Sonication Time Effect towards Stability of Al_2O_3/PAG and SiO_2/PAG Nanolubricants

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ABSTRACT

Ultrasonication is the act of applying sound force to agitate particles in a sample with the frequency of more than 20 kHz. It is an external vibration induced during preparation of nanolubricant that helps the particles to overcome the van der Waals force bonding. Nanolubricant prepared is intended to be used in automotive air conditioning (AAC) system to improve its performance. In this work, stability of Al_2O_3/Polyalkylene glycol (PAG) and SiO_2/PAG nanolubricants of 0.2 % volume concentrations with different sonication time were investigated. Five samples for each nanolubricant were prepared by two-step method process with variation of sonication time from zero to two hours' time with half an hour interval. The stability tests were done by visual sedimentation and UV-vis spectrometer. The optimum sonication time found to be one and half hours and two hours for Al_2O_3/PAG and SiO_2/PAG nanolubricants respectively. Both nanolubricants were found stable for more than two weeks' period.

Keywords: *Nanolubricant, Alumina, Silica, Polyalkylene glycol, Sonication*

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Introduction

In mechanical equipment and machinery, applying lubricant is a must to avoid wear of sliding parts and enhancing the equipment operation consistency. Machinery with sliding contacts usually seizes up due to surface wear at the sliding boundary [1]. Many scientists concentrating their efforts in improving lubricant in enhancing the energy efficiency. Recent popular approach nowadays in enhancing efficiency of lubricant is by dispersing nanoparticle additive in lubricant base to create nanolubricant. Nanolubricants with variation of properties are formed to be employed in various areas like transportation, heavy industries, energy generation, cooling and refrigeration system.

In refrigeration system, researchers such as Bi et al. [2], Henderson et al. [3] and Kumar and Elansezhian [4] have studied the advantages of nanolubricant as a means for increasing efficiencies of air-conditioning and refrigeration equipment. Bi et al. [2] showed that TiO_2 nanolubricant reduce energy consumption by 25 % in domestic refrigerator. While Henderson et al [3] observed that CuO/refrigerant/lubricant mixtures might improve the flow boiling heat transfer by up to 76% and at the same time the lubricant can act as a necessary dispersant. Kumar and Elansezhian [4] studied the vapor compression system by using Al_2O_3 nanolubricant and concluded that enhancement of energy efficiency of 10.32 % was achieved. While Yusof et al. [5] investigated the used of Al_2O_3/POE nanolubricant in domestic refrigerator and found that 2.1 % energy reduction was achieved when compared to base lubricant. Azmi et al [6] and Redhwan et al. [7] reviewed the improvement of nanorefrigerant/nanolubricant, variety of refrigerant based and its performance and energy improvement.

Preparing a stable nanofluid in general is an important step in grapping its optimal potential. The agglomeration of nanoparticles results in the settlement and clogging of channels and may be decreasing its advantages as listed by Bi et al. [8]. The theory behind settlement of agglomeration is best explained by referring to the Stokes law [9] Equation (1) below: -

$$V = \frac{2}{9}\frac{R^2}{\eta}\left(\rho - \rho^{'}\right)g \tag{1}$$

Where V is the sedimentation speed of a particle, R is the radius of particle, η is the dynamic viscosity of the liquid, ρ and ρ' are the density of the particles and density of the liquid respectively. From the equation (1), it shows that the speed is proportioned to the square of the radius of the particle. Therefore, particles with large radius will sediment much faster than small ones.

Till date, two available methods are commonly used; one-step method and two-step method. The one-step method consists of concurrently making and dispersing the particles in the fluid, where few other processes steps were skipped (drying, storage, transportation, and dispersion of nanoparticles), hence the agglomeration of nanoparticles is minimized, and the stability of fluids is increased [10]. The one-step method could give uniformly dispersed nanoparticles, and the particles could be stably suspended in the base fluid. Unfortunately, this kind of process could not be mass produced. The second process is known as two step method. Two-step method is the preferably used for producing nanolubricants. Nanoparticles used in this method are first produced as dry powders. Then, the dry powders is dispersed into a fluid based in the second processing step with the help of intensive magnetic force agitation, ultrasonic agitation and homogenizing. Two-step method is the most viable method to produce nanofluids in large scale due to scale up industrial production level synthesis techniques. But nanoparticles have the tendency to aggregate because of high surface area [11]. Hence, a suitable method on stabilizing the solution is required by obtaining an optimum ultrasonic sonication time. Previously, the effect of sonication time on stability was being done by [12].

