



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

CASE STUDY EFFECT OF TEMPERATURE ON THE NANOPARTICLE STABILITY PROPERTIES

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Engineering Technology (Maintenance Technology)(Hons.)

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DECLARATION

I hereby, declared this report entitled “Case Study Effect of Temperature on The Nanoparticle stability Properties” is the results of my own research except as cited in references.

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APPROVAL

This report is submitted to the Faculty of Engineering Technology of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Mechanical Engineering Technology (Maintenance Technology) with Honours. The member of the supervisory is as follow:



ABSTRAK

Kajian ini memberi tumpuan kepada kesan suhu ke atas kestabilan gerakan nanopartikel. Ini kerana sifat nanopartikel yang mudah untuk bergumpal apabila terhidrat. Selain itu nanopartikel juga menghadapi masalah pemendakan dan keapungan apabila di campur dengan minyak pelincir sebagai bahan tambahan. Prosedur eksperiment ini telah dijalankan untuk mengenalpasti kesan suhu yang berbeza pada sebatian minyak enjin konvensional gred SAE 15W40 ditambah dengan nanopartikel sebagai sampel minyak-nano. Satu set suhu telah ditetapkan dalam kajian ini bermula pada suhu bilik iaitu 30°C, 40°C dan 70°C. Nilai keserapan nano minyak telah diukur menggunakan UV-spektrometer sebagai keputusan kuantitatif. Sampel minyak nano juga diperhatikan dan imej sampel direkodkan sebagai keputusan kualitatif. Nanopartikel yang digunakan dalam eksperiment ini adalah hexagonal boron nitride (hBN), alumina (Al_2O_3) dan zircornia (ZrO_2). Hasil kajian menunjukkan pada suhu tertinggi iaitu 70°C, kestabilan minyak nano dengan hBN, Al_2O_3 dan ZrO_2 nanopartikel adalah lebih baik. Keputusan kajian juga menunjukkan ZrO_2 nanopartikel telah tersebar dengan baik pada setiap suhu yang digunakan. Selain itu, ZrO_2 nanopartikel menunjukkan kestabilan yang baik sehingga dua bulan walaupun ditingalkan pada suhu bilik jika dibandingkan dengan hBN dan Al_2O_3 nanopartikel.

ABSTRACT

This study focused on the effect of the temperature on the nanoparticles stability properties. This is due to the nature of the nanoparticle where it easily to aggregate when hydrated. The nanoparticles also have a suspension or sedimentation problem when it been added in the lubricant as an additive. An experimental procedure was conducted to identify the effect of difference temperature in homogenize the conventional engine oil SAE 15W40 grade mixed with nanoparticles as Nano-oil sample. A set of temperatures were used in this experiment started at room temperature 30°C, 40°C and 70°C. The absorbance values of the nano-oil sample were measured by using UV-spectrometer as quantitative results. The samples also was observed periodically by capturing the image of the Nano-oil as a qualitative result. The nanoparticles used in this study were hexagonal boron nitride (hBN), alumina (Al_2O_3), and zirconia (ZrO_2). The stability of the nano-oil with hBN, Al_2O_3 and ZrO_2 nanoparticles much better at temperature 70°C. Nano-oil with ZrO_2 nanoparticles shows dispersion well at every temperature. Furthermore, it was still stabled up to two months even though was left at room temperature compare to other samples containing hBN and Al_2O_3 nanoparticle.

DEDICATION

To my beloved parents



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First and foremost, I express my profound gratitude to my creator, God almighty for his unfading love upon my life. Apart from that, I would like to thank to my supervisor, Dr Muhammad Ilman Hakimi Chua Bin Abdullah, for his guidance and support through this case study. I cannot thank my family enough for all the prayers they been offering on my behalf, especially my father Abd Manaf Bin Hshim, my only brother Mohd Zulhasnan, my sisters Qairunnisa and Dzuhanisa. I say a big thank you. Lastly, my sincere thanks to all my friends that always with me during good and bad times in the completion of the work of this thesis.

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LIST OF BBREVIATIONS, SYMBOLS AND NOMENCLATURE

hbN	-	Hexagonal Boron Nitride
ZrO ₂	-	Zirconia Oxide
TiO ₂	-	Titanium dioxide
ZnAl ₂ O ₄	-	Zinc Aluminate
Al ₂ O ₃	-	Alumina Oxide
ZnO	-	Zinc Oxide
Fe	-	Ferum
Cu	-	Copper
Co	-	Cobalt
Fe ₃ O ₄	-	Ferum Oxide
SDS	-	Sodium sulfate
SEM	-	Scanning Electron Method
SAE	-	Society Automotive Engineering
PAO	-	Polyalphaolefins
Talc	-	Soapstone
Cp _{nf} /Cp	-	Specific Heat Capacity

CHAPTER 1

INTRODUCTION

1.0 INTRODUCTION

Since the roman era lubricant have been introduced, the often used of lubricant is in term of liquid phase such as engine oil, gear oil including water. The function of the lubricant is to minimize the friction, heat and wear between mechanical parts in contact with each other. There are three types of lubricant which are solid, semi-solid and liquid. Nowadays the study of nanoparticles is receiving much more attention, this is due to their ability when added in lubricating oil which helps to reduce the interfacial friction. More than that, the addition of nanoparticle can improve the ability of the load-bearing capacity of part and can reduce the friction between two parts, due to this advantages nanoparticle has a great potential as lubricant additives. (Zhang et al. 2014). Nanoparticles have been found one of the successful additives into the lubricant. Low concentration of the nanoparticle approximately between 0.2% and 0.3% which been added to lubricant actually can improve the tribological properties. (Qiu et al 1999).

However, introducing nanoparticles in the lubricant is a complicated task due to the size, shape, concentration and materials properties of the nanoparticles itself. Because of the high surface energy regardless of the size nanoparticles are effective to aggregate or coagulate once it added to fluids particularly when they are presented to varieties in temperature and don't move consistently inside a colloidal framework . (Stachowiak, et al, 2014). According to Keller et al. (2010), most of the nanoparticle easy to aggregate when they are hydrated. Beside that Wu et al. 2008 also mention, due to the large ratio of surface area to volume (small particle size) the nanoparticles possess a high surface energy and consequently create an aggregate form to minimize these surface energy. The nanoparticles also tend to sediment due to the gravitational force. Nanoparticle usually collated to the Van der Waals attractions which also a factor causing it is to sediment. At

the point when nanoparticle was added to the diverse arrangement at various temperature sedimentation properties also are different.

Address to mentioned issues, the stability of nanoparticles play an importance role in producing a high-quality lubricant. The nanoparticles stability usually be attained through absorption analysis which refers to the balancing, the goals for repulsive forces is to prevent the different particle from come close end sufficient for the connecting with van der Waals vitality from made agglomerated of atoms on the pigment surface.(H. Amiruddinet all. 2015). Therefore, it is importance to investigate the effect of difference temperature of the nano oil sample on the stability of nanoparticles.

1.1 PROBLEM STATEMENT

Most present lubricant oils that contain petroleum base stocks, which are poisonous to the environment and hard to discard after use. Natural concern proceeds to an increment of contamination from over the excessive lubricant use and transfer, particularly add pollution to the environment. Due to this problem some researchers started to improve the oil by adding the additives. Some nanoparticles were added to the lubricant oil in order to improve the characteristic of the lubricant. The researchers are also started to search renewable and biodegradable lubricants such as oil lubricant, grease or solid lubricants. Some studies show by adding nanoparticles in the lubricant it can help to reduce the coefficient of friction reduces the average wear scar diameter in the oil. According to Gubarevich et al. (2004) by adding Cu nanoparticles into the oil, the COF at room temperature, 37°C, 50°C, 80°C, 110°C, and 140°C were reduced by 5% 8% 10% 15% and 20%. Hence, it also reduces the average WSD of the stationary balls at room temperature by 13% 16% 21% 23% and 25%. Since the 1920s, the used of additives for lubricating oil are increased tremendously. This is belief due to the characteristic of the nanoparticles that able to improve the stability of lubricant.

Nevertheless, there is also have some limited due to the characteristic of the nanoparticle once it has introduced to the lubricant oil as example nanoparticle are easy

to sedimentation or suspension once it introduces to the lubricant oil. According to (Wu et al 2007) and friends, they said that the nanoparticles might disrupt the mechanisms in the mixed and boundary lubrication indirectly and affected the performance of the lubricant. As an example, the particles can affect the oil viscosity, thermal properties, and thermal stability. Another that the particles can also induce abrasive wear which will result in a higher wear rate. Nanoparticle also is easy to coagulate or aggregate once have been introduced to the lubricant oil especially when it exposed to the temperature.

1.2 OBJECTIVE

From the background and the problem statement that have been stated, the objectives of this research are:

- i. To identify the effect of difference temperature in homogenize the nano-oil sample.
- ii. To determine the absorbance of the nano oil when added with nanoparticles

1.3 SCOPE OF WORK

- i. Identify the temperature that needs to be used for the stability test on the nano oil sample.
- ii. Determining the absorbance of the nano oil when added with nanoparticles by using UV-spectrometer

CHAPTER 2

LITERATURE REVIEW

2.0 Lubricant

Lubricant is an important thing in industries, especially when it involve in automotive and machinery. The function of the lubricant is to reduce the wear, heat, and friction between mechanical parts that have contact with each other (Yazawa et al 2014). Since the days of roman era lubricant has been used. They use many types of lubricants including water. Lubricant will act to keep the operating parts from failure and make the operation goes smoothly. In the industries lubricant has been used widely in a various application for example lubricant have been used as transmission lubrication. The advantages of the lubricant are lubricant also, can act as coolant due to their characteristics reduce the production of heat. Another from that lubricant also can reduce the wear and make the operation of a machine more efficient. Lubricants have been classified on the basis of their physical state which contains liquid lubricants, semi-solid lubricants, and solid lubricants.

Liquid lubricant or usually called as lubricating oil also can be classified into three categories which are animal and vegetable oil, mineral and PAO. A good liquid lubricant will consist all this characteristic like low freezing point, high resistance to oxidation, high boiling point, anti- corrosion and stability to the variations at the operating temperatures. The other type of lubricant are solid lubricant and semi-solid lubricant. The solid lubricant is a lubricant that can be found in dry powder. The majority basic solid lubricant is graphite, tungsten disulfide, molybdenum disulfide and zinc oxide as basic material. This lubrications can be applied by rubbing at the surface which can self-lubricate cages in ball bearing with a correct technique (Roberts et al 1990). Usually, this lubricant can hold out with high temperature up to 650°C and can be applied in the continuously operating system. Furthermore, this lubricant also can be used as additive to the minerals oils and grease in order to improve the load

carrying capacity of the lubricant. Other solid lubricants that have been used are soapstone (talc) and mica. Semi-solid lubricants are lubricant from the combination of lubricating oil with thickening agents is named as grease. This grease usually acted as the anti-friction of the roller bearing and other industrial machines (Garcés et al 2014). Another from that this grease usually used for heavy load application at low speed. Compare to lubricating oil the internal resistance of semi-solid lubricants is higher. Because of this, the used of semi-solid lubricants is much better compared to solid lubricants. The most important of lubrication is to guarantee all the machines and motors oil can operate work successfully. Without lubrication oil working parts can't work properly it also can cause the breakdown to the machine.

In order to improve the lubrication some additive have been added. An example of the additive that usually have been used in the lubricant is Cu nanoparticle. According to the (M. Asrul 2013) in tribology, nanomaterials have been added into lubricating oil in order to improve friction reduction properties, anti-wear, and extreme pressure. More than that additive also can act as a protective to the mechanism that can make harmful combustion product and make multi-functioning lubricating oil. However, the importance of additive that should have is anti-oxidant. This is in order reduce the rate of degradation, an extreme pressure that usually used in gear lubricant and detergent or dispersant used in engine oil to prevent carbon deposits. According to Rudnick et al (2009), there is have a lot of lubricant additives that have been introduced in the industries . As an example, the common lubricant additives being used in industries have shown in table 2.1.

Additives		Effect	Common chemicals
Deposit control additives	Anti-oxidants	By preventing lubricant form oxidation, these products prevent the formation of corrosive components. Anti-oxidants act by two different behaviors: Peroxide inhibition and radical scavenging. (Barnes, Bartle, Thibon et al 2001)	-Sulfur-nitrogen compounds -Phosphorous compounds - Sulfur-phosphorous compounds -Aromatic amine compounds -Hindered phenolic (HP) compounds -Organo-copper compounds
	Zinc dithiophosphates (ZDDP)	ZDDP is commonly used as anti-oxidant and wear additive. ZDDP under high temperature and pressure create a glassy phosphate layer on Fe-based surfaces and reduce wear and friction.	ZDDP
	Ashless phosphorusecontaining lubricating oil additives	This group of additives, in contrast to ZDDP, create a smoother and thinner films than can protect surface against wear. They proved to have lower friction than ZDDP (Ribeaud. et al 2006)	_ Phosphate esters: a) Neutal phosphates b) Acid phosphates (Nonethoxylated, Ethoxyalkyloxy) _ Phosphites and phosphonates _ Alkyl or aryl

			phosphites/ phosphonates
	Detergents	Detergents by containing base components neutralize acids that can attack metal surfaces. Other function of detergents is to suspend polar-oxygenated components in oil (Hudson et al 2006).	The metal salts of following acids are being used as detergents: _ arylsulfonic acids _ alkylphenols _ carboxylic acids _ petroleum oxidates
	Dispersants	Combined with detergents, dispersants are designed to suspend the insoluble particles and contaminants in oil and keep the surfaces clean. Having polar groups, dispersants can keep nonpolar molecules suspended in lubricant (Sassiat et al 1995)	
Film-forming additives	Solid lubricants as friction modifiers	solid lubricants considerably reduce friction	_ Graphite _ Molybdenum disulphide

		between surfaces where liquid lubricants do not perform desirably. Five main properties that these group should have are: Yield strength, Adhesion to substrate, Cohesion, Orientation, Plastic flow	_ Boron nitride _ Polytetrafluoroethylene
	Organic friction modifiers	In order to adjust friction characteristics and improve the lubricity and energy efficiency, friction modifiers are added in boundary and/or mixed lubrication conditions. Friction is known to be responsible for about 20e25% of fuel energy consumption (Tang Z, et al 2014)	Organic friction modifiers can be found in the following categories: _ Carboxylic acids or their derivatives, for example, stearic acid and partial esters _ Amides, imides, amines, and their derivatives, for example, oleylamide _ Phosphoric or phosphonic acid derivatives _ Organic polymers
Anti-wear additives and extreme-pressure	Ashless anti-wear and extreme-pressure additives	This term is applied to group of extreme pressure and anti-wear additives	_ Sulfurized Isobutene _ Active-Type

Table 2.1 Common lubricant additives being used in industries

Shahnazar et al 2015

2.1 Liquid as lubricant

The liquid lubricant can be categorized into three types. There are vegetable or animal, PAO and mineral base oil. The lubricant that made from vegetable oil as of now be utilized by human since antiquated time. As stated by (Sharma et al. 2006), since 1650 BC olive oil was used as a lubricant. Nowadays have a lot type of vegetable oils have been produced commercially and it is being utilized as a part of numerous applications particularly in industry and also as food. Vegetable oil is one of liquid lubricant that has gain attention among the current researchers, this is due to their environment friendly characteristic. According to Sharma et al. (2006), vegetable oil is a biodegradability, posse adequate performance in a variety of applications and renewability resource. However, vegetable oil faced a few problems that makes this oil difficult to accept in the industry. As specified by Erhan et al (2006) vegetable infamous because of hydrolytic stabilities and poor low-temperature characteristics, and easy to oxide.

