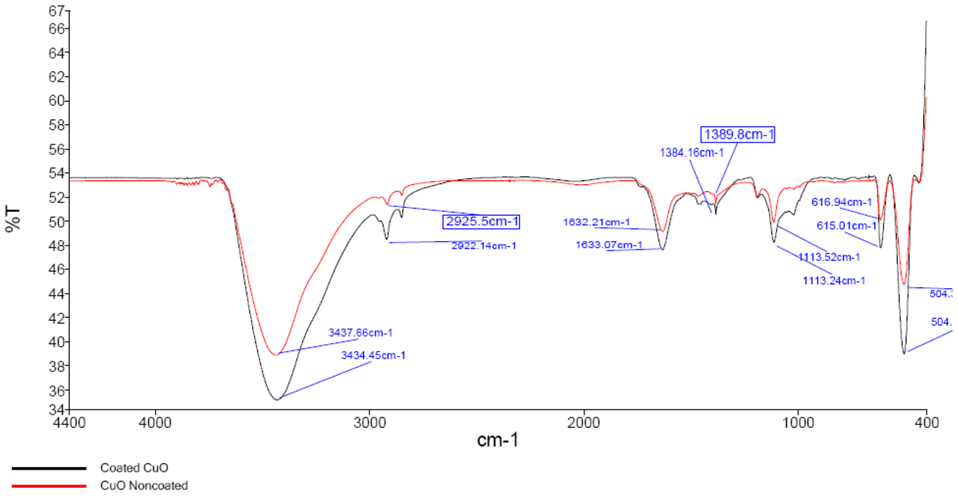


Figure 5.8. (a) FTIR Spectrum of uncoated CuO and Gum Arabic coated CuO Nanoparticles

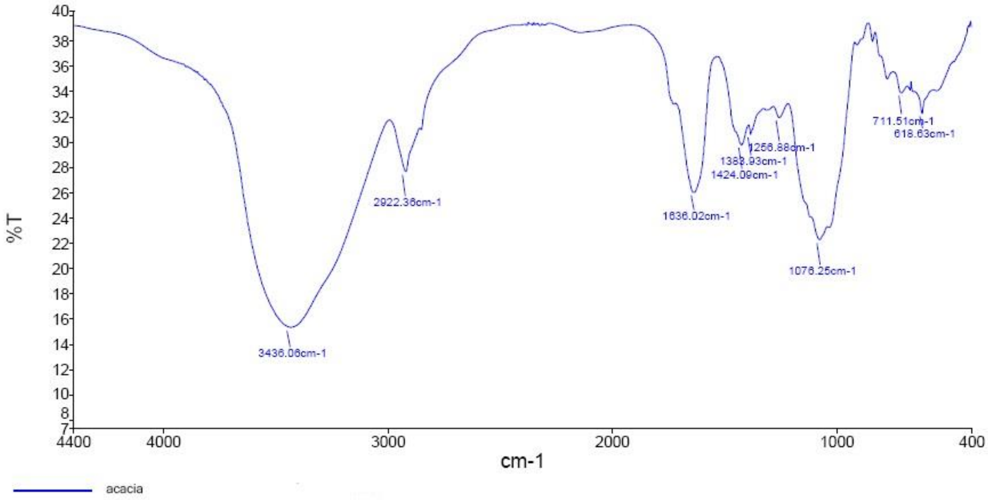


Figure 5.8. (b) FTIR Spectrum of Gum Arabic only

This was done due to capping agent gum arabic. The CuO nanoparticle coated by gum arabic FTIR spectrum shows the major peak at 3434.45 cm^{-1} (O-H symmetric stretching), 2922.1 cm^{-1} (C-H stretching), 1633.1 cm^{-1} (C=O stretching), 1389.8 cm^{-1} (O-H deformation), 1113.52 cm^{-1} (C-OH stretching) and 616.94 cm^{-1} (C-OH stretching) are compared to 3436.06 cm^{-1}, 2922.36 cm^{-1}, 1636.02 cm^{-1}, 1383.93 cm^{-1}, and 618.63 cm^{-1} are correlated to one another [68]. Since there was no occurrence of hydrogen in the copper oxide nanoparticles, there may have been some contamination involved in gum arabic. The following peaks 1113.24 cm^{-1}, 615.01 cm^{-1}, and 504 cm^{-1}, of coated CuO, are related to non-coated CuO. These results are also comparable to the studies mentioned in the literature [69].

