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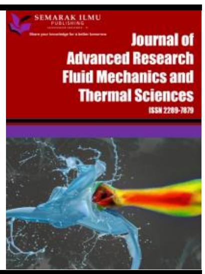

Characterization and Stability of ZrO_2-SiO_2 Nanofluids from Local Minerals Indonesia as Green Nanofluids to Application Radiator Cooling System

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ABSTRACT

In the last few years, there has not been much research on green nanofluids that come from material resources such as parts of palm oil and natural zircon sand. Green nanofluids need to be developed to improve heat transfer performance. The characterization of nanoparticles made from the synthesis process and the stability of ZrO_2-SiO_2 Nanofluids-W/EG, different volume concentrations were investigated. The experiments carried out were characterization of nanoparticles using SEM and FTIR, the stability of green nanofluid was investigated for a volume concentration of 0.1-0.3%, using UV-Vis, Zeta potential and sedimentation observation. The results show that the size of Nano-Zircon particles of 32.984-38.465 nm and Silicate nanoparticles with of 44.002-50.444 nm. The stability of ZrO_2-SiO_2 Nanofluids-W/EG made with the UV-Vis method is stable up to 30 days after preparation with a sonication time of 2 hours with a value of 70-80%. The zeta potential evaluation performed for green ZrO_2-SiO_2 Nanofluids-W/EG obtained a value of 45.37 mV with good stability classification. Sedimentation from this visual observation obtained the absence of agglomeration after 30 days.

1. Introduction

Zircon sand is one of natural mineral widely distributed in Australia, South Africa, India, Mozambique, America, China, Indonesia and other countries used in many industries [1]. It can be derived into nano material which has been extensively gained interest of researcher because of its unique properties deals with its size of 1 – 100 nm. Zircon sand can be synthesized as zirconia nanoparticles (ZrO_2), zircon nano powder ($ZrSiO_4$) and silica nano powder [2]. The zirconia nano particles have been attracted researcher either for its synthesize and for applications due to its remarkable properties in toughness, strength and hardness [3].

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It has led to a wide range of uses in ceramic glass due to its superior mechanical, heat and chemical resistance factors due to inert surface footprint increasing and unsaturated surface area coordinates, fuel cell materials due to its good conductivity properties, nanofluids for heat transfer applications due to their heat resistance and increased thermal conductivity, absorbents due to their high surface area, teeth due to their colourlessness, biocompatible, excellent strength and plasticity, and others biomedicine applications for composite materials [4-11].

Indonesia is the largest producer of palm oil in the world. Indonesia in 2020 produced 44.8 million tons of palm oil with a total area of palm oil plantations of 14.457 million hectares [10,11]. In the palm oil processing process, palm oil is produced as the main product and waste is also produced as a by-product. Waste produced in the form of liquid waste and solid waste. Liquid waste occurs in the processing of fresh fruit bunches, while solid waste is the first waste produced from palm oil processing [12]. A material that has high porosity and a large surface area is silica (SiO_2). There are many uses in medicines, fillers and catalysts [13]. To produce SiO_2 in the industrial environment, sodium silicate is used, where the melting of quartz sand and sodium carbonate requires a lot of energy [14]. Extensive studies have attempted to extract silica from various agricultural waste products such as rice husk, corn and coconut cob ash and palm oil waste [15,16]. Studies on TKKS (empty oil palm bunches) as a source of silica still need to be explored. Recently, nano-silica has been used as a fluorophore carrier for the controlled release of drug molecules as an antiseptic agent and as a biosensor [17-20]. Different silanol groups in nanosilica can change the crystalline behavior of the polymer matrix, increase the hydrogen bonding effect, and change the fluorescence properties. The most important property of polymers is their heat resistance; this affects spectral stability, mechanical properties, age, and other material properties [21,22].

Nanofluids are liquid suspensions containing metal or non-metal nanoparticles with a specific size (1-100 nm) that are dispersed into the base fluid. In 1995, the concept of nanofluids was first introduced by Choi and Eastman [23]. This new method is proven to increase heat transfer by increasing the thermo-physical properties of nanofluids. Nanofluids are known for applications in heating and cooling processes. The main cooling process is an important part of industrial applications such as power plants, chemical processes, microelectronics, transportation, and automotive cooling systems [24-28]. The influence of solid particles can change the thermal properties of the working fluid. Several researchers have conducted research on thermal conductivity, dynamic viscosity and stability of nanofluids produced [29-34]. Green nanofluids or bio-nanofluids, or environmentally friendly nanofluids come from several sources of natural materials [35,36]. Highly colloidally stable green nanofluids as alternative working fluids are very important for their effective use in different thermal systems [37]. Based on previous research, there is very little research on the development of green nanofluids in the world.

