



## Identification of Thermophysical and Rheological Properties of SAE 5w-30 with Addition of Hexagonal Boron Nitride

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### ABSTRACT

This research uses SAE 5W-30 lubricant base material with the addition of Hexagonal Boron Nitride (hBN) nanoparticle additives. This study aims to analyze the thermophysical and rheological properties of lubricants with the addition of nanoparticles. This study used a two-step method where nanoparticles were first prepared separately. Then, the nanoparticles with varying volume fractions of 0.05%, 0.10%, and 0.15% were added to the base fluid in the processing step. It was followed by the stirring process using a magnetic stirrer and ultrasonic homogenizer process. Furthermore, the nano lubricant was tested for thermophysical properties, including viscosity, density, thermal conductivity, and sedimentation. Morphological analysis carried out using an SEM test showed that the surface of the nanoparticles was spherical, like platelets. Phase identification using the XRD test showed a crystalline phase. Functional group analysis using FTIR showed the presence of.....

*Keywords: Oil 5W-30, Hexagonal Boron Nitride, Nanolubricant, Thermophysical Properties, Rheology.*

### ABSTRAK

Penelitian ini menggunakan bahan dasar pelumas SAE 5W-30 dengan penambahan zat aditif nanopartikel *Hexagonal Boron Nitride* (hBN). Penelitian ini bertujuan untuk menganalisis sifat termofisik dan reologi pelumas dengan penambahan nanopartikel. Metode pada penelitian ini menggunakan *two-step method* dimana nanopartikel pertama kali disiapkan secara terpisah, kemudian ditambahkan dengan nanopartikel dengan variasi fraksi volume 0,05%, 0,10%, 0,15% ke dalam cairan dasar sebagai langkah pemrosesan. Tahap selanjutnya adalah proses pengadukan menggunakan *magnetic stirrer*, dan proses *ultrasonic homogenizer*. Selanjutnya *nanolubricant* di uji sifat termofisiknya yang meliputi viskositas, massa jenis, konduktivitas termal, dan sedimentasi. Analisis morfologi menggunakan uji SEM menunjukkan permukaan nanopartikel berbentuk bulat seperti trombosit. Identifikasi fasa menggunakan uji XRD menunjukkan fasa kristalin. Analisis gugus fungsi menggunakan FTIR menunjukkan adanya.....

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Kata Kunci: Oli 5W-30, Hexagonal Boron Nitride, Nanolubricant, Sifat Termofisik, Reologi

### 1. Introduction

The engine or automotive spare parts manufacturing industry always use lubricant for the engines with working principle involving friction, such as during lathe, which induces friction between

the chisel and specimen or piston on the motor vehicle. To lower the effects of friction, as well as wear and tear, the use of lubricant is necessary [1].

There are various types and merk of lubricant circulating on the market. This study used SAE 5W-30 lubricant, an engine lubricant formulated from

synthetic base oil with a selected additive that offers the highest performance compared to the other conventional lubricant. The value within the name of SAE 5W-30 lubricants shows a viscosity of 5 during cold temperatures and 30 viscosity at the perceived 100°C temperature [2].

The addition of nanoparticles as an additive substance on lubricant enhances its thermophysical properties, particularly increasing its viscosity, density, and thermal conductivity. In this study, we added *Hexagonal Boron Nitride* (hBN) nanoparticle since hBN nanoparticle has solid particle that can withstand higher temperatures in their application, where effective lubrication at high temperature is highly required [3].

The hBN nanoparticle presents thermophysical properties, including density, viscosity, and thermal conductivity [4]. Additionally, the addition of nanoparticles also increases the connection between lubricant stress and strain, which correlates with the rheology features. Rheology is a scientific branch that covers fluid phenomena, such as solids, liquids, and gases[5].