5.2.3. Particle Size Analysis CuO nanoparticles

The Zetasizer (Malvern Zetasizer Nano) was to analyze the CuO nanoparticles size. The nanoparticles size is distinguished by using dynamic light scattering (DLS). The CuO nanoparticles were dissolved in water in a disposable polystyrene cuvette and the system temperature was kept at 25°C. The CuO nanoparticle's average size is 55.94 nm with 100% intensity and a standard deviation of 45.14 nm. This is confirmed by [70-72].

5.2.4. Photoluminescence analysis of CuO nanoparticles

Photoluminescence (PL) is the study of the emission of light from the CuO particles after the absorption of photons causing excitation of electrons to a higher energy state. This causes the radiation of a photon when the electron goes back to its lower energy state [73]. The photoluminescence spectrum for CuO particles is given in Figure 5.9, where a test runs from the wavelength of 300 nm to 900 nm.

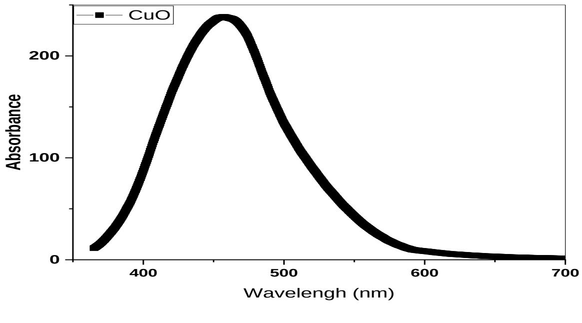


Figure 5.9. Photoluminescence analysis of CuO nanoparticles.

As we can see from Figure 5.9, a considerable spike occurs at the range 455 to

475 nm. The excitation wavelength is set at 230 nm. The spike is flattened at the top due to the limitations of the machine and could be due to the high density of particles. At this range, the CuO nanoparticles have the lowest energy as it releases photons. There is another spike at 750 nm which is ignored as it is due to the dissolving medium or oxygen vacancy. The outcomes are parallel to the data originate in the previous study [74].

5.2.5 UV-Vis Spectroscopy

UV- Vis spectroscopy stands for Ultraviolet-Visible spectroscopy and is often used to measure at which wavelength maximum absorbance of light occurs in both the ultraviolet and visible spectrum. For this report, the PerkinElmer UV-Vis Spectrometer was used. Two disposable polystyrene cuvettes were used, one with the sample dissolved in distilled water and the other being just distilled water to be used as a reference. Figure 5.10 shows the UV-Vis Spectra for the CuO nanoparticles.

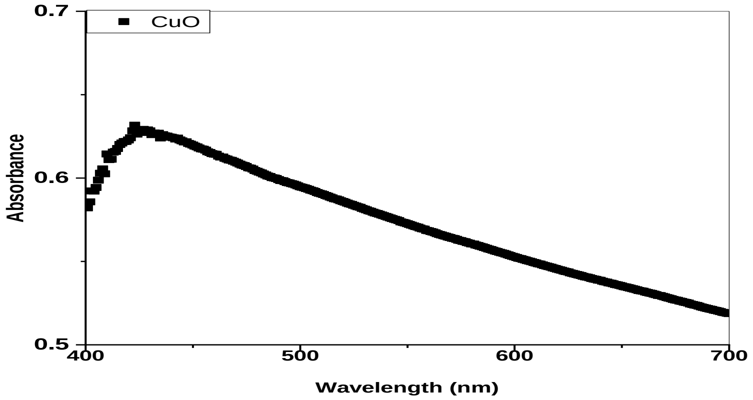


Figure 5.10. UV-Vis absorbance spectrum of CuO nanoparticles.

As we can see, the maximum absorption for the CuO nanoparticles occurs at the wavelength of 430 nm. Using the equation, E = hC/ λ, the band gap value is found to be 2.88 eV. This value seems to be accurate in comparison to the study mentioned in literature [75].

5.3. Rheological study of CuO based nano lubricants

For the rheological tests, the observed factors were the viscosity, shear stress, and the torque on varying temperatures. The temperature of the rheometer was augmented to 80°C and then viscosity, shear stress, and torque were observed while the temperature returned to room temperature. The Shear Rate was kept constant at 1000/s. The temperature controlling part of the rheometer was turned off and the machine could return to room temperature naturally. This was made sure that the temperature goes down slowly, so the observing factors could be recorded at regular intervals instead of temperature moving directly to the set temperature.

