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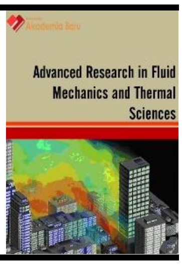

Experimental Investigation on Preparation and Stability of Al_2O_3 Nanofluid in Deionized Water and Ethylene Glycol

Wahaizad Safiei[1,2,*], Md. Mustafizur Rahman[1], M. A. Hadi[2], W. H. Azmi[1], M. N. Arifin[3]

[1] Faculty of Mechanical Engineering, Universiti Malaysia Pahang, 26600 Pekan, Pahang, Malaysia
[2] Faculty of Engineering Technology, Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300 Gambang, Pahang, Malaysia
[3] Faculty of Chemical & Natural Resources Engineering, Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300 Gambang, Pahang, Malaysia

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ABSTRACT

Nanofluid has the potential as a cooling medium for the next generation fluid as it possesses many advantages in many engineering applications. However, one of the main challenges is to establish a well-dispersed nanoparticles system in a base fluid. The preparation technique of nanofluid plays an important part as it influences the measurement of thermal conductivity. Therefore, the objectives of this study are to evaluate the nanoparticle dispersion in different base fluid compositions and to determine the optimized suspension sonication time. In detail, 0.2 wt.%, 0.4 wt.% and 0.6 wt.% of Al_2O_3 nanofluid stability in the three ratios of base fluid (deionized water:ethylene glycol) 80:20, 70:30 and 60:40 were studied. The studies were based on a visual inspection and spectral absorbance analysis. It has clearly shown that the nanofluids prepared in 60:40 base fluid within 3 hours sonication time was the most stable suspension compared to other nanofluids. The visual inspection indicated nanofluid condition remains stable after 30 days. The spectral absorbance of nanofluids was recorded at 100 % for 5 days after preparation and remains above 95 % compared to the initial value, reflecting stable suspension. Hence the novelty of this work lies in the nanofluid stability based on sonication time and base fluid compositions.

1. Introduction

Nanofluid was first discovered by Choi and Eastman [1] in 1995 and since then, there has been much research reporting its plethora of engineering applications which utilize the enhanced thermal properties in radiators, heat exchangers [2,3], medical, nuclear system and electronic cooling system. Thermo-physical properties enhancement has sparked an interest among researchers [4–7] to further explore the potential of nanofluid. Nanofluid has exhibited improved physical properties and rheological performance indicated by the measurement of thermal conductivity and viscosity as well as heat transfer coefficients [8,9] than the base fluid. However, formulating a stable nanofluid suspension in aqueous system is a common challenge among researchers of any backgrounds and

* *Corresponding author.*
E-mail address: wahaizad.safiei@gmail.com

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fields [10,11]. Preparing homogeneous nanofluid is the first and the most critical part in the two-step preparation method, which requires the dispersion of dry nanoparticles into a base fluid for heat transfer enhancement [12]. Many researchers have expressed their concern about this instability issue which affects the validity of physical properties measurement of nanofluid [13–17]. In addition, the nanoparticle aggregation and settling as a result of unstable suspension, are complex phenomena determined by the number of parameters such as concentration, viscosity, density, particle size, type of base fluid and solution temperature [18,19]. Nanoparticles agglomeration not only causing suspension instability but also reducing the thermal conductivity of nanofluid [20,21]. Generally, the three aspects attributed to nanofluid stability are kinetic stability, homogeneous dispersion and chemical reaction. The suspension is considered stable when the dispersion of nanoparticles and the supernatant concentration remain constant significantly for a very long period of time. The dispersion of nanoparticles in a base fluid have strong Brownian motion where colloidal particle moves randomly in the suspension [22]. The liquid temperature and size of nanoparticles and nanoclusters are the main factors dictating the Brownian motion. Brownian motion decreases by increasing size of nano-clusters. Due to this movement, the sedimentation which caused by the gravity force can be delayed in the suspension. The stability of nanofluid is deteriorated over the time due to aggregation formation among particles. The suitability of dispersion between nanoparticles and base fluid is crucial in minimizing chemical reaction. The main idea is to obtain zero chemical reaction for better stability.

To date, there has been inadequate research conducted to understand the formation of agglomeration among nanoparticles in suspension. In the previous study carried out by Lee *et al.,* [23], the nanoparticles remain adhered to agglomerate and formed aggregation of particles due to surface interaction like van der Waals force among particles and molecules, high surface area, high surface activity and gravity force, even after mixing and shaking to purposely break up the clogging [8,23-26]. However, the formation of the cluster among nanoparticles favours high temperature as nanofluid is more stable at high temperature rather than at low temperature due to the surface energy of nanoparticles [27]. In 1939, Derjaguin, Landau, Landau, and Overbeek (DLVO) came out with colloidal stability theory that mentioned when a nanoparticle approaching each other, there are two forces involve namely van der Waals attractive force and electrical double layer repulsive force that could make suspension stable or unstable [28]. Thus, the suspension is in the stable state when the particles have sufficient repulsive force preventing the collision of particles [29]. The collision normally happens when the attractive force of a particle is stronger than repulsive force.

