# PREPARATION OF COPPER NANO LUBRICANT FOR HIGH END APPLICATION

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# PREPARATION OF COPPER NANO LUBRICANT FOR HIGH END APPLICATION

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Report submitted in partial fulfilment of the requirements for the award of the degree of Bachelor of Chemical Engineering

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# SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering.

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## STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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## ABSTRACT

This paper presents a novel two step method for preparing of copper nano lubricants and aluminium nano lubircants by dispersing these two sample with refrigeration lubricant in ultrasonic vibrator. Different concentration of nanofluid were used in this experiment. Kinetic viscosity of nanoparticles inside the mixture was measured by viscometer and transmission electron microscope (TEM). As a result, the nanoparticles size must be in the spec which  $1 \times 10^{-9}$ . This kind of particles can only be measured by TEM. By this way, we can see either these oil has the same viscosity or not. There was no considerable change in the kinematic viscosity of nano-oil at the various volume fractions of nanoparticles, indicating that the kinematic viscosity of nano-oils is a weak function of oil temperature considered. Effect of heating was considered as the temperature used in viscometer is in range 25 - 80°c.

#### ABSTRAK

Kertas kerja ini membentangkan tentang penghasilan minyak nano kuprum dan minyak nano alumina menggunakan kaedah 2 langkah. Minyak yang dihasilkan akan dilarutkan menggunakan getaran ultrasonic. Minyak yang dihasilkan juga adalah dalan kepekatan yang berlainan. Kepekatan zarah-zarah nano dalam larutan ini akan diukur menggunakan mikrsoskop transmisi electron dan viscometer. Hasilnya, zarah-zarah nano akan berada dalam lingkungan saiz  $1 \times 10^{-9}$ . Melalui kaedah ini, kita dapat melihat sama ada minyak yang mengandungi kepekatan yang berlainan ini memiliki kelikatan yang sama atau tidak. Perubahan suhu juga diambil kira dalam eksperiman ini disebabkan zarah-zarah nano dalam kepekatan yang berbeza memiliki karakter tersendiri. Lingkungan suhu yang digunakan semasa ujian viscometer adalah antara 25 - 80°c.

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# LIST OF SYMBOLS

Wt	Weight
%	Percentage
$Al_2O_3$	Aluminium oxide
CuO	Copper oxide
Cu	Copper
К	Thermal conductivity
ср	Centipoise
ml	Mililitre
g	gram
М	Molar
nm	Nanometer

# LIST OF ABBREVIATIONS

- EG Ethylene glycol
- EHD Elctrohydrodynamics
- RPM Revolution per minute

# **CHAPTER 1**

#### **INTRODUCTION**

#### **1.1 Background of Study**

The use of nanoparticles in the lubrication of mechanical systems has become an interesting research line. The replacement of organic molecules by tiny particles of solid material is not straightforward and has only been regarded as a feasible option in recent studies. The main advantage of using nanolubricants is that they are relatively insensitive to temperature and their Tribochemical reactions are limited, compared to traditional additives . Another advantage of the addition of nanoparticles in lubricant oils is that they can pass through the filters. The antiwear mechanism of nanoparticles when they are used as additives in lubricants can be explained in three different ways: nanoparticles may be melted and welded on the rubbing surface, reacted with the specimen to form a protective layer, or tribo-sintered on the surface. However, some authors state that nanoparticles can also act as small bearings on the rubbing surfaces.

The colloidal nanoparticles penetrate into lubricated EHD contacts via a mechanical entrapment mechanism to form a boundary film influencing friction and wear, and these nanoparticles are entrained into sliding contacts only when the film thickness is smaller than the particle size contributing to the film thickness (Castillo.C & Spikes,2002). The above mentioned antiwear mechanisms of the nanoparticles take place only under mixed and boundary lubrication. Results show that nanoparticles can improve the tribological properties of the base oil, displaying good friction and wear reduction characteristics even at concentrations below 2 wt% With the rapid development of nanotechnology,

nanolubricants with metallic additives have also been studied. Experimental results report that the use of metallic nanoparticles as additives to oils can improve the antiwear properties under extreme pressure conditions. The metallic nanoparticles can also act without any corrosive effect and can be used at high temperatures. Therefore, they have the potential to become a new generation of antiwear and extreme pressure additives. The tribological behavior of lubricants with the addition of copper nanoparticles has been studied by some authors. In all these cases, when copper coated nanoparticles (none of them was carbon-coated ones) were tested as oil additives, the results showed that the oils with the addition of these nanoparticles exhibit an excellent antifriction and antiwear performance and high load-carrying capacity. Recently, surface-coated copper nanoparticles also used as oil additives. They showed that surface-coated nano-copper additives can significantly improve the wear resistance and load-carrying abilities of oil, as well as reduce the friction coefficient. They related the results to a soft copper protective film that is formed on the worn surface lubricated with oil containing nano-copper additives, which separates the worn surfaces, avoids their direct contact and reduces friction and adhesive wear. On the other hand, Bin et al. used graphene encapsulated copper nanoparticles as a lubricant additive. In this case, the additive also increased the loadcarrying capacity of the base oil, decreased the friction coefficient and enhanced the antiwear properties. However, they did not compare the tribological behavior of coated nanoparticles used as lubricant additives with the non-coated ones. The objective of this paper is to evaluate the influence of the addition of carbon-coated copper nanoparticles on the tribological properties of a polyalphaolefin (PAO6) and to establish the effect of coating by comparing the results obtained using the same copper nanoparticles with (Cu25C) and without the coating (Cu25).