5.3.1. Viscosity Vs Temperature

Figure 5.11 shows the curves of viscosity vs temperature for all the four CuO blends and Engine Oil for comparison.

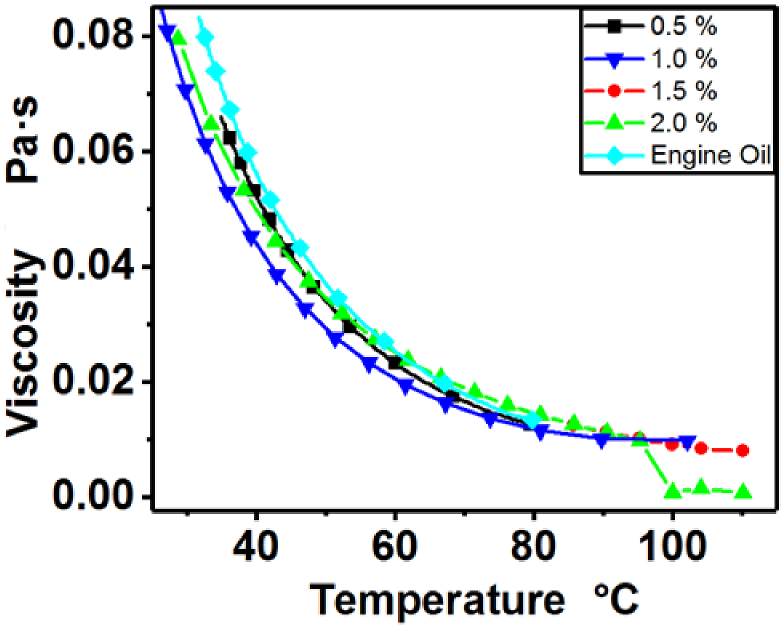


Figure 5.11. Viscosity Vs Temperature for all the four CuO blends and Engine Oil.

The Figure 5.11 shows that all the blends have a lower viscosity as temperature increases compared to engine oil [76]. It is clear from previous literature that the temperature and % concentration of nanoparticles are major factors in evaluating viscosity of nanofluids, and are proved from the widely performed experiments [17, 77]. However, it is detected that the growth in nanoparticles by weight does not necessarily mean more decrease in viscosity. This is seen as 1.0 wt% CuO Engine Oil has the lowest viscosity followed by 2.0% and then 1.5% and then 0.5% finally. For lower temperatures stretching from 25°C to 60°C, there is a considerable difference in viscosity between Engine Oil and 1.0% blend. At

35°C, Engine oil has a viscosity of 0.0825 Pa-s while 1.0% blend has a viscosity of 0.0625 Pa-s. Even 2.0% blend has a huge difference compared to Engine Oil at lower temperatures but at higher temperatures (50°C or higher), engine oil has a lower viscosity. Overall, all the blends have less viscosity compared to just engine oil. With an increase in temperature, the viscosity will be lowered due to the liquidity of lubricant samples. However, the difference lessens at higher temperatures except for the 1.0% CuO - Engine Oil blend. These results seem to agree with the literature [75, 76]. This relationship where viscosity decreases as temperature increases for Engine oil with CuO Nano-additives can also be observed at higher temperatures as found in the literature [59, 78].

5.3.2 Variation of Shear Stress with temperature

Figure 5.12 depicts the graph of shear Stress vs temperature for all the four CuO nanoparticles-Engine Oil blends and Engine Oil for comparison.

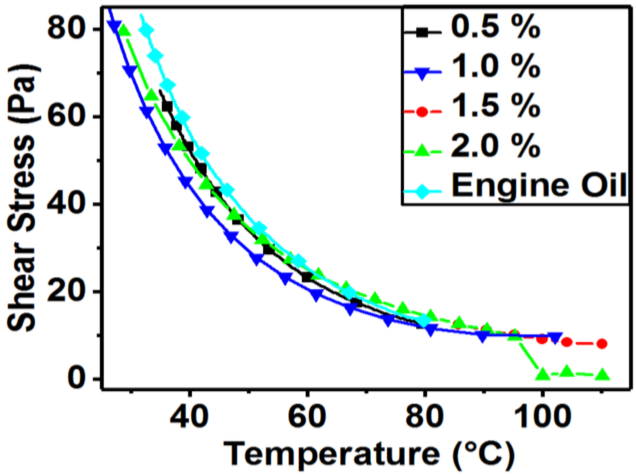


Figure 5.12. Shear Stress Vs Temperature for all the four CuO blends and Engine Oil.

