



Extensive examination of sonication duration impact on stability of Al₂O₃-Polyol ester nanolubricant

A. Nugroho^{a,b,*}, Z. Bo^a, R. Mamat^{a,b}, W.H. Azmi^b, G. Najafi^c, F. Khoirunnisa^d

^a School of Mechanical Engineering, Ningxia University, China

^b College of Engineering, Universiti Malaysia Pahang, Pekan, Pahang, Malaysia

^c Department of Biosystems Engineering, Tarbiat Modares University, Tehran, Iran

^d Department of Chemistry, Indonesia University of Education, Bandung, Indonesia

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ABSTRACT

The sonication technique is one of the effective methods to stabilize nanolubricants. This paper aims to elaborate on the sonication duration's impact on the stability of Al₂O₃-Polyol ester nanolubricants. A two-step method has been performed to prepare nanolubricants. Each sample consisted of a nanoparticle mixed with POE using a magnetic stirrer without surfactant for 30 min. All samples had the same volume concentration, namely 0.02 vol % but received different interlude sonication duration treatments for 0, 40, 60, 80, 100, and 120 min. FESEM with Energy Dispersive X-Ray was used to characterize the Al₂O₃ nanoparticle sample's morphology and element analysis. UV Visible and absolute zeta potential were used to determine Al₂O₃-POE nanolubricants stability. The findings show that the most optimal sonication impact in this study is 80-min sonication. It proofed by the highest absorbance ratio among other samples, which is 0.411, the lowest drop absorbance value, which is 58.9%, and with a zeta potential value of 45 mV. The rheological behavior analysis shows that Al₂O₃-POE nanolubricants show Newtonian behavior.

1. Introduction

It has been well recognized that both mineral and synthetic oil are widely used as lubricants in engineering applications [1–4]. Lubricants' purpose includes reducing friction, wear, the heat between mechanical parts and improving cooling productivity [5]. Conventional coolants like water, ethylene glycol, propylene glycol, and oils possess low thermal conductivity, which hinders improving system thermal efficiency [6,7]. Modern engineering applications are currently requiring an advanced lubricant to improve their machinery productivity and prevent them from the mechanical trip of overheating due to its workloads [8–12]. This issue can be solved by dispersing nanoparticles into the conventional fluid as the base fluid [13–16]. Active and passive cooling methods are the two possible methods for removing heat. An advanced class fluid known as “Nanofluids” was initially introduced by Masuda et al. [17] and Choi [18]. Matsuda et al. dispersed ultra-fine powders of Al₂O₃, SiO₂, and TiO₂ in water as the liquid. Choi reported that an innovative new class of heat transfer fluids could be engineered by suspending metallic nanoparticles in conventional heat transfer fluids. Nanofluids consisting of such particles suspended in liquids

(typically conventional heat transfer liquids) enhanced the thermal conductivity [19] and convective heat transfer performance of the base liquids [20]. The resulting “nanofluids” are expected to exhibit high thermal conductivities compared to those currently used heat transfer fluids [21]. Previous researchers around the globe have done several studies regarding sonication impact on nanoparticle dispersion [20–24] and thermal conductivity of nanofluids improvement [25–30] to improve the heat transfer rate of nanofluids [34,35]. This paper will elaborate on the effect of interlude sonication treatment on the stability of Al₂O₃-POE lubricant without additional surfactant based on the absorbance value of UV visible assessment.

Magnetic stirring and sonication duration are two significant factors that affect nanofluids stability. Karthikeyan et al. [36] suspended CuO nanoparticles by homogenizing them using an ultrasonic horn (Vibronic ultrasonic processor P1–250 W) for 30 min without surfactants. Caixiang et al. [37] reported the stability of CeO₂ and CaCO₃ nanolubricants was developed by employing a magnetic force mixer for two h at 80 °C. Liu et al. [38] demonstrated an enhancement of thermal conductivity with carbon nanotube for nanofluids. The mixture was blended by a magnetic stirrer and mixed up by an ultrasonic homogenizer. Carbon nanotube–synthetic engine oil suspension, thermal conductivity was

* Corresponding author at: School of Mechanical Engineering, Ningxia University, China.

E-mail addresses: ir.agusnug@gmail.com (A. Nugroho), 12712580@qq.com (Z. Bo), rizalman@ump.edu.my (R. Mamat).

Nomenclature

min	Minute
h	Hour
vol%	Volume %
POE	Polyol ester
UV Vis	Ultra Violet visible
T	Temperature (°C)
FESEM	Field-Emission Scanning Electron Microscopy
TEM	Transmission Electron Microscopy

Greek symbols

ϕ	Volume concentration (%)
ρ	Density (g/cm ³)

Subscripts

<i>L</i>	Lubricant
<i>P</i>	Nanoparticle
<i>nf</i>	Nanofluid
<i>bf</i>	Base fluid

Table 1
Al₂O₃ physical nanoparticles properties.

Properties	Metric
Density	3.95 g/cm ³
Molar mass	101.96 g/mol
Diameter	13 nm
Color	White
Melting point	2040 °C
Boiling Point	2977 °C

Table 2
Properties of Polyol ester oil.

Oil	Properties					
	Viscosity at 40 °C	Viscosity at 100 °C	Viscosity Index	Density at 15 °C	Flash Point	Pour Point
Suniso SL 68	70.1 cSt	9.1 cSt	105	0.960 g/cm ³	252° C	-36 °C

enhanced by 30% at a volume fraction of 0.02 vol%. Later, Pang et al. [39] reported that the thermal conductivity of Al₂O₃ and SiO₂ methanol-based nanofluids increased with an increase of the nanoparticle volume fraction, and the enhancement was observed to be 10.74% and 14.29% over the base fluid for the concentration of 0.5 vol% of Al₂O₃ and SiO₂ nanoparticles, respectively. The suspension was dispersed by using the ultrasonic vibration for 2 h to break down the agglomerations and improve the stability. Ruan et al. [40] observed that the sonication process reduced the sizes and length of carbon nanotubes. Asadi et al. [41] reported that 30 min was the optimum sonication time for stability. Beyond that sonication duration, it leads to the destruction of nanofluids' stability and thermal properties.

In the present study, the interlude sonication impact on Al₂O₃-POE nanolubricants samples has been investigated. Most of the recent studies reported that there might be an optimum duration for better dispersion stability. However, this may not necessarily be the most prolonged ultrasonication duration tested. Therefore, this paper aims to elaborate on the extensive examination of sonication impact treatment using the interlude sonication method on newly prepared Al₂O₃-POE nanolubricants systematically by discussing the nanolubricants' absorbance values extensively and their ratio study. The experimental assessment utilizing UV visible spectrophotometer to determine the most optimum

sonication treatment duration. The measurements were taken after sample preparation and after 15 days to all samples.

2. Experimental procedure**2.1. Materials**

Aluminum oxide nanoparticles, 99.99% trace metals basis nanoparticles, were purchased from Sigma-Aldrich, Saint Louis, the USA, for this study. Table 1 shows the Al₂O₃ specification and Al₂O₃ nanoparticles' thermal properties. The polyol ester (POE) lubricant, a synthetic refrigerant oil, was purchased from SUNISO from Belgium. POE lubricant's essential properties are listed in Table 2. The two-step method was employed to prepare nanolubricants as suggested by Ali et al. [42], Mohammed et al. [43], and Nabil et al. [44] with a volume fraction of 0.02% nanoparticles in all samples with the same mixing duration but different ultrasonication ranging from 30 to 120 min with an interlude of 15 min every 40 min cycle.

Al₂O₃ is an essential element of ceramic oxides that has been used by previous researchers in their study [45–48]. Alumina is used in the chemical industry, metallurgy, and other industrial applications due to its performance, morphology, and thermal properties. The surface of mostly ceramic nanoparticles such as alumina is polar and hydrophilic since they do not disperse in organic environments [49].

2.2. Nanolubricants preparation method

The magnetic stirrer device operated without a heater at the rate of 600 rpm. The ultra-sonication device Fisherbrand™ from FisherScientific operated at 37 kHz. Fig. 1 shows how Al₂O₃ nanoparticles are mixed with POE lubricant in the magnetic stirrer and ultrasonication treatment. They are treated in an ultrasonication bath for the agglomeration breakdown process, respectively.