## **1.2 Problem statement**

Nanoparticles as lubricants is a recent idea. High-performance, nanoparticle-based lubricants will soon minimize the labor and materials associated with preserving lubricant and equipment integrity. In oils, greases and solid lubricants, not only were the viability and value of nano-sized particles at issue, but health and environmental concerns need to be addressed. These initial hurdles are being crossed, and commercially available industrial lubricants are on your horizon. Although nano-lubes may reduce the frequency of oil changes, they will increase the value proposition of condition-based maintenance practices. **The purpose of nano-lubes** s actually for the conventional materials, when subdivided to the nanoscale, form nanoparticles, which are measured in nanometers. When particles are so small, their physical and chemical properties differ from those of the bulk material. For example, nanotechnology based extreme-pressure and anti-wear additives were found to have high chemical and physical stability, even under extreme conditions. These properties translate to longer equipment operation, increased efficiency and extended maintenance intervals.

## **1.3** Research Objectives

- 1.3.1 To develop the nanolubricant using two step chemical method.
- 1.3.2 To investigate the characteristics of nanolubricant.
- 1.3.3 To investigate the application of nanolubricant in nowadays technology

## 1.4 Research Question

- 1.4.1 What is the main purpose of the study ?
- 1.4.2 What is the characteristics of nanolubricants ?
- 1.4.3 How about the demand of nanolubricant in the industry ?

### 1.5 Scope of Study

1.5.1 Preparation of copper nano lubricant using two step chemical method for high end application.

#### 1.6 Significance of Study

1.6.1 Make the difference between nanofluid in oil form and the usage of it.

#### **1.7 Expected Outcomes**

1.7.1 The nano lubricants can be used as commercial oil such as being used in vehicles and industry.

### 1.8 Definition of Keyterms

- Copper is a chemical element with the symbol Cu (Latin: *cuprum*) and atomic number
  29. It is a ductile metal, with very high thermal and electrical conductivity.
  Pure copper is rather soft and malleable, and a freshly exposed surface has a
  reddish-orange color. It is used as a thermal conductor, an electrical
  conductor, a building material, and a constituent of various metal alloys.
- Nano lubricants is a new concept of lubrication, based on these nanoparticles, and along with the authors' own research it synthesises the information available on the topic of nanolubrication from existing literature and presents it in a concise form.
- Describes the many advantages and potential applications of nanotechnology in the tribological field.
- Offers a full review of the state-of-the-art as well as much original research that is yet unpublished.
- Includes sections on boundary lubrication by colloïdal systems, nanolubricants made of metal dichalcogenides, carbon-based nanolubricants, overbased detergent salts, nanolubricants made of metals and boron-based solid nanolubricants and lubrication additives.

## **CHAPTER 2**

#### LITERATURE REVIEW

#### 2.1 Overview

Literature review is one of the most impotent part in writing. What is actually the definition of literature review? A literature review is a body of text that aims to review the critical points of current knowledge including substantive findings as well as theoretical and methodological contributions to a particular topic. Literature reviews are secondary sources, and as such, do not report any new or original experimental work.

Most often associated with academic-oriented literature, such as theses, a literature review usually precedes a research proposal and results section. Its ultimate goal is to bring the reader up to date with current literature on a topic and forms the basis for another goal, such as future research that may be needed in the area.

A well-structured literature review is characterized by a logical flow of ideas; current and relevant references with consistent, appropriate referencing style; proper use of terminology; and an unbiased and comprehensive view of the previous research on the topic.

#### 2.2 Review of Copper Nano Lubricant

Copper nano lubricant is a new term in chemical engineering industry and has been developed for many kind of usage. Many research has been done according to this nano particles to improve the performance and stability of this particles.

Fig. 1 shows the thermal conductivity of several RL68H/CuO nanoparticle mixtures as measured with a transient linesource technique (Roder, 2000). Even though the thermal conductivity of CuO, 20 W/m K, (Kwak & Kim, 2005) is two orders of magnitude greater than that of neat RL68H (0.132 W/m.K - 0.001 W/m.K), an improvement in the thermal conductivity significantly beyond that proportional to the volume fraction of the nanofluid was not obtained. For example, the volume fraction used in the boiling experiments (1%) resulted in the nanofluid having a thermal conductivity that is roughly 5% greater than that of the pure base fluid. This proportional improvement is disappointing compared to the 40% improvement in thermal conductivity for a 0.4% volume fraction (Eastman, 2001). However, as shown in Figure 2.1, the solid–liquid thermal conductivity model of Wasp (1977) confirms the measured thermal conductivity of the 1% by volume mixture (0.139 W/m.K - 0.001 W/m.K) to within approximately 4%. The marginal increase in thermal conductivity of the refrigerant mixture as charged may not necessarily translate into marginal improvement of the thermal conductivity of the liquid at the heat transfer surface. The effective enhancement of the lubricant's thermal conductivity may in fact be greater than what the bulk concentration suggests because of the accumulation of nano particles in the lubricant excess layer that exists on the heat transfer surface. The increase in nano particle concentration in the excess layer was supported by observing the darkened lubricant that remained on the test surface after testing and removing the charge from the test apparatus. As a result, the thermal conductivity of the lubricant that resides on the surface and governs the boiling process may be greater than what the bulk nano particles concentration would suggest.