Mineral based oil is one of widely lubricating oil that has been used. In generally mineral oil is an oil that produced from petroleum oil. However, mineral oil also can be acquired from the same sources like tar-stands and oil shales. Usually, lubrication that comes from used oil is fraction from distilling petroleum. Mineral oils have been given into three type there are paraffinic, aromatic and lastly naphthenic. Paraffinic usually produced by solvent extraction process or hydrocracking. The used of paraffinic oils usually for industrial lubricants and as processing oils in rubber, paper industries, industrial lubricants and textile. However, aromatic are results of refining procedure in assembling of paraffinic oils. Commonly aromatic oil is utilized for adhesives, manufacturing seal compounds and also as asphalt production and plasticizers in rubber. While for naphthenic oil is created from crude oil distillates. Naphthenic oils are utilized as a part of moderate temperature applications, mostly to manufacture transformer oils and metal working liquids.

The most popular synthetic lubricant are Polyalphaolefins (PAO). The characteristic and chemical structure of PAO are similar to mineral oils. Furthermore, PAO or also known as synthetic hydrocarbons were produced by alpha olefins (polymerization of hydrocarbon molecules. The advantages PAO oil are thermal

stability, low volatility, good uses lubricity and chemical inertness. Due to this advantages, PAO is good for high temperatures although the operating in high temperature or low temperature. Usually, PAO oil is used for gears compressor and engine oil. PAO have been divide into three categories there are multi-grade, semi-synthetic and fully synthetic.

2.1.1. Fully synthetic

Fully synthetic oils are absolutely manufactured with no mineral oil. They offer the largest amounts of execution and are a fundamental essential requirement for many modern engines. Synthetic lubricants are created from chemical reactions through the temperature from a specific recipe from component and also from the precise application of pressure. The advantages of synthetic lubricants are it can use in extremely higher temperature from refined lubricants without breaking down. Their imperviousness to breakdown likewise permits them to be utilized for long periods compare than refined lubricants.

2.1.2 Semi-synthetic

Semi-synthetic oils are also known as synthetic blends. Semi-synthetic oils are blends from conditional or minerals oils that contain below than 30% of synthetic oils. The advantages of semi-synthetic oils are it can provide engine defense for somewhat higher temperature. Another from that it is suitable for heavy loads and they are not as subject to vanishing as the regular mineral oil. Semi-synthetic oils have been created in order to extend lubricant life and improve the viscosity index for lubricant. It also has been created for reducing the cost of than a full synthetic oil. In 1966, the first semi-synthetic oil has been introduced.

2.1.3 Multi-grade

Multi-grade oil is oil that contains viscosity modifier additives. The function of this additive is to allow the oil to flow in the different condition such as cold and hot. Multi-grade oil has been created into two numbers. The first one number for multi-grade oil is followed by letter W. The meaning of this W are stated for viscosity (weight) of the oil in winter (cold). The second number of multi-grade are meaning for the viscosity of the oil under normal engine temperature. It is critical for the oil to be sufficiently thin to stream effortlessly when cool. This shields the engine parts from undue wear in cool beginning conditions.

2.2 Properties of lubricant

The main properties of lubricants, which are usually indicated in the technical characteristics are corrosion stability, cloud point, and pour point, flash point and fire point, aniline point and viscosity.

Viscosity is a resistance the liquid to flow. Viscosity is the most important thing in order to get a good characteristic of lubricant. When the viscosity of the oil is low it can make the lubricant between two part cannot be maintained. As stated by D et al. (2014), viscosity is the measure of its internal friction. However when the viscosity of the oil is too high it will result in the excessive friction. Furthermore, temperature also plays a big role to the oil viscosity. This is because when the temperature increases the viscosity of the oil will decrease thus when the operating temperature increased, the lubricant oil will be thinner. A good lubricant viscosity will not much affected with temperature. It is in order to use the oil continuously in various temperature conditions. A part from viscosity is Viscosity index (V.I), it is a subjective scale that uses for measure the viscosity oil rate that changes with temperature. The lubricating viscosity of oil will decrease quickly if the lubricating temperature increases, it means the viscosity index for that lubricant is low. However if the viscosity lubricating oil is marginally influenced by increasements of temperature it shows that lubricant has high viscosity index (Noria et al. 2012).

Cloud point happens at the time when the temperature of lubricant oil have been cooled slowly and the oil becomes overcast or foggy in appearance (Huang et al 2006). On the other hand, pour point happens when the lubricant oil ceases to flow with temperature. The functions of cloud point and pour point to demonstrate the suitability of lubricant oil in cold conditions. The lubricant that used on the machine with low temperatures should have low pour point or on the other hand solidification of lubricant will make the machine not function properly and cause the jamming to the machine. This is because when the waxes are contained in the lubricant it will increase the pour point.

In addition, flash point happens when the temperature of the lubricant oil at the lowest temperature and it enough for lubricant vapors that ignite when a tiny flame is close. However for the fire point flame point is the most minimal temperature at which the vapors of the oil blaze consistently for no less than five seconds, when the tiny fire is brought close it. As the same majority case, the flash point is 5° C to 40° C lowest compare to the fire points. Flash point and fire point not affected by lubricating property, however, this is important when oil have been exposed to the high-temperature service. On the other words, a lubricant with the good condition should have a flash point at the point above the temperature which lubricant to be used. This is in order to reduce the risk if the fire occurred when use the lubricant.

Corrosion stability test will be determined by using corrosion test. The procedure to doing this test by polished the copper strip that immersing in the lubricant oil with specific time at particular temperature. After the specifying time, the copper strip will take out from lubricant and the corrosion effect will be examined. If the strip is affected or damaged it show that lubricant contains an active chemical substance that can cause the corrosion to the copper strip. A good lubricant will not affect the copper strip. In order to slow down the corrosion effect of the lubricant oil, the special inhibitors will be added to the lubricant. Usually, inhibitors that have been used are the organic compound. Commonly organic compounds contain Cr, Pb, P, and Bi (Sundeep et al 2014).

2.3 Nanoparticles

Nanoparticles are known as one of additive that can be used in lubricant. Nanoparticle has a great potential in order to improve the characteristic of lubricant as reported by Shahnazar et al (2016). Addition of nanoparticles into the base oil it can improve the certain characteristic of oil such as friction and wear resistant. Lately, there are a lot of studies have proved that by added nanoparticles like metal, metal oxide, metal sulfides, and carbonate, borate carbon material, rare-earth compound and organic material into lubricants it is powerful in decreasing the wear and friction (Zhang et al. 2011). Nanoparticles have large surface area is anticipated that would improve the heat transfer and also stability of the nanofluid. The property of nanoparticle is used to create nanofluids with an unprecedented combination of two features that are highly desirable for heat transfer systems like high thermal conductivity and extreme stability (Agarwal et al 2016). However, a lot of studies need to be done about the effect of this nanoparticle especially about their stability in liquid phase condition. Shahnazar et al (2016) mention that nanoparticles addition can be categorized into four main groups such as metal, metal oxides, carbon nanomaterials nanoparticles and boron base.

Metals nanoparticles is a nanoparticle that has been one of the potential additives. These nanoparticles have been used in many different applications for example semiconductors, magnetics, catalyst and photonic fields. According to Choi et al. (2009), tribological adequacy Cu nanoparticles appeared as an excellent self-repairing properties, plus it friendly to the environment. However due to high surface action, these nanoparticles are just feebly good with base oils. (Shahnazar et al 2016). Nevertheless, this problem still can be improved by surface modification techniques. Base on Padgurskas et al (2013), due to the surface investigation by using Scanning Electron Microscope he found that when Fe, Cu, and Co nanoparticles were added to SAE 10 oil, it shows Fe, Cu, and Co nanoparticle exhibit the different behaviors (figure 2.1). However, Cu nanoparticles are the best effective wear resistance. Other than that, it was proves that the oil that added with nanoparticle will be great and effective compare to the oil did not have nanoparticles.

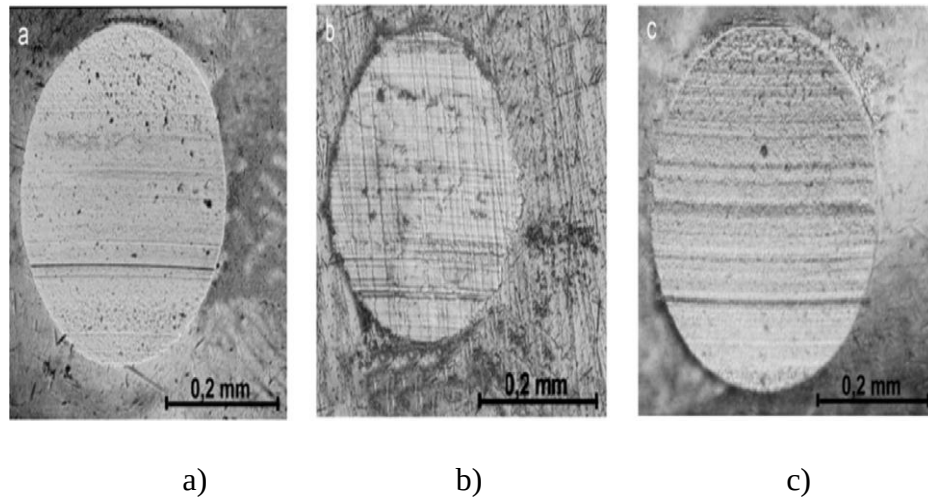


Figure 2.1 SEM images of scar resistance behavior (a) Fe, (b) Cu, (c) Co

(Shahnazar et al 2016).

Metal oxide usually added to lubricant base fluids as added substances, and the subsequent blend utilized is for anti-friction, and also for anti-wear applications. The example of metal oxides nanoparticle is nano-TiO₂, Nano-ZnO, and nano-ZnAl₂O₄. Nano-ZnO, this nano has grabbing considerable attention due to their characteristic of high surface energy, low melting point, large surface area, high diffusion, and easy sintering. (Shahnazar et al 2016). However this nanoparticle has low solubility when added to the oil, their dispersion in the base oil could not be a challenged. (Tang E et al. 2006). In the study, which was conducted by Jianhua et al (2011), they provide ZnO by homogeneous precipitation method and use lauryl sodium sulfate (SDS) as the surfactant. They are also investigating the anti-corrosion, oil solubility, and tribological properties by preparing nano-ZnO to act as a lubricant additive. The result that they get by using SEM showed the average size of ZnO particles reached 125 nm. Considering the solubility, the addition of ZnO from 1.0%, 2.0%, 3.0%, and 4.0%, causing the oil that tested have being clear and unstratified after being left for 10 days. Based on that ZnO nano-scale particles have been proved in order to reduce the wear of direct contact region of grinding by being keeping the sliding surfaces and framing a greasing up layer on moving surfaces. When the addition of surface-modified nano-sized ZnO particles was added to the base oil it can reduce the friction and anti-wear.

Lately, boron based nanoparticles have received a lot of attention among the researchers. This nanoparticle has been investigating because of their behavior due to

anti-wear and load carrying. Besides that this nanoparticle also known as eco-friendly and thermally stable, making them a reliable candidate for use as a lubricant oil additive. According to Hutchinson and Reid et al (2007), they found that boron nitride very excellent with high load and can withstand extremely high temperatures. Boron nitride also the best candidate for electrically insulating, nonreactive, thermally conductive, and it is white in appearance.

Regardless of the past few years, the study on nanoparticles among the researchers have found the nanoparticles have given a lot of advantages in order to improve the lubricant characteristic however it still have some challenges such as preparation in order to maintain homogeneous mixtures of nanostructured particles in oils. This is due to strong of van der Waals force between the particles. Because of this, it can make them aggregate in oils. Furthermore, various modification techniques also have to be used in order to investigate the stabilization of nanoparticles in all groups of base oils. Orderly to produce lubricants that have both physically and chemically stable.

2.4 Stability

The majority of nanoparticles when added inside the lubricant often accounted with suspension and sedimentation issues. One of the causes of this problem is due to the nature pull of gravity itself. In order to maintain the stability, a few type of testing base on surface residual over the area such as stability in suspension, thermal, and dispersion was conducted.

The stability of nanoparticle is also affected by activity of van der Waals. The repulsive force of van der Waals is to prevent another particle from approaching close enough to the attractive van der Waals forces, it can cause agglomeration. The stabilization of nanoparticles will be achieved through the absorption of stabilizing molecules on the pigment. In order to achieve the stabilization of pigment dispersion, there is have two principal mechanisms for this stabilization. Two principal mechanisms for this stabilization are electrostatic stabilization and steric stabilization. Amiruddin et al (2015). Base on the study that conducted by Cesarano et al. (1988) he

uses electrostatic stabilization method, found that suspension can be well-dispersed by using an ionic polymer dispersant and the ionic properties of water. Classic colloidal science explains electrostatic stabilization in terms of an electrical double-layers as shown in Figure 2.2 and Figure 2.3

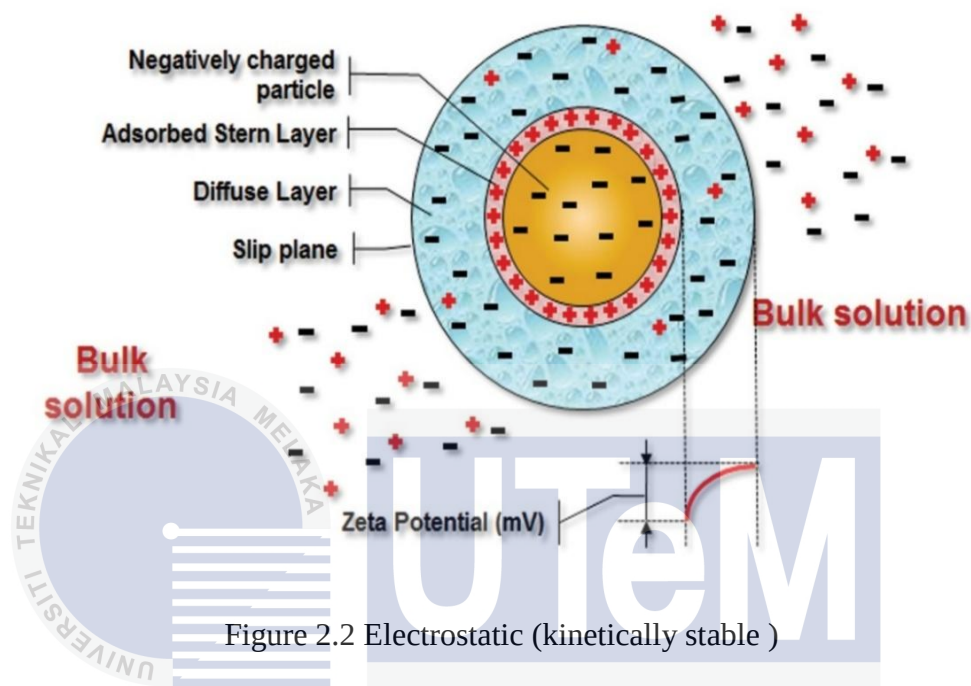


Figure 2.2 Electrostatic (kinetically stable)