Three stages were required to prepare the nanolubricants. The initial stage involved Al₂O₃ nanoparticles and POE lubricant measurement. The nanoparticles were measured using the digital weighing scale Sartorius Entris to determine the required mass of Al₂O₃ nanoparticles and POE lubricant before the mixing process. Previously, the balance was calibrated to reach a reasonable level and adjust the circle centre's levelling bubble. The medial stage involved mixing the base lubricant (POE lubricant) and Al₂O₃ nanoparticles utilizing a magnetic stirrer for 30 min. The third stage involved the ultrasonication process utilizing an ultrasonic bath to breakdown the nanolubricants agglomerations between 30, 40, 60, 80, 100, and 120 min. Eq. (1) is used to calculate the volume fraction of the nanolubricants as suggested by Mahbubul et al. [50], Das et al. [51], Nabil et al. [44], and Elsaid et al. [52]. After each 40-min cycle, a 15-min interlude was applied to prevent nanolubricants' homogeneity damage.

$$\phi = \frac{m_p/\rho_p}{m_p/\rho_p + m_l/\rho_l} \times 100\% \quad (1)$$

Where ϕ is the nanoparticle concentration in volume %; m_p and m_l are the masses of the nanoparticle and lubricant, respectively; and ρ_n and ρ_r are the density of the Al₂O₃ nanoparticle and POE lubricant, respectively.

3. Results and discussion**3.1. Al₂O₃ nanoparticles characterization**

Zawawi et al. [53] and Sadoun et al. [54] employed Field-Emission Scanning Electron Microscopy (FESEM) to characterize the morphology of Al₂O₃ and other nanoparticles. To confirm Al₂O₃ nanoparticles' morphology and shape, in this study, Field-Emission Scanning Electron Microscopy (FESEM) JSM-7800F from JEOL Ltd. was employed

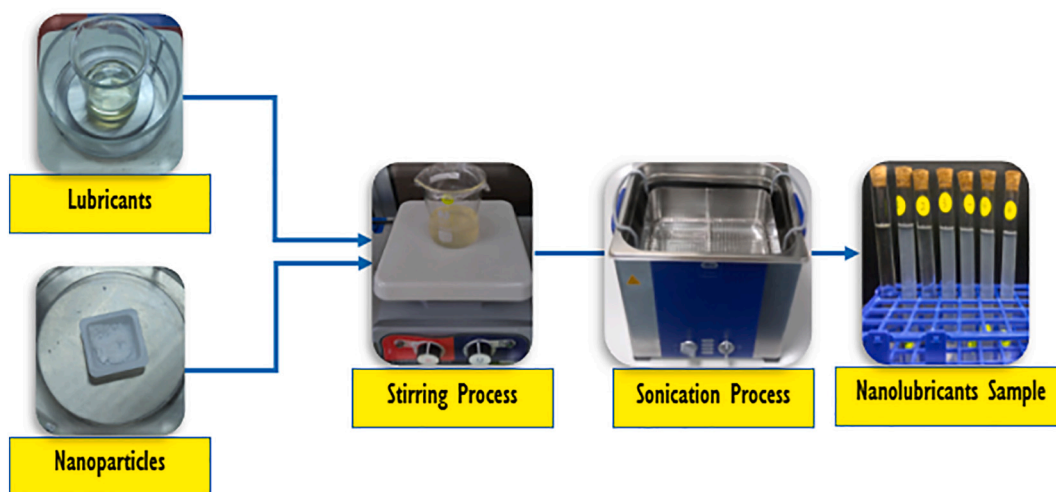


Fig. 1. Al_2O_3 -POE nanolubricants preparation using the two-step method.

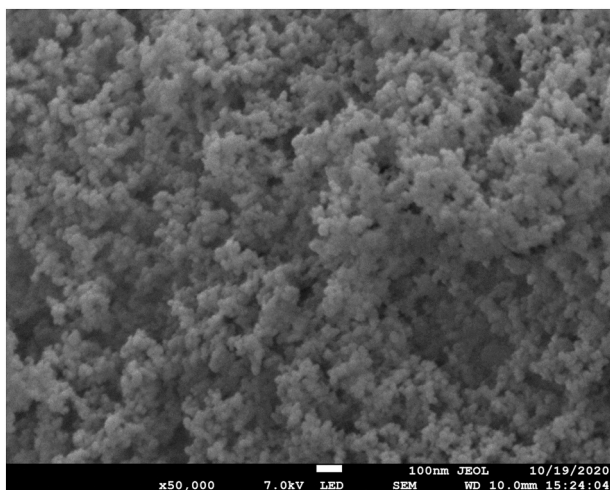


Fig. 2. Al_2O_3 Nanoparticles morphology in 50,000 magnification.

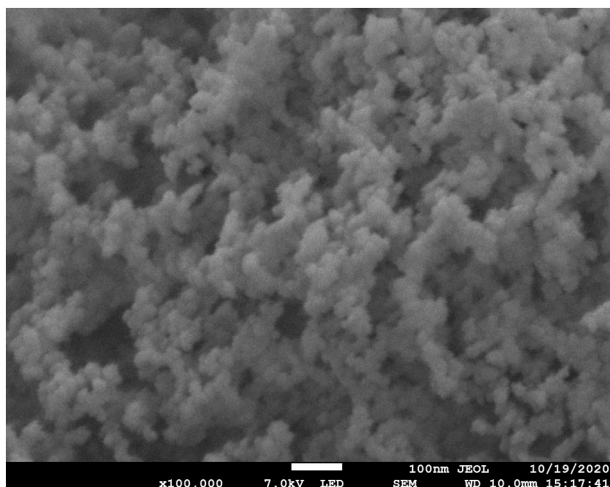


Fig. 3. Al_2O_3 Nanoparticles morphology in 100,000 magnification.

to characterize the morphology, size, and shape of the Al_2O_3 nanoparticles.

The graph was captured using an LED menu of 7.0 kV with a

magnification scale of $50,000\times$ and $100,000\times$, respectively, as shown in Figs. 2 and 3. The data consist of Al_2O_3 nanoparticles microstructure at the molecular level. Instead of macrostructure [55], similarly previous researchers employed FESEM to determine the nanoparticles morphology and its microstructure [56–58].

Based on the results of the FESEM characterization in Fig. 2, it can be seen that the morphology of dry Al_2O_3 has a uniform shape, which is round and has a uniformity level of size with an average grain size of 13.3 nm shown in Fig. 3.

3.2. Al_2O_3 nanoparticles element analysis

In order to confirm the formation of Al_2O_3 nanomaterials, Energy Dispersive X-Ray (EDX) analysis was performed as suggested by Wagih et al. [59], Sofiah et al. [60], Sahoo et al. [61], Pourrajab et al. [62] and other previous researchers [63–65]. During the EDX measurement, different areas were focused, and the corresponding peaks are shown in Fig. 4.

The spectrum shows only major elements Al and O showing, meaning it shows the purity of the nanoparticles. The analysis consists of spectra showing peaks corresponding to the elements making up the actual composition of the sample of Al_2O_3 nanoparticles analyzed, as shown in Fig. 4. According to the EDX spectra result, the larger the peak, the larger the element, representing the particular material element. Similarly, previous researchers [66–68] employed EDX analysis to determine the Al_2O_3 nanoparticles elemental analysis. Sadoun et al. [69] explained that examining element microstructure and morphology of nanoparticles with an XRD-connected FESEM is very accurate.

Table 3. provides the percentage of elements in Al_2O_3 nanoparticles by EDX analysis. Two dominant elements were found in the sample. The elements were oxygen (O) and Aluminum (Al), with a weight percentage of 61.53% and 38.47%. Al_2O_3 was a chemical compound in oxide form, and oxygen element dominated it. Thus, the oxygen percentage of the element was higher than another element [70]. Elements that are not linked to the constituent elements of Al_2O_3 have been overlooked to remove uncertainty in the EDX review. The peak of the highest peak shown in Fig. 4, a carbon element derived from the EDX-detected carbon type.

Because Al_2O_3 was non-conductive material in nature, the surface of the Al_2O_3 nanoparticle acted as an electron trap. Therefore, a sputter coating had been done before the characterization. Otherwise, the increase of electrons (charged) on the nanoparticle's surface would generate extra-white regions on the sample. This extra-white region influenced the image information during the characterization process. Once sputter coating was utilized, the conductive coating created a

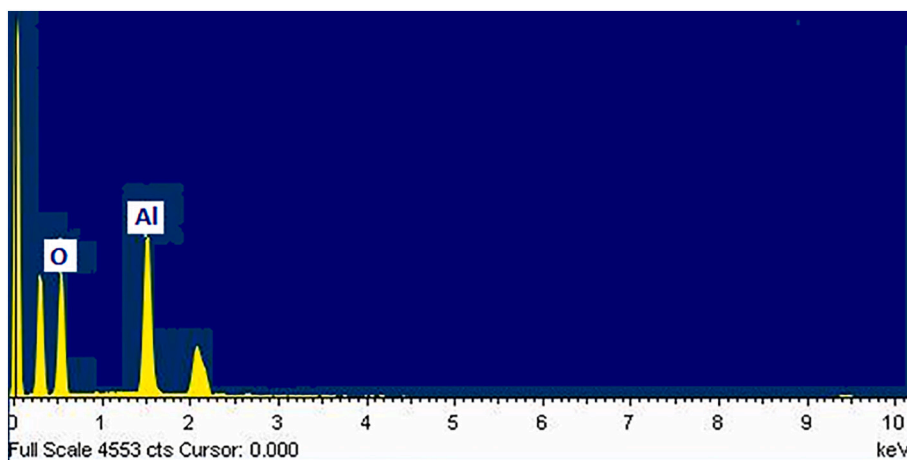


Fig. 4. EDX element analysis of Al₂O₃ Nanoparticle.