Figure 2.1 Thermal conductivity (W/m.K) versus ø

Figure 2.1 shows the kinematic viscosity of nano-oils as a function of volume fraction of fullerene nanoparticles in suspension for temperature ranging from 40°C to 80°C. There was no considerable change in the kinematic viscosity of nano-oil at the various volume fractions of nanoparticles, indicating that the kinematic viscosity of nano-oils is a weak function of oil temperature considered. Fig. 3 shows the change of kinetic viscosity as a function of volume fraction and temperature of the oil. When particles are added, the increase rate of viscosity of the nano-oil is within 1%. In the temperature range for a compressor with time, the viscosity of the nano-oil is about the same as for the mineral oil, but the viscosity of the nano-oil increases by 7% at 208°C in comparison with the mineral oil.



Figure 2.2 Kinematic viscosity (mm<sup>2</sup>/s) versus particle concentration



Figure 2.3 Kinetic viscosity (cSt) versus temperature (°C)

Many interesting properties of nanofluids have been reported in the review. In the previous studies, thermal conductivity has received the maximum attention, but many researchers have recently initiated studies on other thermo-physical properties as well. The use of nanofluids in a wide variety of applications appears promising. But the development of the field is hindered by (i) lack of agreement of results obtained by different researchers; (ii) poor characterization of suspensions; (iii) lack of theoretical understanding of the mechanisms responsible for changes in properties. Therefore, this paper highlighted several important issues that should receive greater attention in the near future.

For the long term stability if nanoparticles, there are a few problem which encountered. The preparation of homogeneous suspension remains a technical challenge since the nanoparticles always form aggregates due to very strong van der Waals interactions. To get stable nanofluids, physical or chemical treatment have been conducted such as an addition of surfactant, surface modification of the suspended particles or applying strong force on the clusters of the suspended particles. Dispersing agents, surfaceactive agents, have been used to disperse fine particles of hydrophobic materials in aqueous solution On the other hand, if the heat exchanger operates under laminar conditions, the use of nanofluids seems advantageous, the only disadvantages so far being their high price and the potential instability of the suspension. Generally, long term stability of nanoparticles dispersion is one of the basic requirements of nanofluids applications. Stability of nanofluids has good corresponding relationship with the enhancement of thermal conductivity where the better the dispersion behavior, the higher the thermal conductivity of nanofluids.

As a result, thermal conductivity of nanofluids is eventually affected. Eastman et al. revealed that, thermal conductivity of ethylene glycol based nanofluids containing 0.3% copper nanoparticles is decreased with time. In their study, the thermal conductivity of nanofluids was measured twice: first was within 2 days and second was 2 months after the preparation. It was found that fresh nanofluids exhibited slightly higher thermal conductivities than nanofluids that were stored up to 2 months. This might be due to reduced dispersion stability of nanoparticles with respect to time. Nanoparticles may tend to agglomerate when kept for long period of time. The  $Al_2O_3$  nanofluids stability was compared visually over time span. It was found that nanofluids kept for 30 days exhibit some settlement and concentration gradient compared to fresh nanofluids (Lee & Mudawar, 2005). It indicated that long term degradation in thermal performance of nanofluids could be happened. Particles settling must be examined carefully since it may lead to clogging of coolant passages. It has been reported that the excess quantity of surfactant has a harmful effect on viscosity, thermal property, chemical stability, and thus it is strongly recommended to control the addition of the surfactant with great care. However, the addition of surfactant would make the particle surface coated, thereby resulting in the screening effect on the heat transfer performance of nanoparticles (Choi, 2002) Authors also mentioned that the surfactant may cause physical and/or chemical instability problems.

In contrast to other common base fluids such as water or ethylene glycol, a remarkably rapid agglomeration and settling of common nanoparticles was observed in refrigerants.



**Fig. 2.4.** Samples of  $Al_2O_3$  nanofluids (without any stabilizer) stability change with time.

## 2.3 Viscosity of Nano Lubricant

Cooling and lubricating are important in many industries, especially in transportation and energy production. Advanced developments in operating high-speed, high-power, and high efficiency engines and turbines with significantly higher thermal loads require more efficient cooling and lubricating technology. In order to increase heat dissipation, the usual approach is to increase the surface area available for the lubrication fluid. This approach, however, is not desirable and accurate because a larger heat exchange system is required which acts as a primary barrier to the development of energy-efficient compact heat exchangers. Therefore the development of more efficient and advance heat transfer fluids with higher thermal conductivity is considered urgent. Over the years, many researchers focused their attention to create new kinds of heat transfer fluids by suspending tiny (micrometer sized) particles in the conventional liquids to enhance their heat transport performances. Choi et al. in 1995 created Nanofluids by suspending nanoparticles in common heat transfer fluids (viz, water, ethylene glycol etc.) and reported dramatic enhancement of their thermal properties.