Literature review states that nanoparticles cause an anti-wear boundary film which causes decreasing shear stress [79]. The results prove this as we can see from the graph, all the CuO nanoparticle- base engine oil blends have lower shear stress as temperature increases related to base oil. The CuO and base Oil blend with 1.0% CuO by wt has the lowest shear rate followed by 2.0% CuO by wt. These results are confirmed by literature [78, 80].

5.3.3. Torque Vs Temperature

Figure 5.13 shows the graph of Torque vs Temperature for CuO nanoparticles-Engine Oil blends and Engine Oil for comparison.

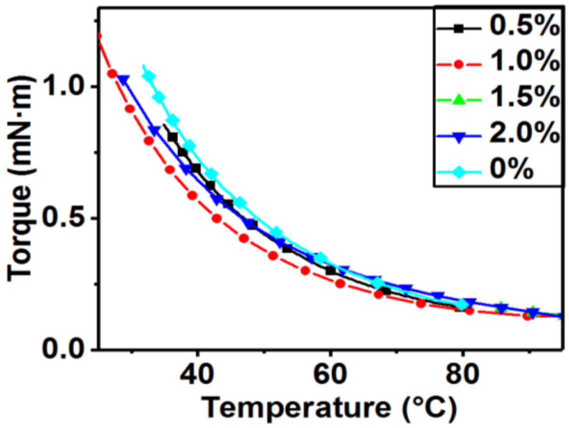


Figure 5.13. Torque Vs Temperature for all the four CuO blends and Engine Oil.

As we can see from the graph, at lower temperatures, all the CuO nanoparticles-Engine Oil blends have lower torque compared to engine oil. In fact, 1.0% blend has a torque of 0.55 mN-m at 40°C which is half of what engine oil has at 40°C. This is a very significant reduction in torque. However, this difference in torque seems to diminish and lessen as temperature rises and is mostly non-existent at high 70°C and 80°C at which point the CuO nanoparticles- Engine Oil blends and Engine Oil have similar torques. The graph shows that a rise in temperature causes a reduction in torque. The decline in torque is steep at inferior temperatures and flattens at advanced temperatures. These outcomes seem to match with the literature [72, 81, 82].

5.4. Friction and Wear Characterization

A total of two tests were conducted. The first test was carried out on 5W30 mineral engine oil. The second test was carried out on 1% by wt. CuO blended engine oil. The pin-on-disc tribotester (Model No.-DUCOM TR-20LE-CHM-400) has been used to evaluate various tribological properties of different blends of nano lubricants. This tribotester has a spindle, a chuck used for carrying steel rotating disc, a lever & arm attachment for holding test pin, and some attachment for permitting the test pin to be forced against the steel test disc with some constant load. A circular wear track is made on a test disc, which is having several wears passes onto the same track. The coefficient of friction is determined by a system that has a load cell as a friction force measuring device. The pins were made of aluminum-silicon alloy to be used on a pin-on-disc tribotester. It consisted of 93% aluminum and 7% silicon and the revolving disc was made of steel. The pins used for each test experienced a weight loss for each stage. This can be seen in Table 5.2. The five different stages of each test would run for a total of 3 kilometers and the difference in time would affect the track diameter. The total sliding distance of the whole test was 3 km.

Table 5.2.

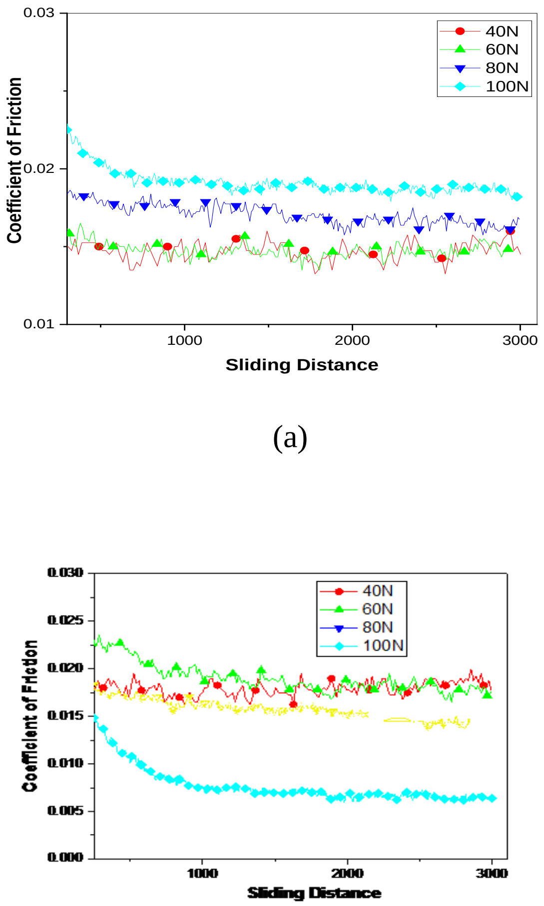
Pin weight loss after analysis.