The nanofluid preparation method is important to minimize the agglomeration of nanoparticles so as to increase stability. The most common processes used in the manufacture of nanofluids are one-step and two-step methods. The one-step method is the process of synthesizing nanoparticles and simultaneously dispersing them in an alkaline liquid. Another nanofluid preparation method is known as the two-step method. There are two processes in this method, namely (i) synthesis of nanoparticles in powder form (ii) dispersion of nanoparticles into the base liquid to form a stable and homogeneous solution [38,39]. The stability of the nanofluid and the size of the nanoclusters affect the thermal conductivity parameters [32,40-43]. Nanofluid stability is defined as the resistance of nanoparticles to aggregation. Factors such as Van der Waals attraction cause aggregation, which results in the formation of nanoclusters contained in nanofluids [44-46]. The formation of nanoclusters depends on their size, which causes the sedimentation of particles in the nanofluid. The stability of nanofluids has a significant influence. There are two testing methods, quantitative and qualitative. UV-Vis spectrophotometric measurements have been used to quantitatively characterize the stability of nanoparticles from dispersions [42,47]. Can be applied to all base fluids, while zeta potential analysis has the limitation of base fluid viscosity [48]. In other literature studies, different techniques are used to stabilize nanoparticles in liquids such as ultra-sonication, additional surfactants, surface modifiers and pH adjustment [49,50]. For qualitative testing by visually seeing the sedimentation from the nanofluid made [51-53].

The characterization and stability of ZrO_2-SiO_2 nanofluid as a green nanofluid is very important to study. This aims to determine preparations with nanoparticles from local natural resources in Indonesia. In order to synthesize nanoparticles into green nanofluids that are used for cooling system applications. The stability and behaviour of the nanofluids as well as the factors that affect the stability of the nanofluids that has been made. Based on the information obtained by the authors, studies of nanoparticle-influenced preparations and stability of green nanofluids are limited to the literature. Based on these problems, this research was conducted by emphasizing the stability effect of Nano-zircon (ZrO_2) from zircon sand in Indonesia and Nano-silicate (SiO_2) from empty palm oil shells (EPS) on the stability of green nanofluids for application to radiator cooling systems.

2. Methodology

2.1 Preparation of Green Nanofluids from ZrO_2 and SiO_2 Nanoparticles

The production of green nanofluids involves mono nanofluid, namely ZrO_2 nanoparticles or Nano-Zircon and SiO_2 nanoparticles or Nano-Silicate, each of which comes from local natural materials found in Indonesia. Both are mixed and dispersed in a base fluid mixed with water/EG (60:40) ratio. Nano-Zircon is made through the Caustic Fusion method and Nano-Silicate is obtained from Empty Palm Oil Shell (EPS) which is made using the Ultra-Sonication Method. The sizes of Nano-Zircon and Nano-Silicate nanoparticles are 32 nm and 44 nm, respectively. The characteristics of Nano-Zircon and Nano-Silicate are given in Table 1.

Table 1
Properties of ZrO_2 and SiO_2 nanoparticles from synthesis process

Properties	ZrO_2	SiO_2
Molecular mass, g mol^{-1}	231.891	60.08
Average particle diameter, nm	32	44
Density, kg m^{-3}	5680	2220
Thermal conductivity, W m^{-1} K^{-1}	2.8	1.4
Specific heat, J kg^{-1} K^{-1}	418	745

A two-step method is used for the preparation of green nanofluids. Green nanofluid is prepared by mixing Nano-Zircon and Nano-Silicate (50:50) ratio with water/EG mixture (60:40), and sonication. The production of green nanofluid begins with the calculation of the required volume according to its concentration. In this research, green nanofluids were made with volume concentrations of 0.1, 0.2, and 0.3%. The nanofluid was first prepared at the highest concentration, 0.3% and then diluted to a lower concentration. Green nanofluid preparation process based on Figure 1.

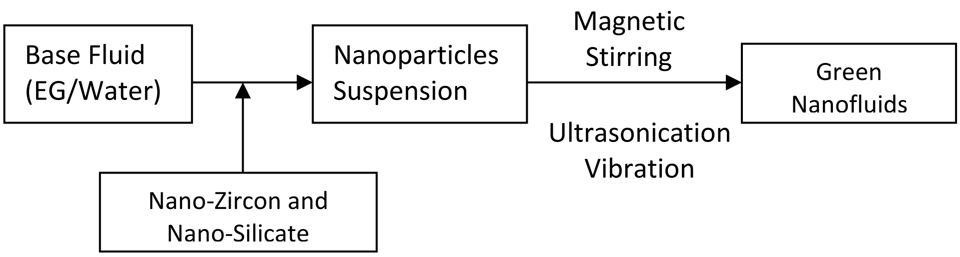


Fig. 1. The preparation of green nanofluids with ZrO_2 and SiO_2 nanoparticles

ZrO_2 Nanoparticles or Nano-Zircon and SiO_2 Nanoparticles or Nano-Silicate are available in powder form with a weight concentration of 22% for ZrO_2 and 25% for SiO_2. Eq. (1) is used to convert weight concentration into volume concentration [36,47]. Dilution from a higher volume concentration to a lower volume concentration using Eq. (2) by adding the base fluid (*ΔV*) [51].