(Sources <http://www.drillingcontractor.org/>)

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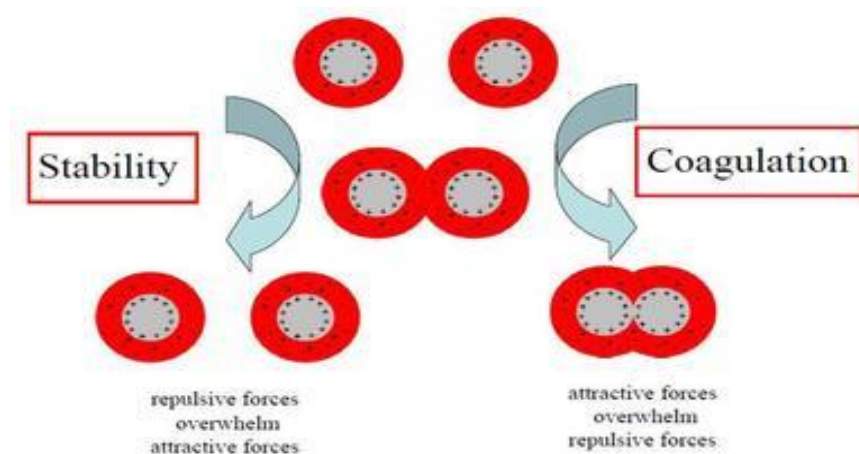


Figure 2.3 Repulsive and attractive forces

(Sources <https://imk209.wikispaces.com>)

Based on their high surface energy in most liquid usually, will face a few challenges especially when they are subjected to variations in temperature or pressure. This is due to their characteristic some nanoparticles will not disperse well once be added to the liquid phase or base oil. As example base on the study that conducted by Zhang et al (2014) he found that inorganic fullerenes are mineral particles that have the size between 50nm to 150nm and their shape such as cylindrical or spherical is difficult to disperse once introduced to the oil. This is due to their properties is different so they need surfactants to help particle well disperse. Thermal conductivity is an important parameter in order enhancing the heat transfer performance of a base fluid. However, there is still have a lot of study need to do about thermal stability according to various factor especially effect of temperature on nano oil, type of particles and size of particles.

2.4.1 Effect of temperature on the stability

Based on the past, the study on the effect of temperature on the stability of nano oil is one of the important things. According to Stachowiak et al. (2004) nanoparticles are easy to aggregate cluster or coagulate once it added to the most liquid, especially when they are subjected to variations in temperature. Hong et al. (2006) studied the effect of the agglomeration of nanoparticles on the thermal conductivity of nanofluids. They suggest that the clustering of nanoparticles is responsible for the large increment of the thermal conductivity of Fe nanofluids. The thermal conductivity of nanofluids increases with increasing volume concentration. The viscosity of lubricant also affected by nanoparticle and temperature this is according to Asrul et al. (2013), he found at higher temperature the viscosity of the lubricant is very low where the increment of nanoparticle contents in the suspension make the mixture more viscous in higher and lower temperature.

2.4.2 Temperature control

In order to achieve the stability of nanoparticle temperature control to the nano, oil is an important parameter. Based on the experiment that conducted Kulkarni et al (2006) he used Julabo temperature-controlled bath like in figure 2.3.2. In order to control the temperature Cu nanofluid experiment, the sample temperature raised to 50 °C, then gradually decreased to 5 °C in increments of 5 °C. Viscosity was measured at every 5 °C interval. The precision of temperature control was ± 0.1 °C. Another case study that conducted by Asango et al (2014) he used Brookfield DV-II pro rotary viscometer in which a concentric cylindrical arrangement with an inverted cone tip of the rotary spindle, in order to measure viscosity and temperature control for the SAE 5W-30 lubricant oil.

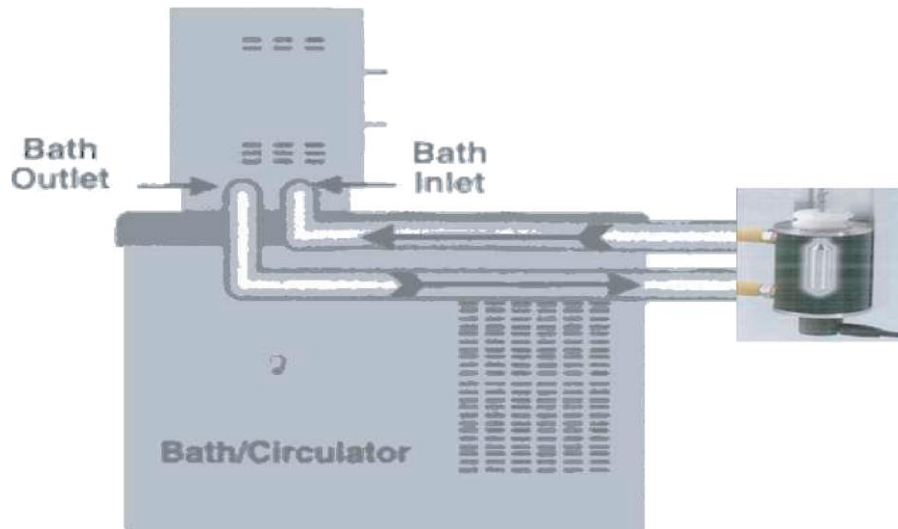


Figure 2.3.2: Experimental set-up for measuring temperature dependency on the viscosity of nanofluids. Kulkarni et al. (2006)

2.4.3 Specific heat

An understanding of the thermal conductivity and specific heat capacity is important in order to understand the characteristics of heat transfer in nanofluids (Angayarkanni et al 2015). This is because specific heat capacity with temperature, different concentrations and the ratio of the specific heat capacity of nanofluid. Nanofluids, which has a high heat capacity is very necessary because it will increase the efficiency of heat transfer. According to Wen et al (2004), the specific heat capacity will determine the rate at which the material will be cold or hot. Usually, the model for water based are used to predict specific heat of nano lubricants but their accuracy not to accurate. Nanofluids have lower specific heat than their base fluids can be estimated by using equation 1. (Lorenzo et al 2014)

$$C_p(nl) = \phi \cdot c_p(p) + (1 - \phi) \cdot c_p(f) \quad (1)$$

Specific heat diminished if the volume concentration of nanoparticles expanded. Specific heat also additionally expanded with expansion in temperatures (Vajjha and Das, 2009). Tests led by Paul et al. (2010) used a double hot-wire technique to measure the effective specific heat for different types of nano fluids. Their

study presumed that liquids with nanoparticles had lower specific heat than their base liquids, what's more, that the qualities for specific heat diminished with expanding volume division of the nanoparticles. Based on the result from Angayarkanni et al. (2015) in his study show the variation specific heat capacity with temperature for different concentrations. The ratio of the specific heat capacity of nanofluid with respect to the C_p of base fluid for kerosene based nanofluids like in Figures 2.3.3(a) and (b). The concentration of nanoparticle is increased because the specific heat capacity of the kerosene-based Fe_3O_4 nanofluid is found to decrease.

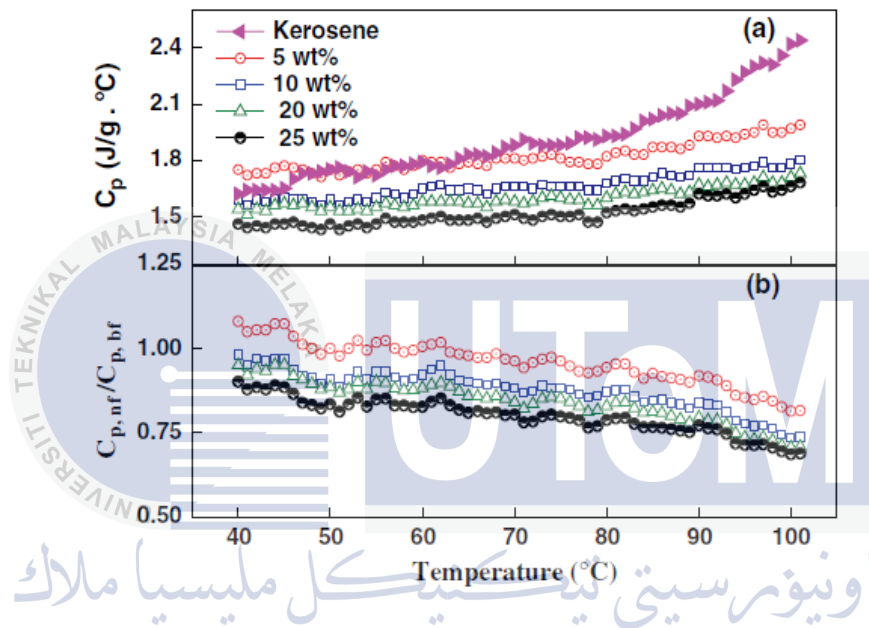


Figure 2.3.3. (a) The variation of specific heat capacity with temperature for different concentration (wt%) of kerosene based Fe_3O_4 nanofluids containing nanoparticles of average size ~ 8 nm. (b) Specific heat capacity ratio $C_{p,nf}/C_{p,bf}$ as a function of temperature for different concentration of Fe_3O_4 nanoparticles. Angayarkanni et al. (2015)

2.5 Stability specific test on nano oil

Stability specific test on nano oil will be characterized by two type of analysis, it is qualitative analysis and quantitative analysis. The type of this analysis is utilized to pick up an understanding of hidden reasons, conclusions, and motivations. It gives a little bit about knowledge into the issue. Qualitative analysis is used to show the

patterns in thought and opinions. Another from that, qualitative analysis is accumulation strategies using semi-structured techniques or unstructured. Contrary to the quantitative analysis, it is utilized to evaluate the issue by a method for producing numerical information or information that can be changed into useable insights. It is utilized to measure opinions, device deeper into the problem and opinions. Another from that sum up the results from a bigger specimen. Quantitative Research uses a quantifiable information to plan the reveal designs in an examination. Due to their advantages, two type of this analysis will be used in this experiment. Upon to the qualitative and quantitative examination on nano-oil security, UV-spectrometer has been chosen to characterize and to contemplate the quantitative investigation towards the nano-oil stability. For subjective investigation towards the nano-oil stability, systematic observation has been chosen. This technique has to be chosen because it can show the progressions of physical normal for the nano-oil.

2.5.1 UV – spectrometer

In order to test the stability of nanoparticle UV spectrometer will be used, this is because UV-spectrometer able to measure the stability of nano oil. According to Sarsam et al. (2016) UV-vis spectrophotometry can provide a measurable characterization of stability by evaluating the light absorbance of a suspension. UV-spectrometer can imply the conditions of measurement such as sample path length, sample concentration, wavelength, and solvent. This testing strategy covers the estimation of the ultraviolet absorption of a variety of petroleum products. UV-spectrometers covers the absorbance of fluids or the absorptivity of fluids and solids, or both, at wavelengths in the district from 220 to 400 nm of the range. Furthermore, UV-spectrometer also can decipher to the quantitative concentration of nano-oil. Lee et al. (2009) expressed the stability of nano-oil was emphatically influenced by the qualities of the suspended particles and the base liquid, for example, molecule morphology (framing by blends). As stated by K. Omri et al. (2014) absorption spectroscopy is an excellent technique in order to explore the optical properties of nanoparticles. The ingestion spectra of ZnO nanoparticles for various thermal treatment temperatures in the UV and visible range are shown in Fig. 2.5.1. All spectra

demonstrated adsorption edges around 385 nm, which related to the optical band gap of ZnO.

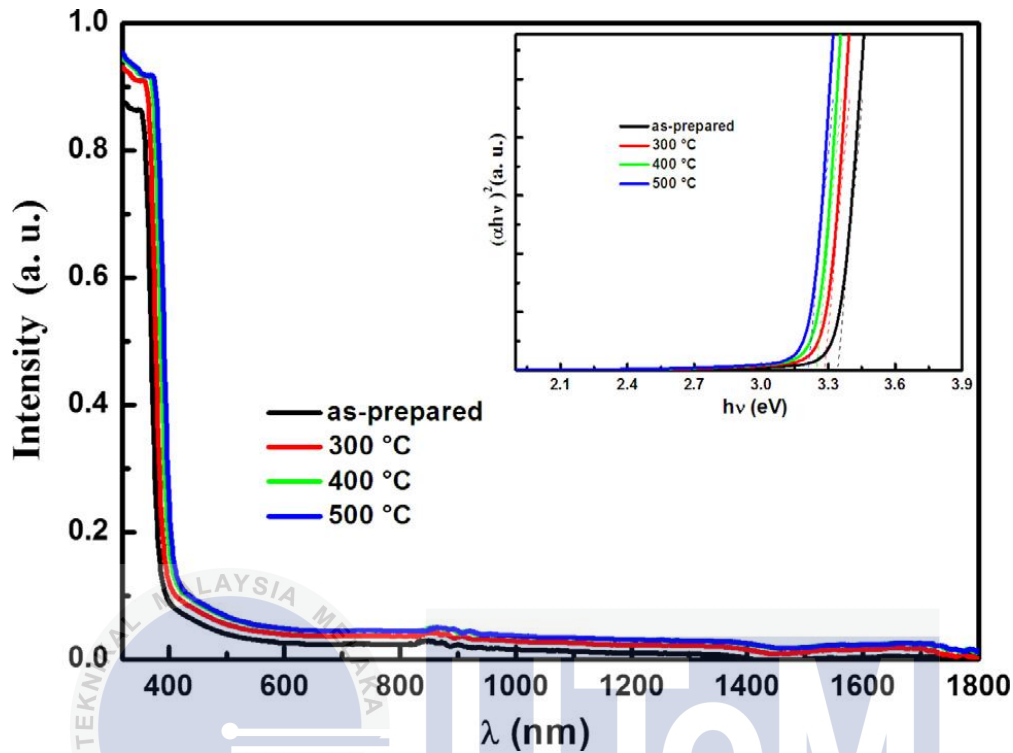


Fig. 2.5.1 UV-vis-IR absorption spectrum of ZnO nanoparticles for different annealing temperatures. The inset showing the E_g spectrum for different temperatures. K. Omri et al. (2014)

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2.5.2 Comparing (Qualitative)

In sequence to see the characteristic of nano oil will show in the data Qualitative analysis. This data will get from the comparison of nano oil after formation of nanoparticle over time and the temperature. Using this analysis it will show properties of the oil and at the same time can see the nanoparticle stability at the variations temperature. Have a lot of researchers use this method in order to see their stability of nano oil. As the example the study that conducted by Amiruddin et al

(2015) , in his study on the stability of nano oil by pH control he using qualitative analysis in order to show the formation of hBN nanoparticles layer over time in different pH values like in figure 2.5.2b. Another research that conducted by Cremaschi et al (2014) he uses qualitative analysis to show sedimentation of nano-lubricant that use same Al_2O_3 nanoparticles but used a different surfactant such as in figure 2.5.2a