S. No.	Rotation per minute (rpm)	Time (min)	Wear track Dia. (mm)	Load (N)	Pin Weight (g) (after test) 5W-30 semi-synthetic engine oil Initial M =13.714 g	Pin Weight (g) (after test) Oil + 1% CuO Initial M =13.4986 g
Symbol→	N	T	D	F	M	M
1	900	10	106.16	20	13.296	13.4983
2	900	15	70.77	40	13.401	13.4979
3	900	20	53.08	60	13.513	13.4976

4	900	25	42.46	80	13.678	13.4974
5	900	30	35.39	100	13.706	13.4970

5.4.1 Coefficient of Friction and Wear

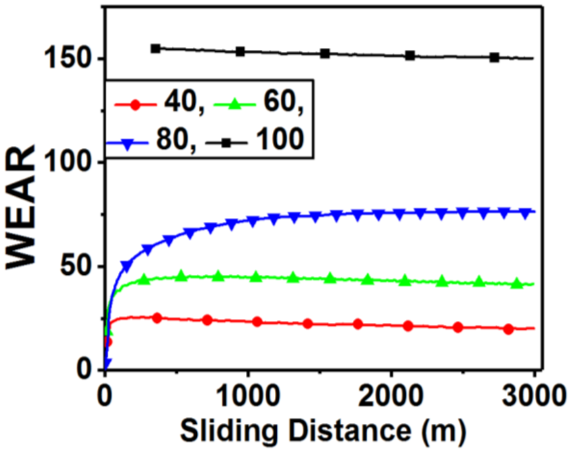
It was noticed that coefficient of friction and wear would decline with CuO nanoparticle. It can be seen from Figures 5.14 (a) and 5.15 (a) for pure oil compared from Figures 5.14 (b) & 5.15 (b) for oil with CuO nanoparticles. The coefficient of friction varies with different loads.



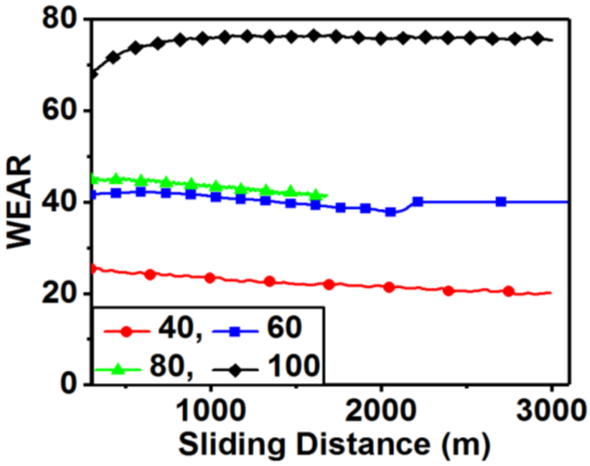
(a)

(b)

Figure 5.14. Coefficient of friction Vs sliding distance for (a) Pure oil (b) 1% CuO blended oil.



(a)



(b)

Figure 5.15. Wear Vs sliding distance for (a) Pure oil (b) 1% CuO blended oil.