Nanofluid stability can be assessed by evaluating the clustering effects on nanofluid using various techniques such as visual inspection, UV-visible spectrophotometer, pH value control and zeta potential measurement [24]. A different technique and its parameters use for nanofluid preparation have adversely affected the nanoparticle dispersion in the base fluid. In addition, different nanofluid has a specific mixing time and sonication time that is essential to control in order to get stable suspension [30]. However, nanoparticle and nano-cluster size measurement also can be employed in order to find the stability of nanofluid. For example, Kwak and Kim [31] found that 9 hours sonication time of CuO nanofluid in ethylene glycol was more stable over 1-hour sonication time by comparing the nanofluid stability using TURBISCAN Lab transmission analysis and after evaluating the particle size for 100 days. Sadeghi *et al.,* [32] investigated stability of gamma-Al_2O_3 nanofluid using UV-Vis Spectrophometer and dynamic light scattering (DLS). The results showed by increasing sonication time, size of nano-cluster decreases and zeta potential value increases which attributed to the stability of suspension. Ultrasonication is a vital and common technique for boosting the stability of the nanoparticles in nanofluid. Sonication fragments the nanoparticle clusters and disperses nanoparticles in the base fluid by oscillating the agglomerated particles using ultrasonic waves [33].

Moreover, optimum sonication time is essential when preparing the nanofluid, otherwise higher temperature that gradually increase during sonication can diminish the thermal properties of nanofluids [34]. Rouxel *et al.,* [35] presented that the average size of nanoparticles significantly reduced in the suspension by sonicating 1 g/L concentration of 13 nm Al_2O_3 nanoparticles in water with 30% amplitude. This is an important indication of a considerable nanoparticle stability enhancement that aligned with the Stoke’s law.

Usri *et al.,* [36] investigated the properties of nanofluid in the different mixture of distilled water and ethylene glycol, however, visual observation technique was applied to evaluate the nanofluid stability. Said *et al.,* [37] evaluated the stability characterization of different nanofluids based on Dynamic Light Scattering (DLS). However, it just shows the size of the nanoparticle in the solution which did not reflect the nanofluid stability condition. Optimum sonication time is essential when preparing the nanofluid, otherwise higher temperature that gradually increase during sonication can diminish the thermal properties of nanofluids.

From the literature studies, a comprehensive study that employ visual observation, UV–visible spectrum and sonication time effect on different base fluid ratio has not yet been carried out to investigate Al_2O_3 nanofluid stability. Therefore, the main objective of this study is to determine the nanofluid stability among different ratios of the base fluid at 60:40, 70:30 and 80:20 of deionized water and ethylene glycol respectively. The optimum sonication time that effect on nanofluid stability would be determined from 1 hour to 3 hours with half an hour interval for the different ratios of the base fluid. The base fluid conditions with different ratio have significantly influence on the dispersibility of nanoparticle.

2. Experimental Strategy

In this study, the stability of samples was tested by comparing nanoparticle settlement through daily observation in the test tubes and a spectral absorbance ratio of nanofluid. To evaluate the absorption spectra of nanofluids, UV-vis spectrophotometer (Model UV-1800), Figure 1, manufactured by Shimadzu Scientific Instrumentation was used. The validation of wavelength accuracy, stray light and resolution for instrument was performed in scheduled maintenance by technical personnel. The instrument was validated to ensure the accuracy of the absorbance data.

Fig. 1. Shimadzu UV-Visibile spectrophotometer (Model: UV-1800)

Spectral absorption analysis is an efficient way to study nanofluid stability by examining the nanoparticles absorbance, with respect to stability condition of colloidal solutions. The spectral absorbance was then used to correlate the nanoparticles concentration by employing Beer-Lambert's law to develop a linear correlation between colloidal suspension concentration and absorbance intensity as shown in Eq. (1) [38].

$$A = log(\frac{i_o}{i}) = \varepsilon CL \quad (1)$$

i_o is the initial intensity of light, i is the final intensity of light, ε is the molar absorptivity, C is the concentration, and L is the path of length through the sample. The nanofluid can be prepared using a single-step method or two-step method. The single-step method combines two stages of preparation, nanoparticles and nanofluid, simultaneously [39]. Nanoparticles are prepared by physical vapor deposition (PVD) and liquid phase methods. On the other hand, in the two-step method, the nanofluids were prepared by dispersing the nanoparticles into a base fluid. It was found that, for oxide nanoparticles like Al_2O_3, two-step method produces better results than a one-step method, whereas for metal nanoparticles could get less successful [15]. Therefore, in this study the Al_2O_3 nanofluids were prepared using a two-step method which dry nanopowder with 13 nm size from Sigma-Aldrich were dispersed into the base fluid. Figure 2 shows preparation processes of Al_2O_3 nanofluid by applying two-step method.