The lubricant that has been added with nanoparticles is named nano lubricant. Nanolubricant is a dispersion of nanoparticle and base oil, with the nanoparticle at 1-100 nm size. Nanolubricant is commonly used on heat transfer instruments to improve their thermal efficiency, which includes conductivity, viscosity, and thermal diffusivity than the basic lubricant [6].

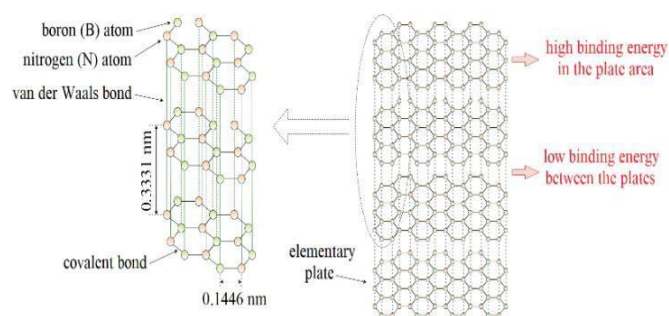
According to the above explanation, this study used an experimental method by modifying the *hexagonal boron nitride* (hBN) nanoparticle to improve its density, viscosity, thermal conductivity, and rheology to identify the best stability for the nano lubricant. The results of the research were expected to be used for future studies on the application of *nano lubricant Hexagonal Boron Nitride* (hBN).

## 2. Methodology of Research

### 2.1. Nanoparticle

*Boron nitride* (BN) is an isoelectronic and isostructural chemical compound toward carbon with the same composition of boron and nitrogen atoms. The hBN nanoparticle presents a similarity with carbon. The boron nitride was produced in the form of amorf and crystal. From its crystalline form, boron nitride is divided into three types, namely *sphalerite boron nitride* ( $\beta$ -BN), which resembles a cubic diamond, *wurtzite boron nitride* ( $\gamma$ -BN) resembling a hexagonal diamond, and *hexagonal boron nitride* (hBN) resembling graphite [7].

In addition, Hexagonal Boron Nitride (hBN) is a type of polymorphic form of boron nitride. In the last few years, the production of hBN has increased dramatically due to the increasing interest of researchers in this compound. This increase is induced by the excellent properties of hBN, including its great chemical endurance, thermal stability, and thermal conductivity without electrical conductivity [4]. According to the tribology perspective, the lamellar structure of hexagonal nitride is highly tight, as illustrated in Figure 1.



**Figure 1.** Hexagonal Structure of Boron Nitride

### 2.2. Characterization of Material

The characterization of the material was carried out to identify the property of the material being investigated. This study used three types of characterization tests. The first test was carried out using a *scanning electron microscope* (SEM) to investigate the surface morphology of the particle as the instrument facilitated detailed particle investigation [9]. The second test was completed using *X-ray Diffraction* (XRD) to examine the properties of the material, which included its crystal structure, crystallite size, and strain [10]. The third test was *Fourier Transform Infrared* (FTIR) to identify the characteristic of the primary functional group using the spectrum infrared to scan the sample [11].

### 2.3. Preparation of Nanolubricant

The nano lubricant was prepared using SAE 5W-30 lubricant as the base oil and three samples of hBN nanoparticle with volume fractions of 0.05, 0.10, and 0.15%. Further, the mixture of lubricant and nanoparticle was stirred using *Thermo Scientific* and juga *magnetic bar* at 1250 rpm speed for 20 minutes at room temperature. Then, we conducted homogenization using an *ultrasonic homogenizer* for 30 minutes. After that, the nano lubricant was ready to be used. The results of nanolubricant preparation are shown in Figure 2.



**Figure 2.** Results of Nanolubricant Preparation with Volume Fractions of 0.05, 0.10, and 0.15%

**2.4. Analysis of Thermophysical Property**

The thermophysical properties of a nano lubricant are highly essential for improving the heat transfer process [12]. Meanwhile, the thermophysical property of a material is highly affected by its type and specific content. The parameter of the nano lubricant’s thermophysical property test included the tests of viscosity, density, thermal conductivity, and sedimentation.