Table 3
Element percentage of Al₂O₃ by EDX.

Element	Weight %	Atomic %
Oxygen	61.53	72.95
Aluminum	38.47	27.05
Total	100.00	–

channel that allowed the charging electrons to be detached from the nanoparticle surface.

Platinum sputter coating was used in this study. There are some advantages of using Platinum; (i) it has a finer grain size than gold or gold-palladium, (ii) it is more suitable for higher magnifications applications, (iii) it is resistant to “stress cracking” when oxygen is present (a porous sample may be a source of oxygen). Dry samples of Al₂O₃ nanoparticles had been mounted on the carbon type. The sample positioning was done very carefully and did not press the sample’s surface to avoid changes in the surface morphology of the sample particles.

The coating thickness was between 2 and 3 nm due to the optimum thickness; otherwise, the platinum element would dominate the primary element of Al₂O₃ nanoparticles if it was beyond 3 nm or would be less conductive if it was less than 2 nm.

3.3. Visual stability analysis

Nanolubricants sample comparison is shown in Fig. 5. It is to observe the level of nanolubricants stability as done by previous researchers [71–74]. Observations were made to observe whether agglomeration and sedimentation occurred and determined the sample stability visually. Fig. 5 (a) shows the Al₂O₃/POE nanolubricants samples just after the preparation.

From the samples, it can be seen that Al₂O₃ nanoparticles are very well dispersed with POE lubricants. Sample 0 was a POE lubricant without any nanoparticles addition, and sample 1 was Al₂O₃/POE nanolubricants that stirred for 30 min without sonication treatment. Meanwhile, samples 2, 3, 4, 5, and 6 were Al₂O₃-POE nanolubricants samples that experienced 30 min stirring and ultrasonic treatment for 40, 60, 80, 100, and 120 min respectively. The interlude sonication treatment process is shown in Fig. 6.

Fig. 5(b) shows the display of the Al₂O₃/POE nanolubricants sample on day 15th. There are different visualizations on day 15th. Sedimentation and agglomeration in sample 1 were significant, as nanoparticles were deposited at the experimental channel’s bottom. Different conditions were also in samples 2, 5, and 6. This agglomeration phenomenon in nanolubricants was due to Van der Waals’ force between

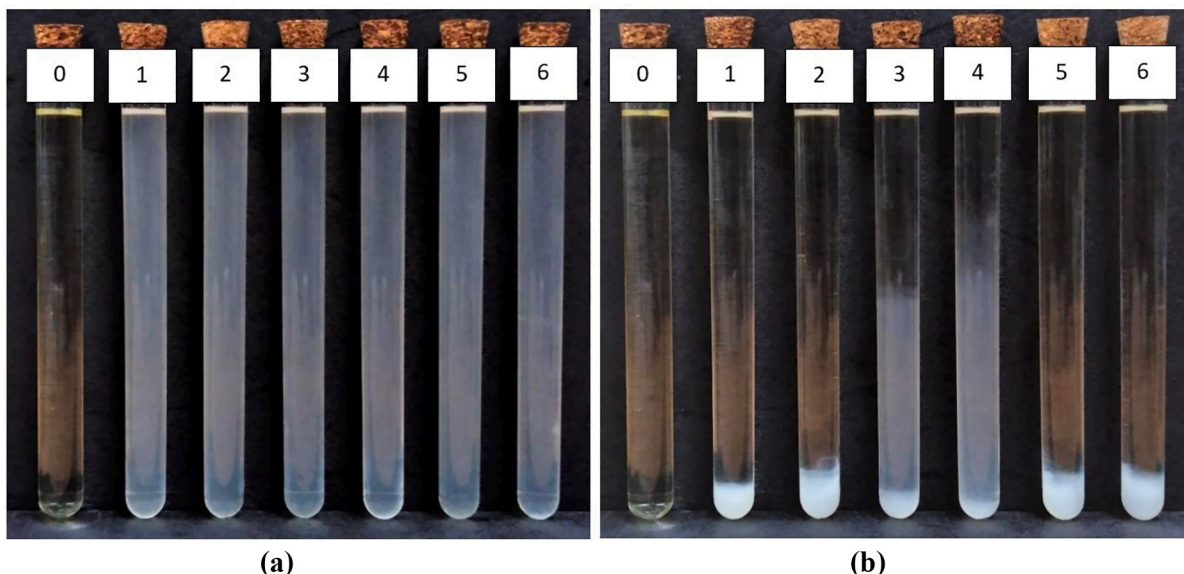


Fig. 5. Al₂O₃/POE nanolubricants sample in (a) day 1 and (b) day 15.



Fig. 6. Ultrasonication treatment process on Al₂O₃/POE nanolubricants.

nanoparticles at the atomic level [75,76]. Samples 3 and 4 looked better than the rest of the samples. Agglomeration and sedimentation occurred throughout samples 3 and 4 but did not drop to the maximum level at the experimental tube's bottom. It concludes that a good sonication treatment duration under these conditions is between 80 and 100 min.

Nevertheless, it cannot be said that these two samples are stable even though they visually show better conditions than samples 1, 2, 5, and 6. Previous researchers had consistently proposed obtaining stable nanolubricants before apparatus implementation [77,78]. Visual observation for 15 days and UV visual examination was performed in this study to establish the stability of the Al₂O₃-POE nanolubricants as the previous researchers [79,80] did. In this analysis, unlike previous researchers, nanoparticles were well dispersed on a specially formulated synthetic lubricant for electric motors application. Nanolubricants stability could be predicted by analyzing the result of UV visible analysis. These two approaches are commonly used in literature by various researchers [81,82].

3.4. Absorbance studies analysis

Understanding the definition of absorbance and transmittance is crucial before the experiment. Absorbance is the quantity of light absorbed by a nanolubricant, whereas transmittance is the quantity of light that passes through nanolubricants. The schematic of absorbance studies utilizing UV visible is shown in Fig. 9.

Absorbance analysis utilizing a UV visible spectrophotometer approach is one of the most potent methods to assess the stability of nanolubricants by the absorption of transmitted light passing nanolubricants [83].

The visible UV provides the desired spectrum of light wavelength. Second, the lens transmits a straight beam of light (photons) that passes through a monochrome to separate it into many wavelength components (spectrum). The wavelength selector then transmits only the desired wavelengths, as shown in Fig. 7. After the target spectrum of light wavelength passes through the nanolubricants in the cuvette, the

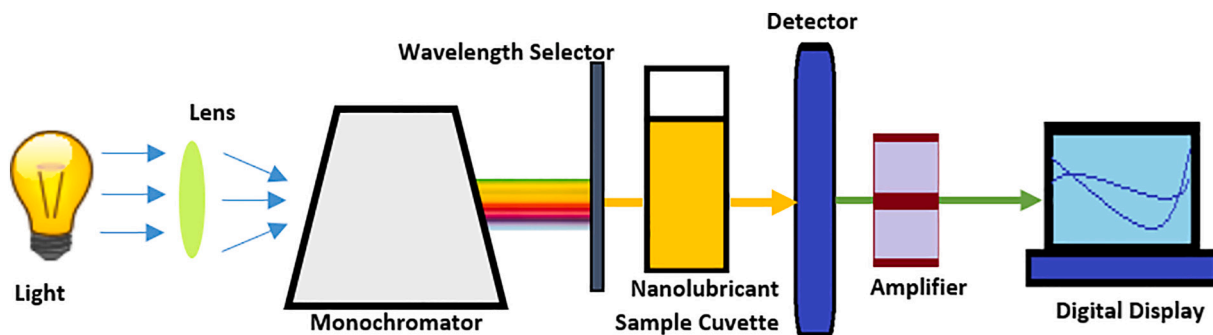


Fig. 7. Schematic diagram of absorbance process in UV Vis Spectrophotometer.

photometer measures the photons' sum. The photons are absorbed, and then they send a signal to the galvanometer or digital display.