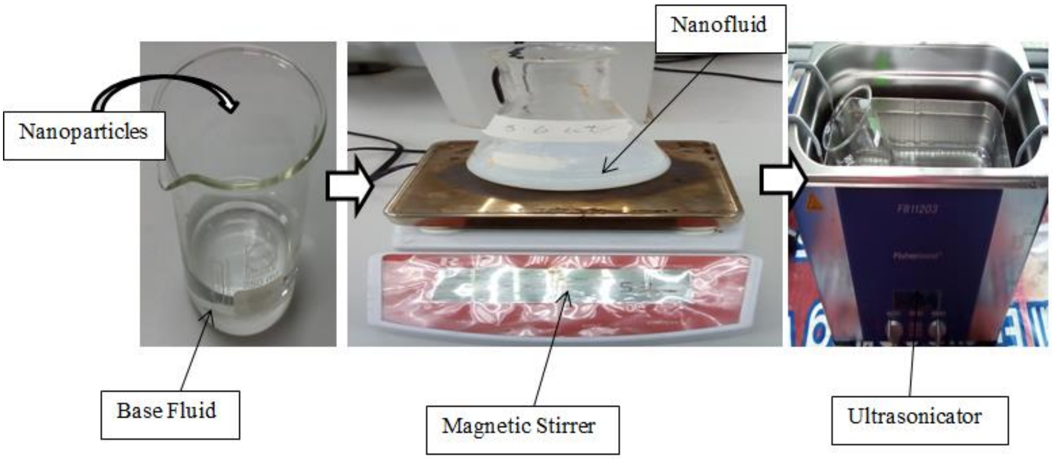


Fig. 2. Step by step process of Al_2O_3 nanofluid preparation

Three different volume ratios of base fluid were prepared, 60:40, 70:30, 80:20, by mixing the deionized water and ethylene glycol. Whereas the nanoparticles were prepared in three weight concentration of 0.2 wt.%, 0.6 wt.%, and 1.0 wt.%. After the mixing of nanoparticle in the base fluid, the suspensions were stirred for half an hour using a magnetic stirrer to enhance the mass transfer of dispersed particles. Then, the solutions were placed in an ultrasonic bath for one to three hours of sonication time as suggested by Redhwan *et al.,* [30] and Razak *et al.,* [25] for single nanofluid. The samples were ultrasonicated at a power of 330 W and the frequency of 50 Hz. The use of surfactant is an effective way to improve the nanoparticles dispersibility in the suspension [37,38], however, no surfactant was used in preparing the stable Al_2O_3 nanofluid in this study. This is to ensure no external factor interrupt the stability condition of nanofluid in this study. As mentioned earlier, base fluid, sonication time and concentration are the factor studied of Al_2O_3 nanofluid stability. The details of Al_2O_3 nanoparticle and base fluid properties are shown in Table 1.

Table 1
Thermal and physical properties of Al_2O_3 and deionized water/ethylene glycol base fluid

Nanoparticle/ Base fluid	Thermal conductivity (W/mK)	Electrical conductivity σ, (µS/cm)	Dielectric constant ε	Density ρ, (kg/m^3)	Reference
Al_2O_3	36	10-8	9.1-9.3	4000	[48]
Ethylene Glycol	0.252	1.07	38	1110	[48]
Deionized Water	0.58 at 25ºC	0.055-0.1	78.4-80.1	999	[40– 42]

In order to minimize the sedimentation and agglomeration, Stokes' Law can be referred as a guide for nanofluid preparation [30,42]. There are three main factors affecting the sedimentation time according to Stokes' Law which are nanoparticle size, base fluid viscosity and density difference between nanoparticle and base fluid. Hence, by referring to Stokes' Law in Eq. (2), the sedimentation time of nanofluid can be minimized effectively by applying three strategies.

i) Disperse nanoparticle using the smallest particle size
ii) Increase viscosity of the base fluid
iii) Ensure both nanoparticle and base fluid having a minimal delta of density value.

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

where V is the sedimentation speed of a particle, R is the radius of a particle, η is the dynamic viscosity of the liquid, ρ and ρ' are the density of the particle and the liquid, respectively.

By applying the Stokes' Law principle of a particle sedimentation velocity in Eq. (1), the base fluid dynamic viscosity based on three different ratios were measured as shown in Figure 3. Brookfield LV-DVII viscometer was used to measure the base fluid dynamic viscosity. The 40 vol. % ethylene glycol base fluid apparently gives higher viscosity than the other two ratios due to the dominant glycol phase. Through the proposed analysis, both, the suitable base fluid ratio and the optimum sonication time are obtained, thus improving suspension stability. However, the heat transfer capability of nanofluid is affected if the base fluid viscosity increases or the difference density decreases [25].