Viscosity represents the thickness of a fluid. Higher viscosity means thicker fluid and more incredible difficulty for the fluid to flow, while more watery fluid can flow more easily [13]

This study used Viscometer NDJ-8S to measure the dynamic viscosity, using rotor Number 1 and various speeds of 6 rpm, 12 rpm, 30 rpm, and 60 rpm, at 30°C to 100°C temperature.

In addition, density represents the measured mass per volume unit of fluid. A higher density of a fluid reflects its greater mass per volume [14]. The density of fluid changes following the change in temperature and is least affected by the pressure, except for the compressible fluid [15]. The density is symbolized by rho ( $\rho$ ) with  $\text{kg/m}^3$  unit. The density was calculated using Formula 1.

$$\rho = \frac{m}{V} \tag{1}$$

- Description:
- $\rho$  = density of the fluid ( $\text{kg/m}^3$ )
  - $m$  = mass (kg)
  - $V$  = Volume ( $\text{m}^3$ )

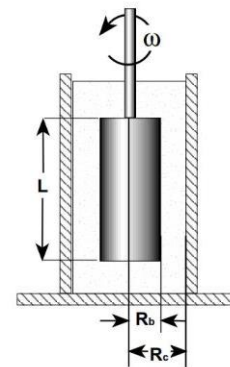
Thermal conductivity shows the capacity of a material to transfer heat from one side to another side. A material with greater thermal conductivity is commonly known as a conductor, while a material with low thermal conductivity is popular as an isolator. Thermal conductivity level reflects the speed of heat flow within a material [16]. The thermal conductivity test was carried out using KD2 Pro *Thermal Properties Analyzer* with an accuracy of 5-10% resistivity.

Meanwhile, sedimentation is a natural process of phase separation due to the gravitation force and density difference. Sedimentation has been frequently implemented in the industry sector. The sedimentation method has been reported as the simplest method for determining the nano lubricant stability as it facilitates investigation of nano lubricant being set aside at a specific period [17].

**2.5. Analysis of Rheology Property**

Rheology is a scientific branch that discusses the phenomena of material flow, such as solid, fluid, and gasses, particularly the ones involving the relation between shear rate, shear stress, and time [18]

In rheology measurement, the deformation force was used as the stress or the force per unit area. The deformation degree implemented on a material is known as strain [19]. The rheology measurement was carried out using a spindle and vessel, as illustrated in Figure 3.



**Figure 3.** Spindle and Vessels for Rheology Measurement

The shear rate represents the rate of fluid performance during the stream. The shear rate is determined by the geometry and stream speed. The shear rate was calculated using Formula 2.

$$\gamma = \frac{2\omega R_c^2 R_b^2}{R^2(R_c^2 - R_b^2)} \tag{2}$$

- Description:
- $\gamma$  = Shear Rate (/s)
  - $\omega$  = angular velocity of the spindle (*spindle*) (rad/sec)
  - $R_c$  = Vessel radius (cm)
  - $R_b$  = Spindle radius (*spindle*) (cm)

*Shear stress* is a multiplication result between the shear rate and dynamic viscosity. The shear stress was calculated using Formula 3.