When adding the nanoparticle into the lubricant, the coefficient of friction experiences a 96% decrease in 20N, 89% decrease in 40N, 90% decrease in 60N, 88% decrease in 80N and a reduction of 76% in 100N loads. These tribological test findings recommend that friction as well as wear both can be significantly enhanced for the blend of optimum % weight of CuO and base oil. When pin and disc both made contact with each other, rubbing of their surface hills, microscopically, generates friction between two surfaces. When CuO is added to engine oil, it fills up small nanogaps of these two mating surfaces hence direct contact of these surfaces is avoided and friction is reduced. A thin continuous film consisting of CuO was created in the oil of these rubbing surfaces. In the case of CuO mixed nanofluids, the sliding mechanism is the main mechanism that reduces friction. As CuO has planar geometry, easily sliding occurs between the surfaces in oil. Best results could be achieved for the optimal % weight of CuO added in pure oil. If there is an increase in the percentage concentration of CuO furthermore, the aggregation and coagulation of CuO nanoparticles will result, so we will notice an increment in wear and friction between surfaces. Wear scar diameter of the steel disc is decreased evidently due to penetration of 2-3 nm CuO into the contact surfaces and the antiwear property is enhanced. The effect of roller bearing is a key mechanism of lubrication for reduced friction. After tribological tests, wear scar surface analysis revealed that CuO's superior lubrication presentation is due to their small size and extremely thin coated microstructure, which allows CuO nanoparticles to reach the contacting area readily, preventing direct contact with higher rough surfaces. The nanoparticles of CuO will first fill up the microgaps of rubbing surfaces during surfaces contact, so a self-assembled thin lubricating film is formed. The coefficient of friction is significantly affected for 60 N force, and value agrees with the graph. This effect is caused due to sphere-like nanoparticles rolling between rubbing or contacting surfaces and hence we get reduced friction. When nanoparticles concentration is increased, the coefficient of friction is also increased. But this increment cannot be more than that of friction coefficient of pure base oil. Due to the accumulation of CuO nanosized particles, this effect takes place. Due to this accumulation of CuO onto a worn surface, the anti-wear mechanism is endorsed. It can decrease the resistance to shearing and hence tribological properties are improved. When adding 1% CuO nanoparticle into the lubricant, the wear experiences a 10% decrease at 40N, 8% decrease at 60N, 42% decrease at 80N and a reduction of 50% at 100N loads. Not only there was a decrement in friction coefficient and wear on the disc surface, but the scratching pin also experienced weight loss as shown in table 1. The initial weights of the pins were 13.714 gm for the first experiment and 13.4986 gm for the second experiment. As seen in Table 5.1, the difference in 20N and 40N loads for the second experiment, the weight of the pin did not change. Even though friction can never be avoided; there was no significant decrease in wear on the surface of the pin. This is confirmed when looked at wear vs. sliding distance graph figure 5.14. Wear is determined with the help of a linear variable differential transformer (LVDT) and friction is determined by using sensors. These results agree with noted works of literature [83, 84].

5.4.2. Worn surface characterization

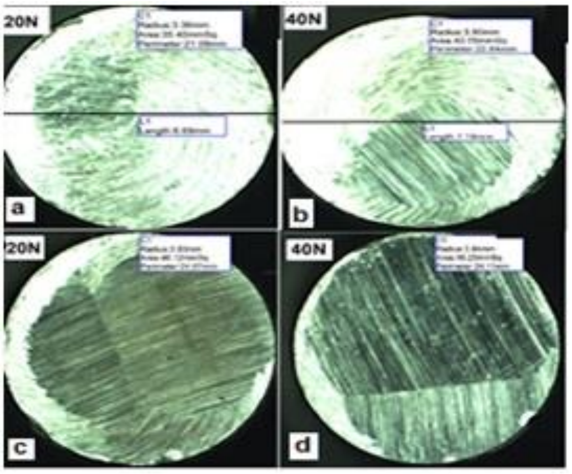


Figure 5.16**.** Surface images of the pin at (a) 20N and (b) 40 N for pure oil, (c) 20N and (d) 40N for Oil + 1% CuO.

Figure 5.16 (a-d) show the worn surface morphology using the metallurgical microscope equipped with view 7 software for the samples at variable loads [20 N and 40 N]. Figure 5.16 (a and b) shows the worn surfaces detected when mineral base oil was used. Innate twin indentations are developed on the surface. Under heavy load, the film of lubricant produced by the base oil does not sustain for a longer duration. Figure 5.16 (c and d) illustrates the worn surfaces when 1% CuO nanoparticles are mixed to the lubricant and in this duration uniform surface finish is observed with reference to the base oil. Some parallel lighter indentations are made onto the surface. It can be due to the accumulation of CuO nanosized particles onto the worn surfaces when sliding contact took place. With the help of this indication, we confirm that the oil + 1% CuO blend has an ability to produce a shielding film of lubricant which sustains stable for longer duration when there is sliding contact between two surfaces. It is noticed that the wear of the surfaces is increased when the concentration of nanoparticles rises. The formation of shallow and rough grooves is there onto the worn surfaces when the concentration of nanoparticles rises to 1.5% and 2%. The nanoparticles got entrapped into the grooves (valley), so it prevents the motion further between these surfaces and a higher stress concentration is received. These trends give rise to the abrasive wear of surfaces and both friction and wear are increased [85].