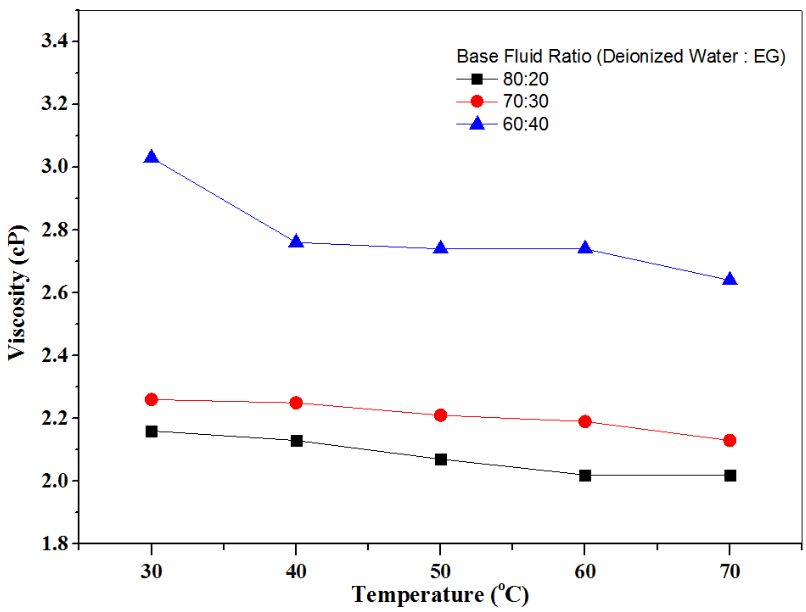


Fig. 3. Base fluid dynamic viscosity based on three different ratios of deionized water and ethylene glycol

3. Results

3.1 Nanofluid Stability Evaluation (Visual Inspection)

Visual inspection is a basic method to assess the sedimentation and agglomeration of nanoparticles in the suspension. Nanofluid can be considered stable when there is no sedimentation found in the test tube. The formation of different layers of colors and transparency can be observed easily using this technique, indicating the inhomogeneity of the solution. Figure 4 shows the results of Al_2O_3 nanofluid after one month, in three different ratios of base fluid (60:40, 70:30 and 80:20) and sonication times.

Fig. 4. Al_2O_3 nanofluids in (a) 60:40 (b) 70:30 and (c) 80:20 base fluid (deionized water: ethylene glycol) at different sonication times after one hour and one month

On the first day of preparing the samples, there were no sedimentations observed at the bottom of the test tubes irrespective of the base fluid ratio and sonication time. The samples seem stable as the nanoparticles were well dispersed in the suspension. The conditions remain steady for two weeks. It was very difficult to distinguish the presence of sedimentation especially within two weeks after nanofluid preparation because there was no formation of nanoparticles clusters that can been seen. However, a marginal sedimentation of particles was observed after three weeks of preparation, particularly for 80:20 base fluid. After one month of samples preparation, more particles settling down especially for 80:20 and 70:30 (Figure 4(b)) base fluid suspensions. However, Al_2O_3 nanofluid in 60:40 base fluid suspensions remain homogenous after one month (sonication time of 3 and 2.5 hours). This finding can be explained by the minimization of agglomeration by forming smaller clusters in the suspension with the effect of temperature and sonication which substantially increased the nanofluid stability.

3.2 Nanofluid Stability Evaluation (Spectral Absorbency Analysis)

The relationship between spectral absorbance and Al_2O_3 nanofluid concentration is illustrated in Figure 5. It has clearly shown that the findings are in line with the Beer-Lambert's law for all base fluid ratios.