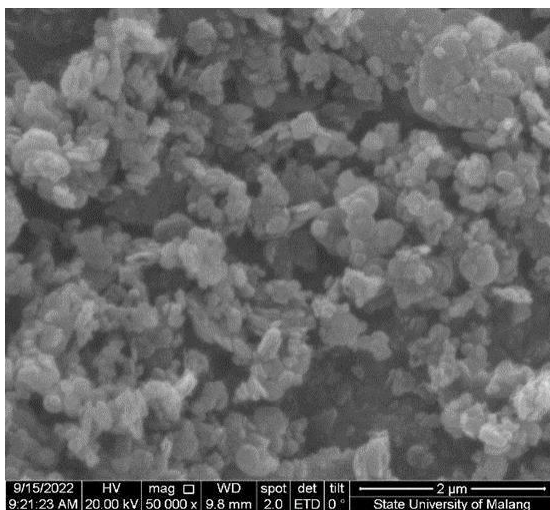
$$\tau = \gamma\mu \tag{3}$$

- Description:
- $\tau$  = *Shear stress* (mPa.s)
  - $\gamma$  = *Shear rate* (/s)
  - $\mu$  = *Dynamic viscosity* (kg/m.s)

### 3. Result and Discussion

#### 3.1 Scanning Electron Microscope (SEM) Analysis of Hexagonal Boron Nitride (hBN)

The Scanning Electron Microscopy (SEM) test aims to provide information on the surface morphological structure of nanoparticles. The results of the SEM characterization test of Hexagonal Boron Nitride nanoparticles are shown in Figure 4 below.



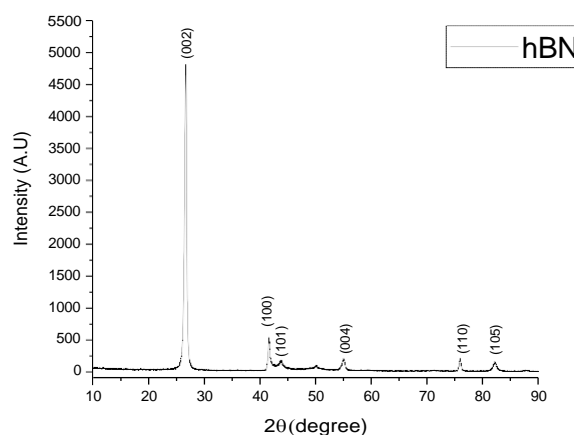
**Figure 4.** SEM results of Hexagonal Boron Nitride (hBN) Nanoparticles Magnification 50,000×.

Based on the results of the SEM test analysis. Hexagonal Boron Nitride (hBN) nanoparticles with a magnification of 50,000×, it is known that Hexagonal Boron Nitride (hBN) nanoparticles have a spherical and agglomerated surface morphology. Where these results are in line with the opinion of [20], that hBN nanoparticles have a platelet structure with a spherical morphology and also lead to clumping. The measurement results of hBN nanoparticles using Image J software showed results of 41 nm, 49 nm, 59 nm, 72 nm, and 89 nm. The measurement results are also reinforced by the statement of [21], where the average particle size distribution of hBN nanoparticles shows an average size of 64.87 nm and 68.97 nm which is below 100nm.

#### 3.2 X-Ray Diffraction (XRD) Analysis of Hexagonal Boron Nitride (hBN).

XRD testing in this study was carried out to identify the phase, grain shape, and crystallinity formed on Hexagonal Boron Nitride (hBN)

nanoparticles through the diffraction peaks that occur on the XRD graph.



**Figure 5** Graph analysis of hBN XRD testing

Based on Figure 5 it shows the hBN graph has the highest peak intensity of 4723.83 at  $2\theta = 26.7^\circ$ . This is in accordance with the opinion of [22] that the highest range is in the value of  $26.5-26.8^\circ$ . The crystal form of Hexagonal Boron Nitride (hBN) nanoparticles was analyzed using MATCH software, the results of the analysis showed that the crystal form of hBN is hexagonal, the data is in line with research [23], that the crystal form of hBN is hexagonal. Meanwhile, to find out the size of the crystal, you can use the Scherrer equation as follows [24].

$$D = \frac{K \lambda}{\beta \cos \theta}$$

information:

- D = Crystal Diameter (nm)
- K = Crystal Form Factor (0.9)
- $\lambda$  = X-Ray Wavelength (0.15406)
- $\beta$  = FWHM Value (Full Width at Half Maximum) (rads)
- $\theta$  = Diffraction Angle ( $^\circ$ )