The absorbency is proportional to the percentage of the particles contained in the nanolubricants. The nanolubricants sample is exposed to incident light. The higher absorbance value indicates the higher concentration of nanoparticles in a particular scanned area. UV visible assessment has been performed for all nanolubricants samples and the findings are shown in Fig. 9. UV visible assessment of Al₂O₃-POE nanolubricants was conducted using Thermo Scientific™ GENESYS™ 50 UV Visible spectrophotometer as shown in Fig. 8. It is a single-cell configuration UV visible measurement that assesses sample absorbance value up to 5 points of absorbance unit.

UV visible assessment was performed using scan mode, and here the wavelength was scanned while the ordinate value was recorded to generate a spectrum. The spectrophotometer scanned from the longest wavelength (1100 nm) to the shortest wavelength (200 nm). This was to reduce the sample's cause of photo-decomposition. The assessment was conducted on the first day immediately after the preparation of nanolubricants as suggested by Mamat et al., [84] and Gallego et al. [85].

It was observed that the absorbance in nanolubricants first increased and then decreased after reaching a peak at a particular wavelength, as shown in Fig. 9. Fig. 9 shows the experimental result of Al₂O₃-POE nanolubricants based on screen-printed Al₂O₃ of every sample. It indicates a broad absorption spectrum in the UV-Visible region and shows significant absorbance as reported Kawai et al. [86] and Xian et al. [87]. The display can see the effects of the calculation of the absorbance of Al₂O₃-POE nanolubricants. The graph shows that all of the sample charts travel from the longest wavelength of 1100 nm to the shortest wavelength of 200 nm. In general, there is no substantial increase in all wavelength samples from 1200 to 370 because the samples have the same concentration, which is 0.02 vol%. However, both samples undergo their respective absorption peaks at wavelengths 360, 302, 278, and 218 nm are 2, 1, 4, and 2 points, respectively. It can, therefore, be argued that the Al₂O₃-POE nanolubricants exhibits significant absorbance. Fig. 10 demonstrates the correlation between the wavelength

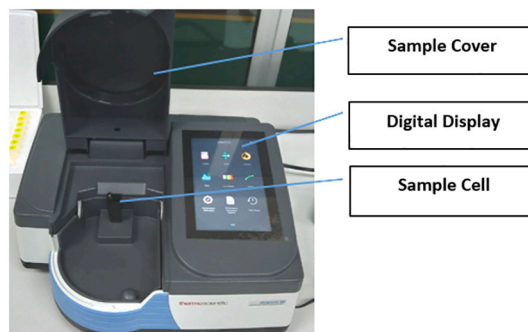


Fig. 8. Thermo Scientific™ GENESYS™ 50 UV Visible spectrophotometer.

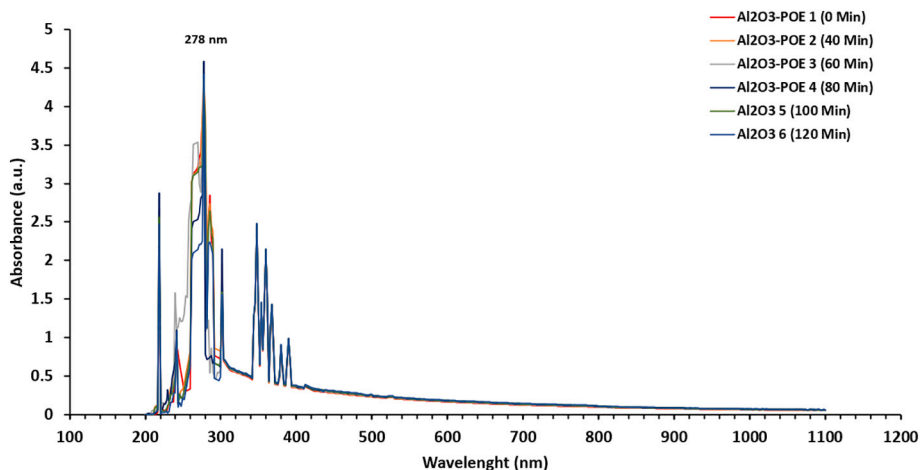


Fig. 9. Absorbance distribution of Al₂O₃-POE in every test on day 1.

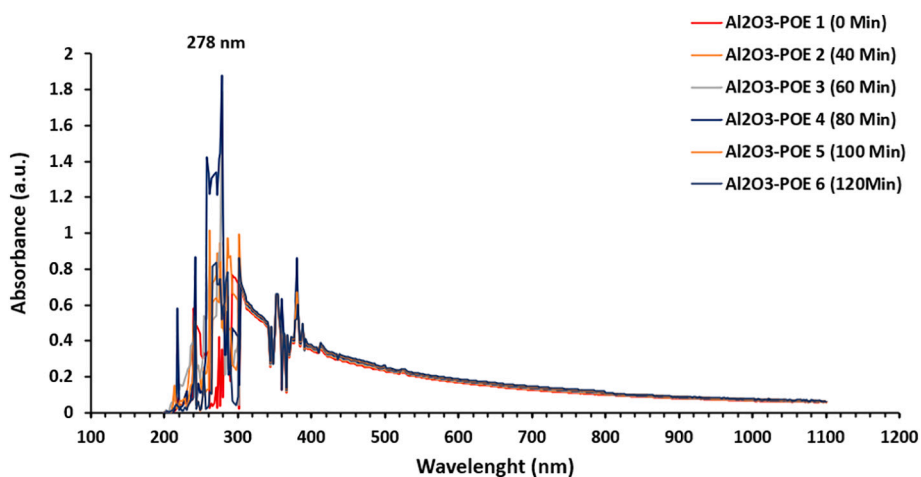


Fig. 10. Absorbance distribution of Al₂O₃-POE in every test on day 15.

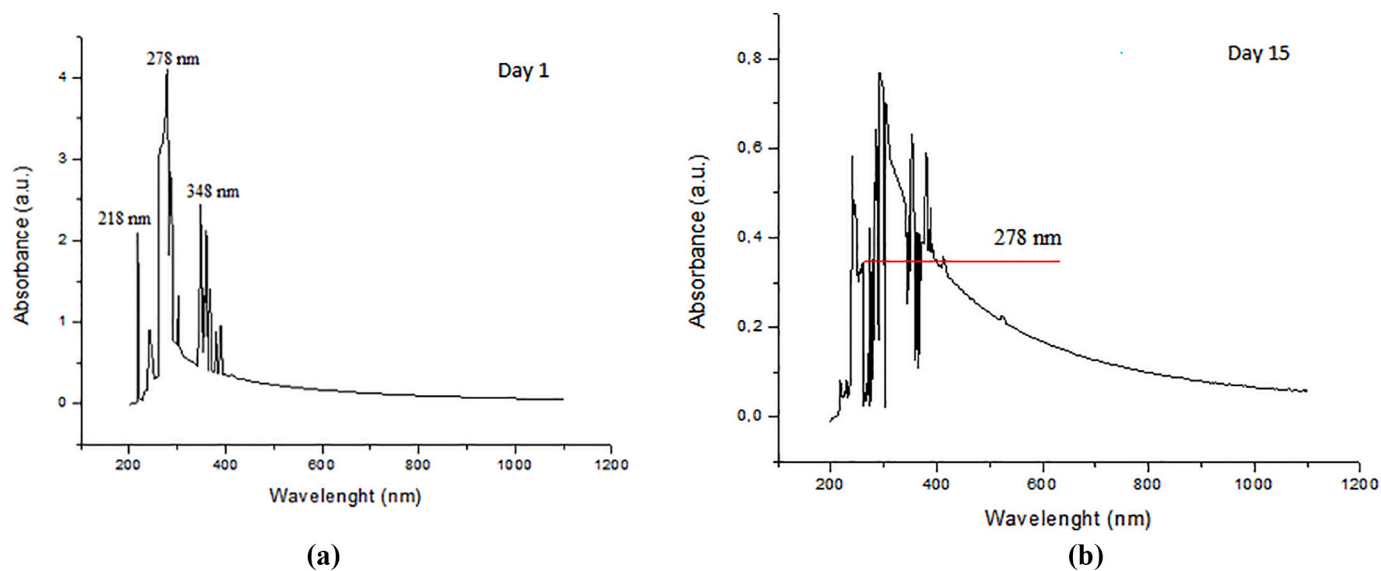


Fig. 11. The absorbance value of Al₂O₃-POE nanolubricants with 0 min sonication (a) day 1 (b) and day 15.