Since then, most of the published reports on nanofluids concentrated on the heat transfer behavior including thermal conduction, phase change (boiling) heat transfer, and convective heat transfer. In comparison, very few studies, however, have been devoted on the rheological behavior of nanofluids. A number of review articles emphasized the significance of investigating the viscosity of nanofluids and it is believed that viscosity is as critical as thermal conductivity in establishing adequate pumping power as well as the heat transfer coefficient in engineering systems that employ fluid flow. This is because; pumping power is proportional to the pressure drop, which in turn is related to fluid viscosity and also both Reynolds and Prandtl numbers depend upon the viscosity.

Prasher et al. reported effects of shear rate, nanoparticle size, volume fraction and temperature on the viscosity of alumina based nanofluids. Their data demonstrated that viscosity is independent of shear rate, proving that the nanofluids are Newtonian in nature. Namburu et al. studied the viscosity of copper oxide nanoparticles dispersed in ethylene glycol (EG) and water mixture. They also concluded that copper oxide nanofluids exhibit Newtonian behavior in EG water mixture for particle volume fraction varying up to 0.0612. Viscosity of EG based nanofluids containing titania nanoparticles was measured by Chen et al. and Newtonian behavior was observed over a wide shear rate range at temperatures between 293K and 333K. Kwak et al. studied the viscosity and thermal conductivity of copper oxide EG nanofluids, containing rod shaped particles having an aspect ratio of about three. Kulkarni et al. observed that copper oxide nanoparticles with volume fractions between 0.05 and 0.15 in water behaved as non-Newtonian fluids in the temperature range of 5 - 50 °C. Phuoc et al. reported the effects of the shear rate and particle volume fraction on the shear stress and the viscosity of Fe2O3 distilled water nanofluid and confirmed the existence of yield stress even at low volume concentration. At higher volume fraction (>0.02), the fluid became non-Newtonian with shear thinning behavior. Kole et al. investigated  $Al_2O_3$  car engine coolant nanofluid and confirmed that the Brownian motion of the nanoparticles in the fluid plays an important role in understanding the viscosity of nanofluids.

Although nanofluids have reasonable application potential in transportation and lubrication engineering, till recently major attention have been focused primarily to the preparation and evaluation of the thermophysical properties of water and EG based nanofluids. Most of the reported works on oil based nanofluids concentrated on the thermal conductivity of transformer oil/silicon oil/poly alphaolefin oil (PAO) as base fluids with  $Al_2O_3/AlN/$  Cu nanoparticles and to our knowledge, there exists no data on the nanofluids with gear oil as base fluid. Further, it is generally observed that the enhancement of thermal conductivity for both water and EG based nanofluids with CuO nanoparticles as dispersant is much higher than that with alumina/other metallic oxides. In view of the above, the present work is undertaken to explore CuO gear oil nanofluids, with emphasis on its synthesis and the measurement of effective viscosity as a function of CuO volume fraction, shear rates and temperatures. The results obtained are evaluated in light of the contemporary theoretical models and mechanisms.

#### 2.4 Usage of Nano Lubricant

Firstly, according to the method for preparing a mixed nano-lubricating oil of the present invention, it is possible to prepare a mixed nano-lubricating oil having excellent extreme pressure load resistance and wear resistance by performing the wet pulverization and the surface modification at the same time and mixing at least two nano-lubricating oils prepared using physically and chemically different nanopowders prepared by substituting a solvent to oil by vacuum concentration.

Secondly, since the method for preparing a mixed nano-lubricating oil of the present invention dilutes each nano-lubricating oil with a pure oil, it is possible to freely determine the mixing ratio and select the composition according to the application.

Thirdly, the method for preparing a mixed nano-lubricating oil of the present invention can be used in the preparation of a high efficiency cooling nanofluid using a metal, an alloy, an oxide, or a non-metal nanopowder. Fourthly, according to the method for preparing a mixed nano-lubricating oil of the present invention, it is possible to obtain the lubricating properties of an equivalent level even the use of a lubricating oil having a viscosity lower than that of the lubricating oil used at preset. Moreover, since the mixed nano-lubricating oil according to the present invention has low viscosity, it has relatively high cooling properties, and thus it is possible to reduce the operating rate or the volume of a circulation pump and a cooling fan, thus reducing the amount of energy consumed.

Fifthly, according to the method for preparing a mixed nano-lubricating oil of the present invention, it is possible to effectively reduce the friction heat, and thus it is possible to extend the durability of the lubricating oil by preventing the temperature of the lubricating oil from rising. Moreover, it is possible to extend the durability of an expensive large gear and prevent a failure by effectively prevent the wear of the parts such as the gear during operation under heavy load.

## **CHAPTER 3**

## **RESEARCH METHODOLOGY**

#### **3.1 Chemical Substance**

- Isopropanol
- Copper oxide
- Aluminium oxide
- Refrigerant oil RL68H

## **3.2 Procedure**

All the reagents used in our experiments were of analytical purity and were used without further purification. The beakers used in this procedure were cleaned by an ultrasonic cleaner in an ultrasonic bath. In this procedure, 75 ml of isopropanol solution was taken in a 500 ml beaker. 1.2 grams of copper oxide powder was added into isopropanol. The reaction mixture was subjected to magnetic stirring for 1 hour in a magnetic stirrer and dispersed in ultrasonic cleaner once more for 1 hour. The experiment was proceed with 1.8 grams ,2.4 grams and 3.0 grams of copper powder. Step 1 until 6 were repeated using aluminium oxide powder with weight of 1.5 grams, 2.3 grams, 3 grams, 3.8 grams.10 ml of base oil using refrigerant oil RL68H was added for each sample for viscometer testing.