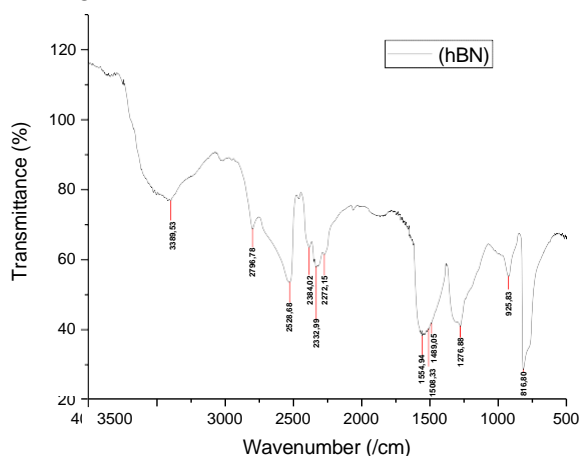
The crystal size value obtained using the Scherrer equation is obtained at the peak of  $2\theta = 26.72^\circ$  with a value of 54.20 nm.

#### 3.3 Fourier Transform Infrared (FTIR) Analysis of Hexagonal Boron Nitride (hBN)

Fourier Transform Infrared (FTIR) testing of Hexagonal Boron Nitride (hBN) nanoparticles has the objective of determining its elemental content and functional groups. The results of the FTIR test are in the form of a graph with a wavelength range of 4000-500.



Based on Figure 6. shows the hBN FTIR spectra curve in the wavenumber range of 4000-500 cm<sup>-1</sup>. The peak of 3389.53 cm<sup>-1</sup> has a wide absorption range due to O-H stretching [25]. The peak band of 2796.78 indicates adsorption of -NH<sub>2</sub> and -OH groups cm<sup>-1</sup>, the peak bands of 2528.68 and 2384.02 cm<sup>-1</sup> indicate B-H stretching [26]. The peak band of 1554.94 shows B-N [27]. The peak band of 1508.33 indicates B-O stretching, the peak band of 925.83 indicates B-O Out Of Plane Bending [28], while the peak band of 816.80 indicates B-N-B Out Of Plane Bending [29].



**Figure 6.** FTIR Graphic Analysis of Hexagonal Boron Nitride (hBN)

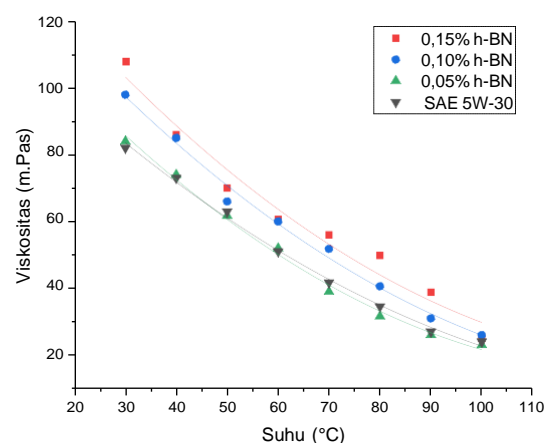
### 3.4 Sedimentation

Sedimentation testing is one of the easiest tests to do to see the stability of the nanofluid, the nanofluid sample to be tested for sedimentation is placed in a cup. Over time, the nanofluid will settle due to the gravitational force that influences it. The force of attraction between nanoparticles can create agglomeration which accelerates the sedimentation process [30]. Large nanoparticles precipitate faster than small nanoparticles. A nanofluid will be considered stable when the concentration between the particles remains constant [31].

Based on the results of the nanolubricant sedimentation test, it showed that the SAE 5W-30 lubricant nanolubricant with the addition of volume of the hexagonal boron nitride fraction for a duration of 0, 7, 14, 21, and 28 days resulted in a change in sedimentation in each sample. Based on visual observations on each nanolubricant sample it is known that on the 14th day precipitation has occurred marked by the presence of a white precipitate at the bottom of the nanolubricant.