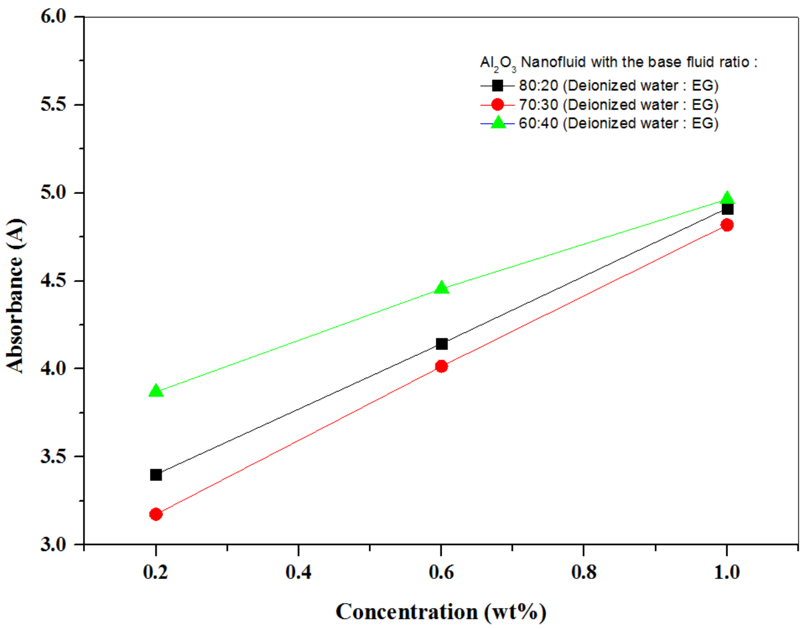
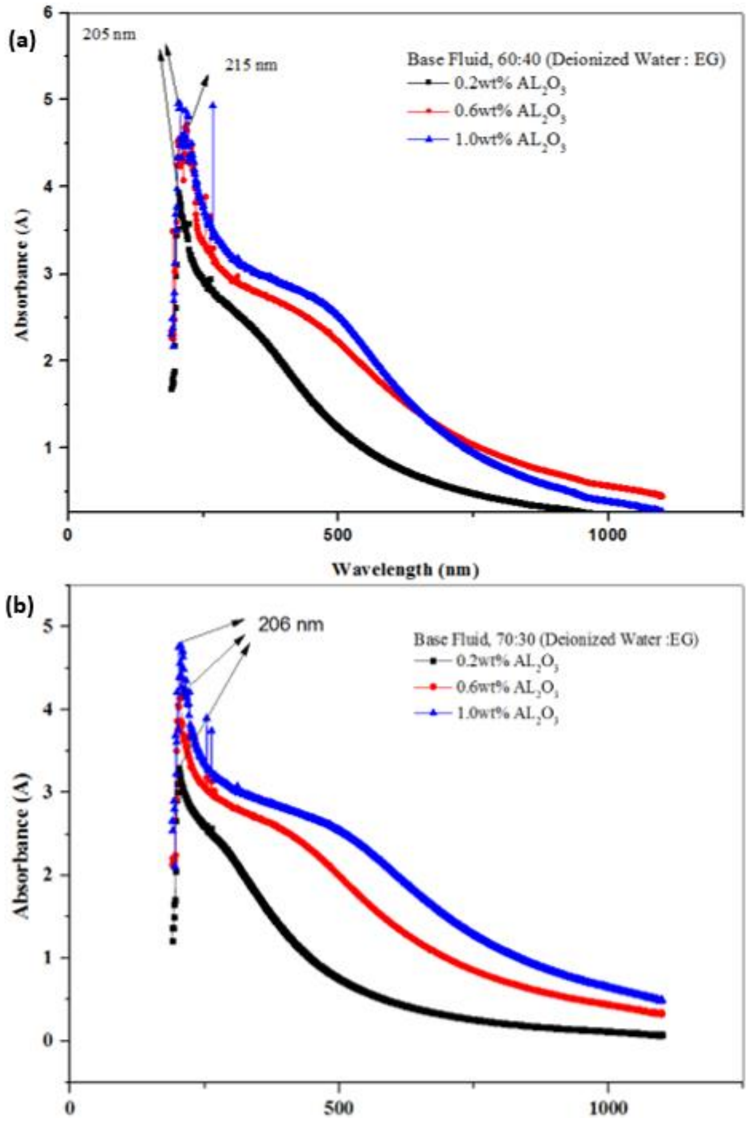


Fig. 5. Nanofluid spectral absorbance with respect to different Al_2O_3 concentration (wt. %)

The variation of a supernatant particle concentration in a suspension over the time can be determined by measuring the absorption of nanofluid at a certain wavelength of spectral. As suggested by Zawawi *et al.,* [38] and Sharif *et al.,* [43], the maximum nanofluid absorbance can be obtained by scanning the nanofluid from 190 to 1100 nm wavelength. This is the peak value of wavelength which nanofluid has maximum absorbance when light intensity passed through it. The peak value of wavelength also acts as a reference point for measuring the absorbance of nanofluid for subsequent days.

The suspension absorbance versus wavelength for different base mixture fluid at 0.2 wt.%, 0.4 wt.% and 0.6 wt.% is illustrated in Figure 6. The results revealed that the maximum absorbance peak of Al_2O_3 in 60:40, 70:30 and 80:20 nanofluid were recorded at 205 nm, 206 nm and 217 nm of wavelength respectively. However, the maximum absorbance peak of 1.0 wt.% Al_2O_3 in 60:40 nanofluid slightly shifted to a higher value, 215 nm. Suspension having a high degree of aggregation and active nanoparticles movement during the spectral absorbance measurement, is believed to produce this outlier. It can be seen from the graphs that the absorbance value is varying at a different wavelength and also corresponding to its concentration even at the same peak of wavelength.



