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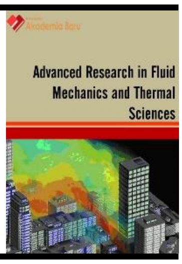


# Optimization on the Nanoparticles Stability in Liquid Phased Condition by Using Taguchi Analysis

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**ABSTRACT**

The problem arises from the application of nanofluid is that the nanoparticles tend to agglomerate and sedimentation which affect the stability of nanofluid. The aim of the study is to investigate the effect of different surfactant agents and homogenize time on the stability of nanoparticle (SAE 15W 40). In this study, the Nano-oil was prepared by dispersing the nanoparticles with an optimal composition of 0.5 vol.% 70 nm graphite, $Al_2O_3$ and $ZrO_2$ in conventional engine oil (SAE 15W 40) grade by using ultrasonic homogenizer for 10-30 minutes. In order to determine the stability of the dispersion, Oleic Acid, SDBS Salt and Sodium chloride were utilized as a surfactant agent with an optimal composition of 0.3 vol. %. The stability test was conducted by using UV-spectrophotometer as quantitative test and observation of sedimentation by using the traditional method as a qualitative test. The collected data were analysed by using the Taguchi method to determine the optimum value of nanoparticle stability. The results of Taguchi analysis show that zirconia nanoparticle with SDBS agent is more stable compared with another sample. Unfortunately, Taguchi analysis analysed on the alumina nanoparticle with an oleic acid agent is a less stable sample.



## 1. Introduction

Nanoparticles as an additive in lubricants are found to give better performance in tribological properties and increment of heat transfer. Lubricants are the substances utilized to decrease the effect of friction between the two moving sliding surfaces. Generally, lubricants can be classified based on their physical states such as a solid lubricant, semi-solid lubricant and liquid lubricant. Utilizing nanoparticles as an added substance in fluids to expand the thermal performance of fluids has already begun in the 1990s. The lubricant is vital in any industry because it helps to reduce the frictional resistance, wear, surface deformation, provide protection against corrosion, and also

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improve the efficiency of the engine component. Besides, a lubricant with nanoparticle enhanced the load-carrying capacity, friction reduction properties and anti-wear. Kong *et al.,* [1] found that the mechanism of lubricant may be accessible when nanoparticles were utilized as additives in lubricants such as polishing effect, rolling of Nano-spheres, self-repairing effect and tribochemical.

The nanoparticle as an additive in the lubricant was also called as a nanofluid. According to Khairul *et al.,* [2], the mixtures of base fluid and nanoparticles have produced the nanofluid without the chemical reaction between these two components. The existence of nanoparticles in the lubricants contributes to better flow combination and excellent thermal conductivity compared to pure fluid. The typical size of producing nanofluid usually below 100 nm by the dispersion of metallic or nonmetallic nanoparticles into the mixture [3, 4]. Nanofluids have superior thermophysical properties over the base fluids in terms of thermal conductivity, viscosity, thermal diffusivity and convective heat transfer coefficients. The high surface area of nanoparticles improves the thermal conductivity of nanofluids from the heat transfer takes place on the particle surface. The number of atoms existing on the nanoparticles surface is bigger compared with the interior. In addition, the nanoparticle may reduce erosion and clog due to the smaller size which contributed to the stability of the nanoparticles [5, 6].

Generally, one of the fundamental prerequisites of nanofluid features is the long-term stability generated through nanoparticles dispersion. The stability of nanofluid is crucial to ensure the nanofluid is in good condition. Yue and Xie [7], stated that the stability was indicated by those particles which do not aggregate at a significant rate. The nanoparticle aggregation rate is determined by the frequency of collision caused by the probability of cohesion during the collision and Brownian motion. Nanoparticles tend to aggregate because of the high surface region and surface movement [1]. Nanofluids stability is determined by the amount of van der Waals forces and electrostatic repulsion force between nanoparticles dispersed living in nanofluids. When the electrostatic repulsion force is greater than the forces of attraction then the stability will be achieved. Another researcher also found that the repulsion force from the repel of particles forms van der Waals which leads to agglomeration and this is when stabilization has been reached through the permeation of stable particle on the surface of the pigment [8]. In order to increase the repulsion force between nanoparticles, two mechanisms are used like steric mechanism and electrostatic mechanism. Nevertheless, the field of this study focuses only on the steric mechanism.