The sonication time may vary according to the types of base fluid. For example, Beck et al. [13] prepared Al_2O_3/ ethylene glycol in ultrasonic mixer for few minutes to gain homogeneous dispersion. They observed that the resulting dispersions remained homogeneous for the period of the experiments because of the surface charges on the particles. Jung et al. [14] produced two types of water-based alumina nanofluids with / without polyvinyl alcohol (PVA), using a horn-type ultrasonic disrupter for 2 hours. They observed that the aggregated particles were stably suspended for more than 1 month. Hung et al. [15] prepared Al_2O_3/water nanofluid via a homogenizer working at 8000 rpm for 30 min, followed by an electromagnetic agitator operating at 600 rpm for 90 min, then using an ultrasonic vibrator running at 400 W for 60 min. They concluded that after 2 weeks the initial and final concentrations differences of Al_2O_3/ water nanofluid was less than 5 % representing the stability of the prepared nanofluids. Esmaeilzadeh et al. [16] stabilized alumina water nanofluids through a 4 hours process of ultrasonication with 170W and 50Hz and electromagnetic stirring. They came up with the observation that no sedimentation was occurred and the concluded that the alumina water nanofluid was stable.

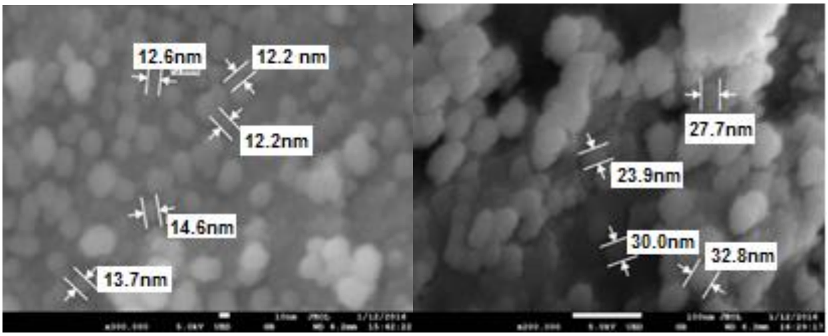
While, the effect of sonication time on stability of SiO_2 was being investigated by several researchers [17-20]. Fazeli et al. [17] dispersed SiO_2 nanoparticles in distilled water, and then the suspension was sonicated by an ultrasonic bath for 90 min. They observed that silica nanofluids stayed stable for a period of 72 hours without any noticeable settlement. Pang et al. [18]

mixed nanoparticles SiO_2 /pure methanol by using ultrasonic vibration with 750 W and 20 kHz for 2 hours to crack down the agglomeration. The measured zeta potential of SiO_2 nanofluids was over 30 mV, which indicates the good stability of the nanofluid. The photo of visualization showed that methanol based nanofluids was well dispersed. Bolukbasi and Ciloglu [19] prepared SiO_2 nanofluids by using magnetic stirrer then sonicated continuously for 2 hours by using an ultrasonic vibrator (600 W and 40 kHz). They found that no sedimentation was observed during the period of experiment. Darzi et al. [20] added distilled water to a specified amount of SiO_2 nanoparticles and mixed together by magnetic stirrer for half an hour. After that, it was dispersed by ultrasonic vibrator for 2 hours to get the constant suspension. Nevertheless, through literature reviews and to the researchers' knowledge, no specific research on ultrasonication time towards stability of Al_2O_3/PAG and SiO_2/ PAG nanolubricants were been done.

In the recent study, Al_2O_3 and SiO_2 nanoparticles dispersed in PAG lubricants were to be tested in the automotive air conditioning system [21]. Hence the purpose of this paper is to investigate the Al_2O_3/PAG and SiO_2 /PAG nanolubricants stability on varies sonication times by using visual sedimentation analysis and UV-Vis spectrometer absorbance analysis.