This study used a two-step method of nanolubricant synthesis. According to [32], synthesis with a two-step process can also affect the stability of the nanolubricant because it tends to agglomerate and also has limited control over the size and shape of the particles during synthesis.

### 3.5 Viscosity



**Figure 7.** Viscosity Value of SAE 5W-30 and Nanolubricant with Volume Fraction Variations of 0.05%, 0.10% and 0.15%

Based on the chart above. SAE 5W-30 base oil with the addition of 0.15% hBN has the highest viscosity value. The highest dynamic viscosity value was obtained by adding SAE 5W-30 0.15% hBN base-oil at 30°C with a spindle speed of 30 rpm with a yield of 108 mPa.s. while the lowest dynamic viscosity value was found in SAE 5W-30 lubricant obtained by adding 20 mPa.s of 0.05% hBN volume fraction at 100 °C and 6 rpm spindle speed. The viscosity value of SAE 5W-30 with variations in the addition of volume fractions did not increase significantly at higher temperatures, because the viscosity of the nano-lubricant was close to that of base oil. This is due to the weakening of the van der Waals forces between molecules [37].

According to the argument [38], the value of viscosity is influenced by several factors such as volume fraction, nanoparticle size, particle shape, temperature and shear rate. In this study, the viscosity value decreased with increasing temperature. This is in accordance with the argument of [39] that the higher the temperature value being treated in a nanofluid, the lower the viscosity value due to the weakening of intermolecular interactions. The increase in temperature helps the particles overcome van der Waals attractions. Van der Waals forces can

break down clusters of nanoparticles suspended in alkaline solution, weakening intermolecular interactions, and this phenomenon causes a decrease in viscosity.

### 3.6 Density

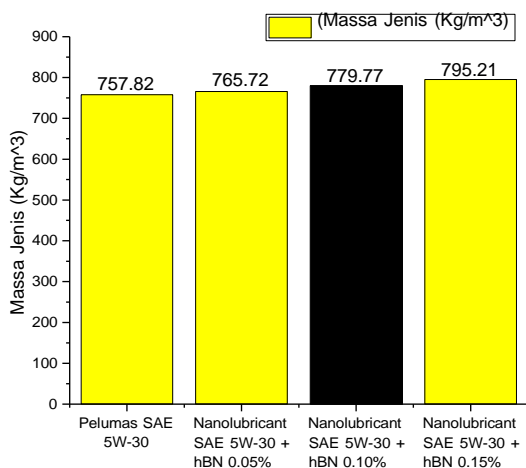


Figure 8. Results of the Nanolubricant Density Test

Density or density is a measurement of the mass of each unit volume of fluid. The higher the density value of a fluid, the greater the mass of each volume [14]. The density of a fluid changes little with temperature and very little with pressure unless the liquid is highly compressible [15].

The graphical image of the test results above explains the density of SAE 5W-30 lubricant with the addition of a volume fraction of 0.05%, 0.10%, 0.15% hBN. The highest density value was obtained with SAE 5W-30 + 0.15% hBN lubricant with a value of 795.21, while the lowest density value was obtained with 5W-30 base oil with a value of 757.82. In the opinion of [35]. The addition of h-BN nanoparticles with various mass fractions of 0.01% and 0.08% at 0.1 wt.% significantly increased the tribological behavior and density. This is in line with the opinion of [36], nanofluids have a higher density value than base lubricants. This is because the density of the nanoparticles contained in the base oil is higher. In addition, temperature can affect density.