To obtain better concentration and stability of the nanofluid, physical or chemical treatment has been conducted in which surfactant agent is added into the lubricant. The utilization of surfactants is the imperative strategy to upgrade the stability of nanoparticles in liquids condition [9, 16]. The usefulness of the surfactants under high temperature is another major concern, particularly for high-temperature applications. In addition, a homogenizer is utilized in this investigation as a mechanism to stabilize the nanofluids with surfactant agents. The intermolecular interactions between particles can be disrupted by sonication while the agglomeration in the nanofluid can be decreased by homogenization processes [10, 15, 17]. Hence for this study, the effect of difference surfactant agents, temperature and homogenize time on the nanoparticle stability inside SAE 15W 40 conventional engine oil grade were optimized and analyzed. The results expected can conclude the suitable surfactants, and optimum parameters to obtain the most stable nanoparticles inside liquid phased condition.

## 2. Methodology

### *2.1 Design of Experiment 9DOE Taguchi L9*

The Taguchi method used in this study comprise of L9 orthogonal arrays with nine rows based on the number of tests and three columns at three levels. It is utilized for sampling, testing, and analysis of the results. The L9 orthogonal array has eight Degrees of Freedom (DOF). Six of DOF are assigned to three factors which each one has two DOF and two of DOF were assigned to errors. Furthermore, to observe the level of significance of the outline parameters, three outline parameters were determined as homogenizer time, types of nanoparticles, and type of surfactant agents which referred to Table 1. The outcome was analyzed by utilizing the analysis of signal-to-noise (SN) ratio to obtain the optimal values for design parameter and quantitatively measured the importance of each primary factor. Table 2 showed the Design of Experiment with L9 orthogonal arrays.

**Table 1**
DOE parameters and levels

| Level | Parameter | | |
|---|---|---|---|
| | Nanoparticle | Surfactant | Time |
| 1 | Graphite, (A) | Oleic Acid (A) | 10 minutes (A) |
| 2 | Alumina, (B) | SDBS (B) | 20 minutes (B) |
| 3 | Zirconia, (C) | Sodium Chloride (C) | 30 minutes(C) |

**Table 2**
DOE with L9 orthogonal arrays

| Test | Factor | | |
|---|---|---|---|
| No. | Nanoparticle | Surfactant | Time |
| 1 | Graphite | Oleic Acid | 10 minutes |
| 2 | Graphite | SDBS | 20 minutes |
| 3 | Graphite | Sodium Chloride | 30 minutes |
| 4 | Alumina | Oleic Acid | 20 minutes |
| 5 | Alumina | SDBS | 30 minutes |
| 6 | Alumina | Sodium Chloride | 10 minutes |
| 7 | Zirconia | Oleic Acid | 30 minutes |
| 8 | Zirconia | SDBS | 10 minutes |
| 9 | Zirconia | Sodium Chloride | 20 minutes |

### *2.2 Sample Preparation*

The sample of Nano-oil in this study was prepared in 100 ml each according to Table 1. The Nano-oil was prepared by dispersing the nanoparticles with an optimal composition of 0.5 vol. % graphite, $Al_2O_3$ and $ZrO_2$ in conventional engine oil (SAE 15W 40) grade using ultrasonic homogenizer which shown in Figure 1. Oleic Acid, SDBS Salt and Sodium chloride were utilized as a surfactant agent with an optimal composition of 0.3 vol. %. During the homogenizing process, the sample temperature controlled not to exceed 70°C.