Sample	Concentration (M)
	0.2
	0.3
	0.4
Copper Nanofluid	0.5
	0.2
	0.3
	0.4
Aluminium Nanofluid	0.5

Table 3.1	Sample	concentration
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#### **3.3 List of Equipment**

- Transmission Electron Microscope
- Ultrasonic Cleaner
- Viscometer
- Thermal Conductivity (KD2 Pro)

## 3.3.1 Transmission Electron Microscope

As opposed to a light microscope, the wavelength of illumination that is produced by an energized beam of electrons in TEM increases greatly the resolving capabilities. So, the main use of this technique is to examine the specimen structure, composition or properties in submicroscopic details so that this microscopy technique is significantly involved in numerous fields such as biological components, chemistry, materials science or electronics.

Using an electron microscope offers the advantage of increasing both the magnification of an object and the resolution over other imaging tools.



Figure 3.1 Transmission Electron Microscope

Resolution is defined as the ability to distinguish two separate items from one another. In a light microscope, to magnify the image, the deflection of the illumination from its path is accomplished through the use of glass lenses where the properties are determined by its shape and index of refraction. So, as the light travels into the lens, refraction occurs when it is travelling through a medium with a different refractive index. The situation in a TEM differs as there is no change in the refractive index of the medium when the illumination beam is deflected, the vacuum in the lens is the same as the vacuum in the column. Deflection is in this case only due to the electromagnetic properties of the lens which are defined by electromagnetic plates that are only able to influence the path direction of the electrons, since all of the electrons carry a negative charge. Then, they have similar function to glass lenses in that they do produce a deviation in the trajectory of the electrons from a point source which causes them to converge at a single focal point. By this way, the electron beam is influenced in order to focus it precisely.

There are essentially three types of lenses used to form the final image in the TEM. These are the condenser, objective, and projector lenses. The mean function of the condenser lens is to concentrate and focus the beam of electrons coming off of the filament onto the sample to give a uniformly illuminated sample. The objective lens and its associated pole pieces is the heart of the TEM and the most critical of all the lenses. It forms the initial enlarged image of the illuminated portion of the specimen in a plane that is suitable for further enlargement by the projector lens. Also, the TEM builds an image by way of differential contrast. Those electrons that pass through the sample go on to form the image while those that are stopped or deflected by dense atoms in the specimen are subtracted from the image. In this way a black and white image is formed. Some electrons pass close to a heavy atom and are thus only slightly deflected.

Thus many of these "scattered" electrons eventually make their way down the column and contribute to the image. In order to eliminate these scattered electrons from the image we can place an aperture in the objective lens that will stop all those electrons that have deviated from the optical path. The smaller the aperture we use the more of these scattered electrons we will stop and the greater will be our image contrast. Finally, one uses the projector lens to project the final magnified image onto the phosphor screen or photographic emulsion. It is in the projector lens that the majority of the magnification occurs. Thus total magnification is a product of the objective and projector magnifications. For higher magnifications an intermediate lens is often added between the objective and projector lenses. This lens serves to further magnify the image. The image is then projected onto either the fluorescent screen or onto the photographic film. Remember that the image is focused up at the objective lens. It is the focused image that is projected so the plane in which the final image appears is not critical and the image remains in focus regardless. What does change is the relative size of the projected image and thus the magnification on the screen and that on the photographic film will differ. Another important element of the TEM is the vacuum system. There are three main reasons why the microscope column must be operated under very high vacuum. The first of these is to avoid collisions between electrons of the beam and stray molecules. Such collisions can result in a spreading or diffusing of the beam or more seriously can result in volatization event if the molecule is organic in nature. Such volatizations can severely contaminate the microscope column especially in finely machined regions such as apertures and pole pieces that will serve to degrade the image.

When examination of a specimen has to be performed, the extreme conditions that exists inside the TEM have to be considered, as for ex. high vacuum or intense heat generated by the beam of electrons. For a conventional TEM analysis, a specimen has to be reasonably dried and thin for ensuring electron transparency. In general, a sample has to follow certain conditions including a complete lack of water (as high vacuum conditions are used), an ability to remain stable when exposed to e-beam damage and the presence of both electron transparency and electron opacity zones. For that, specific specimen preparation steps are usually employed, including the use of fixatives, embedding resins and ultramicrotomes for shaving off electron transparent slices of material. As part of this, it is important to try and keep the sample in as near a natural state as is possible.

#### **3.3.2 Ultrasonic Cleaner**

Ultrasonic cleaning uses high frequency sound waves to agitate in a liquid. Cavitation bubbles induced by the agitation act on contaminants adhering to substrates like metals, plastics, glass, rubber, and ceramics. This action also penetrates blind holes, cracks, and recesses. The intention is to thoroughly remove all traces of contamination tightly adhering or embedded onto solid surfaces. Water or other solvents can be used, depending on the type of contamination and the work piece. Contaminants can include dust, dirt, oil, pigments, rust, grease, algae, fungus, bacteria, lime scale, polishing compounds, flux agents, fingerprints, soot wax and mold release agents, biological soil like blood, and so on. Ultrasonic cleaning can be used for a wide range of work piece shapes, sizes and materials, and may not require the part to be disassembled prior to cleaning. Objects must not be allowed to rest on the bottom of the device during the cleaning process, because that will prevent cavitation from taking place on the part of the object not in contact with water.