Material

Alumina (Al_2O_3) and silica (SiO_2) nanoparticles used in the present study are in the nano powder form. Al_2O_3 is in 13nm primary particle size, 99.8 % trace metal basis with white in colour. While SiO_2 is in 30nm primary particle size, 99.5 % trace metal basis and white in colour. Both Al_2O_3 and SiO_2 nanoparticle average size were verified with FESEM analysis as depicted in Figure 1



(a) (b)

Figure 1. FESEM analysis of (a) 13nm average size of Al_2O_3 (b) 30nm average size of SiO_2 nanoparticles.

In general, properties of both Al_2O_3 and SiO_2 nanoparticles are shown in Table 1. While for the base, Polyalkylene glycol (PAG) lubricant is specially designed to be used in R134a refrigerant automotive air conditioning compressor. PAG have been used mainly in automotive air-conditioning systems due to the compatibility characteristic with most of elastomers [22]. The properties of the PAG lubricant are shown in Table 2.

Table 1. Properties Al_2O_3 and SiO_2 nanoparticles. [23-25]

Property	**Al_2O_3**	**SiO_2**
Molecular mass, g mol^{-1}	101.96	60.08
Average Particle diameter, nm	13	30
Density, kg m^{-3}	4000	2220
Thermal Conductivity, W m^{-1} k^{-1}	40	1.4
Specific heat, J kg^{-1} K^{-1}	773	745

Table 2. Properties of PAG 46 lubricant. [26, 27]

Property	PAG 46
Density, $g.cm^{-3}$@ 20.0°C	0.9954
Flash Point, °C	174
Kinematic viscosity, cSt @ 40°C	41.4-50.6
Pour Point, °C	-51

Methodology

In this section preparation of five samples of nanolubricant will be elaborated. Then, stability evaluation by using qualitative and quantitative method will be discussed in detailed.

Preparation of Nanolubricant

In the present study, two-step process was used in preparing nanolubricant. Equation (2) introduced by Azmi et al. [28] was used to determined volume concentration of nanolubricant.

$$\phi = \frac{m_p / \rho_p}{m_p / \rho_p + m_L / \rho_L} \times 100 \tag{2}$$

Where, ϕ is the volume concentration in percent, m_p and m_L are the masses of the nanoparticle and lubricant, respectively; and ρ_p and ρ_L are the density of the nanoparticle and density of the lubricant, respectively. Other fellow scholars [29-31]are also using the same equation. Five samples of Al_2O_3 nanoparticles were mixed with PAG by using a magnetic stirrer for 30 minutes at room temperature with the volume concentration of 0.2 %. Then Four samples namely 0.5S, 1.0S, 1.5S and 2.0S were dispersed continuously for half, one, one-and half and two hours respectively. One sample namely 0.0S was not been sonicated. Ultrasonic bath vibrator Fisherbrand (model: FB15051) is generating ultrasonic pulses of 230V at 50 kHz as shown in Figure 2 was used. Ultrasonication is an external vibration induced that helps the particles to overcome the van der Waals force bonding. Even though the sonication cannot break nanoparticles individually, the cluster of nanoparticles breaks into smaller clusters resulting in the smaller clusters with higher stability [32]. In all samples, surfactant was not being used. The procedures then been repeated by using SiO_2 nanoparticles.

Stability Evaluation

In this paper, the stability of the 0.2 % volume concentration suspension of both nanolubricants were determined by observing the relative stability of colloidal using sedimentation photograph capturing methods. The observation was carried for a period of two weeks. This is the primary method that been used in determining stability of colloidal [33]. The qualitative observation data then been validated by using quantitative measurement.The second method was using UV-Vis Spectrophotometer analysis method as shown in Figure 3.

Figure 2. FB15051 Fisherbrand Ultrasonic bath vibrator

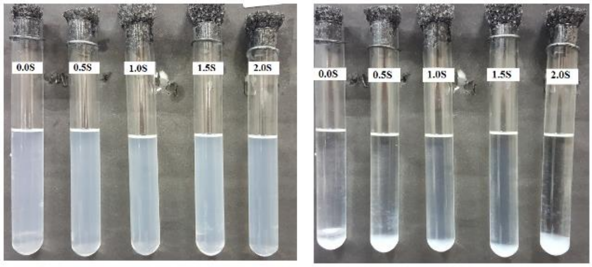
Figure 3. Drawell FDU 8200 UV-Vis Spectrophotometer.