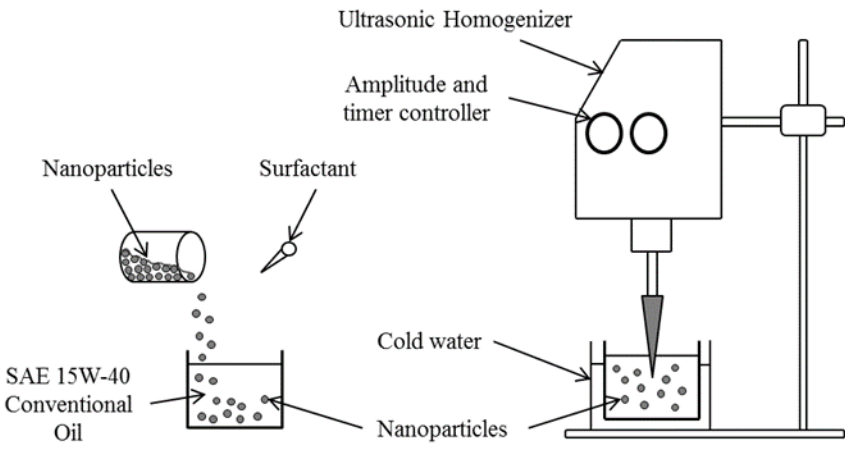


**Fig. 1**. Schematic diagram sample preparation by using ultrasonic method

*2.3 Sample Testing by Utilized UV-Spectrophotometer*

The stability testing of nanofluid was performed using UV-Vis spectrophotometer. The absorbency value measure by UV-Vis Spectrometer was recorded as quantitative analysis. The samples were held by glass cuvette with 1 mm thickness. The absorption of Nano-oil was measured at 200 - 600 nm wavelength. The level of absorbance is relative to the measure of the particles per unit volume, hence it can indicate the scattering strength of the particles in the solution. Figure 2 below shows the schematic diagram for UV-Vis Spectrophotometer.

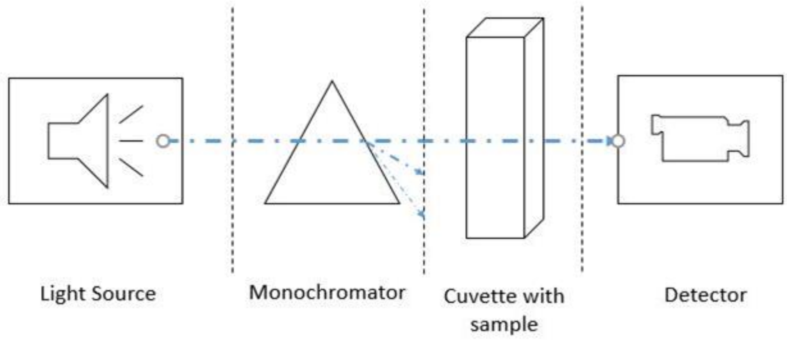


**Fig. 2.** Schematic diagram UV-Vis Spectrophotometer Sedimentation Photograph Capturing Methods

The stability testing of nanofluid was also performed with a normal method which is an observation of sedimentation by capturing the picture for a 2-month period. Sedimentation photograph capturing is a qualitative analysis for measuring the dispersion stability. The dispersion stability was determined by observing the nanoparticles developed at the bottom of the bottle.