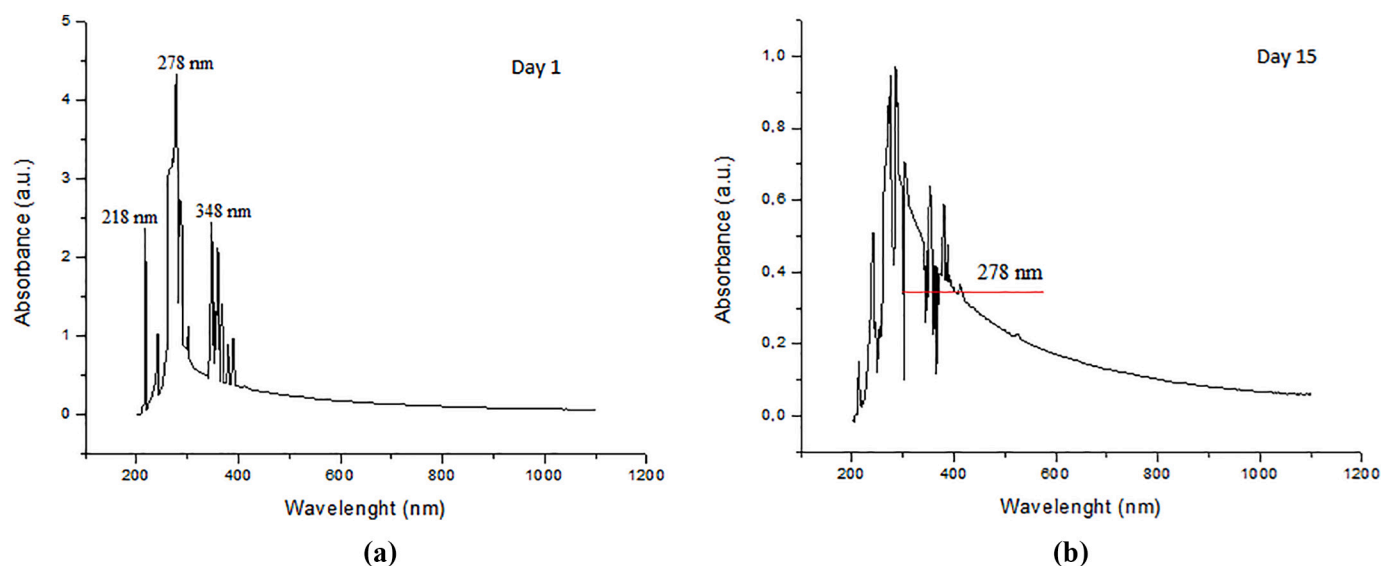


Fig. 12. The absorbance value of Al_2O_3 -POE nanolubricants with 40 min sonication (a) day 1 (b) and day 15.

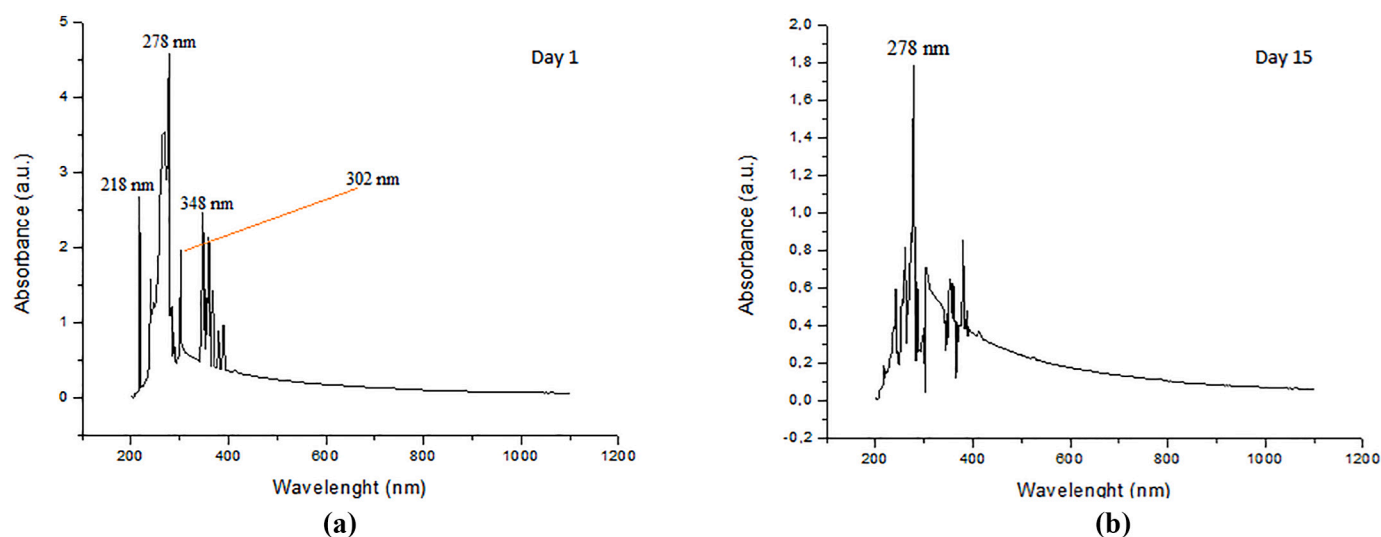


Fig. 13. The absorbance value of Al_2O_3 -POE nanolubricants with 60 min sonication on (a) Day 1 (b) and Day 15.

scan's outcome and the absorbance of the Al_2O_3 -POE nanolubricants on day 15. It can be seen that the absorbance decreases in each sample and that there is an increase in agglomeration.

Further analysis regarding the absorbance ratio of this present work will be more elaborated in the next section. To compare the stability of the nanolubricants, the researchers previously considered two key factors: (i) the selection of the most prominent absorbance peaks among the wavelength peaks that had been scanned and (ii) the selection of the absorbance peak at the shortest wavelength position [88]. According to these two factors, the wavelength of 278 nm was chosen as the reference point for evaluating the stability of the nanolubricants in the next UV visible assessment.

Fig. 11a demonstrates the relationship between the wavelength and the absorbance value of the nanolubricants Al_2O_3 -POE that is not treated with ultrasound. There is a slight increase in absorbance from scanning at a wavelength of 1200–370 nm. The absorbance value appears to increase by 368 nm after passing through the wavelength. There are three major absorbance value peaks, respectively 2450, 4114, and 2102 absorbance units, each at 348 nm, 278 nm, and 218 nm. Meanwhile, Fig. 11b is the relationship between the absorbance values of the Al_2O_3 -

POE nanolubricants on day 15. There is a decrease in the significant spectra 1.165, 3.760, and 1.020. The highest decrease occurs in a wavelength of 278 nm, which is 91%. It is significantly dropped since this sample does not get any treatment of sonication.

These three peaks and other small peaks show the absorbance values. It also illustrates how large the nanoparticles absorb the particular light spectrum in the nanolubricants. While the number of peaks also shows the number of agglomeration and aggregation of nanoparticles in the nanolubricants.

The major three peaks and others reflect the absorbance values of the nanolubricants. These peaks also demonstrate how often nanoparticles can absorb the light that reaches them in the nanolubricants. The number of peaks on the graph also indicates the amount of agglomeration and aggregation, as reported by Xian et al. [87]. Both agglomeration and aggregation occur in the nanolubricants due to the attractive forces between the nanoparticle atomic bonding upon its nanolubricants due to Van der Waals forces, as Jiang et al., [76] and Mahbulul et al. [89] stated. The result of the Al_2O_3 -POE nanolubricants ultrasonic treatment procedure for 40 min is shown in Fig. 12a and b.

Based on the graph in Fig. 12a, the similar pattern as Fig. 12b

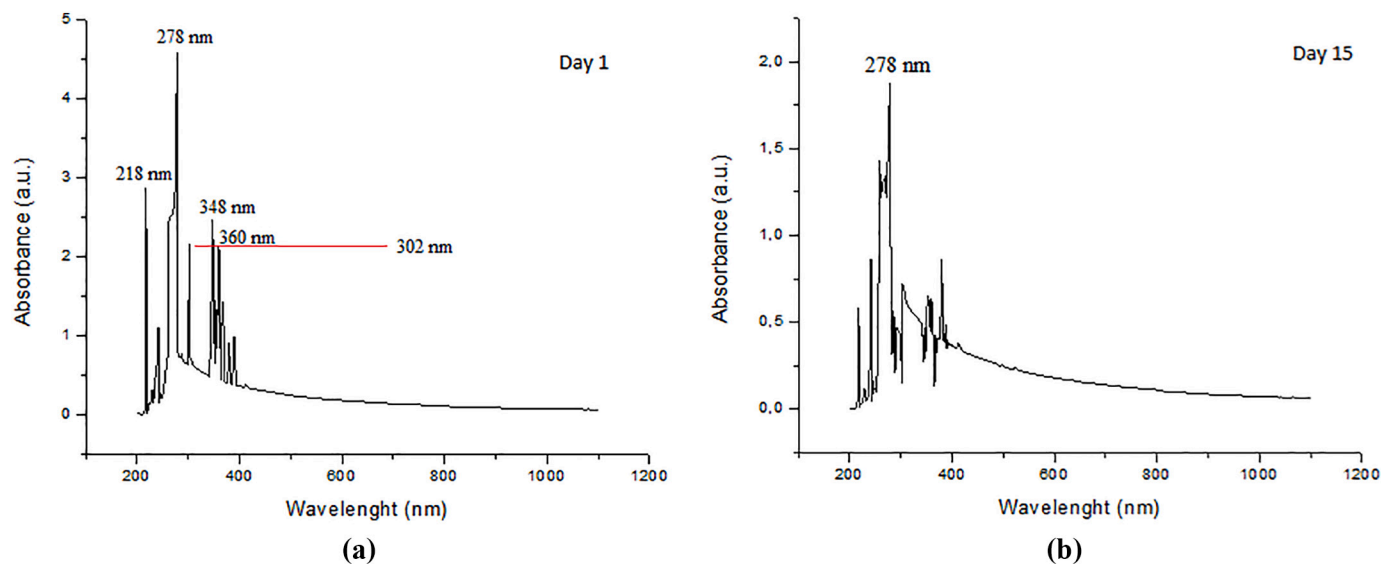


Fig. 14. The absorbance value of Al_2O_3 -POE nanolubricants with 80 min sonication on (a) day one and (b) on day 15.