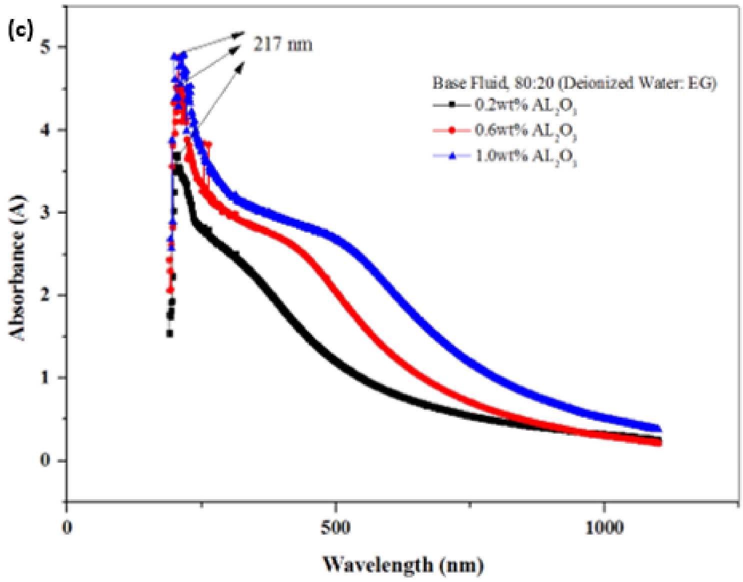
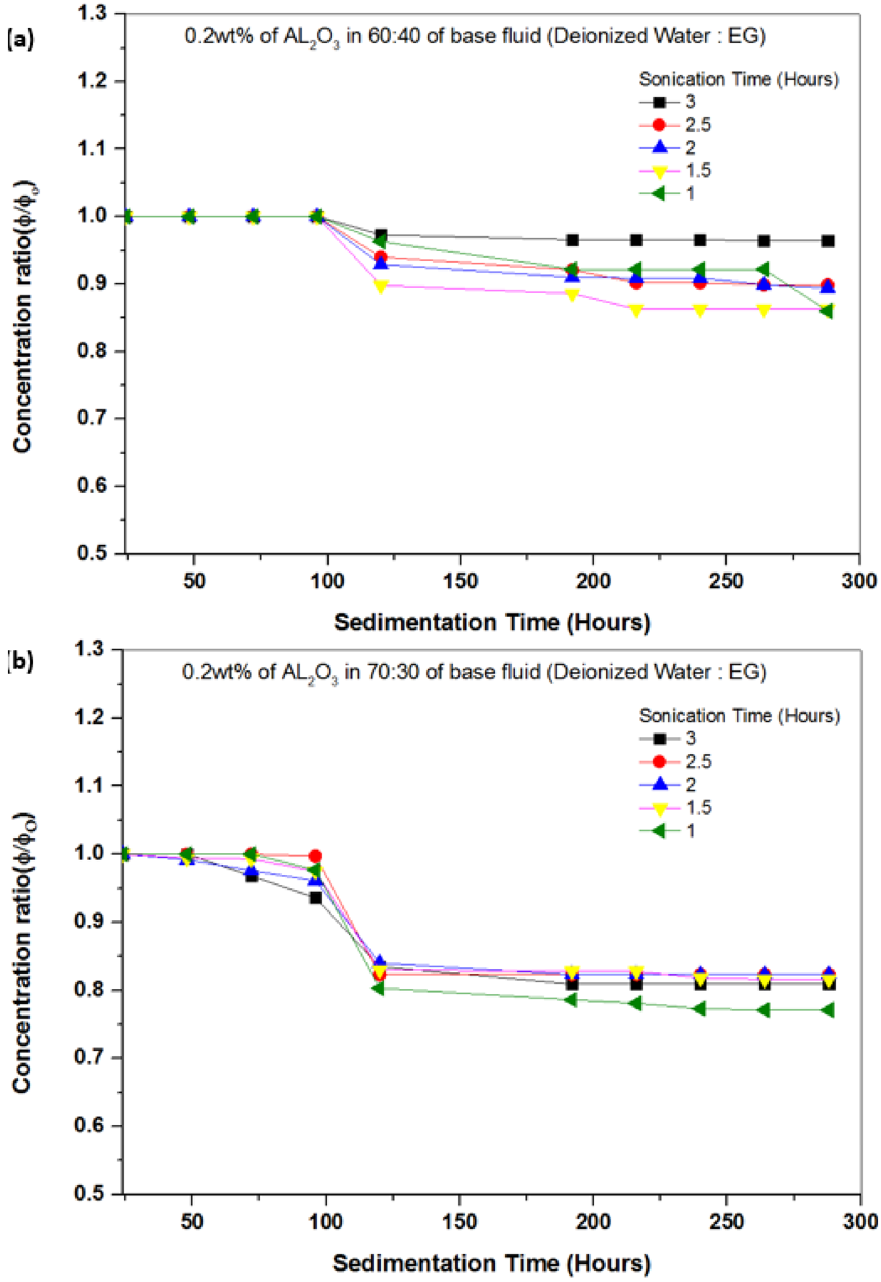


Fig. 6. Peak absorbance for 0.2 wt.%, 0.6 wt.% and 1.0 wt.% Al_2O_3 in (a) 60:40 (b) 70:30 (c) 80:20 nanofluids (Deionized water:EG)

In order to obtain the absorbance concentration, the UV light is radiated through the nanofluids and the absorbance value was recorded. The absorbance value was recorded once in a day for three weeks. Hence, the present absorbance value can be compared with the initial value prior to obtain the ratio. Higher spectral absorbency of UV transmittance means higher mass fraction particles in the suspension which is attributed to better dispersion of nanoparticles in base fluid [44]. This is a common phenomenon due to the presence of nanoparticles with greater concentration have high tendency for the UV-light being absorbed rather than passing the solution.