## 3. Results and Discussion

*3.1 Analysis of the S/N Ratio*

Figure 3 shows an optimal condition for process parameters given by main effect plots for S/N ratios. Based on the analysis, the highest S/N ratio obtained was level-3 for nanoparticle (A), level-2 for surfactant agent (B) and level-1 for the homogenize time (C), respectively. Therefore, the optimal condition for process parameters was obtained to be A3-B2-C1 which are zirconia nanoparticle, surfactant agent SDBS and homogenize time at 10 minutes as shown in Figure 3. As seen in Figure 3, the optimum parameters obtained may be due to the stability of the nanoparticle dispersion in oil and the effectiveness of SDBS agent. SDBS as surfactant agent was found to be very effective to acquire a uniform sizes of nanoparticles. This result is also consistent with that reported by Suleiman *et al.,* [11]. Addition of SDBS surfactants reduces surface tension between solid and liquid and increases particle absorption. In addition, nanoparticles can be dispersed between oil molecules due to the lack of van der Waals power caused by SDBS. Furthermore, the worst S/N ratio obtained was level-2 for nanoparticle (A), level-1 for surfactant agent (B) and level-2 for the homogenize time (C), respectively.

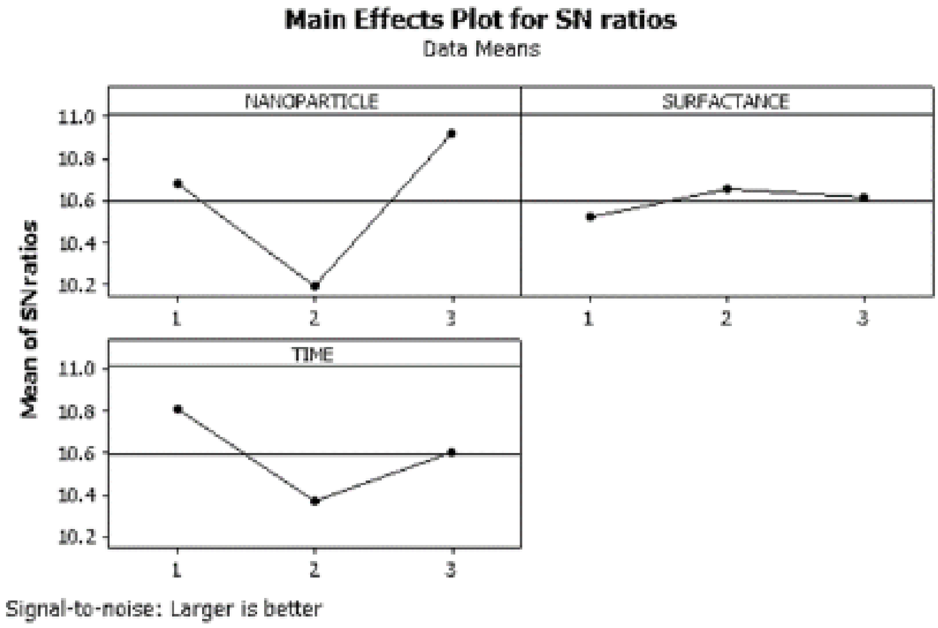


**Fig. 3.** Main effect plot for S/N ratio's effect on nanoparticle stability

Therefore, the worst condition for process parameters was obtained to be A2-B1-C2 which is alumina nanoparticle, surfactant agent Oleic acid and homogenize time at 20 minutes. The result showed clearly that the nanoparticles with surfactant oleic acid were not well dispersed in the oil may be due to the influence of shorted homogenizing time. Mahbubul *et al.,* [12] also agreed that the ultrasonication period of fewer than 30 minutes is insufficient to provide dispersing force to overcome the agglomeration of nanoparticles and nanoparticles remain in clustered conditions. This phenomenon may cause the absorption of oleic acid as a surfactant in nanofluid cannot survive and is not applicable for prolonged stability.

### *3.2 Effect of Difference Surfactant Agent and Time on the Graphite Nanoparticle Stability*

The photographs SAE 15W 40 on Graphite nanoparticle with three different Surfactant Agent shows in Table 3. Dark colours of graphite make it difficult to observe and evaluate the sedimentation of Nano-oils. This is due to the nature of the graphite that contains carbon. Therefore, Nano oil samples appear to be quite dark or black from the first day of the Nano-oil produced and up to 2 months observation which quite difficult to compare for qualitative analysis. Hwang *et al.,* [13] expressing a very concentrated nanofluid or dark colour is unsuitable for using

UV-vis spectrophotometers because high concentrated nanofluids leading to high absorption light and decrease scattered light intensity that consequences reducing the quality of data.