Ultrasonic activity (cavitation) helps the solution to do its job; plain water would not normally be effective. The cleaning solution contains ingredients designed to make ultrasonic cleaning more effective. For example, reduction of surface tension increases cavitation levels, so the solution contains a good wetting agent (surfactant). Aqueous cleaning solutions contain detergents, wetting agents and other components, and have a large influence on the cleaning process. Correct composition of the solution is very dependent upon the item cleaned. Solutions are mostly used warm, at about 50–65 °C (122–149 °F), however, in medical applications it is generally accepted that cleaning should be at temperatures below 38 °C (100 °F) to prevent protein coagulation.



Figure 3.2 Ultrasonic cleaner

Water-based solutions are more limited in their ability to remove contaminants by chemical action alone than solvent solutions; e.g. for delicate parts covered with thick grease. The effort required to design an effective aqueous-cleaning system for a particular purpose is much greater than for a solvent system.

Some better machines (which are not unduly large) recycle the hydrocarbon cleaning fluids. Three tanks are used in a cascade. The lower tank containing dirty fluid is heated causing the fluid to evaporate. At the top of the machine there is a refrigeration coil. Fluid condenses on the coil and falls into the upper tank. The upper tank eventually overflows and clean fluid runs into the work tank where the cleaning takes place. Purchase price is higher than simpler machines, but such machines are economical in the long run. The same fluid can be reused many times, minimizing wastage and pollution.

#### 3.3.3 Viscometer

The Brookfield Viscometer or Rheometer is a precise torque meter which is driven at discrete rotational speeds. The torque measuring system, which consists of a calibrated beryllium-copper spring connecting the drive mechanism to a rotating cone, senses the resistance to rotation caused by the presence of sample fluid between the cone and a stationary flat plate.

The resistance to the rotation of the cone produces a torque that is proportional to the shear stress in the fluid. This reading is easily converted to absolute centipoise units  $(mPa \cdot s)$  from pre-calculated range charts. Alternatively, viscosity can be calculated from the known geometric constants of the cone, the rate of rotation, and the stress related torque.

The correct relative position of cone and plate is obtained by following a simple mechanical procedure without the need for external gauges or supplementary instrumentation. The stationary plate forms the bottom of a sample cup which can be removed, filled with .5 ml to 2.0 ml of sample fluid (depending on cone in use), and remounted without disturbing the calibration. The sample cup is jacketed and has tube fittings for connection to a constant temperature circulating bath.

The system is accurate to within  $\pm 1.0\%$  of full scale range. Reproducibility is to within  $\pm 0.2\%$ . Working temperature range is from 0°C to 100°C. Various cone spindles are available for use with the Brookfield instruments. One cone spindle is provided with the instrument and is calibrated for use with the sample cup. Additional cone spindles may be purchased and will be calibrated for use with the same sample cup.

This equipment provides a wide variety of shear rates and viscosity ranges, which can be further extended by the use of interchangeable cone spindles. Different models can be selected to meet the specific range of viscosities and shear rates required. The small sample volume required permits rheological evaluations to be made on materials where sample availability is limited, such as biological fluids and thick film coatings that contain precious metals. All wetted parts are stainless steel for corrosion resistance and ease of cleaning. Optional purge fitting, luer fitting, and embedded temperature probe available.

# **CHAPTER 4**

# **RESULTS AND DISCUSSION**

# 4.1 Transmission Electron Microscope Result



Figure 4.1. Copper nanofluid

Composition : 1.2 grams copper oxide , 75 ml isopropanol Concentration : 0.2M Magnification : 100k Nano particle size : 16.12nm



Figure 4.2 Copper nanofluid

Composition : 1.2 grams copper oxide , 75 ml isopropanol Concentration : 0.2M Magnification : 200k Nano particle size : 13.18nm



Figure 4.3 Copper nanofluid

Composition : 1.2 grams copper oxide , 75 ml isopropanol Concentration : 0.2M Magnification : 300k Nano particle size : 21.88 nm



Figure 4.4 Aluminium nanofluid

Composition : 1.5 grams aluminum oxide , 75 ml isopropanol Concentration : 0.2M Magnification : 100k Nano particle size : 11.40n



Figure 4.5 Aluminium nanofluid

Composition : 1.5 grams aluminium oxide , 75 ml isopropanol Concentration : 0.005M Magnification : 200k Nano particle size : 14.37nm



Figure 4.6 Aluminium nanofluid

Composition : 1.5 grams aluminium oxide , 75 ml isopropanol Concentration : 0.2M Magnification : 300k Nano particle size : 15.45 nm

# 4.2 Viscometer Result

# 4.2.1 Copper nano oil

Copper 0.2M		
RPM	temperature (°c)	cp (mPa·s)
60	25	170.9
60	30	162.8
60	35	150.5
60	40	144.3
60	45	132.7
60	50	119.8
60	55	110
60	60	97.6
60	65	88.9
60	70	76.5
60	75	64.6
60	80	55.6

 Table 4.1 Centipoise value for copper nano lubricant at 0.2M

Copper 0.3M		
RPM	temperature (°c)	cp (mPa·s)
60	25	254.5
60	30	246.7
60	35	233
60	40	221.5
60	45	210.6
60	50	200.9
60	55	193.8
60	60	185.3
60	65	171
60	70	165.6
60	75	148.9
60	80	133.3