UV–vis spectrophotometer measures the absorption and the scattering of light by comparing the intensity of the light of the nanolubricant with the base lubricant as it reference [34]. Further, the absorption and dispersions in the nanolubricant or nanofluid generally measured in the range of 200-900 nm wavelength [35]. Normally, nanosuspension stability is analyzed by measuring the sediment volume and concentration ratio versus the sediment time [36]. The absorbance for five different ultrasonication times with 0.2 % volume concentration will be observed in this experiment for over period of two weeks.

Results and Discussion

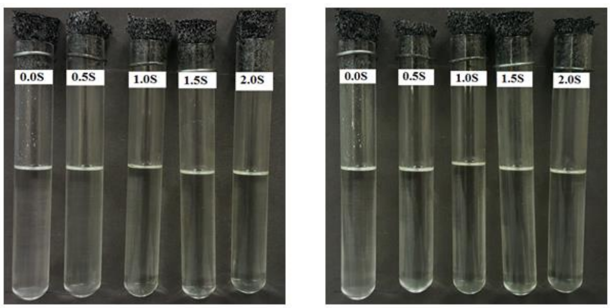
Figure 4 depicted the sedimentation photograph of Al_2O_3/PAG nanolubricant with five different sonication times just after preparation and after 14 days of preparation. From the figure, it shows that 1.5S sample was having minimum sedimentation compared to other samples. Sample 1.0S also show good stability with minimum sedimentation observed. On the other hand, it was clearly observed that the 2.0S sample had poor stability with total drop in sedimentation during the test period.

While, Figure 5 shows the sedimentation photograph of SiO_2/PAG nanolubricant with five different ultrasonication time just after preparation and two weeks after preparation. Referring to the figure, the 2.0S sample reflects to 2 hours' sonication time show minimum sedimentation occurs after period of two weeks compared to other four samples.



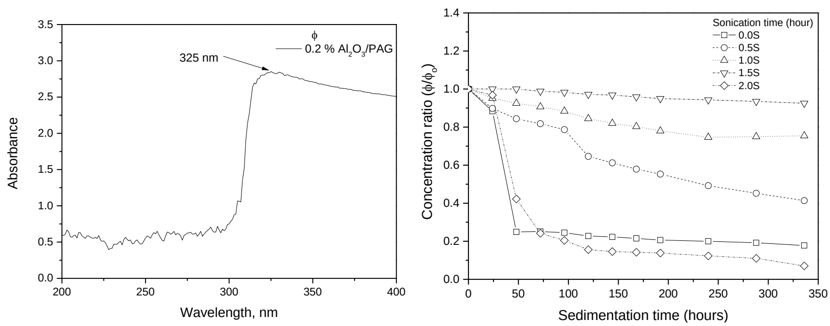
(a) (b)

Figure 4. Al_2O_3/PAG nanolubricant (a) After preparation (b) After 14 days



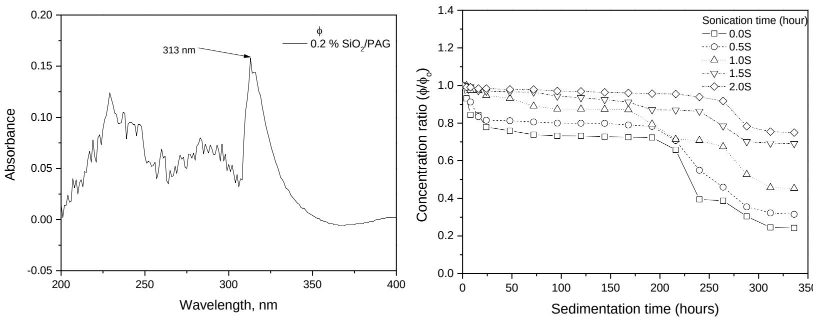
(a) (b)