From the UV-vis spectrophotometer analysis, absorbance values clearly indicate the occurrence of sedimentation processes in Nano oil on the second day after preparation. After 56 days, it was clear evidence that Nano-oil with oleic acid agents have better oil properties than Nano-oils with SDBS and sodium chloride agents.

**Table 3**
Picture of SAE 15W 40 on Graphite with three Different Surfactant Agent

| Picture of SAE 15W 40 + Graphite + Surfactant Agent | | | | | | |
|---|---|---|---|---|---|---|
| DAY | Oleic Acid (10 minute) | abs | SDBS (20 minute) | abs | Sodium Chloride (30 minute) | abs |
| 01 |  | 3.9999 |  | 3.9999 |  | 3.9999 |
| 02 |  | 3.8402 |  | 3.5194 |  | 3.5194 |
| 03 |  | 3.6898 |  | 3.4270 |  | 3.3917 |
| 04 |  | 3.5649 |  | 3.3345 |  | 3.2639 |
| 07 (Week 1) |  | 3.5291 |  | 3.2977 |  | 3.2412 |
| 14 (Week 2) |  | 3.5085 |  | 3.1980 |  | 3.1980 |
| 21 (Week 3) |  | 3.4803 |  | 3.1203 |  | 3.1203 |
| 28 (1 Month) |  | 3.4597 |  | 3.0425 |  | 3.0425 |
| 56 (2 Month) |  | 3.2421 |  | 2.9428 |  | 2.9993 |

Figure 4 shows the image and line graph of graphite nanoparticle blend with three types of surfactant agent which are oleic acid, SDBS, and sodium chloride. Based on the line graph, it shows a drastic decreasing trend for the first week for all samples. It gives information that sedimentation process occurred and nanoparticle starts to fall in the bottom of the bottle.