Table 4.2 Centipoise value for copper nano lubricant at 0.3M

Copper 0.4M		
RPM	temperature (°c)	cp (mPa·s)
60	25	357.6
60	30	344.5
60	35	332
60	40	321.3
60	45	311.9
60	50	300.7
60	55	294.7
60	60	284.3
60	65	275.7
60	70	262.1
60	75	251.8
60	80	239.9

Table 4.3 Centipoise value for copper nano lubricant at 0.4M

Copper 0.5M		
RPM	temperature (°c)	cp (mPa·s)
60	25	480.7
60	30	466.5
60	35	452.3
60	40	437.6
60	45	422.1
60	50	412.9
60	55	400.7
60	60	391.8
60	65	383.6
60	70	374.4
60	75	358.9
60	80	342.1

 Table 4.4 Centipoise value for copper nano lubricant at 0.5M

Petronas gear oil		
RPM	temperature (°c)	cp(mPa·s)
60	25	118.2
60	30	116.9
60	35	115.8
60	40	110
60	45	107.8
60	50	101.8
60	55	90.9
60	60	83.4
60	65	74.8
60	70	69.7
60	75	57.7
60	80	50.4

 Table 4.5 Centipoise value for PETRONAS gear oil



**Figure 4.7** Graph of centipoise (mPa.s) versus temperature (°C)

Graph above shown the vary concentration of copper nano oil and gear oil versus temperature from range 25 - 80°c. Pattern of the graph shown that the value of centipoise is decreasing due to the increasing of temperature which means the viscosity of the sample is decreasing when the temperature was raised. For the conclusion centipoises of copper nano oil and gear oil are inversely proportional with temperature.

# 4.2.2 Aluminium nano oil

Aluminium 0.2M		
RPM	temperature (°c) cp (mPa-	
60	25	165.4
60	30	153.2
60	35	144.7
60	40	137.7
60	45	121.4
60	50	110.7
60	55	101.2
60	60	90.3
60	65	82.1
60	70	70.9
60	75	63.8
60	80	51.1

Table 4.6 Centipoise value for aluminium nano lubricant at 0.2M

Aluminium 0.3M		
RPM	temperature (°c)	cp (mPa·s)
60	25	238
60	30	225.6
60	35	210.8
60	40	200.9
60	45	193.2
60	50	184
60	55	170.7
60	60	163.3
60	65	154.5
60	70	141.1
60	75	130.9
60	80	122.5

 Table 4.7 Centipoise value for aluminium nano lubricant at 0.3M

Aluminium 0.4M		
RPM	temperature (°c)	cp (mPa·s)
60	25	324.5
60	30	310.9
60	35	300.7
60	40	291.9
60	45	284.3
60	50	272.7
60	55	260.6
60	60	254.4
60	65	241
60	70	220.9
60	75	203.9
60	80	190.6

Table 4.8 Centipoise value for aluminium nano lubricant at 0.4M

Aluminium 0.5M		
RPM	temperature (°c) cp (mPa-	
60	25	440.9
60	30	421.7
60	35	404.3
60	40	392.1
60	45	380.8
60	50	366.5
60	55	349
60	60	330.8
60	65	324.4
60	70	306.7
60	75	289
60	80	274.4

Table 4.9 Centipoise value for aluminium nano lubricant at 0.5M

Petronas gear oil		
RPM	temperature (°c) cp(mPa·s	
60	25	118.2
60	30	116.9
60	35	115.8
60	40	110
60	45	107.8
60	50	101.8
60	55	90.9
60	60	83.4
60	65	74.8
60	70	69.7
60	75	57.7
60	80	50.4

Table 4.10 Centipoise value for PETRONAS gear oil



**Figure 4.8** Graph of centipoise (mPa.s) versus temperature (°C)

Graph above shown the vary concentration of aluminium nano oil and gear oil vs temperature from range 25 - 80°c. Pattern of the graph shown that the value of centipoise is decreasing due to the increasing of temperature which means the viscosity of the sample is decreasing when the temperature was raised. For the conclusion centipoises of aluminium nano oil and gear oil are inversely proportional with temperature.

# 4.3 Thermal Conductivity Result

Concentration (M)	Temperature (°C)	Thermal Conductivity , k ( W/m.K)
0.2	27.83	0.131
0.2	27.84	0.134
0.2	27.35	0.135
0.2	27.13	0.137
0.2	27.13	0.135
average	27.456	0.1344

# 4.3.1 Aluminium Nanofluid

Table 4.11 Thermal conductivity, k (W/m.k) value for aluminium nanofluid at 0.2M



Figure 4.9 Graph of thermal conductivity,k (W/m.k) versus time (min)

Figure 4.9 shown the value of thermal conductivity,K versus time. For the first 15 minutes until 60 minutes, value of k is increasing. Peak value of k for aluminium nano fluid is 0.137W/m.k. For the last 15 minutes, the value is decreasing which the reading is being stabilized.