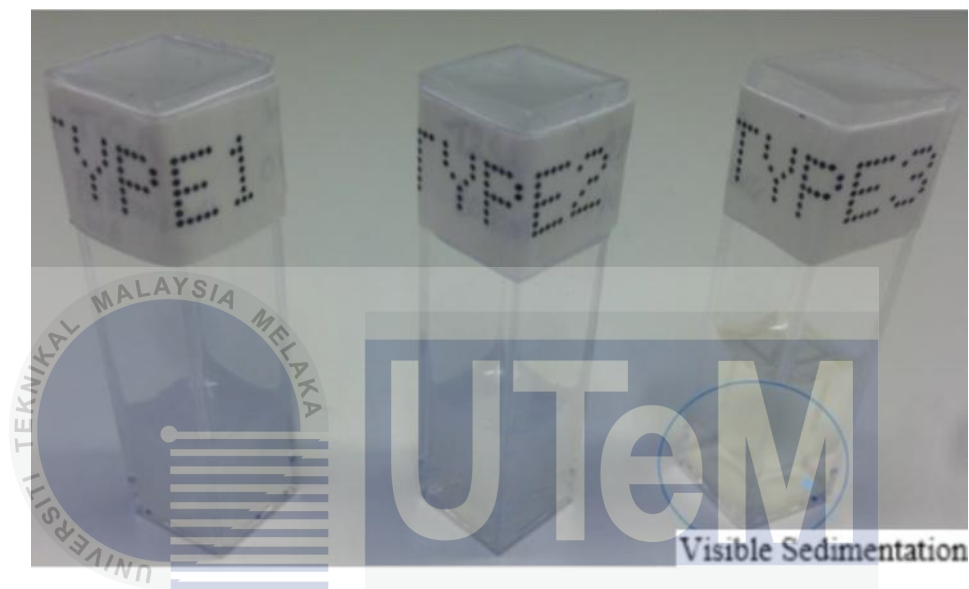


Figure 2.5.2a : visual observation of sedimentation for type 3
nano lubricant. Cremaschi et al (2014)

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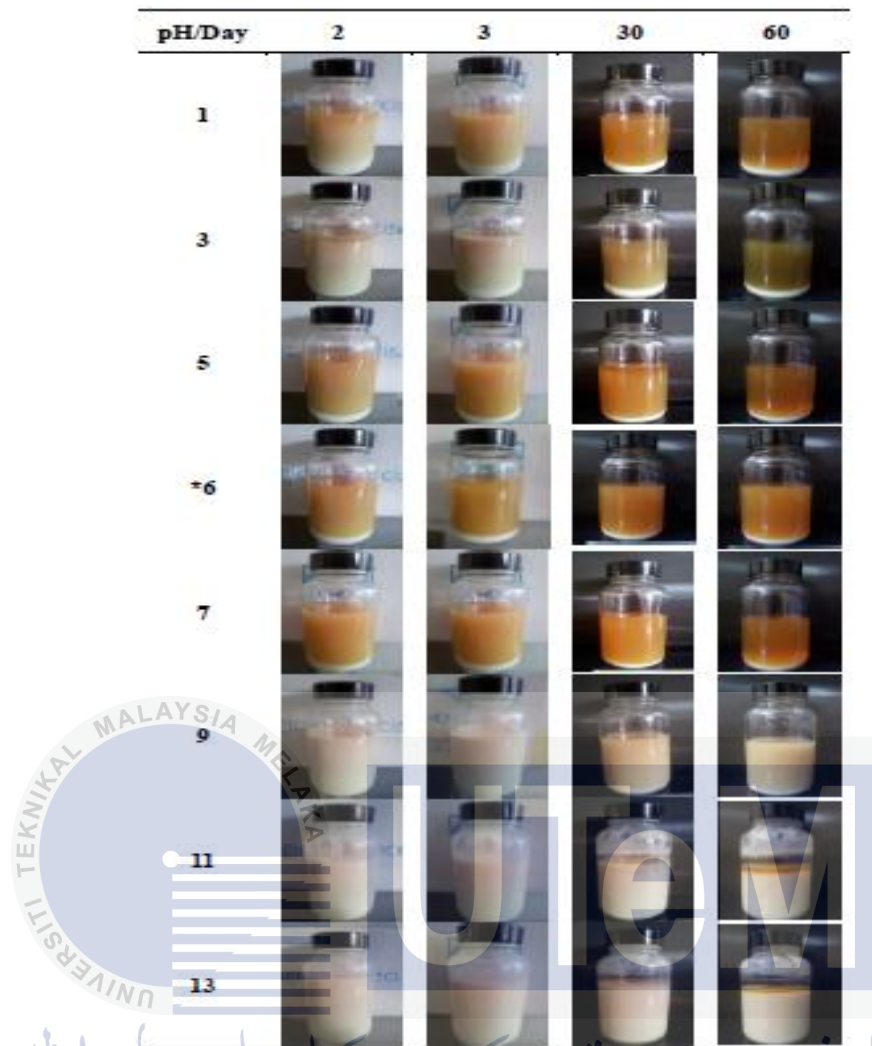


Figure 2.5.2b : Photo of formation of hBN nanoparticles layer over time in different pH values. Amiruddin et al (2015)

CHAPTER 3

METHODOLOGY

3.0 Introduction

In this chapter, an experiment procedure was conducted by prepared and test the stability of the nano-oil. The process started with sample preparation, where the conventional engine oil SAE 15W40 grade is added with nanoparticles and sodium chloride as nano-oil. Three type nanoparticles were be used are hexagonal boron nitride (hBN), alumina (Al_2O_3), and zirconia (ZrO_2). The nano-oil was be stabilized with the addition of an appropriate amount of sodium chloride (surfactant). Usually, nanoparticle will be agglomerated suspension and sedimentation when it has been introduced to the oil. Due to this problem, ultrasonic homogenizer has been used to homogenize the particle in order to ensure a homogeneous dispersion of the nanoparticle in the oil. The preparation of nano oil will are controlled by the several sets of temperature begin with room temperature 30°C until 70°C . The effect of temperature and the absorbency of nano oil are measured by using UV- spectrometer. After all the data has gathered and shown in the qualitative analysis to make a comparison with all nanoparticle that has been used. Lastly, toward the end of the procedure, the assessment of the outcome information and examination are measured where the finish of the examination will be finished. Figure 3.0 have shown the methodology flow chart.

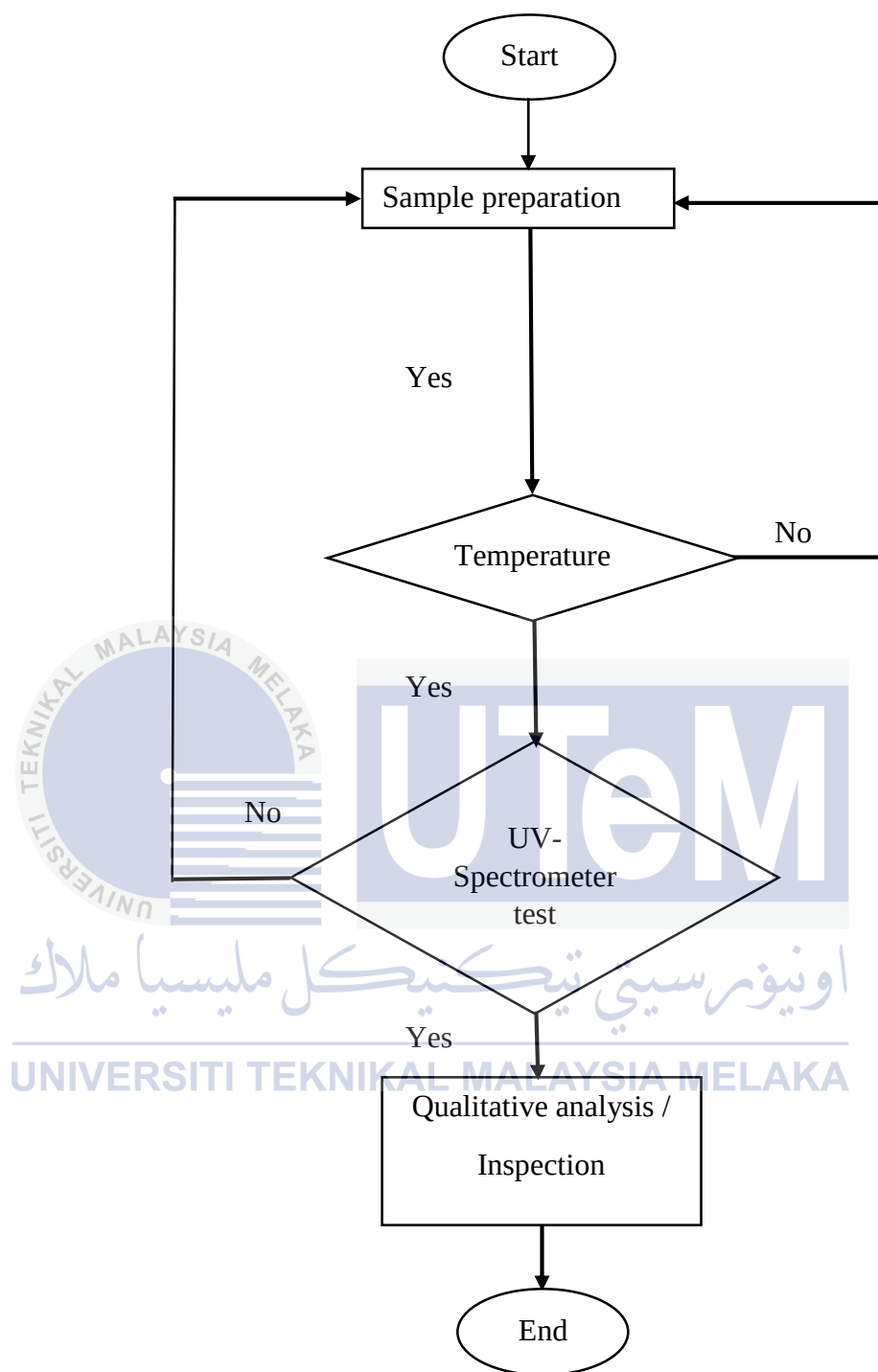


Figure 3.0 Methodology Flow Chart

3.1 Material Selection

The conventional oil and nanoparticles were sourced from professional suppliers. For the preparations of the nano-oil, the material that will be used are SAE 15W40 conventional oil, sodium chloride (surfactant), Hexagonal boron nitride (hBn) nanoparticles, Alumina Oxide (Al_2O_3) nanoparticles, and Zirconia Oxide (ZrO_2).

3.1.1 Lubricant (Conventional oil SAE 15W40)

SAE 15W40 conventional engine oil usually clarify as diesel engine lubricating oil. The alphabet and number show the level of the viscosity of the oil. The viscosity of the oil when it cold is 15W and SAE 40 show the viscosity when it hot. The properties of this oil were shown in Table 3.1

Table 3.1 the properties of conventional oil SAE 15W40

Source (ETS Product Specification)

Oil Property	Unit	Test method	Result
Kinematic viscosity @ 100 °C	mm ² /s (cst)	ASTM D445 15.5	15.5
Viscosity Index (VI)		ASTM D2270	135
Density @ 15 °C	Kg/L	ASTM D1298	0.875
Flash Point	°C	ASTM D92	210
Pour Point	°C	ASTM D97	-27
TBN	Mg KOH/gr	ASTM D4739	12
CCS @ -20 °C	cP	ASTM D5293	≤ 7000

3.1.2 Nanoparticle

In this study, three type of nanoparticles was be used which are hexagonal boron nitride (hBN), alumina oxide (Al_2O_3), and zirconia (ZrO_2).

Hexagonal boron nitride is one of the excellent solid lubricants. Their structure quietly similar to graphite. Hexagonal boron nitride has good properties of high temperature although at normal temperature it is considered less effective than graphite and MoS₂ (Kimura et al 1999). The properties of this nanoparticle have been shown in Table 3.2.

Alumina (Al_2O_3) and Zirconia (ZrO_2) are metal oxide composite nanoparticles. When Alumina acts as additive to the lubricant, it shows excellent of friction and anti-wear mechanism in the tribological application (Shahnazar et al 2015). Apart from that, Al_2O_3 , ZrO_2 is effectively efficient when been modified to aluminum zirconium and used as lubricating oil. It shows a significant reduction in friction coefficient (Li et al 2011). The properties of Alumina (Al_2O_3) and Zirconia (ZrO_2) nanoparticle are given in Table 3.3 and Table 3.4.

Figure 3.2 The properties of Hexagonal boron nitride (hbN)

Source Abdullah et al (2014)

Characteristic	Value
Purity	99%
Average Particle Size	70nm
Bulk Density	0.3g/cm ³
True Density	2.3 g/cm ³
Appearance	White powder
Maximum used temperature °c	1800
Thermal conductivity (Wm ⁻¹ K ⁻¹)	27

Table 3.3 The properties of Alumina oxide (Al_2O_3)

Source Abdullah et al (2014)

Characteristic	Value
Appearance	White powder
Maximum used temperature °c	1750
Thermal conductivity ($\text{Wm}^{-1} \text{K}^{-1}$)	30
Density g/cm^3	3.97
Average Particle Size	70nm
Thermal expansion coefficient @25°C-1000°C	2×10^{-3}

Table 3.4 The properties of Zirconia (ZrO_2)

Source (ferrocera.com/zirconia.htm)

Characteristic	Value
Appearance	white
Density g/cm^3	6.04
Crystal structure	tetragonal
Specific Surface Area	0.10 $\text{cal/g}^\circ\text{C}$ @ R.T.
Thermal Conductivity W/m-K @ R.T	2.7

3.1.3 Ultrasonification setting parameter

To deagglomeration the nanoparticle when added to the oil, ultrasonic was used to break down the particle. By using ultrasonic, nanoparticle in the SAE1540 conventional oil was homogenized with fix amplitude and cycle it is 0.5. This nano-oil is stirred by using ultrasonic for 30 minutes. From the ultrasonic cycle, it will produce heat. The preparation of nano oil is controlled at different temperature begin with room temperature 30°C until 70°C. Why only at 70°C it is because when nano oil has tastes above 90°C the temperature can change the nanoparticles properties. In order to control the temperature, cool water from water bath will be added under nano oil and the temperature of nano oil will be observed by using a thermometer. The schematic diagram of ultrasonic has shown in figure 3.1.