Moreover, the absorbance value decreased over the time passes due to nanoparticles are settling down to the bottom side. Therefore, by comparing the present absorbance value to initial value, the stability of nanofluid can be measured. Furthermore, the method for preparing Al_2O_3 nanofluid with better stability can be identified. Figure 7 shows the concentration ratio over sedimentation time for 0.2 wt.% of Al_2O_3 nanofluid in 60:40, 70:30 and 80:20 of the base fluid (Deionized water: EG) in different sonication times. In terms of absorbance value analysis, nanofluid is considered stable when there is no drastically decrease of concentration ratio value from initial 1.0 over sedimentation time of nanoparticles. Hence, the Al_2O_3 nanofluid that was prepared in 60:40 of the base fluid for 3 hours sonication time was found stable as shown in Figure 7(a). After 96 hours, the concentration ratio is decreasing below 100% regardless of sonication times in 60:40 of the base fluid. However, the nanofluids remain stable even after 300 hours of sedimentation time. This reflects that the nanofluids were stabilized by 3 hours sonication during samples preparation. In addition, during sonication, the sound energy agitates the base fluid to disperse the nanoparticles. When the temperature is applied and gradually increased, the strength of chemical bonding among molecules are decreasing, thus, a nanoparticle is getting more space for dispersion. The trend of absorbance ratio decreasing is much faster in 70:30 and 80:20 of base fluid as observed in Figure 7(b) and Figure 7(c) respectively. In fact, there is a sudden drop in terms of absorbance ratio after 96 hours for the suspensions that containing 70:30 and 80:20 of deionized water and ethylene glycol indicates unstable suspensions.


(a)
0.2wt% of AL_2O_3 in 60:40 of base fluid (Deionized Water : EG)
Sonication Time (Hours)
3
2.5
2
1.5
1
Concentration ratio(ϕ/ϕ_o)
Sedimentation Time (Hours)
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
50
100
150
200
250
300
(b)
0.2wt% of AL_2O_3 in 70:30 of base fluid (Deionized Water : EG)
Sonication Time (Hours)
3
2.5
2
1.5
1
Concentration ratio(ϕ/ϕ_O)
Sedimentation Time (Hours)

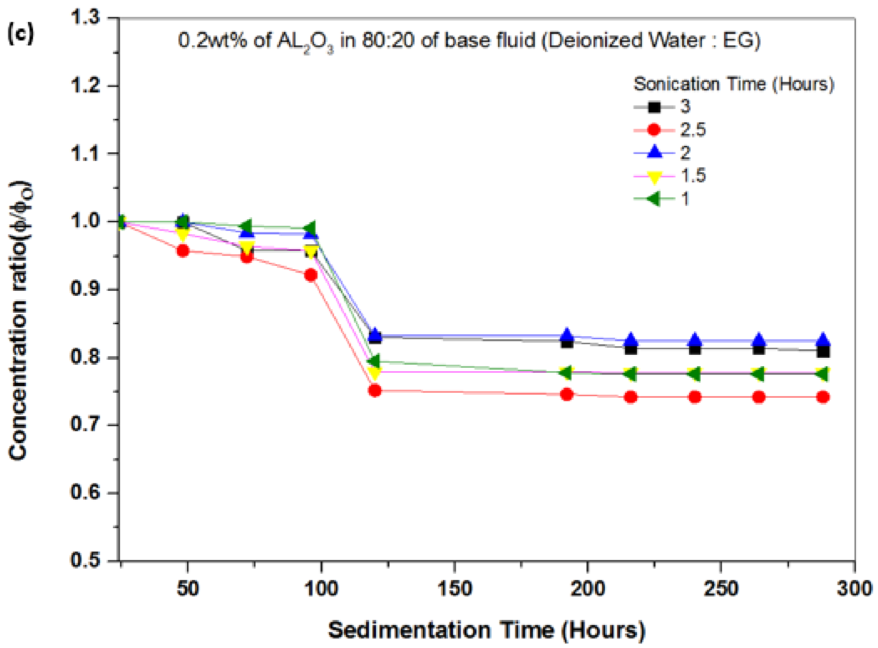


Fig. 7. Concentration ratio over sedimentation time for 0.2 wt% of Al_2O_3 nanofluid in (a) 60:40 (b) 70:30 (c) 80:20 of the base fluid (Deionized water: EG) at different sonication times

4. Discussion

Figure 8 shows the results of spectral absorbance based on Al_2O_3 nanofluid in different concentration and base fluid. The similar trends of Al_2O_3 nanofluid stability can be seen on nanofluid that dispersed in the ratio 60:40 of deionized water: ethylene glycol and PAG lubricant respectively. It shows that the spectral absorbance is above 95% within 400 hours of sedimentation time for both suspensions. In order words, water-based nanofluid exhibits better dispersibility for 0.2 wt.% of Al_2O_3 nanofluid especially for the first 7 days of study even though it contains 60% of deionized water. However, this result was based on 3 hours sonication time. On the other hand, a higher concentration of Al_2O_3 nanofluid at 1 vol.% exhibits less dispersion of the nanoparticles for a longer of sedimentation time. Furthermore, it can be seen that more volume percentage of deionized water in the nanofluid also reveal the similar trends as the particles are pretended to settling down faster rather than moving randomly and well-dispersed in the suspension.