Concentration (M)	Temperature (°C)	Thermal Conductivity , k ( W/m.K)
0.3	27.84	0.134
0.3	27.52	0.138
0.3	27.17	0.138
0.3	27.37	0.138
average	27.475	0.137

Table 4.12 Thermal conductivity, k (W/m.k) value for aluminium nanofluid at 0.3M



Figure 4.10 Graph of thermal conductivity, k (W/m.k) versus time (min)

Figure 4.10 shown the value of thermal conductivity,K versus time. For the first 15 minutes until 30 minutes, value of k is increasing from 0.134 to 0.138. Peak value of k for aluminium nano fluid at 0.3M is 0.138W/m.k. Value of K is constant from minutes 30 until 60. This means the reading is stable already.

Concentration (M)	Temperature (°C)	Thermal Conductivity , k ( W/m.K)
0.4	28.11	0.138
0.4	28.85	0.139
0.4	29.18	0.138
0.4	29.22	0.139
Average	28.84	0.1385

Table 4.13 Thermal conductivity, k (W/m.k) value for aluminium nanofluid at 0.4M



Figure 4.11 Graph of thermal conductivity,k (W/m.k) versus time (min)

Figure 4.11 shown the value of thermal conductivity, K versus time. For the first 15 minutes, value of K is increasing from 0.138W/m.k. 15 minutes later, K value is increasing to 0.139W/m.k. At 60 minutes, value of K is decreasing once more to 0.138W/m.k. At 75 minutes, value of K is at 0.139W/m.k which means the thermal conductivity in the fluid is already stable.

Concentration (M)	Temperature (°C)	Thermal Conductivity , k ( W/m.K)
0.5	28.57	0.128
0.5	28.1	0.134
0.5	27.65	0.133
0.5	27.88	0.134
average	28.05	0.13225

Table 4.14 Thermal conductivity, k (W/m.k) value for aluminium nanofluid at 0.5M



Figure 4.12 Graph of thermal conductivity,k (W/m.k) versus time (min)

Figure 4.12 shown the value of thermal conductivity, K versus time. For the first 15 minutes until 30 minutes, value of k is increasing from 0.128W/m.k to 0.134W/m.k. From 30 minutes until 45 minutes, K value is decreasing from 0.134W/m.k to 0.133Wm.k. For the last 15 minutes, K value is increasing to 0.134W/m.k.

## **CHAPTER 5**

## CONCLUSION AND RECOMMENDATIONS

#### 5.1 Conclusion

As the conclusion, the invention of nano lubricant is a very recent and brilliant idea for the development of lubrication industry. Besides that, the usage of the nano lubricant is the most important factor that make this type of oil becomes more familiar in the industry and other usage. Theoretically, nano oil can simply replace the standard gear oil as its behavior in viscosity. Besides that, the preparation of nano oil is quite simple than standard gear oil.

# 5.2 Recommendations

For the recommendation, invention of nano oil in industry must be highlighted as its capability in viscosity. Different concentration of nano fluid give effect to the value of centipoises. In viscometer testing, the heating medium of it is jacket of the cylinder. So to maximize the effect of temperature on this experiment, we can extend the time of the testing to get accurate reading. Besides that, usage of ultrasonic cleaner can be maximized by increasing the time of dispersing. As example, we actually took time for about 30 minutes to dispersed the nano fluid. So we can add it up to 1 hour to make sure that the nano fluid is perfectly mixed. Other than that, while using transmission electron microscope the measurement of the nano particle size can be made several times to get the

best reading. Snapping a lot of samples' picture is one of the best way to make sure that the nano particles are perferctly dispersed in base fluid.

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## **APPENDIX A**

## **Sample Concetration**

Copper Oxide, CuO  $\frac{1.2g \ CuO}{79.55g/mol} = 0.015 \ \text{mol CuO}$   $\frac{0.015 \ mol \ CuO}{0.075L} = 0.2 \text{M}$   $\frac{1.8g \ CuO}{79.55g/mol} = 0.023 \ \text{mol CuO}$   $\frac{0.023 \ mol \ CuO}{0.075L} = 0.03 \ \text{mol CuO}$   $\frac{2.4g \ CuO}{79.55g/mol} = 0.03 \ \text{mol CuO}$   $\frac{0.03 \ mol \ CuO}{0.075L} = 0.4 \ \text{M}$   $\frac{3.0g \ CuO}{79.55g/mol} = 0.038 \ \text{mol CuO}$   $\frac{0.038 \ mol \ CuO}{0.075L} = 0.5 \text{M}$ 

Concentration = mol/volume (L)

Aluminium Oxide,  $Al_2O_3$  $\frac{1.5g \ Al_2O_3}{101.96g/mol} = 0.015 \ \text{mol} \ Al_2O_3$   $\frac{0.015mol \ Al_2O_3}{0.075L} = 0.2M$   $\frac{2.3g \ Al_2O_3}{101.96g/mol} = 0.023 \ \text{mol} \ Al_2O_3$   $\frac{0.023mol \ Al_2O_3}{0.075L} = 0.3M$   $\frac{3.0g \ Al_2O_3}{101.96g/mol} = 0.029 \ \text{mol} \ Al_2O_3$ 

$$\frac{0.029mol\ Al_2O_3}{0.075L} = 0.4 \text{ M}$$

$$\frac{3.8g \, Al_2 O_3}{101.96g/mol} = 0.037 \, \text{mol} \, Al_2 O_3$$

 $\frac{0.037mol\ Al_2O_3}{0.075L} = 0.5M$