CHAPTER 6
CONCLUSIONS AND SUGGESTIONS

The study's findings were given in this chapter, and they were compared to the goals set forth in Chapter 1. The findings are reported point by point while the objectives are kept in line with the study's goal. This chapter also discusses future work recommendations, publications resulting from this effort, and scientific knowledge gained.

6.1. Conclusion involving different metal oxide nano lubricants

- Characterization and rheological studies of five nanoparticles are addressed in this study. Five different nano lubricant samples were identified using XRD and photoluminescence spectroscopy, and the rheological characteristics of the nano lubricants were investigated. The nano lubricants Newtonian behaviour was validated by the shear stress and shear rate data.
- The Newtonian behaviour of these five nano lubricants was further validated by the shear rate's independence from viscosity.
- The viscosity of the nano lubricants employed in this study was unaffected by the shear rate or the type of nano lubricants employed, as it was in most other investigations in this field.
- Shear viscosity was also found to be temperature dependent. The results revealed a nonlinear reduction in shear viscosity with increasing temperature. This nanolubricant's nonlinear behaviour opens up a plethora of high-temperature uses in the automobile industry.

6.2. Conclusion involving CuO based nano lubricants

- CuO nanoparticles were characterized using particle size analyzer, Photoluminescence and UV visible spectroscopy. The maximum emission of CuO nanoparticles was shown to be 455 to 475 nm using photoluminescence. While on the other hand, UV-Vis Spectroscopy shows maximum absorbance at 430 nm having a band gap energy value of 2.88 eV. After experiments, two surfactants out of the selected four surfactants showed little to no increase in the dispersion of nanoparticles in oil.

- Coating CuO nanoparticles with gum arabic showed a significant increase in dispersion. The samples were sonicated for 90 minutes followed by ultrasonic vibrator for 30 minutes and left aside for 2 days. The stability was accessed by the sedimentation method. All CuO - Engine Oil blends showed a higher decrease in viscosity, shear stress, and torque compared to Engine Oil with a rise in temperature. The 1.0 wt% CuO in Engine Oil blend seems to show the most promising results with the highest reduction in viscosity, shear rate, and torque.
- The CuO nanoparticles used in the semi-synthetic engine oil showed good anti-friction and anti-wear performances, coefficient of friction reduced by 75-90%. This decrease varied from 20N – 100N loads on the pin-on-disc machine. With the reduction of the friction coefficient, the wear would also decrease. Using Debye Scherrer's equation, an obtained XRD analysis helped evaluate the values of coated and non-coated CuO nanoparticles size. The crystallized particles had a volume of 15.3 nm and 18.21 nm for a non-coated nanoparticle. These results indicate the dispersion in the lubrication.
- The FTIR result shows the functioning between coated and non-coated CuO nanoparticle. Stating that there was a strong presence of gum arabic in the coated mixture. Having enough gum arabic created higher levels of dispersion as in this case it was 6:1 ratio. Adding heavier loads increases the wear on the surface of the object. On the other hand, the wear has appreciably reduced by adding 1% CuO nanosized particle additive in the base lubricant.
- CuO particles hold the attributes of good solubility and stability in a particular lubricant**.** Hence, CuO nanoparticles are fully qualified to be adopted as a multifunctional additive.

6.3. Contribution to scientific knowledge

The use of nano lubricants as a replacement for engine oil has shown to be a beneficial process. During their rheological and tribological analysis on a pin on disc tribometer, the nano lubricant provided an improvement during their analysis under various operating conditions. The scientific knowledge obtained from this study are:

- During the rheological characterization, nano lubricants showed a better behavior at all concentration in comparison to the base oil.
- During the sliding motion between the surfaces in contact, the considered parameters load, speed and lubricant influenced effectively their friction and wear characteristics. Higher load, and weight percentage concentration of nanoparticles to the engine oil adversely affected the tribological performance of the surfaces in contact.
- The wear occurred during the sliding motion when surfaces are in contact follows the Archards adhesive wear law while considering the application of the applied loads. It can be stated that maximum amount of stress is produced when load is increased on the surface of the lubricant film.

6.4. Future work recommendations

The findings of this study make a significant addition to the lubricant industry by examining nano lubricants as an alternative to engine oil during their rheological and tribological characterisation. The investigation of nano lubricants improved the friction and wear qualities of surfaces during their sliding motion significantly.

The application of the nano lubricants used in this study is restricted to the parameters described during the research. So, while considering nano lubricants for tribological investigation and making them viable on a wide scale, more progress is required.