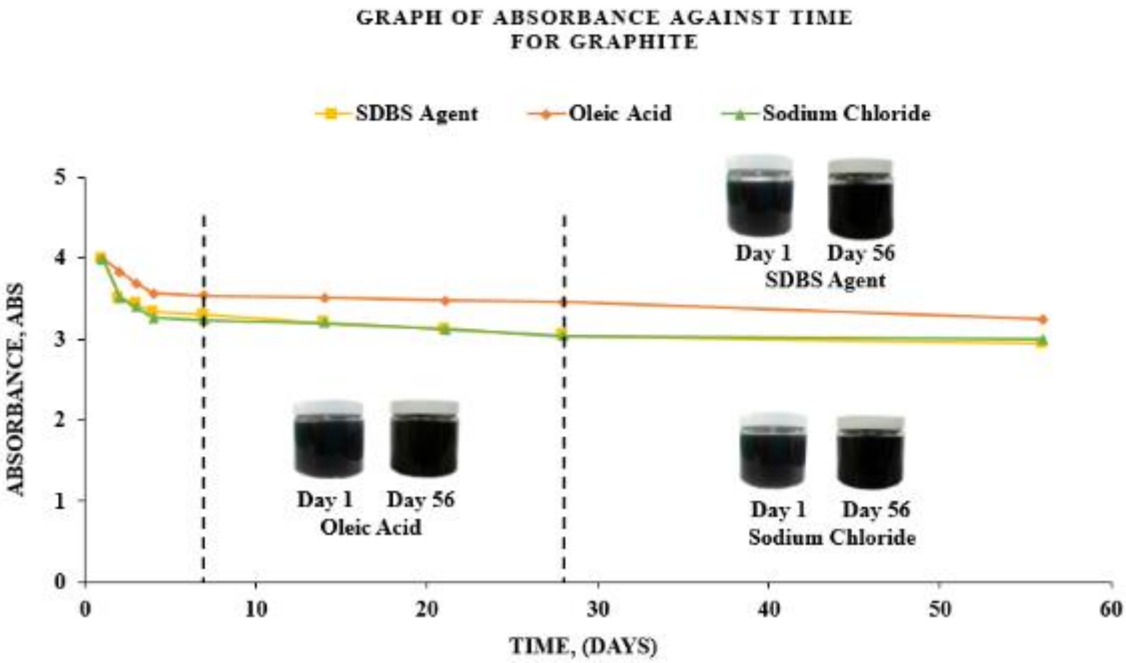


**Fig. 4.** Picture and graph of SAE 15W 40 for Stability of Graphite Nanoparticle with Oleic Acid, SDBS and Sodium Chloride

### *3.3 Effect of Difference Surfactant Agent and Time on the Alumina Nanoparticle Stability*

Table 4 showed the photographs of SAE 15W 40 on Alumina nanoparticle with three different surfactant agents that were taken after the Nano-oil was kept at room temperature. Based on the analysis of the images, alumina nanoparticles do not show any significant changes. In addition, the formation of the bottom of the bottle can be seen with a coarse eye on oleic acid sample agent and sodium chloride agent with alumina nanoparticle on the second day and continued until the period of 2 months.

SDBS agent with Nano-oil exhibit higher absorbance reading from the first day after preparation and last for 2 months. This showed that the addition of SDBS agents has provided dispersion stability on the Nano-oil. This addition of surfactants also may increase the stability of nanoparticles in aqueous suspension [3]. The spread of surface active agents has been used to alter hydrophobic substances to allow dispersion in aqueous solution [13].

The absorption value of oleic acid agent with Nano-oil indicates low readings compared to sodium chloride agent from the first day period of 2 months. Based on the observations it was found that there was a layer formed on the bottom of the bottle after 56 days. This phenomenon believed due to the attractive force between the oleic acid and nanoparticle which produces high surface energy and makes the suspension unstable. Therefore, it causes an aggregation and sedimentation which result in the decline in suspension characteristics. Even though the sample of sodium chloride also showed a significant layer formation at the bottom of the bottle after 2 months, the absorbance value shows the sample in more stable and this proves that the surfactant agent is well-collaborated in Nano-oil. Figure 5 shows the absorbance trend for all Nano oil sample with three surfactant agents.

**Table 4**
Picture of SAE 15W 40 on Alumina with three Different Surfactant Agent

| Picture of SAE 15W 40 + Alumina + Surfactant Agent | | | | | | |
|---|---|---|---|---|---|---|
| DAY | Oleic Acid (20 minute) | abs | SDBS (30 minute) | abs | Sodium Chloride (10 minute) | abs |
| 01 | | 3.1620 | | 3.6277 | | 3.6277 |
| 02 | | 3.1575 | | 3.5536 | | 3.5085 |
| 03 | | 3.2189 | | 3.4830 | | 3.4830 |
| 04 | | 3.1174 | | 3.4072 | | 3.4718 |
| 07 (Week 1) | | 2.9963 | | 3.3391 | | 3.2412 |
| 14 (Week 2) | | 2.9435 | | 3.2710 | | 3.0812 |
| 21 (Week 3) | | 2.8906 | | 3.2598 | | 3.0769 |
| 28 (1 Month) | | 2.7637 | | 3.2485 | | 3.0726 |
| 56 (2 Month) | | 2.7618 | | 3.0277 | | 2.9391 |