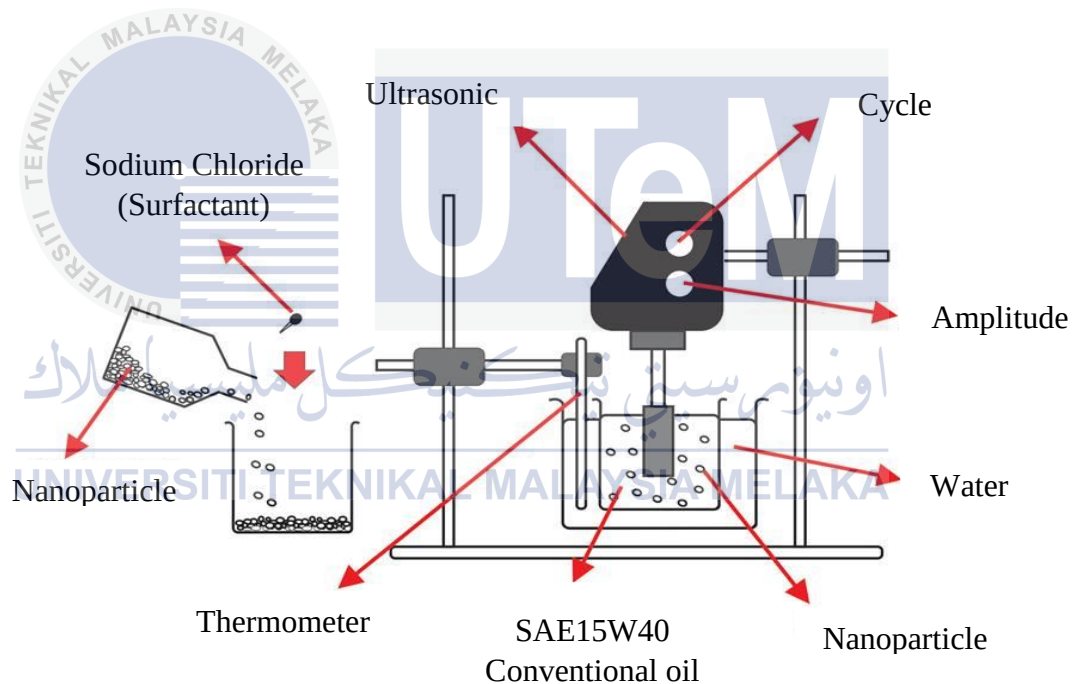


Figure 3.1 Schematic diagram for sample preparations.

3.2 Sample preparation

In this study, the process started with sample preparation, where the conventional engine oil SAE 15W40 grade will be added with nanoparticles as nano-oil. Nano-oil will be stabilized using sodium chloride. After that nano-oil is stirred manually before use ultrasonic. It is to prevent nanoparticle from the stick at the beaker. The preparation of nano oil is controlled at the different temperature begin with room temperature. The preparation of nano oil sample was prepared for 200ml including nanoparticle and sodium chloride. The composition for nanoparticle is 0.5vol% and the composition of sodium chloride is 0.3%. Figure 3.2 have shown the flow chart of sample preparation for nano-oil.

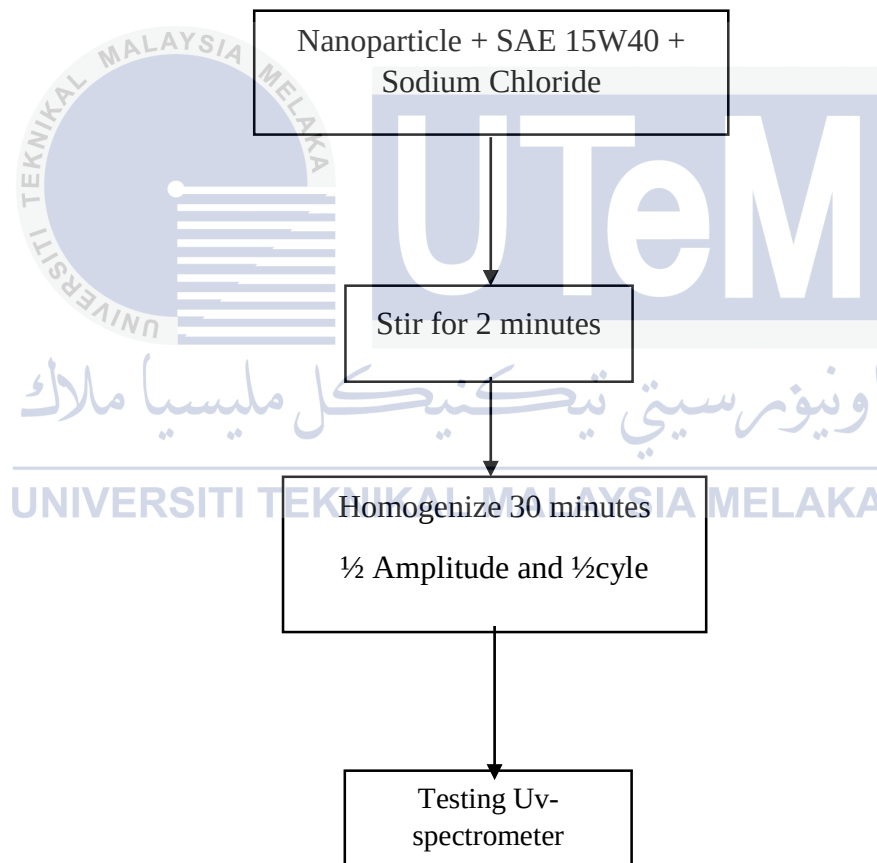


Figure 3.2 Sample preparation for nano-oils

3.2.1 Sample composition

The total volume of nano oil prepared are 200ml including nanoparticle and sodium chloride. The composition for nanoparticle to be mixed in the SAE 15W40 conventional oil is 0.5vol% and composition for sodium chloride is 0.3vol%. However, the nanoparticle composition will be calculated in the form of mass as to facilitate the measurement of a nanoparticle. The formula to be used is $\rho = m/v$, where ρ is nanoparticle density (g/cm³), m is mass (g), and v is volume (ml). The example of calculation has shown below.

Composition for hexagonal boron nitride (hBN)= 0.5% vol.

$$=1.0 \text{ ml}$$

Density of (hBN), $\rho_{\text{hBN}} = 2.3 \text{ g/cm}^3$

$$\begin{aligned} & \rho = m/v \\ & 2.3 \text{ g/cm}^3 = m/1.0 \text{ ml} \\ & (2.3 \text{ g/cm}^3)(1.0 \text{ ml}) = m \\ & \rightarrow 2.3 \text{ g/cm}^3 \times \frac{1 \text{ cm}^3}{1 \text{ ml}} = 2.3 \text{ g/ml} \\ & (2.3 \text{ g/ml})(1.0 \text{ ml}) = m \\ & 2.3 \text{ g} = m_{\text{hBN}} \end{aligned}$$

3.2.2 Sampling setup

The sampling setup for 200ml nano oil has shown below in the table 3.2.2

Table 3.2.2

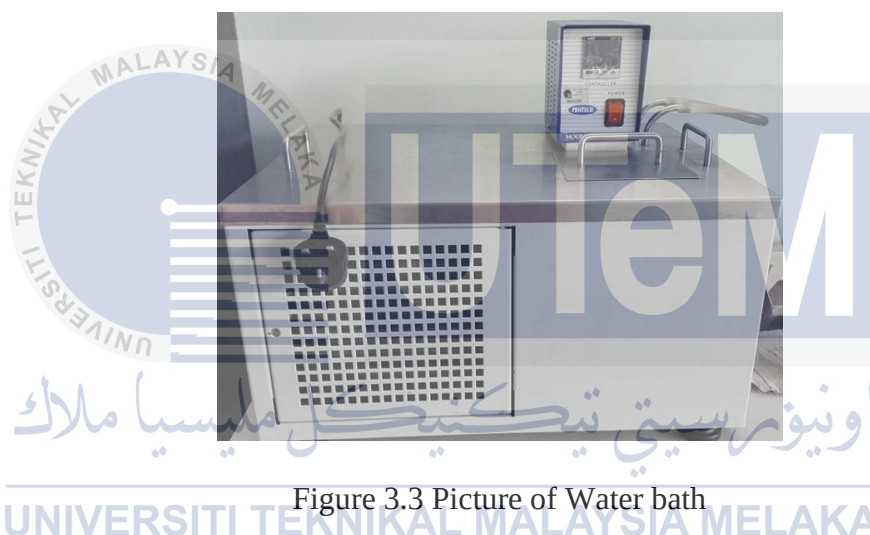
Type	Temperature test (°c)	Concentration Nanoparticle 0.5vol% @ g	Volume SAE15W40 (ml)	concentration sodium chloride 0.3vol% @ ml	Mixing Time (minute)
Hexagonal	30	2.3g			
Boron	40				
Nitride	70				
Alumina	30	3.97g			
Oxide	40		198.4ml	0.6 ml	30minutes
Zirconia	70				
	30	6.04g			
	40				
	70				

3.3 Testing

The stability test of nanoparticle that contained in the nano oil sample, was carried out by using UV-spectrometer. Before testing the stability of the nanoparticle, it was controlled by several sets of temperature started with room temperature 30 up to 70. The nano oil sample was controlled by placed the oil in the water, where the water temperature has been set by using a water bath.

3.3.1 Temperature control

Temperature control for this nano oil is done by added the water under the nano-oil sample. Two step procedure are used to control the temperature. Firstly the water from the water bath was heated to the 30°C and put under the nano-oil sample. After that water from water bath are cooled 5°C. When water that placed under the oil sample showed an increase in temperature, the cold water from the water bath will be continuously added until the temperature of the water become $30\pm 2^{\circ}\text{C}$. The same step will be used for other sample nano-oil and every nano-oil sample was tested at 30°C, 40°C, and 70°C. Figure 3.2 show a picture of the water bath.



3.3.2 UV-Spectrometer

The stability of nano-oil is measured by using UV-spectrometer. UV-vis spectrophotometry can provide a measurable characterization of stability by evaluating the light absorbance of a suspension. UV-spectrometer can imply the conditions of measurement such as sample path length, sample concentration, wavelength, and solvent. This testing strategy covers the estimation of the ultraviolet absorption of a variety of petroleum products. UV-spectrometers covers the absorbance of fluids or

the absorptivity of fluids and solids, or both, at wavelengths in the district from 220 to 400 nm of the range. Figure 3.3 show a picture of UV-spectrometer.



Figure 3.3.2 Picture of UV-spectrometer.

3.3.3 Verification

Qualitative analysis is used to verify this process. The process was identified by compared the characteristic of nano oil for two months after left it at room temperature. Lastly, the changes in the nano-oil sample like sedimentation of nanoparticle was observed every day. At the same time, the picture was taken for every day in order to compare the oil sample and interpret it in qualitative data. The verification process is very important in order to identify the best stability of nanoparticle between three type nanoparticles.

CHAPTER 4

RESULT & DISCUSSION

4.0 RESULT AND DISCUSSION

In this chapter, results for stability test by using UV-spectrometer are presented. The result has been showed in table form and graph. The outcomes result included the stability of the three types of nanoparticle with time (day) at difference temperature. Three type of nanoparticle that involved in this experiment are hexagonal boron nitride (hBN), Alumina Oxide (Al_2O_3) and Zirconia (ZrO_2). The measured stability of hexagonal boron nitride (hBN), Alumina Oxide (Al_2O_3) and Zirconia (ZrO_2) are presented against the absorbance and temperature. All this result are done based on the procedure in methodology at chapter 3. The aims of this study are to investigate the effect of the temperature on the nanoparticle stability properties.

4.1 Quantitative Analysis

The result from the UV-spectrometer is interpreted in quantitative analysis in order to provide a measurable characterization of stability by evaluating the light absorbance of a suspension. UV-spectrometer is able to show the stability of the nanoparticles by covers the absorbance of fluids and solids, or both, at wavelengths in the district from 220 to 600 nm of the range. Quantitative analysis is one of the methods that is very verifiable and measurable in order to present the stability of the nanoparticle. In addition UV- spectrometer will facilitate to comparative among all sample. It is because by used the quantitative analysis it can measuring or counting attributes (i.e. quantities). Indirectly it makes the result that comes out easy to understand and compared each other. Based on the experiment that conducted by Shipway et al (2000), quantitative analysis to display the contact of nanoparticles that

show a Plasmon absorbance guide to the appearance of an absorbance band. The effect of the temperature on the nanoparticle stability properties was the focus of the study. The absorbance value versus day starting with the first day until two months was presented in Figure 4.1, 4.2 and 4.3. Tables 4.1, 4.2 and 4.3 display the recorded of the nanoparticle for three type nanoparticle was controlled at three level temperature 30°C, 40°C, 70°C and leave for two months at the room temperature. Next, the sample has been tested by using UV-spectrometer.

4.1.1 Analysis of nanoparticles stability at temperature 30°C

Table 4.1 show the result absorbance of three type nanoparticle that controlled at temperature 30°C. The reading on the first day for hexagonal boron nitride, Alumina Oxide, and Zirconia was 3.9813 abs, 3.3419 abs, and 3.4998 abs. The absorbance value for hexagonal boron nitride clearly shows the highest value. According to the Amiruddinn et al (2015), better dispersion of nanoparticle should have a greater absorbency. However, the absorbance value for hexagonal boron nitrite starting to decrease at day 2 and continued until two months.

Table 4.1 Result of absorbance for hBN, Al₂O₃ and ZrO₂ at temperature 30°C

UV-Spectrometer absorbance of nanoparticle at 30°C						
Days	hBN		Al ₂ O ₃		ZrO ₂	
	Absorbance	Standard Deviation	Absorbance	Standard Deviation	Absorbance	Standard Deviation
1	3.9813	0.0000	3.3419	0.0027	3.4998	0.0208
2	3.5244	0.1384	3.1865	0.0021	-	-
3	3.4870	0.3640	3.1251	0.0853	-	-
4	3.3462	0.0173	3.0609	0.0593	-	-
5	3.1886	0.0000	3.0532	0.0003	3.4878	0.0398
7	3.1681	0.1034	3.0375	0.1204	3.4383	0.0127
14	3.1078	0.0799	2.9877	0.1291	3.4183	0.0579
21	3.0867	0.0000	2.9019	0.0228	3.4072	0.0000
28	3.0774	0.0199	2.8813	0.0000	3.3953	0.0000
60	3.0028	0.0138	2.7739	0.0419	3.2446	0.0000

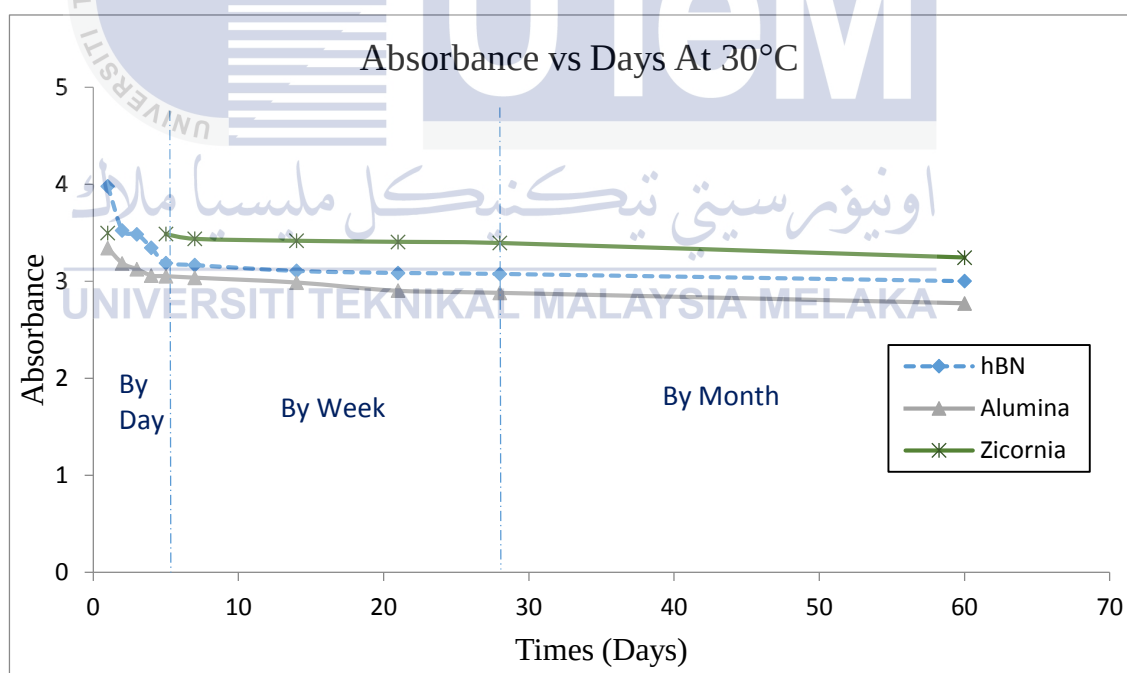


Figure 4.1 Graph of Absorbance against Days at 30°C.