To make nanolubricants a viable alternative to currently available engine oil, several qualities must be improved. These are connected to their oxidation stability at higher temperatures and the degradation of characteristics as the chemically modified oils are blended further. Certain commercially available additives can help to improve these characteristics. Anti-wear additives, extreme pressure additives, and anti-friction additives are among them, and more research is being done on the use of nano-additives to improve lubricity qualities.

6.5. Publications obtained from this work

6.5.1 Peer-reviewed journal

- Harsh Gupta, Santosh Kumar Rai, Piyush Kuchhal and Gagan Anand 2020. “Characterization and experimental investigation of rheological behavior of oxide nanolubricants”. Particulate Science and Technology, DOI: https://doi.org/10.1080/02726351.2020.1792018.

  (SCI Indexed)

- Harsh Gupta, Santosh Kumar Rai, Satya Krishna Nippani and Gagan Anand 2021. “The Effect of Copper Oxide Nanoparticle additives on the Rheological and Tribological properties of engine oil”. Dispersion Science and Technology, Vol. 42, Issue 4. Pages 622-632.
  DOI: https://doi.org/10.1080/01932691.2020.1844017

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(2) Two papers in reputed international conferences.
(3) One book chapter in Springer Singapore Ltd.
(4) One paper in reputed national conference (SMTST 2020), published in Springer book series.

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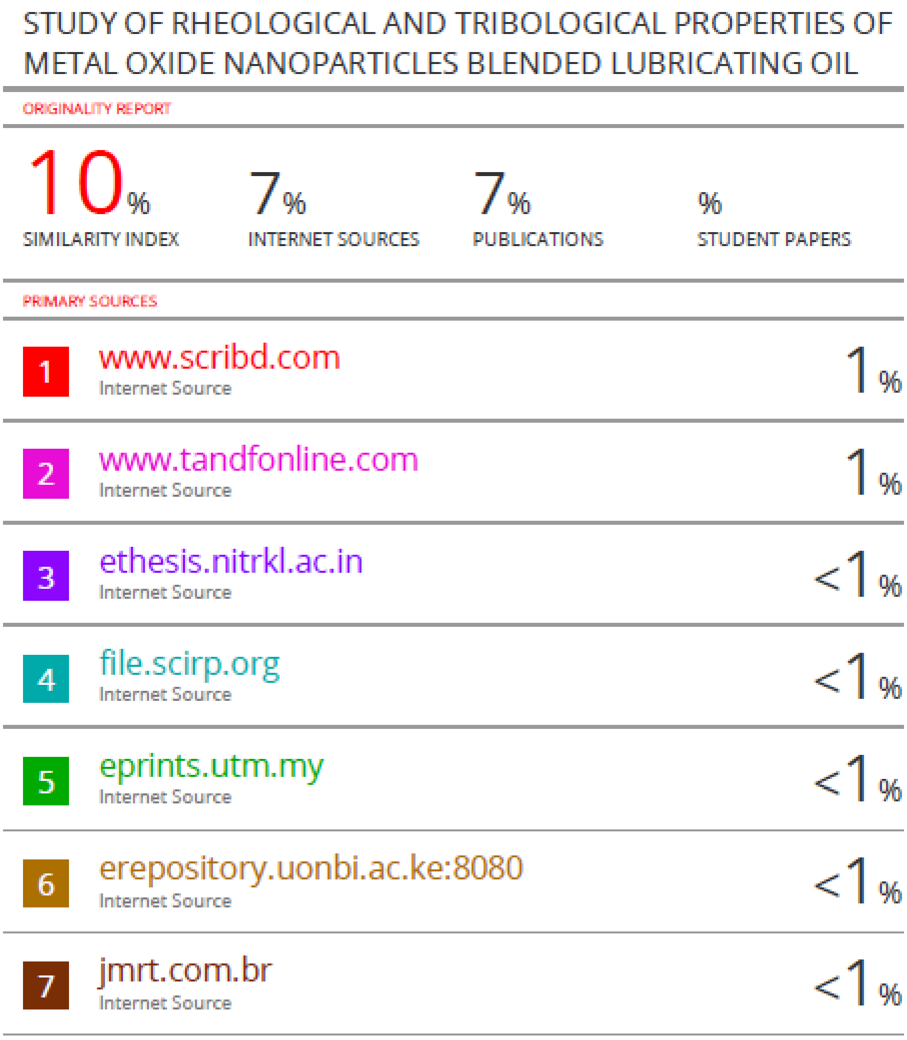

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