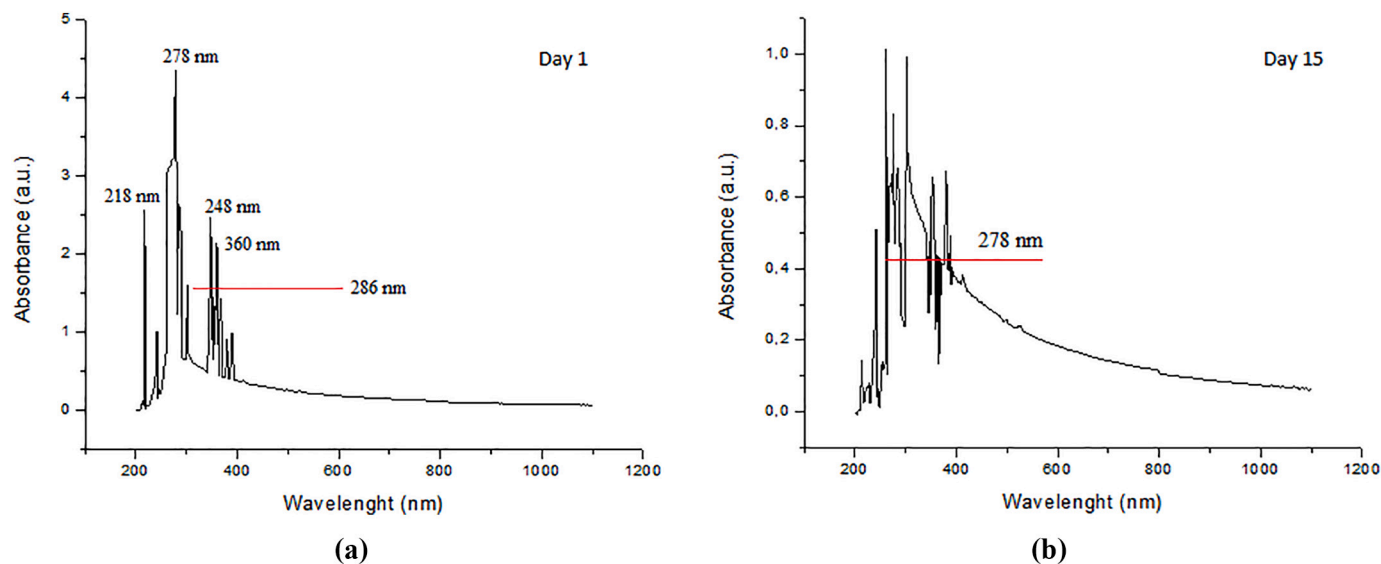


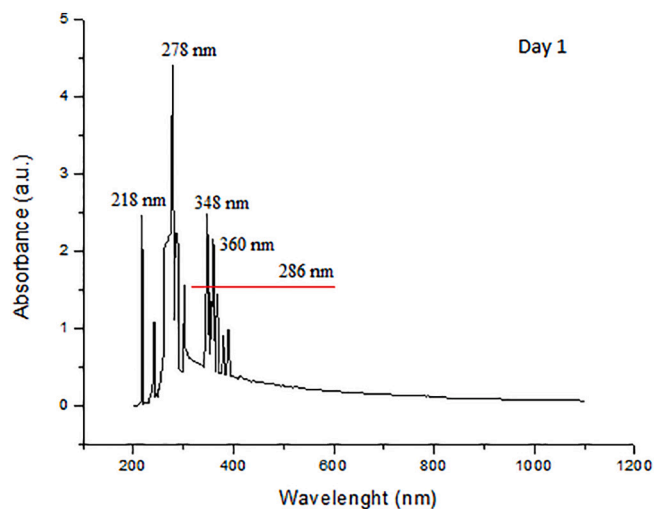
Fig. 15. The absorbance value of Al_2O_3 -POE nanolubricants with 100 min sonication on (a) day one and (b) day 15.

remains the same. There has been an insignificant rise in absorption from the initial scan point at 1100–200 nm. There are three significant peaks at 348 nm, 278 nm, and 218 nm, with absorbance values at 2.456, 4.326, and 2383, respectively. It can be seen that the absorbance values at these three points are typically increased. The wavelength 278 nm is the highest peak compared with the UV visible assessment in Day 15, as shown in Fig. 12b. It indicates decreased absorbance value in wavelength 278 nm to 0.536. Meaning, the absorbance value of sample 2's UV visible result is dropped by 3.79 absorbance unit or 87.6%. Fig. 13a shows the absorbance value of Al_2O_3 -POE nanolubricants that has been treated with sonication for 60 min on day 1. The absorbance value of the highest peak is 4.579, which is shown on 278 wavelengths. On day 15, the absorbance value drops to 1.789. Thus, there is a 60% decrease in absorbance value. A 60-min-treatment was able to boost the amount of light absorption and increase the amount of agglomeration that occurred in the nanolubricants.

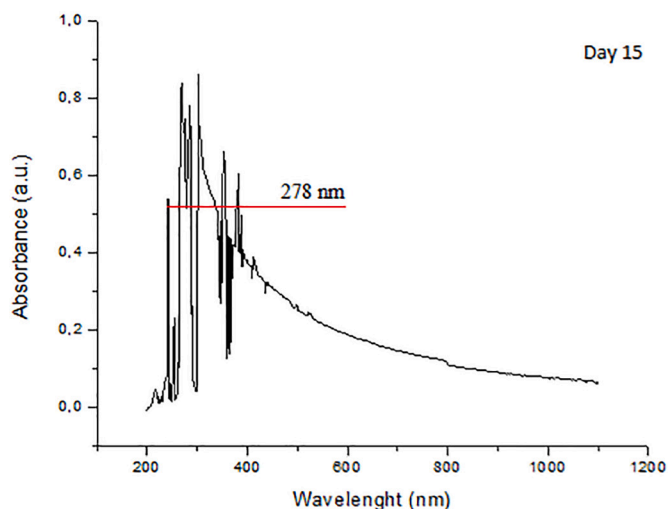
Fig. 14a shows five significant peaks, i.e. peak at wavelengths of 360 nm, 348 nm, 302 nm, 278 nm, and 218 nm, with each absorbance value being 2142, 2464, 2153, 4582, and 2873, respectively. However, there

are several other peaks in other areas that demonstrate the absorbance capacity of the nanolubricants. Some of these peaks do not draw attention because they are not dominant even if the location is in the longer wavelength.

The absorbance value at the highest wavelength at 278 nm is 1.882. Thus, there is a decrease in the absorbance value of 58.9%. Based on Fig. 15a, there are five absorbance peaks considered. They are at the wavelength of 360 nm, 348 nm, 286 nm, 278 nm, and 218 nm. The values for each wavelength are 2.148, 2.474, 2.644, 4.352, and 2.562. The absorbance value at the maximum wavelength at 278 nm is 0.468. Thus, there is an 89.2% drop in absorbance value. Fig. 16a shows the absorbance value of the nanolubricants Al_2O_3 -POE. The nanolubricants was ultrasonicated for 120 min on day one and day 15 (Fig. 16b). A dramatic decrease at a wavelength of 278 nm is 3042. The absorbance value drops significantly at the wavelength of 348 and 218 by 1213 and 1429, respectively. Major agglomeration occurs in the wavelength ranged between 400 and 358 nm after 15 days, as shown in Fig. 16b. The absorbance value, at the maximum wavelength at 278 nm, is 0.468. Thus, there is a drop of 88.3% of absorbance value. This decline can be



(a)



(b)

Fig. 16. The absorbance value of Al₂O₃-POE nanolubricants with 120 min sonication on (a) day one and (b) day 15.