Figure 4.1 shows the graph trend of the nanoparticle absorbance against day controlled at 30°C and left at room temperature for two months. The line graph for the hexagonal boron nitride shows the drastically decrement starting at day two compare to the alumina oxide and the zirconia nanoparticle. Where the slope value for line graph hexagonal boron nitride was 0.0166 and the highest value. While the slope value for alumina oxide and zirconia are 0.022 and 0.004. Although that, after a week the decline in absorbance value for the three type nanoparticle becomes more slowly. From this result absolutely show hexagonal boron nitride was affected at the temperature 30°C. This due to the changes of particle size in nanofluid indirectly increase thermal conductivity. As stated by Solangi et al. (2015) due to the particle size it will affect the temperatures and cause the thermal conductivity to increase. Nonetheless, at 30°C temperature hexagonal boron nitride will have the extensive agglomeration due to low thermal conductivity. However, nanoparticle stability for the Alumina oxide and the Zirconia nanoparticle does not affect too much at this temperature especially the Zirconia nanoparticle.

4.1.2 Analysis of nanoparticles stability at temperature 40°C

The stability test against three type nanoparticle also done at temperature 40°C. Nanoparticle stability was measured based on the absorbance of the UV light when it passes through the sample. The result was recorded as in table 4.2. On the first day, the absorbance value to three type nanoparticle was taken shown hexagonal boron nitride have a good stability. The absorbance value for hexagonal boron nitride is 3.9937 abs and followed by alumina oxide 3.6571 abs lastly zirconia 3.5099 abs. This shows that hexagonal boron nitride was dispersed well in the lubricant after have been homogenize by using ultrasonic compare to another nanoparticle. However, at the second day the absorbance value for hexagonal boron nitride starting to decrease. Compare to the alumina oxide the absorbance value for zirconia nanoparticle showed just a slightly changed. As study conducted by kouloulis et al (2016) the good suspension of Al_2O_3 nanoparticle concerning its physical properties.

Table 4.2 Result of absorbance for hBN, Al₂O₃ and ZrO₂ at temperature 40°C

UV- Spectrometer absorbance of nanoparticle at 40°C						
Days	hBN		Alumina		Zirconia	
	Absorbance	Standard Deviation	Absorbance	Standard Deviation	Absorbance	Standard Deviation
1	3.9937	0.0862	3.6571	0.0091	3.5099	0.0050
2	3.8065	0.0832	3.1927	0.0000	-	-
3	3.7463	0.0832	3.1603	0.1995	-	-
4	3.6796	0.0327	3.1025	0.0000	-	-
5	3.2196	0.0675	3.0854	0.0008	-	-
7	3.1730	0.0128	3.0729	0.0001	3.5062	0.1058
14	3.1624	0.0263	3.0105	0.0006	3.3434	0.0063
21	3.0983	0.0053	2.9761	0.0421	3.3043	0.0000
28	3.0657	0.0729	2.9501	0.0003	3.2790	0.2144
60	2.9885	0.0015	2.9354	0.0000	3.1879	0.0000

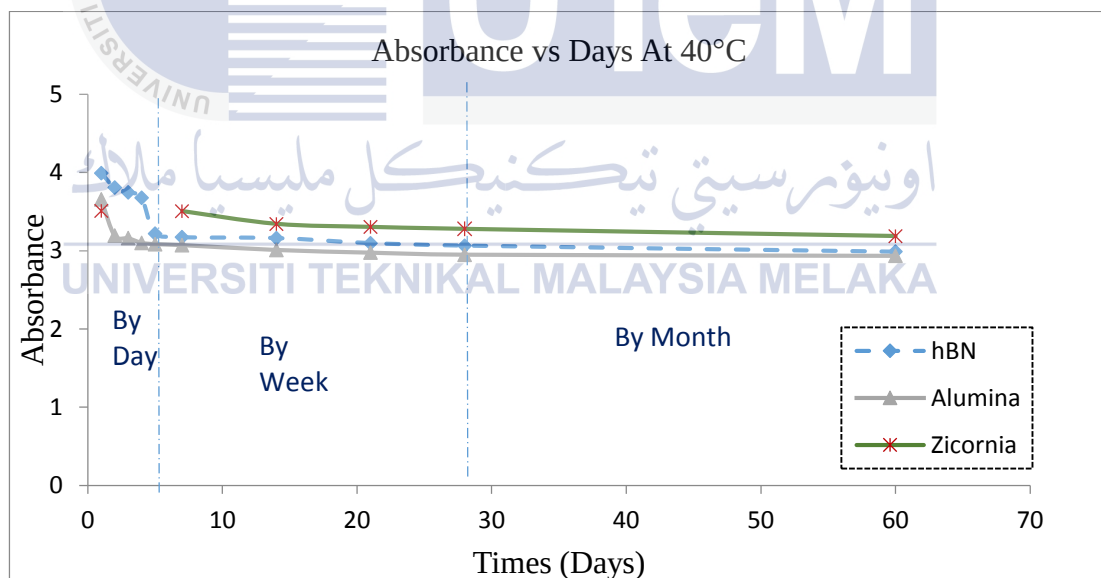


Figure 4.2 Graph of Absorbance against Days at 40°C

The line graph trend for three type nanoparticle are shown in figure 4.2. Based on the line graph shown zirconia nanoparticle is able to maintain the stability until 2 months. The value slope for zirconia was only 0.01435. This explained zirconia

nanoparticle are not affected by the temperature at 40°C. This is maybe due to the physical properties of zirconia nanoparticle itself which is not easily affected by temperature. However, different with stability for sample that containing hexagonal boron nitride nanoparticles. The line slope value for hexagonal boron nitride nanoparticles showed the highest value is 0.1368. This is prove that the stability for hexagonal boron nitride nanoparticles is affected at this temperature. This is possibly due to temperature can induce structural changes to the nanoparticle make it easy to agglomerate and sedimentation occur. Contrary to the alumina oxide nanoparticle, the absorbance value is drastically decline only at day two. However at third day until day 60 the absorbance value shown, only the slightly decrement. Alumina oxide nanoparticle are not really affected by temperature. The Physical properties of alumina oxide like a glass may be a factor why this nanoparticle also not affected by temperature. More than, the surfactant also the cause the alumina oxide can stabilize and sedimentation does not occur. According to Kedienhon et al (2012), Surfactants have been used as agents to prevent the agglomeration of nanoparticles in aqueous and non-aqueous systems.

4.1.3 Analysis of nanoparticles stability at temperature 70°C

— Hexagonal boron nitride still has the higher absorbance value on the first day, after have been homogenized by using ultrasonic and controlled at 70°C. The value of UV absorbance for hexagonal boron nitride is 3.9999abs. Followed by zirconia nanoparticle is 3.5923abs and alumina oxide is 3.6724abs. All the data are shown in table 4.3. Although, on the first day of hexagonal boron nitride, has dispersed well but it began to decrease on the second day as shown in figure 4.3. This shows that hexagonal boron nitride begins to agglomerate with each other when the high temperature was imposed and leave at room temperature. This is due to the changes of temperature from high temperature to low temperature. It is also agreed by Prasher et al (2006) which reported changed of thermal conductivity in nano-fluid cause the nanoparticle begin to agglomerate. In the other hand, the line graph for zirconia nanoparticle was the most stable as can see in the graph. Impairment of absorbance on line graphs zirconia almost did not happen. This shows zirconia nanoparticle very

unimpressed with temperature. In addition, it may be the effect of mixed surfactant effective against zirconia nanoparticle. According to (Vaisman et al. 2006) addition of the surfactant agent can reduce the tendency to agglomerate in the continuous phase solvent.

Table 4.3 Result of absorbance for hBN, Al₂O₃ and ZrO₂ at temperature 70°C

UV absorbance of nanoparticle At 70°C						
Days	hBN		Alumina		Zicornia	
	Average	Standard Deviation	Average	Standard Deviation	Average	Standard Deviation
1	3.9999	0.0000	3.6724	0.0127	3.5923	0.1027
2	3.7293	0.2343	3.2880	0.0398	-	
3	3.6508	0.0985	3.2672	0.0335	-	
4	3.5701	0.3722	3.2072	0.1132	-	
5	3.5263	0.1529	3.1926	0.0283	-	
7	3.4956	0.1634	3.1445	0.0000	3.4878	0.0398
14	3.4878	0.1880	3.1432	0.0005	3.4728	0.0238
21	3.3218	0.0561	2.9501	0.0156	3.3454	0.0000
28	3.1687	0.0047	2.9472	0.0189	3.3399	0.0003
60	3.0553	0.0655	2.9466	0.0000	3.3218	0.0000

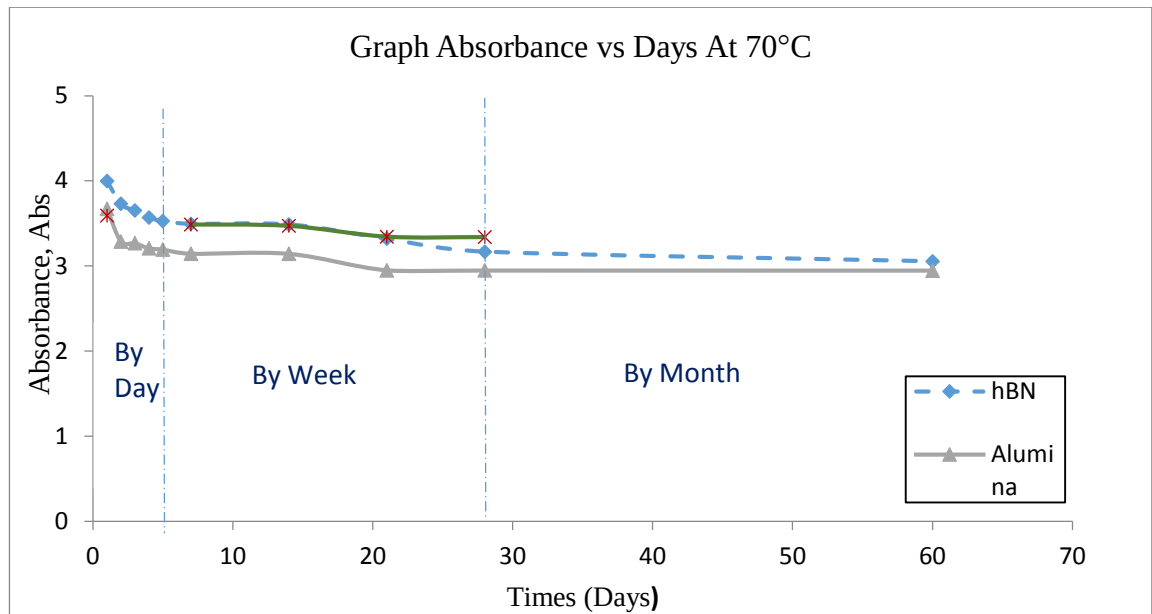


Figure 4.3 Graph of Absorbance against Days at 70°C

4.2 Qualitative Analysis

Qualitative analysis has been used in order to verify the process for this experiment. By using qualitative analysis it can identify the characteristic of the nano-oil after left at room temperature for 60 days. From this analysis, it can show the formation or sedimentation of a nanoparticle. The formation result for three type of nanoparticle was shown in Table 4.4, 4.5 and 4.6.

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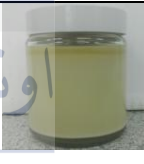
4.2.1 Effect of difference temperature on the hexagonal Boron Nitride, (hBN) nanoparticles stability.

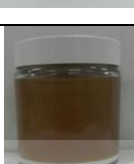
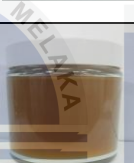
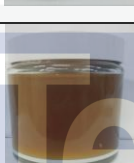
Table 4.3 show the formation of the hexagonal boron nitride sedimentation at the bottom of the bottle for a period 60 days. In this experiment, nanoparticle was controlled at different temperature at the first day begin with room temperature 30°C until 70°C. After that nanoparticle was kept at room temperature for 60 days. Hexagonal boron nitride shows the highest value for absorbance value at the first day after controlled at the different temperature. However, the sedimentation of hexagonal Boron nitride was occurred as early at the second day. It can see in Table 4.4 the color changes of the hexagonal boron nitride. As can see the changes of the color starting at

day two until day 60. The oil sample becomes brighter compared to the first day of oil have been mixed.

This happens maybe due to the changes of the temperature. When temperature cooled down the enthalpy will be changed to negative. In order to balance the changes, the system will try to maximize the entropy by separating molecules from the "large" nanoparticles (Angayarkanni, et al 2015). Despite that hexagonal boron nitride controlled at 70° was the most stable among two temperature that was used. This is because at this temperature hexagonal boron nitride has a higher value of absorbance compare to the others temperature that been used in this experiment.

Table 4.4 Picture of SAE15W40 + hBN nanoparticle at different temperature

Picture of SAE15W40 + hBN nanoparticle at different temperature						
Days	Abs	30°C	Abs	40°C	Abs	70°C
1	3.9813		3.9937		3.9999	
2	3.5244		3.8065		3.7293	
3	3.4870		3.7463		3.6508	
4	3.3462		3.6796		3.5701	
5	3.1886		3.2196		3.5263	

7	3.1681		3.1730		3.4956	
14	3.1078		3.1624		3.4878	
21	3.0867		3.0983		3.3218	
28	3.0774		3.0657		3.1687	
60	3.0028		2.9885		3.0553	

4.2.2 Effect of difference temperature on the Alumina (Al_2O_3) nanoparticles stability

The images of alumina oxides nano-oil was kept at room temperature for two months is display at Table 4.4. The observation has been done starting from day one until day 60. Based on observation the image of the sample for alumina oxide did not show significant changes. The changes of the alumina oxide nanoparticle only can be detected by using UV- spectrometer. At a temperature of 70°C alumina oxide nanoparticle showed the highest stability compared to other temperatures. Alumina oxide nanoparticle can only be seen with the naked eye only on the seventh day where nanoparticle starting to form at the bottom of the bottle.