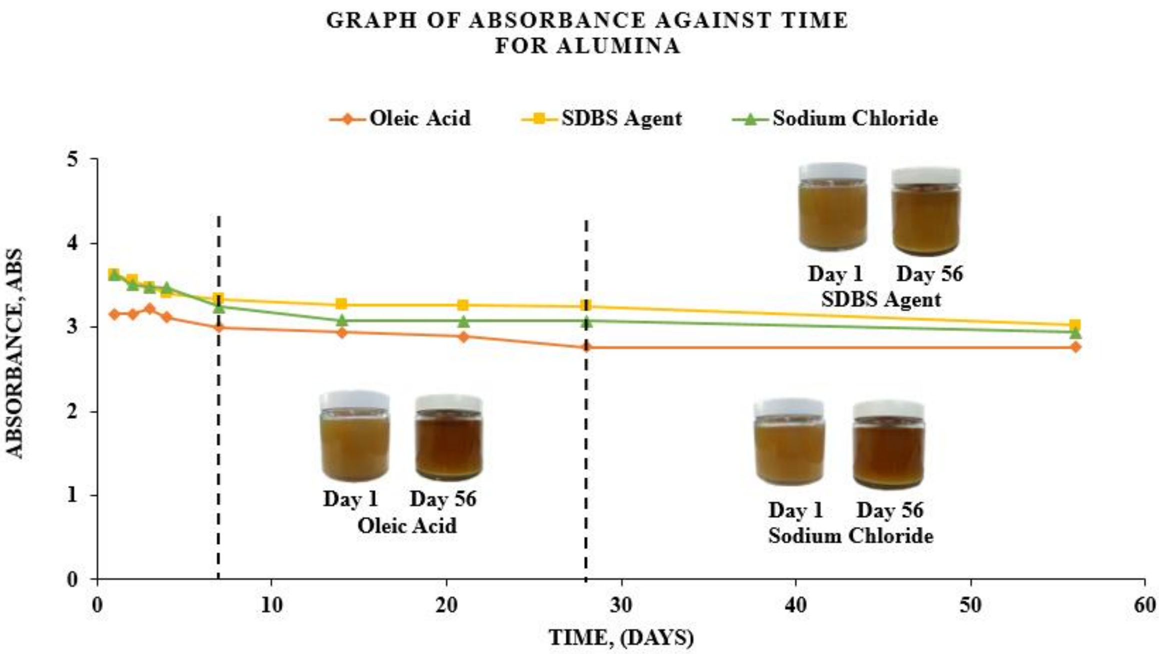


**Fig. 5.** Picture and graph of SAE 15W 40 for Stability of Alumina Nanoparticle with Oleic Acid, SDBS and Sodium Chloride

### *3.4 Effect of Difference Surfactant Agent and Time on the Zirconia Nanoparticle Stability*

Table 5 shows the photographs of SAE 15W 40 on Zirconia nanoparticle with three different surfactant agents. Based on the analysis of the images, zirconia nanoparticles do not show any significant changes in colour. All zirconia samples with three different surfactant agent's show a bright colour and some changes can be seen in the first week after the preparation of the sample. An oleic acid agent with Nano-oil shows the formation of yellowish-brown layers over the surface of Nano-oil samples while samples containing SDBS and sodium chloride agents show the formation of a layer under the bottle. The formation continues over a period of 2 months. From the absorbance value, all surfactant agent sample with Nano-oil exhibit higher reading from the first day after preparation until the last testing after 56 days. Figure 6 shows less difference in absorbance between these three samples.

**Table 5**
Picture of SAE 15W 40 on Zirconia with three Different Surfactant Agent

| Picture of SAE 15W 40 + Zirconia + Surfactant Agent | | | | | | |
|---|---|---|---|---|---|---|
| DAY | Oleic Acid (30 minute) | abs | SDBS (10 minute) | abs | Sodium Chloride (20 minute) | abs |
| 01 |  | 3.9580 |  | 3.9779 |  | 3.9581 |
| 02 |  | 3.7792 |  | 3.7808 |  | 3.7449 |
| 03 |  | 3.6721 |  | 3.7404 |  | 3.7013 |
| 04 |  | 3.5649 |  | 3.5546 |  | 3.6430 |
| 07 (Week 1) |  | 3.2977 |  | 3.4738 |  | 3.5847 |
| 14 (Week 2) |  | 3.2597 |  | 3.3477 |  | 3.5085 |
| 21 (Week 3) |  | 3.2216 |  | 3.2216 |  | 3.3975 |
| 28 (1 Month) |  | 3.2225 |  | 3.1742 |  | 3.2186 |
| 56 (2 Month) |  | 3.0468 |  | 2.9219 |  | 3.0450 |