The similar findings also revealed by Sadeghi *et al.,* [32] that in order to get the most stable γ-Al_2O_3 nanofluid in de-ionized water, the optimum ultrasonic optimum vibration time was found within 150 min and 200 min for 1%, 2% and 3% of volume concentration. The results were based on zeta potential measurement, dynamic light scattering and UV-V is spectrum methods. Mahbubul *et al.,* [45] found that the nanofluid stability was greater at lower concentration 0.5 vol.%. The average particle size rapidly reduced in the first 3 hours of sonication time but remains constant after 3 hours at 210 nm for 5 vol.% of ZnO nanofluid in ethylene glycol. Thus, Yu *et al.,* [46] dispersed the nanoparticles for 3 hours of sonication time in preparing ZnO nanofluid in order to get a well-dispersed suspension. In short, longer sonication time able to reduce aggregated size of nanoparticles which resulted better stability of nanofluid [47]. The findings of this study are really significant for the appropriate selection of base fluid ratio, concentration and sonication time in preparing single and hybrid nanofluid. This is an attempt to obtain related and important information about the factors affecting nanofluid stability in initial phase of nanofluid preparation by conducting systematic experimental investigation.

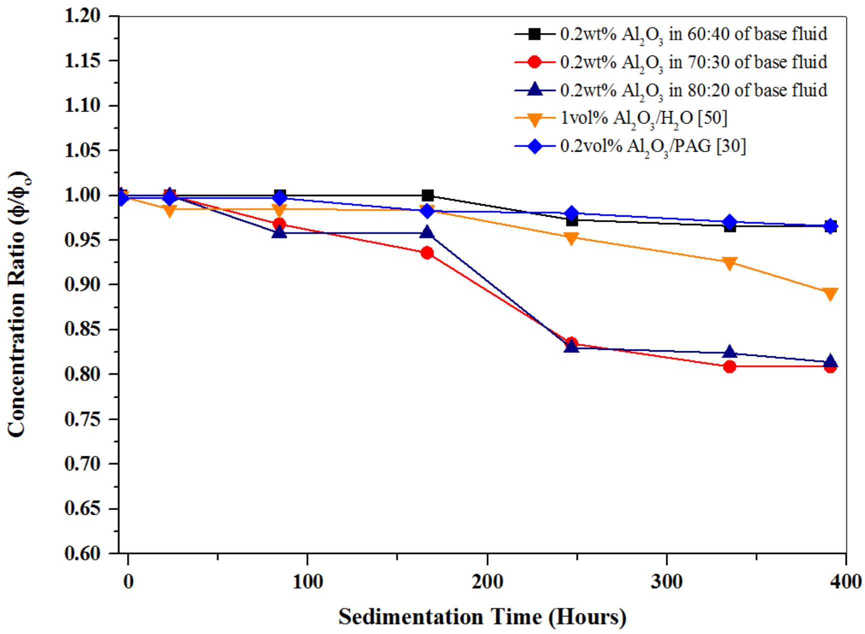


Fig. 8. Concentration ratio over sedimentation time for 0.2 wt% of Al_2O_3 for 3 hours sonication time in three conditions of base fluid ratio

5. Conclusion

Nanofluid synthesis plays an important role to obtain stable suspension. Nanofluid thermal conductivity is greatly affected by the stability condition of nanofluid which can be obtained by determine the base fluid ratio, sonication time and concentration of nanoparticles. These are novel areas which are significant to the chemical colloidal and interfacial tension in the suspension. Through this study, it can be concluded that

i. The most stable nanofluid was prepared in 60 to 40 base fluid, with sonication time of 3 hours. This is the optimized composition mixture for preparing Al_2O_3 nanofluid. The spectral absorbance remains above 95% compared to initial concentration over 96 hours. Through observation, the same nanofluid remains stable even after 30 days.
ii. After 3 hours sonication time, the stability of samples slightly dropped for 70 and 80 vol.% deionized water in ethylene glycol base fluid but remain above 95% of spectral absorbance. The similar trends observed for both samples when the stability further dropped after 96 hours but remains stable for long sedimentation time.
iii. The absorbance ratio drastically dropped after 96 hours for 70:30 and 80:20 suspensions. But for 60:40 base fluid, the absorbance ratio remains constant after 96 hours of sedimentation time.
iv. The viscosity of base fluid plays a key role to establish stable nanofluid as obtained for 60:40 base fluid (Deionized water: EG) which exhibited a good dispersibility of nanoparticles. This is because the base fluid with 40% of ethylene glycol shows higher viscosity and a particle is well-dispersed in this ratio of base fluid. Moreover, this finding is aligned with the Stoke Law theory that the sedimentation speed of a nanoparticle is reduced when the dynamic viscosity of the base fluid is higher.
v. For future recommendation, other factors influencing nanofluid stability such as sonication temperature and different weight concentration can be explored. Since hybrid

nanofluid receives wider attention nowadays, a comprehensive study of different nanoparticles dispersion into base fluid also can be conducted.

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