### 3.7 Thermal Conductivity

Based on Figure 7 shows the value of the thermal conductivity of base-oil and nanolubricant with volume variations that experience an increase or decrease that is not significant. Factors that affect the thermal conductivity of nanolubricant include the size

of the nanoparticles, the shape of the nanoparticles, the type of base-oil used, and also the temperature of the nanofluid [33]. This study used hBN powder of various sizes dispersed into nanolubricant, which included 41 nm, 49 nm, 59 nm, 72 nm, and 89 nm, thus affecting the conductivity value of the nanolubricant. This is in line with the statement [34].

That the size of the nanoparticles can cause agglomeration and will cause a decrease in the stability of the nanoparticles.

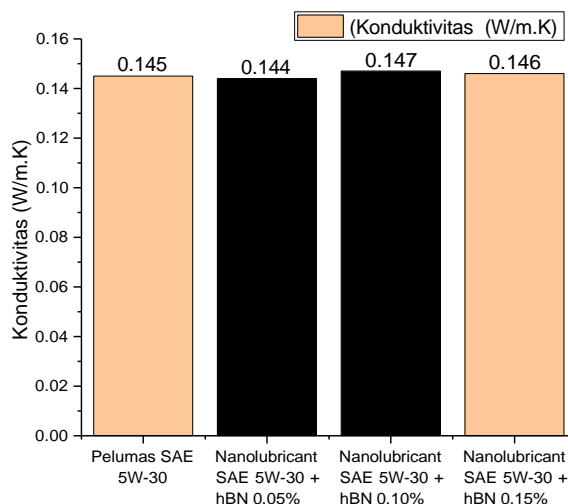
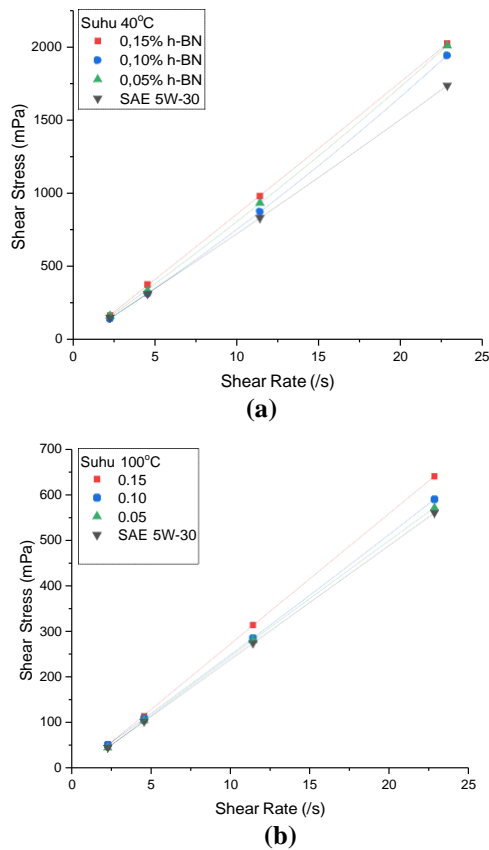


Figure 9. Results of the Nanolubricant Thermal Conductivity Test

### 3.7 Rheology

Based on a comparison of the shear stress value to the shear rate value of SAE 5W-30 lubricant and nanolubricant with variations in volume fraction of 0.05%, 0.10% and 0.15% at 40°C and 100°C. Based on the graph of Figure 9 above. This indicates that SAE 5W-30 base-oil and nanolubricant with different volume fractions exhibit Newtonian flow behavior [40]. Newtonian flow is excellent for the use of nanofluids in thermal applications such as convective heat transfer, for example in internal combustion engines [41]. SAE 5W-30 oil has a lower shear stress value than SAE 5W-30 nanolubricant and the increase in volume fraction varies. The highest shear stress value was found in the volume fraction nanolubricant 0.15 hBN at 30°C with a spindle speed of 60 rpm with a value of 2280.52 mPa.s, while the lowest shear stress value was obtained for SAE 5W-30 base lubricant of 45.65 mPa.s at a temperature of 100°C with a spindle speed of 6 rpm. Figure 8. (a) and (b) shows that the shear stress value decreases with increasing temperature from 40°C to 100°C. this

explains why van der Waals forces decrease at high temperatures [42].