This indicates that the sedimentation process occurs very little on zirconia nanoparticles. Sedimentation is the tendency of nanoparticles to settle from base fluids in which they are scattered and ultimately rest with external forces [14]. Furthermore, sedimentation occurs because nanoparticles have high surface energy, aggregation, and clusters that increase Van der Waals's attraction on the surface of the particles. This attractive force tends to attract other particles to form clusters known as agglomeration. The formation of agglomerates has a negative effect on the stability of Nano-oil suspension [7]. After that, the particles will clog and formed a layer in the bottom of the bottle.

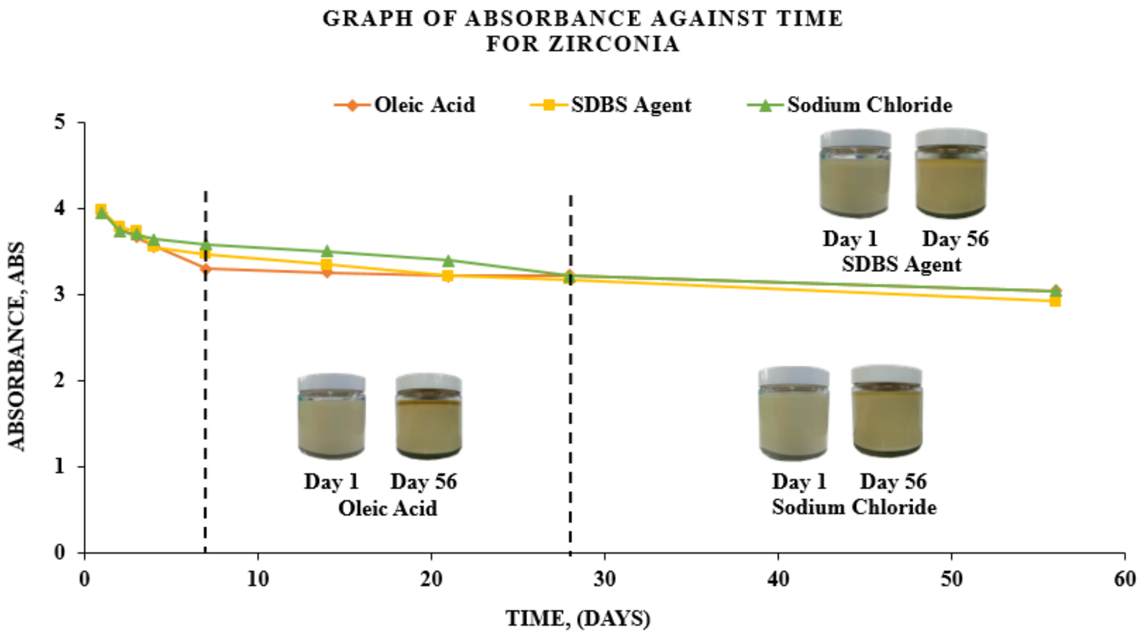


**Fig. 6.** Picture and graph of SAE 15W 40 for Stability of Zirconia Nanoparticle with Oleic Acid, SDBS and Sodium Chloride

## 4. Conclusions

In conclusion, Taguchi analysis showed the zirconia nanoparticle with SDBS agent and 10 minutes homogenize time is the most optimum parameter compared with the other samples in determining the stability of Nano-oil. This result was consistent with the qualitative and quantitative analysis obtained from the photograph and UV-Vis spectrometer test.

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