Although that the sedimentation process for alumina not bad such as hexagonal boron nitride. This shows that alumina oxide nanoparticle is not affected by the temperature. This is maybe the existing properties in alumina was like glass. The agglomeration at the bottom of the bottle maybe due to the gravitational force or

affected by the activity of van der Waals (Abdullah et al 2014). In the other hand, maybe this is the effect the addition of the surfactant not suitable with this alumina oxide nanoparticle.

Table 4.5 Picture of SAE15W40 + Al₂O₃ nanoparticle at different temperature

Picture of SAE15W40 + Al ₂ O ₃ nanoparticle at different temperature						
Days	Abs	30°C	Abs	40°C	Abs	70°C
1	3.3419		3.6571		3.6724	
2	3.1865		3.1927		3.2880	
3	3.1251		3.1603		3.2672	
4	3.0609		3.1025		3.2072	
5	3.0532		3.0854		3.1926	
7	3.0375		3.0729		3.1445	
14	2.9877		3.0105		3.1432	

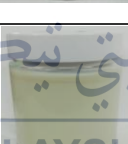
21	2.9019		2.9761		2.9501	
28	2.8813		2.9501		2.9472	
60	2.7739		2.9354		2.9466	

4.2.3 Effect of difference temperature on the Zirconia, nanoparticles stability

Among all nanoparticle that has been tested in this experiment zirconia nanoparticle is the most stable. Although on the first day absorbance readings were taken showed the most stable were hexagonal boron nitride but on the second day, it already showed instability. Compare to the zirconia oxide although at the start of the absorbance readings are lower than the hexagonal boron nitride but its stability can be maintained right up to day 60. The decrease in the absorbance reading has almost not happened, as shown in the picture in table 4.6.

The color change indicates that almost did not happen due to the sedimentation of nanoparticle occurs was very little. Zirconia nanoparticle was the same group with alumina oxide it is a metal oxide (Shahnaz et al 2016). Probably because of this, zirconia nanoparticle was not easily affected by temperature. And surfactant that has mixed with zirconia nanoparticle was functioned properly. When compared to the same nanoparticle but at different temperatures, at 70°C nanoparticle showed the highest stability because it has the highest absorbance value.

Table 4.6 Picture of SAE15W40 + ZrO₂ nanoparticle at different temperature

Picture of SAE15W40 + ZrO ₂ nanoparticle at different temperature						
Days	Abs	30°C	Abs	40°C	Abs	70°C
1	3.4998		3.5099		3.5923	
2	-		-		-	
3	-		-		-	
4	-		-		-	
5	3.4878		-		-	
7	3.4383		3.5062		3.4878	
14	3.4183		3.3434		3.4728	
21	3.4072		3.3043		3.3454	
28	3.3953		3.2790		3.3399	

60	3.2446		3.1879		3.3218	
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4.3 Impact of thermal properties on the nanoparticles stability

Thermal properties have affected on the stability of the nanoparticles. According to the Alexander (2006) the sedimentation of the nanoparticle was due to the thermal conductivity that happens in the lubricant. Thermal conductivity has a dependence on particle size and/or temperature. This is because thermal conductivity is one of an important parameter to enhanced heat transfer of a base fluid (Sridhara 2015).

4.3.1 Effect of low temperature on the nanoparticle sedimentation

The image result at Figure 4.7 for hexagonal boron nitride show a lot of formation sedimentation nanoparticle which a white layer at the bottom of the bottle compares to another nanoparticle. The absorbance value of the line graph shows the trend of hexagonal boron nitride nanoparticle have decreased drastically over time (day). The color changes of the hexagonal boron nitride can see as early on day two but it more clearly after a week. The changes of the color of hexagonal boron nitride still happen after 60 days. This is due to the natural pull gravity itself. When temperature subjected to the nano-fluid it will cause hexagonal boron nitride to vibrate and cause the changes in particle size. More than it will result in a smaller particle size and occur higher relative surface thus agglomeration may occur.

However, it is different from the condition of the alumina oxide and the Zirconia. As can see in figure 4.7 the line graph for the absorbance of alumina oxide show the drastically decrement starting at day 2 but at day 3 until day 60 the decrement of absorbance become slow. The picture of the alumina oxide in figure 4.7 show where the color obviously started to change. For zirconia nanoparticle the line graph for

absorbance show trend that almost evenly. The decrement still happens but it too little. The changes of the color for zirconia do not happen until day 60. The formation of the layer has happened at the top of the lubricant. This is maybe due to the suspension of the zirconia nanoparticle in the lubricant. Overall at low temperature, nanoparticle more easy to sedimentation at the bottom of the bottle because at low temperature the movement of nanoparticle are to slow (Mahbubul et al 2015).





4.3.2 Effect of high temperature on the nanoparticle sedimentation

The effect of high temperature on the nanoparticles sedimentation is shown in 4.8. The figure shows the combination of the graph and picture of SAE 15W 40 was added with three type of nanoparticle. At this temperature, the stability of zirconia was the most satisfying compare to another nanoparticle. Although at the first day the three type of this nanoparticle was be tested showed hexagonal boron nitride have the highest value of absorbance. But starting at day 2 the agglomeration at the bottom of the bottle for hexagonal boron nitride was very bad. As can see in figure 4.8 the line graph for hexagonal boron nitride was sharply decrease with time. The picture of clear sedimentation that happens on the hexagonal boron nitride was can see at day 7 as shown in figure 4.8. As discussed in 4.3.1 temperature is very easy to influence the nature of physical Hexagonal boron nitride especially with high temperature. When nanoparticle started to agglomerate the sedimentation will occur cause by *gravitational* force.

The color changes for alumina oxide was hardly occurred. However the little of sedimentation at the bottom of the bottle still can see with the naked eye as shown in Figure 4.8. The verification of stability the alumina oxide was interpreted by using UV spectrometer. Based on the result that gets from UV-spectrometer for alumina oxide, at the first day was the best stability for this nanoparticle. Although so, started at day 2 the sediment of the nanoparticle was started to occur. In accordance with the result that shows in figure 4.8, zirconia nanoparticle was the most stable dispersion at this temperature. When the stable dispersion of particles can be improved it may also increase the heat transfer capability of the fluid foundation. Based on the study that conducted by Devraj et al (2016) the increment of temperature and volume fraction will increase thermal conductivity. Alumina oxide was the second place has a good stability at this temperature. The characteristic of this two type nanoparticle almost the same this is maybe they was at same group namely metal oxide. According to Hendraningrat et. al (2015), the aggregation appears for metal oxide at room conditions which mean that the higher temperature does not significantly affect the stability of this nanoparticle.



CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.0 CONCLUSION

In this study, the stability of 15 w40 oil based hBN, Al_2O_3 and ZrO_2 nano lubricants were characterized by using UV- spectrometer (quantitative analysis) and image analysis (qualitative analysis). Sodium chloride is used to stabilize nano-oil. All nano lubricants were prepared with the help of ultrasonic homogenizer. In order to study the effect of temperature on the nanoparticle stability properties several sets of temperature are used. Started at room temperature 30°C, 40°C and 70°C.

The nanoparticle stability study for the hBN, Al_2O_3 and ZrO_2 were carried out by observing the changes color and sedimentation of nanoparticle with time. The concentration of nanoparticle was verified by analyzing the value of light that pass through nano-oil sample by using UV-Spectrometer. The absorbance value that obtained from UV-Spectrometer indicated that the temperature can affect the stability of hexagonal boron nitride. Either at low temperature or at high temperature. Different with alumina oxide and zirconia nanoparticle the stability of nanoparticle still stable even after charging with high temperature and left at room temperature. Although that, at the 70°C all nanoparticle more dispersed well in lubricant SAE15 W40 compared to the other temperature. This may be due to the increment of thermal conductivity in the nano-oil. The atomic movement of nanoparticle will more aggressive and prevent nanoparticle from easy to agglomerate. Besides that, UV-Spectrometer also proves that nanoparticles can be efficiently dispersed in the lubricant with adequate homogenize. This is due to the absorbance value was higher after homogenize time.

5.1 Recommendation

In order to further this experiment, there is a few suggestion in the future experiments.

1. This experiment can be extended with different surfactants. Surfactants have been proving extensively used as agents to prevent agglomeration of nanoparticles in lubricant.
2. The researcher needs to understand the physical and chemistry properties of the nanoparticles. This is because every type of nanoparticle has their own characteristic once introduce to temperature.
3. The researcher need to find another method other than UV-Spectrometer. This is because of UV-Spectrometer not able to verify the stability of nanoparticle when nano-oil to viscous.
4. This study can also be extended to the microstructure analysis characterization by using scanning electron microscopy (SEM) before and after nanoparticle imposed to temperature. In order to understand the average size of nanoparticle once introduce temperature.

REFERENCES

- Agarwal, D. K., Vaidyanathan, A., & Kumar, S. S. (2016). Experimental investigation on thermal performance of kerosene–graphene nanofluid. *Experimental Thermal and Fluid Science*, 71, 126-137. doi:10.1016/j.expthermflusci.2015.10.028
- Angayarkanni, S. A., Sunny, V., & Philip, J. (2015). Effect of Nanoparticle Size, Morphology and Concentration on Specific Heat Capacity and Thermal Conductivity of Nanofluids. *J Nanofluids Journal of Nanofluids*, 4(3), 302-309. doi:10.1166/jon.2015.1167
- Asango, A., La Rocca, A., and Shayler, P., "Investigating the Effect of Carbon Nanoparticles on the Viscosity of Lubricant Oil from Light Duty Automotive Diesel Engines," SAE Technical Paper 2014-01-1481, 2014, doi:10.4271/2014-01-1481.
- Asrul, M., Zulkifli, N., Masjuki, H., & Kalam, M. (2013). Tribological Properties and Lubricant Mechanism of Nanoparticle in Engine Oil. *Procedia Engineering*, 68, 320-325. doi:10.1016/j.proeng.2013.12.186
- Asrul, M., Zulkifli, N., Masjuki, H., & Kalam, M. (2013). Tribological Properties and Lubricant Mechanism of Nanoparticle in Engine Oil. *Procedia Engineering*, 68, 320-325. doi:10.1016/j.proeng.2013.12.186
- Cesarano III J., Aksay I., "Processing of Highly Concentrated Aqueous Alumina Suspensions Stabilized with Polyelectrolytes." *Journal of The American Ceramic Society*, 71, No: 12 (1988), 1062-1067
- Choi Y., Lee K., Hwang Y., Cheong S., et al., "Understanding the Role of Nanoparticles in Nano-oil Lubrication," *Tribol. Lett.* 35(2):127-131, 2009.
- Cremaschi, Lorenzo; Wong, Thiam; and Bigi, Andrea A. M., "Thermodynamic and Heat Transfer Properties of Al₂O₃ Nanolubricants" (2014). *International Refrigeration and Air Conditioning Conference*. Paper 1500. <http://docs.lib.purdue.edu/iracc/1500>

D, M., P., K., P, M. R., &N.h., J. (2014). Analysis of lubrication properties of zinc-dialkyl-dithio-phosphate (ZDDP) additive on Karanja oil (Pongamiapinnatta) as a green lubricant. IJER International Journal of Engineering Research,3(8), 494-496. doi:10.17950/ijer/v3s8/804

D.Sundeeep,Use of Nanotechnology in Reduction of Friction and Wear 2014 2349-2163

Erhan, S. Z., Sharma, B. K., & Perez, J. M. (2006). Oxidation and low temperature stability of vegetable oil-based lubricants. Industrial Crops and Products,24(3), 292-299. doi:10.1016/j.indcrop.2006.06.008

Garcés, R., Martínez-Force, E., & Salas, J. J. (2011). Vegetable oil basestocks for lubricants. Grasas Y Aceites,62(1), 21-28. doi:10.3989/gya.045210.

Gubarevich, A., Usuba, V., Kakudate, Y., Tanaka, A., &Odawara, O. (2004). Diamond powders less than 100 nm in diameter as effective solid lubricants in vacuum, Japanese journal of applied physics, 43 (7A), 920–923

H. Amiruddin.,M.F.B.Abdollah.,AM. Idris.,M.I.H.C.Abdullah., N.Tamaldin. (2015). Stability of nano-oil by pH control in stationary conditions. Proceedings of Mechanical Engineering Research Day 2015, pp. 55-56, March 2015

Hendraningrat, L., &Torsæter, O. (2014). Metal oxide-based nanoparticles: Revealing their potential to enhance oil recovery in different wettability systems. Appl Nanosci Applied Nanoscience,5(2), 181-199. doi:10.1007/s13204-014-0305-6.

Hong, K. S., Hong, T., & Yang, H. (2006). Thermal conductivity of Fe nanofluids depending on the cluster size of nanoparticles. Appl. Phys. Lett. Applied Physics Letters,88(3), 031901. doi:10.1063/1.2166199

Huang, H.D.; Tu, J.P.; Gan, L.P.; Li, C.Z. An investigation on tribological properties of graphite nanosheets as oil additive. Wear 2006, 261, 140– 144.

Hudson LK, Eastoe J, Dowding PJ. Nanotechnology in action:overbasednanodetergents as lubricant oil additives. Adv Colloid Interface Sci 2006;123e126(0):425e31.

Hutchinson R, Reid S. Lubricant compositions; 2007, Google Patents.

Jianhua, Nozawa R, Ferdows M, Murakami K, Ota M. Effects of cyclodextrin solutions on methane hydrate formation. In: Proceedings of the Asme/Jsme thermal engineering summer heat transfer conference 2007, Vol 2; 2007.p. 655e9.

Keller AA, Wang H, Zhou D, Lenihan HS, Cherr G, Cardinale BJ, Miller R, Ji Z (2010) Stability and aggregation of metal oxide nanoparticles in natural aqueous matrices. *Environ Sci Technol* 44(6):1962–1967.

Kimura, Y., Wakabayashi, T., Okada, K., Wada, T., and Nishikawa, H., Boron nitride as a lubricant additive, *Wear*, 232 (1999) 199–206.

Kouloulis, K., Sergis, A., & Hardalupas, Y. (2016). Sedimentation in nanofluids during a natural convection experiment. *International Journal of Heat and Mass Transfer*, 101, 1193-1203. doi:10.1016/j.ijheatmasstransfer.2016.05.113

Kulkarni, D. P., Das, D. K., & Chukwu, G. A. (2006). Temperature Dependent Rheological Property of Copper Oxide Nanoparticles Suspension (Nanofluid). *Journal of Nano science and Nanotechnology J. Nano sci. Nanotech.*, 6(4), 1150-1154. doi:10.1166/jnn.2006.187

Lee, J., Cho, S., Hwang, Y., Cho, H., Lee, C., Choi, Y., . . . Kim, S. H. (2009). Application of fullerene-added nano-oil for lubrication enhancement in friction surfaces. *Tribology International*, 42(3), 440-447. doi:10.1016/j.triboint.2008.08.003

Li W, Zheng S, Cao B, Ma S. Friction and wear properties of ZrO₂/SiO₂ composite nanoparticles. *J Nanoparticle Res* 2011;13(5):2129e37

Lorenzo & T. W. (2014). Thermodynamic and Heat Transfer Properties of Al₂O₃ Nanolubricants. 2463,, Page 1. Retrieved May 15, 2016. Oklahoma State University, School of Mechanical and Aerospace Engineering Stillwater, OK, USA

Mahbubul, I., Shahrul, I., Khaleduzzaman, S., Saidur, R., Amalina, M., & Turgut, A. (2015). Experimental investigation on effect of ultrasonication duration on colloidal dispersion and thermophysical properties of alumina–water nanofluid. *International*

Journal of Heat and Mass Transfer, 88, 73-81.

doi:10.1016/j.ijheatmasstransfer.2015.04.048.