**Figure 10.** Comparison of Shear Stress Values, Shear Rates of SAE 5W-30 Lubricant and Nanolubricant with Variations in Volume Fractions of 0.05%, 0.10% and 0.15% Against (a) Temperature 40°C and (b) Temperature 100 °C

Based on a comparison of the shear stress value to the shear rate value of SAE 5W-30 lubricant and nanolubricant with variations in volume fraction of 0.05%, 0.10% and 0.15% at 40°C and 100°C. Based on the graph of Figure 9 above. This indicates that SAE 5W-30 base-oil and nanolubricant with different volume fractions exhibit Newtonian flow behavior [40]. Newtonian flow is excellent for the use of nanofluids in thermal applications such as convective heat transfer, for example in internal combustion engines [41]. SAE 5W-30 oil has a lower shear stress value than SAE 5W-30 nanolubricant and the increase in volume fraction varies. The highest shear stress value was found in the volume fraction nanolubricant 0.15 hBN at 30°C with a spindle speed of 60 rpm with a value of 2280.52 mPa.s, while the lowest shear stress value was obtained for SAE 5W-30 base lubricant of 45.65 mPa .s at a temperature of 100°C with a spindle speed of 6 rpm. Figure 8. (a) and (b) shows that the shear stress value decreases with

increasing temperature from 40°C to 100°C. this explains why van der Waals forces decrease at high temperatures [42].

#### 4. Conclusion

Based on the results of the analysis discussed in this study using the experimental method, it can be concluded as follows.

The surface morphology of Hexagonal Boron Nitride (hBN) nanoparticles has crystalline properties, and the shape of rounded particles is like platelets with particle sizes of 41 nm, 49 nm, 59 nm, 72 nm, and 89 nm. Hexagonal Boron Nitride (hBN) nanoparticles undergo agglomeration due to sintering temperature and sintering time. The crystal size value of Hexagonal Boron Nitride (hBN) nanoparticles obtained a peak of  $2\theta = 26.72^\circ$  with a value of 54.20 nm. It is known that the functional groups of Hexagonal Boron Nitride (hBN) nanoparticles are located at the peaks at 816.80 and 925.83  $\text{cm}^{-1}$  Out Of Plane Bending. Nanolubricant based on SAE 5W-30 lubricant with the addition of volume fractions of 0.05%, 0.10%, 0.15% has good stability because the nanoparticles are well dispersed, but over time it will precipitate. This will affect the stability of the nanolubricant to the value of thermal conductivity. The highest dynamic viscosity value was obtained by adding SAE 5W-30 0.15% hBN base-oil at 30°C with a spindle speed of 30 rpm with a yield of 108 mPa.s. while the lowest dynamic viscosity value was found in SAE 5W-30 lubricant obtained by adding 20 mPa.s of 0.05% hBN volume fraction at 100 °C and 6 rpm spindle speed. Density value of nanolubricant increases with increasing volume fraction of nanoparticles with SAE 5W-30 lubricant. The highest density value was achieved at 0.15% volume fraction with a value of 795.21  $\text{kg/m}^3$ , while the lowest density value was obtained in SAE 5W-30 base oil with a value of 757.82  $\text{kg/m}^3$ . In addition, the viscosity value increased with increasing volume of the fraction and decreased with increasing temperature. The addition of Hexagonal Boron Nitride (hBN) nanoparticles to SAE 5W-30 lubricant shows a relationship between shear rate and shear stress, so that it can be said to be Newtonian flow. At 40°C and 100°C the value of the shear stress increases. The increase was caused by an increase in the volume fraction in the base-oil. The shear rate of

the nanolubricant at 40°C is lower than the shear rate at 100°C because the van der Waals forces decrease at high temperatures.

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