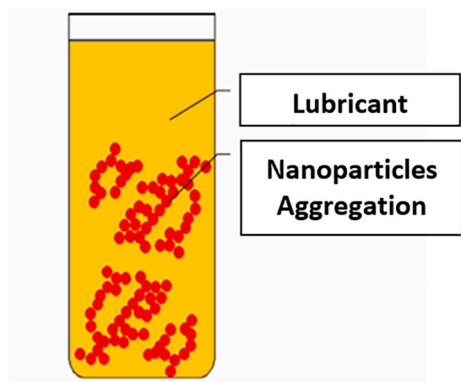


Fig. 17. Nanoparticle aggregation illustration in lubricant.

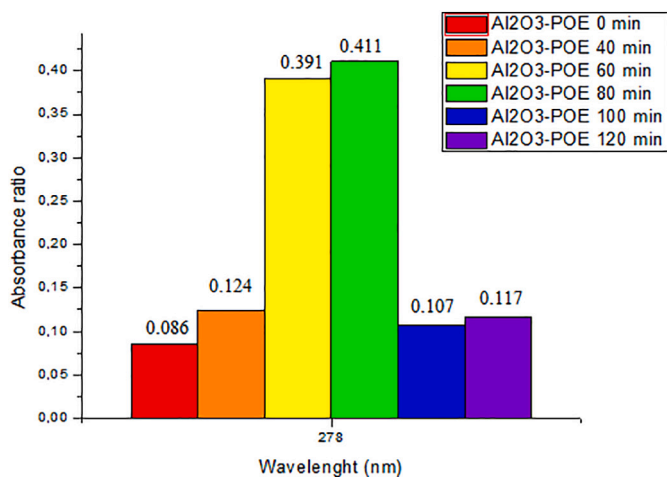


Fig. 18. Absorbance ratio of each Al₂O₃-POE nanolubricants sample.

argued due to the intense attraction between nanoparticles at the atomic level. The intense attraction generates agglomeration and aggregation of nanoparticles in the lubricant, as illustrated in Fig. 17.

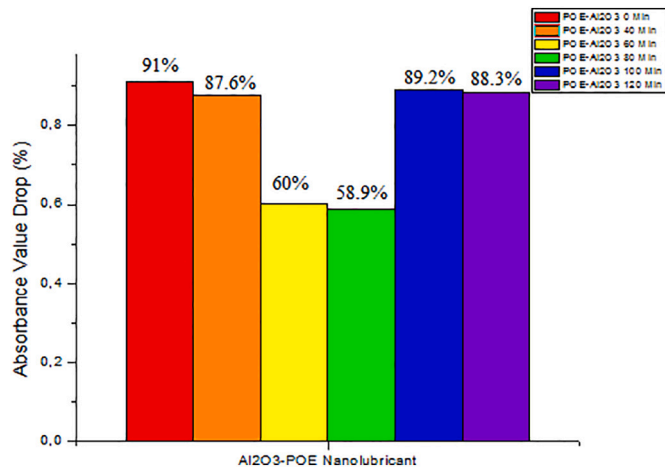


Fig. 19. Absorbance value drop percentage in every Al₂O₃-POE nanolubricants sample.

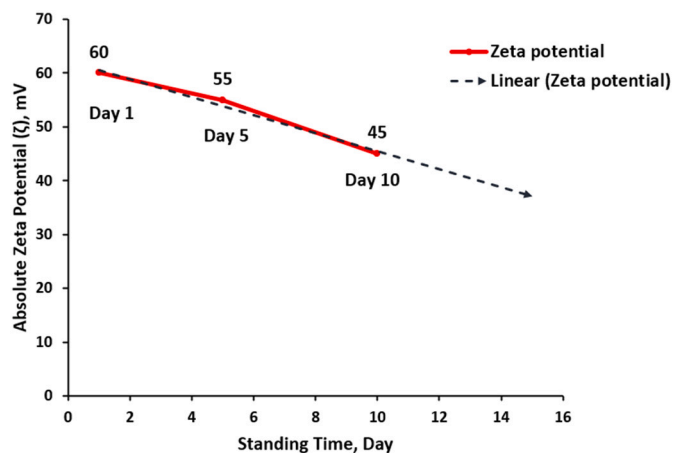


Fig. 20. Absolute Zeta Potential of Al₂O₃-POE nanolubricant as a function of time.

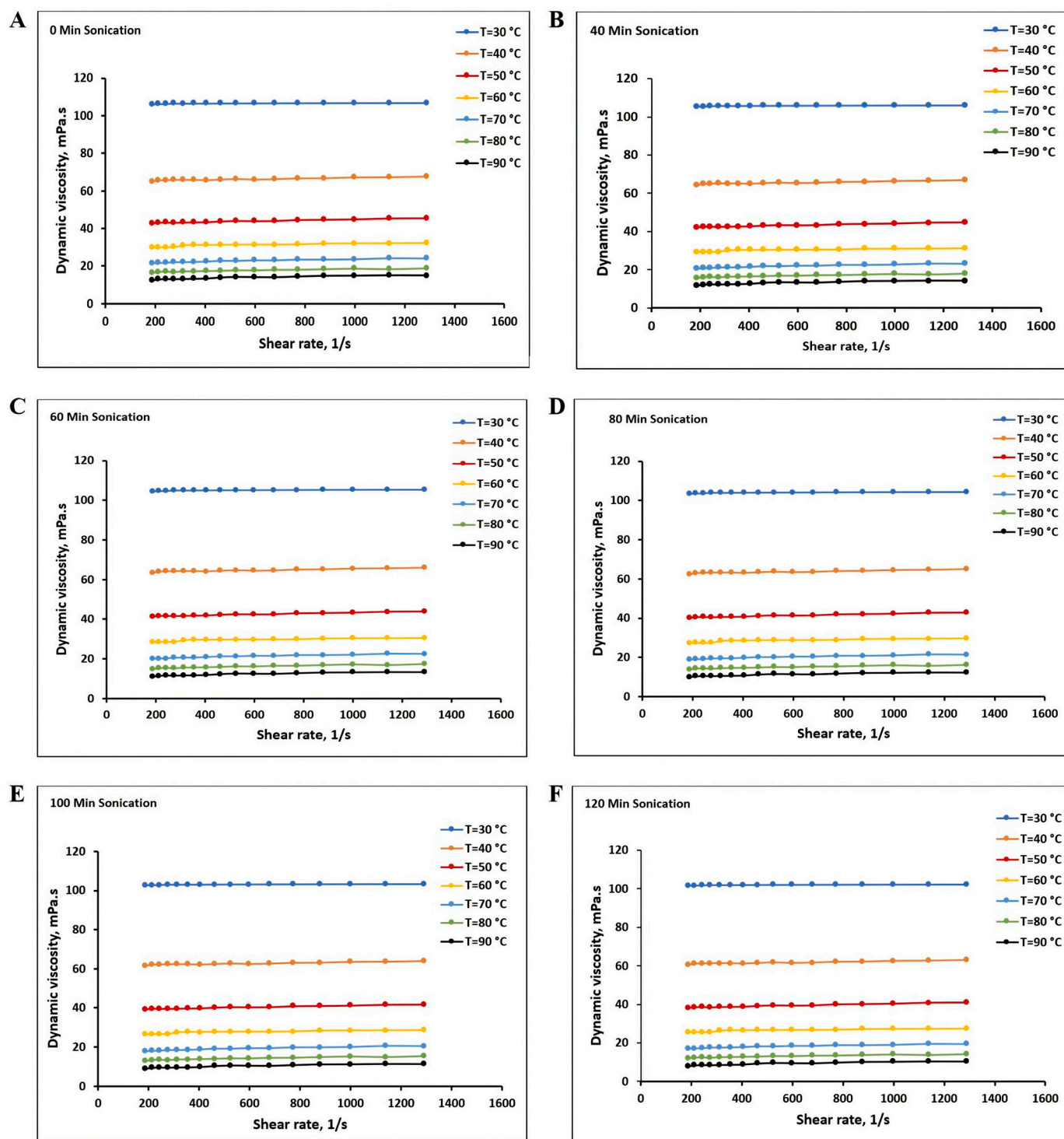


Fig. 21. (a) 0 min sonication duration effect on dynamic viscosity at different shear rate. (b) 40 min sonication duration effect on dynamic viscosity at different shear rate. (c) 60 min sonication duration effect on dynamic viscosity at different shear rate. (d) 80 min sonication duration effect on dynamic viscosity at different shear rate. (e) 100 min sonication duration effect on dynamic viscosity at different shear rate. (f) 120 min sonication duration effect on dynamic viscosity.