Mohamad SA, Ahmed NS, Hassanein SM, Rashad AM (2012). Investigation of polyacrylates copolymers as lube oil viscosity index improvers. *J Petroleum Sci Eng* 2012;100(0):173e7.

Noria (2012) Oil Viscosity - How It's Measured and Reported. (n.d.). Retrieved May 16, 2016, from <http://www.machinerylubrication.com/Read/411/oil-viscosity>

Omri, K., Najeh, I., Dhahri, R., Ghoul, J. E., & Mir, L. E. (2014). Effects of temperature on the optical and electrical properties of ZnO nanoparticles synthesized by sol-gel method. *Microelectronic Engineering*, 128, 53-58. doi:10.1016/j.mee.2014.05.029

Padgurskas J, Rukui_za R, Kreivaitis R, Asadauskas SJ, Braz_inskiene_ D. Tribologic behaviour and suspension stability of iron and copper nanoparticles in rapeseed and mineral oils. *Tribol e Mater Surfaces Interfaces* 2009;3(3):97e102.

Paul, G., Chopkar, M., Manna, I., & Das, P. (2010). Techniques for measuring the thermal conductivity of nanofluids: A review. *Renewable and Sustainable Energy Reviews*, 14(7), 1913-1924. doi:10.1016/j.rser.2010.03.017

Prasher, R., Phelan, P. E., & Bhattacharya, P. (2006). Effect of Aggregation Kinetics on the Thermal Conductivity of Nanoscale Colloidal Solutions (Nanofluid). *Nano Letters*, 6(7), 1529-1534. doi:10.1021/nl060992s

Qui S, Zhou Z, Dong J, Chen G. Preparation of Ni nanoparticles and evaluation of their tribological performance as potential additives in oils. *Journal of Tribology* 1999;123:441-443.

Refining the grip on nature's fine grains - Drilling Contractor. (2012). Retrieved May 27, 2016, from <http://www.drillingcontractor.org/refining-the-grip-on-natures-fine-grains-15705>

Roberts, E. (1990). Thin solid lubricant films in space. *Tribology International*, 23(2), 95-104. doi:10.1016/0301-679x(90)90042-n

Rudnick, L. (2009). Additives for Industrial Lubricant Applications. Lubricant Additives Chemical Industries Chemistry and Applications, Second Edition, 493-509. doi:10.1201/9781420059656-c20

Sadri, R., Ahmadi, G., Togun, H., Dahari, M., Kazi, S., Sadeghinezhad, E., & Zubir, N. (2014). An experimental study on thermal conductivity and viscosity of nanofluids containing carbon nanotubes. *Nanoscale Res Lett Nanoscale Research Letters*, 9(1), 151. doi:10.1186/1556-276x-9-151

Sarsam, W. S., Amiri, A., Zubir, M. N., Yarmand, H., Kazi, S., & Badarudin, A. (2016). Stability and thermophysical properties of water-based nanofluids containing triethanolamine-treated graphenenanoplatelets with different specific surface areas. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 500, 17-31. doi:10.1016/j.colsurfa.2016.04.016

Sassiat P, Machtalere G, Hui F, Kolodziejczyk H, Rosset R. Liquid chromatographic determination of base oil composition and content in lubricating oils containing dispersants of the polybutenylsuccinimide type. *Anal Chim Acta* 1995;306(1):73e9.

Sridhara, V., & Satapathy, L. (2015). Effect of Nanoparticles on Thermal Properties Enhancement in Different Oils – A Review. *Critical Reviews in Solid State and Materials Sciences*, 40(6), 399-424. doi:10.1080/10408436.2015.1068159

Shahnazar, S., Bagheri, S., & Hamid, S. B. (2016). Enhancing lubricant properties by nanoparticle additives. *International Journal of Hydrogen Energy*, 41(4), 3153-3170. doi:10.1016/j.ijhydene.2015.12.040

Sharma, B. K., Adhvaryu, A., Liu, Z., & Erhan, S. Z. (2006). Chemical modification of vegetable oils for lubricant applications. *Journal of the American Oil Chemists' Society J Amer Oil Chem Soc*, 83(2), 129-136. doi:10.1007/s11746-006-1185-z

Shipway, A. N., Lahav, M., Gabai, R., & Willner, I. (2000). Investigations into the Electrostatically Induced Aggregation of Au Nanoparticles. *Langmuir*, 16(23), 8789-8795. doi:10.1021/la000316k.

Solangi, K., Kazi, S., Luhur, M., Badarudin, A., Amiri, A., Sadri, R., . . . Teng, K. (2015). A comprehensive review of thermo-physical properties and convective heat transfer to nanofluids. *Energy*, 89, 1065-1086. doi:10.1016/j.energy.2015.06.105

Stachowiak, G.W.; Batchelor, A.W.; Stachowiak, G.B. *Experimental Methods in Tribology*; Elsevier: Amsterdam, The Netherlands, 2004; Volume 44.

StachowiakGW, BatchelorAW, editors Chapter 3-lubricants and their composition. In: *Engineering tribology*. 4th ed. Boston: Butterworth-Heinemann; 2014. p. 51e104.

Tang E, Cheng G, Ma X, Pang X, Zhao Q. Surface modification of zinc oxide nanoparticle by PMAA and its dispersion in aqueous system. *Appl Surf Sci* 2006;252(14):5227e32.

Tang Z, Li S. A review of recent developments of friction modifiers for liquid lubricants (2007epresent). *CurrOpin Solid State Mater Sci* 2014;18(3):119e39. Forbes ES. Antiwear and extreme pressure additives for lubricants. *Tribology* 1970;3(3):145e52.

Vajjha, R. S. and D. K. Das (2009), Specific Heat Measurement of Three Nanofluids and Development of New Correlations, *J. of Heat Transfer-Transactions of the ASME*, 131(7): 1-7.

Vaisman, H. D. Wagner, G. Marom, 2006 The role of surfactants in dispersion of carbon nanotubes *Advances in Colloid and Interface Science* 128-130 37 46.

Wen, D., & Ding, Y. (2004). Experimental investigation into convective heat transfer of nanofluids at the entrance region under laminar flow conditions. *International Journal of Heat and Mass Transfer*,47(24), 5181-5188 doi:10.1016/j.ijheatmasstransfer.2004.07.012

Wu W, He Q, Jiang C et al (2008) Magnetic iron oxide nanoparticles: synthesis and surface functionalization strategies. *Nano scale Res Lett* 3:397–415.

Wu YY, Tsui WC, Liu TC. Experimental analysis of tribological properties of lubricating oils with nanoparticle additives. *Wear*. 2007;262:819–825.

Xialong, D., Dave, R., Yin, X., & Sundaresan, S. (2009). Deagglomeration of nanoparticle aggregates via rapid expansion of supercritical or high-pressure suspensions. *AIChE Journal*, 55(11), 2807-2826. doi:10.1002/aic.11887.

Yazawa, S., Minami, I., & Prakash, B. (2014). Reducing Friction and Wear of Tribological Systems through Hybrid Tribofilm Consisting of Coating and Lubricants. *Lubricants*, 2(2), 90-112. doi:10.3390/lubricants2020090.

Zhang BS, Xu BS, Xu Y, Gao F, Shi PJ, Wu YX. CU nanoparticles effect on the tribological properties of hydrosilicate powders as lubricant additive for steel-steel contacts. *TribolInt* 2011;44(7-8):878-86.

Zhang, Z., Simionesie, D., & Schaschke, C. (2014). Graphite and Hybrid Nanomaterials as Lubricant Additives. *Lubricants*, 2(2), 44-65. doi:10.3390/lubricants2020044

Zirconia | Zirconia Grinding | Ceramic Zirconia | Zirconia Properties. (n.d.). Retrieved May 08, 2016, from <http://www.ferroc ceramic.com/zirconia.htm>.

APPENDIX

Hexagonal boron nitride at 30°C

hBN 30	1 st day		2 nd day		3 rd day		4 th day	5 th day	1 week	2week	3week	1month 21/9	2month 19/10							
	wv	abs	wv	abs	wv	abs	Wv	abs	wv	abs	wv	abs	wv	abs						
1 st reading	439	3.9813	488	3.4800	493	3.2947	541	3.3662	541	3.1886	541	3.2845	491	3.9999	526	3.0687	543	3.0634	586	2.9876
2 nd reading	464	3.9813	493	3.4800	484	3.9069	576	3.3362	541	3.1886	541	3.1331	494	3.1378	526	3.1002	527	3.1002	571	3.0063
3 rd reading	457	3.9813	484	3.4136	489	3.2595	571	3.3362	541	3.1886	541	3.0867	494	3.1378	526	3.0787	538	3.0687	574	3.0146
Average		3.9813		3.5244		3.4870		3.3462		3.1886		3.1681		3.1078		3.0867		3.0774		3.0028

Hexagonal boron nitride at 40°C

hBN 40	1 st day		2 nd day		3 rd day		4 th day		5 th day		1 week		2week		3week		1month		2month	
	wv	abs	wv	abs	wv	abs	Wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs
1 st reading	439	3.9999	448	3.9069	452	3.3337	526	3.6796	526	3.3602	528	3.1825	537	3.1542	541	3.1321	555	3.1321	571	2.8100
2 nd reading	440	3.9813	472	3.6058	448	3.9055	527	3.6796	540	3.3499	526	3.1825	527	3.1765	537	3.0929	546	3.0087	571	2.9125
3 rd reading	439	3.9999	562	3.9069	517	3.9999	527	3.6796	526	2.9407	524	3.1542	532	3.1542	537	3.0565	540	3.0656	537	2.8838
Average	3.9937		3.8065		3.7463		3.6796		3.2169		3.1730		3.1624		3.0983		3.0657		2.9885	

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Hexagonal Boron Nitride at 70°C

hBN 70	1 st day		2 nd day		3 rd day		4 th day		5 th day		1 week		2week		3week		1month		2month	
	wv	abs	wv	abs	wv	abs	Wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs
1 st reading	300	3.9999	440	3.9999	440	3.5940	439	3.3606	440	3.5232	452	3.4641	464	3.4800	478	3.2897	500	3.1660	538	3.0876
2 nd reading	440	3.9999	440	3.5940	442	3.7646	439	3.9999	440	3.3750	433	3.6725	464	3.3038	442	3.3866	436	3.1742	539	3.0543
3 rd reading	440	3.9999	440	3.5940	440	3.5940	441	3.3499	440	3.6807	448	3.3503	440	3.6796	440	3.2893	448	3.1660	538	3.0653
Averag	3.9999		3.7293		3.6508		3.5701		3.5263		3.4956		3.4878		3.3218		3.1687		3.0553	

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Alumina Oxide at 30°C

Alumina 70	1 st day		2 nd day		3 rd day		4 th day		5 th day		1 week		2week		3week		1month 4/10		2month 1/11	
	wv	abs	wv	abs	wv	abs	Wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs
1 st reading	441	3.5768	523	3.0856	523	3.1943	526	3.1098	521	3.0083	526	3.0681	546	2.9100	528	3.2095	432	3.3346	527	2.9366
2 nd reading	465	3.4209	527	2.8615	527	3.1934	526	3.3215	526	2.9498	525	3.0684	541	3.0076	528	2.0618	435	3.3566	527	2.1174
3 rd reading	465	3.5786	521	3.0845	521	3.1903	526	3.0123	525	2.9904	523	3.1710	561	2.9838	526	2.2095	432	3.3346	532	2.1647
Average	3.6724		3.2880		3.2672		3.2072		3.1926		3.1445		3.1432		2.9501		2.9472		2.9466	

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Alumina Oxide at 70°C

Alumina 30	1 st day		2 nd day		3 rd day		4 th day		5 th day		1 week		2week		3week		1month		2month	
	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs
1 st reading	439	3.3346	439	3.2095	440	3.0865	442	3.1917	515	3.0859	521	3.0375	522	2.9882	520	2.9141	528	2.8718	527	2.7654
2 nd reading	440	3.3566	440	3.2095	438	3.1445	440	2.9956	490	3.0369	528	3.0375	523	2.9879	528	2.8843	538	2.8727	564	2.6543
3 rd reading	434	3.3346	434	3.1406	437	3.1445	439	2.9956	484	3.0369	516	3.0375	526	2.9872	523	2.9073	523	2.9049	527	2.8727
Average		3.3419		3.1865		3.1251		3.0609		3.0532		3.0375		2.9877		2.9019		2.8831		2.7739

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Zirconia at 30°C

Zirconia 30	1 st day		2 nd day		3 rd day		4 th day		5 th day		1 week		2week		3week		1month 17/10		2month 14/11	
	wv	abs	wv	abs	wv	abs	Wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs
1 st reading	569	3.5239	-	-	-	-	-	-	546	3.4946	537	3.2876		3.1440	-	-	438	3.4749	578	3.3953
2 nd reading	553	3.4878	-	-	-	-	-	-	546	3.5239	540	3.2345		3.1691	-	-	439	3.4209	590	3.3953
3 rd reading	541	3.4878	-	-	-	-	-	-	546	3.4451	538	3.3879		3.1532	-	-	550	3.3591	561	3.3953
Average		3.4998								3.4878		3.4383		3.1554			475	3.4183		3.3953

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Zirconiaat 40°C

Zirconia 40	1 st day		2 nd day		3 rd day		4 th day		5 th day		1 week		2week		3week		1month		2month	
	wv	abs	wv	abs	wv	abs	Wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs
1 st reading	537	3.4473	-	-	-	-	-	-	-	-	449	3.3398	561	3.4451		3.2456	530	3.2453	530	3.0067
2 nd reading	537	3.6284	-	-	-	-	-	-	-	-	452	3.7507	537	3.0371		3.1256	526	3.1451	526	3.2435
3 rd reading	586	3.4539	-	-	-	-	-	-	-	-	449	3.3398	541	3.3549		3.4657	526	3.2284	526	3.0987
Average		3.5099	-	-	-	-	-	-	-	-		3.5062		3.3434		3.3043		3.2790		3.1879

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Zirconia at 70°C

Zirconia 70	1 st day		2 nd day		3 rd day		4 th day		5 th day	1 week	2week	3week	1month	2month					
	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs	wv	abs			
1 st reading	429	3.6284	-	-	-	-	-	-	-	449	3.4946	537	3.5239	561	3.3274	561	3.3398	578	3.2791
2 nd reading	453	3.6284	-	-	-	-	-	-	-	448	3.5239	543	3.4451	537	3.3724	537	3.3403	590	3.2791
3 rd reading	449	3.5201	-	-	-	-	-	-	-	449	3.4451	538	3.6284	563	3.3364	563	3.3398	561	3.1234
Average		3.5923									3.4878		3.4728		3.3454		3.3399		3.3218

- Wv- Wavelength
- Abs- Absorbance



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