Figure 5. SiO_2/PAG nanolubricant (a) After preparation (b) After 14 days



(a) (b)

Figure 6. (a) Absorbance value against wavelength (b) Concentration ratio against sedimentation time of Al_2O_3/PAG nanolubricant



(a) (b)

Figure 7. (a) Absorbance value against wavelength (b) Concentration ratio against sedimentation time of nanolubricant of SiO_2/PAG nanolubricant

Concurrent to the photographed sedimentation method, UV-vis spectrophotometer was also being carried out. In the UV-vis spectrophotometer test, the peak absorbance wavelength of the Al_2O_3/PAG and SiO_2/PAG nanolubricants at 0.2 % volume concentration first needs to be determined. The scanning of the UV-vis spectrophotometer in the range of 200 nm to 400 nm result was shown in Figure 6(a) and Figure 7(a). Referring the Figure 6(a), the scanning results show that the peak absorbance for Al_2O_3/PAG lies at 325 nm wavelength with the absorbance value of 2.852. The colloidal stability of these five samples then were tested based on 325 nm wavelengths. The linear relationship between absorbance and concentration was seek and proofed that it follows the Lambert equation. Then the ratio of immediate concentration reading over initial concentration against the sedimentation time was plotted and depicted in Figure 6(b). From the graph, it clearly shows that the 1.5S sample was very stables. After 14 days (336 hours), the relative concentration was maintained above 90 % compared to initial concentration. 1.0S sample also shows good stability and keep maintaining above 80 % concentration ratio even after 336 hours. The graph also shows that without sonication, the 0.0S sample had poor stability with relative concentration value drop to 25 % just after 48 hours. Interestingly to observe through concentration ratio reading, 2.0S shows very poor stability with just 20 % absorbance ratio after day 4 (96 hours). The ratio value was below the 0.0S non-sonication sample. Thus, ultrasonication initially would give positive effect towards stability but above 1.5 hours, it gave negative impact towards Al_2O_3/PAG nanolubricant stability. This is supported the argument by Ghadimi et. al [33] where they mentioned after exceeding the optimized period of sonication process, it will cause more severe complications in clogging and agglomeration causing in fast sedimentation. 2.0 hours sonication initially will further reduce the size of agglomeration. But the smaller nanoparticles have higher surface energy, hence it will increase the possibility of agglomeration of nanoparticle [37]. This is the possible reason why the 2.0 hours sonication time has higher agglomeration and deteriorate the stability of Al_2O_3/PAG nanolubricant.

Referring the graph as depicted in Figure 7(a), the scanning results of SiO_2/PAG nanolubricant show that the peak of absorbance lies at 313 nm wavelength with the absorbance value of 0.158. Hence, the colloidal stability of five samples of SiO_2/PAG nanolubricant then were tested based on 313 nm wavelengths. Figure 7(b) depicted the immediate concentration reading over initial concentration ratio against the sedimentation time for SiO_2/PAG nanolubricant. From the graph, it clearly shows that the 2.0S sample was stable and concentration ratio was above 75 % even after 336 hours (two weeks). 1.5S sample also show good sign of stability throughout experiment but in the end, could not hold above 70 % absorbance ratio after 288 hours (day 12). Other samples show deterioration of concentration ratio over sedimentation time and could not able to maintain the stability above 50 % in two-week time periods. It shows that the other samples had poor stability. SiO_2 nanoparticle has lower density and bigger in size. This may the reason why 2.0 hours sonication of SiO_2/PAG nanolubricant is still stable compared to 2.0 hours sonication of Al_2O_3/PAG nanolubricant.

Conclusions

In this study, the effect of ultrasonication time towards stability of Al_2O_3/PAG and SiO_2/PAG nanolubricants have been evaluated. Ultrasonication process up to certain time improves the stability of the Al_2O_3/PAG nanolubricant. 1.5S sample reflecting in 1.5 hours of sonication time show the best result in its stability. But elongated sonication time would jeopardise the stability of Al_2O_3/PAG nanolubricant. While 2-hours sonication time is proven to be sufficient in preparing stable SiO_2/PAG nanolubricant. In summary, it is essential to control the time of sonication in order to gain best stability of these nanolubricants.

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