3.5. Absorbance ratio of Al₂O₃-POE Nanolubricants studies

The absorbance ratio method is a simultaneous measurement method for two components with an absorbance ratio at any two wavelengths. It is a constant value regardless of the concentration or the length of the wavelength. Previous researchers have used this standard method to determine the stability of nanolubricants analytically. Picking the highest peak at the shortest wavelength based on the nanolubricants

scan data of Al₂O₃-POE in each sample [90,91]. The absorbance ratio (A_r) is defined as that initial absorbance (A) is over final absorbance (A_0), as shown in Eq. (2).

$$A_r = \frac{A}{A_0} \tag{2}$$

Fig. 18 shows the absorbance ratio of each Al₂O₃-POE nanolubricants sample by comparing the highest peak absorbance value at a wavelength

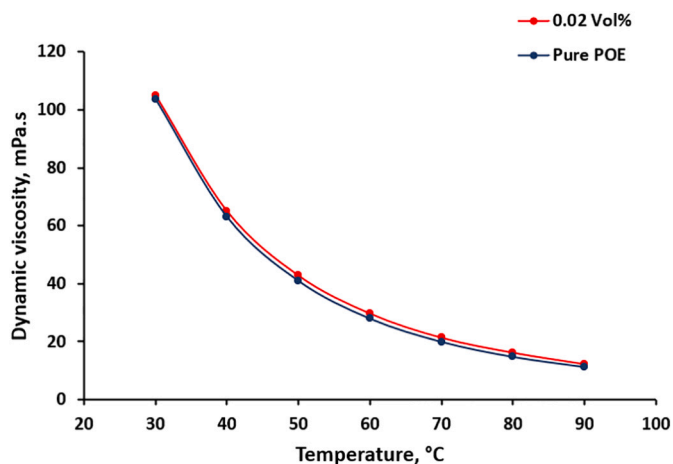


Fig. 22. Dynamic viscosity of Al_2O_3 -POE nanolubricants and POE lubricants at different temperature.

of 278 nm. Al_2O_3 -POE nanolubricants were treated for 60 and 80 min. The nanolubricants had a higher absorbance than the other samples. As a result of this treatment, the absorbance ratio is 0.391 and 0.411, respectively. This phenomenon confirms the visual observation conducted, as shown in Fig. 7b. It shows the stability of the Al_2O_3 -POE nanolubricants sample.

The other four samples have a low absorbance ratio due to a high degree of agglomeration, accumulation, and sedimentation. Besides, it can be argued that the high agglomeration, aggregation, and sedimentation of Al_2O_3 -POE is because Al_2O_3 is an oxide-based nanoparticle with a hydroxyl group on its surface. Thus, the Al_2O_3 is a naturally hydrophilic form that appears to be unstable with this type of lubricant [92,93]. Therefore, the modification of the Al_2O_3 surface is required to increase the wettability of the Al_2O_3 surface to oil, as discussed by Liu et al., [94,95].

However, for this present analysis, sample 4 is the most stable sample accompanied by sample 3 with an absorbance value of 0.411 and 0.391. In which, these two samples have the lowest absorbance value drop, as shown in Fig. 19.

3.6. Absolute zeta potential analysis

The interaction between the particles and the particle-lubricant interface over time determines the stability of the colloid system, and the stability of the Al_2O_3 -POE nanolubricant is closely related to the density of its surface charge. As a result, after determining the results of the absorbance ratio, this stability analysis must be performed using the zeta potential measurement method to verify the stability level of the prepared nanolubricants.

The absorbance ratio's main output as shown in Fig. 20 was used as the main reference. In the zeta potential test, the sample with the highest absorbance ratio was used for further analysis as suggested by Sharif et al. [96] and Zawawi et al. [97].

Fig. 20 depicts the results of the 240-h absolute zeta potential evaluation (10 days). The measurements were taken on the first day, day 5, and day 10. The Al_2O_3 -POE nanolubricant had a zeta potential of 60 mV on day 1, 55 mV on day 5, and 45 mV on day 10. According to the zeta potential examination, the prepared Al_2O_3 -POE nanolubricant can be classified as having acceptable stability based on these findings since it ranges between 30 mV – 60 mV [98].

3.7. Various sonication duration effect on dynamic viscosity

Various sonication duration was used to prepare nanolubricants. The rheological behavior of the nanolubricant prepared without sonication

is shown in Fig. 21a (0 sonication duration). Fig. 21b, c, d, e, and f demonstrate nanolubricants rheological behavior prepared by sonication for 40, 60, 80, 100, and 120 min, respectively. According to the graph, the sonication duration has no significant effect on the nanolubricant behavior. In all samples of different sonication durations, the nanolubricant behaves in Newtonian fluid. This phenomenon demonstrates that shear rate changes in each sample do not increase nanolubricant dynamic viscosity significantly. The Al_2O_3 -POE dynamic viscosity graph tends to be flat with a very small increment rate.

These graphs demonstrate two Newtonian fluid behavior. Specifically, the behavior of the nanolubricant thickening viscosity, which shows a small rise in dynamic viscosity at high shear rate values and a thinning viscosity at low shear rates.

Fig. 22 depicts the effect of adding Al_2O_3 nanoparticles to a POE lubricant tested in various temperature, which results in an increase in the nanolubricant's viscosity. The dynamic viscosity increases as the nanoparticle added to the lubricant. The effect of the nanoparticle density, which is higher than the lubricant induces this phenomenon. Furthermore, this phenomenon is caused by the van der Waals force, which causes the lubricant's viscosity to increase [75]. The graph demonstrates the dynamic viscosity decreases as the temperature increased. At this stage, the presence of a small proportion of nanoparticles indicates Newtonian behavior as shown in Fig. 2, in which the viscosity is determined by the shear stress or the applied shear rate.

Shear thickening occurs at higher stresses, according to Norman et al. [99] once the critical shear stress is reached, viscosity increases rapidly and sometimes discontinuously. The presence of dynamic viscosity in the nanolubricants under our investigation contradicts our previous experimental findings. Even though there is shear thickening at higher shear stresses and rates. Viscosity appears to level off at much higher shear rates. The viscosity will start to rise above a certain shear-rate threshold [100]. However, the graphs depict that the dynamic viscosity does not increase rapidly and significantly, as shown in Fig. 21. As a result, this nanolubricant's behavior can be classified as Newtonian fluid [101].

4. Conclusions

According to the results of the assessment that had been carried out, a range of essential points can be drawn as follow:

The influence of sonication duration leads to the stability of the Al_2O_3 -POE nanolubricants. The stability is determined by the absorbance value and the absorbance ratio of the Al_2O_3 -POE nanolubricants. Every sample has a maximum drop on the 15th day with an absorbance ratio range of 58.9% and 91%.

In this study, the interlude sonication period gives the most optimal impact at 80-min sonication. The highest absorbance ratio evidences it among other samples, which is 0.411, and the lowest drop absorbance value, which is 58.9%. The absolute zeta potential evaluation result for 240 h is 45 mV. The Al_2O_3 -POE Nanolubricant was found to have an acceptable degree of stability within the examination period.

Agglomeration, aggregation, and sedimentation are three parameters that reduce the absorbance value of nanolubricants. This state is due to Van der Waals' attraction between the elements of alumina at the atomic level. This attractive force makes two or more atoms together form a single atomic unit cell and tends to fall, ultimately creating Al_2O_3 sedimentation at the bottom of the experimental tube.

The various sonication duration on Al_2O_3 -POE nanolubricants does not give significant enhancement on dynamic viscosity. Even though there is shear thickening at higher shear stresses and rates. The dynamic viscosity slightly appears to level up at much higher shear rates. Thus, the rheological behavior of Al_2O_3 -POE nanolubricants show Newtonian behavior in all examined samples.

Another element that stimulates the instability of the Al_2O_3 -POE nanolubricants is the surface of Al_2O_3 itself. Al_2O_3 is an oxide-based nanoparticle with many hydroxyls groups on its surface. This state

allows Al_2O_3 to become a hydrophilic compound that is hardly stable for an extended period when combined with oil-based fluid. An active treatment such as a functionalization nanoparticles surface modification should be performed to Al_2O_3 for future investigation. The surface modification leads to the Al_2O_3 's surface wettability enhancement when it disperses into the lubricant.

Authorship statement

All persons who meet authorship criteria are listed as authors, and all authors certify that they have participated sufficiently in the work to take public responsibility for the content, including participation in the concept, design, analysis, writing, or revision of the manuscript. Furthermore, each author certifies that this material or similar material has not been and will not be submitted to or published in any other publication before its appearance in the International Communications in Heat and Mass Transfer.

Declaration of Competing Interest

The authors declare that they have not known competing for financial interests or personal relationships that could have influenced the work reported in this paper.

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