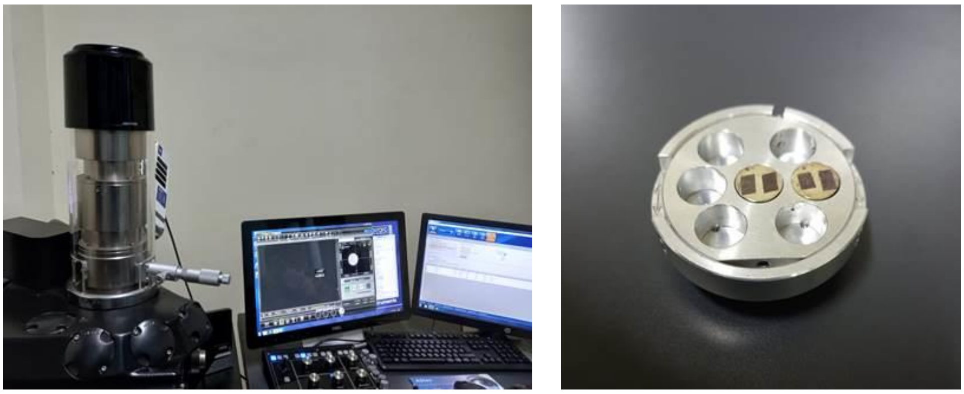
$$\phi = \frac{\omega \rho_w}{\frac{\omega}{100}\rho_w + \left(1 - \frac{\omega}{100}\right)\rho_p} \tag{1}$$

$$\Delta V = (V_2 - V_1) = V_1\left(\frac{\phi_1}{\phi_2} - 1\right) \tag{2}$$

Nano-Zircon and Nano-Silica are mixed together with a mixture of Water/EG with a ratio of 60:40 to become green nanofluids. A total volume of 100 mL was prepared for nanofluids concentration. The nanofluids solution was mixed together using a magnetic stirrer for 15 minutes. The solution is then sonicated for 2 hours using ultrasonic immersion to increase stability.

2.2 Characterization of Nanoparticles with SEM and FTIR

Characterization components of the two nanoparticles, one of which can study the morphology of Nano-Zircon and Nano-silicate, used a scanning electron microscope (SEM) series JSM-IT300 at 20.0 kV. Morphological images of Nano-Zircon and Nano-Silicate captured at 5,000X magnification. Due to the unique chamber configuration, the sample is mounted on the sample holder and imaged directly without pretreatment, even when hydrated. ImageJ software was used to calculate the diameter based on SEM images. The SEM analysis was carried out by the National Research and Innovation Agency (Figure 2). This characterization testing process is done in exactly the same way as in the previous research [26-28].



(a) (b)

Fig. 2. (a) Scanning electron microscope (SEM) series JSM-IT300, (b) samples of Nano-Zircon and Nano-Silicate

Further, the FTIR Spectra of Zircon and Silicate nanoparticles were collected using a Fourier Transform Infrared Spectrometer (FTIR) Carry 630 FTIR (Figure 3). Samples were measured using KBr pellets in the range of 4000-400 cm-1. The same method was used by previous research, to determine and identify the characterization of prepared Zircon and Silicate nanoparticles [29-33].

Fig. 3. Carry 630 FTIR for check sample of Zircon and Silicate nanoparticles

2.3 Stability of Green Nanofluids

Investigation of the stability of nanofluids in this research is done through the measurement of UV-Vis spectrophotometer, Zeta potential. UV-Vis, and visual observation was done for 30 days (720 hours) with varying sonication times. The wavelength of the UV-Vis spectrophotometer is set at 850 nm following the research of Azmi *et al.,* [29] and Hamid *et al.,* [54]. UV-Vis measures the absorption and light scattering intensity of the nanofluid by comparing the intensity level with the base fluid (Figure 4). The absorbance ratio of the sonication time is different during the sedimentation time at a constant wavelength (λ) of 850 nm. Stability evaluation with UV-Vis was also used by previous research [37,42,44,45]. Figure 5 illustrates measurement with zeta potential performed using Zetasizer Nano ZS (Malvern Instruments Ltd., GB) [54,55]. Sedimentation with visual observation is done for up to 30 days. Nanofluids will be considered stable when its concentration is constant. Previously, the same method to observe the visual sedimentation of nanofluids was prepared by Choudhary *et al.,* [35].

Fig. 4. Stability measurement of ZrO_2-SiO_2 nanofluids with UV-Vis spectrophotometer

Fig. 5. Zeta potential measurement of ZrO_2-SiO_2 nanofluids with Zetasizer Nano ZS

3. Results

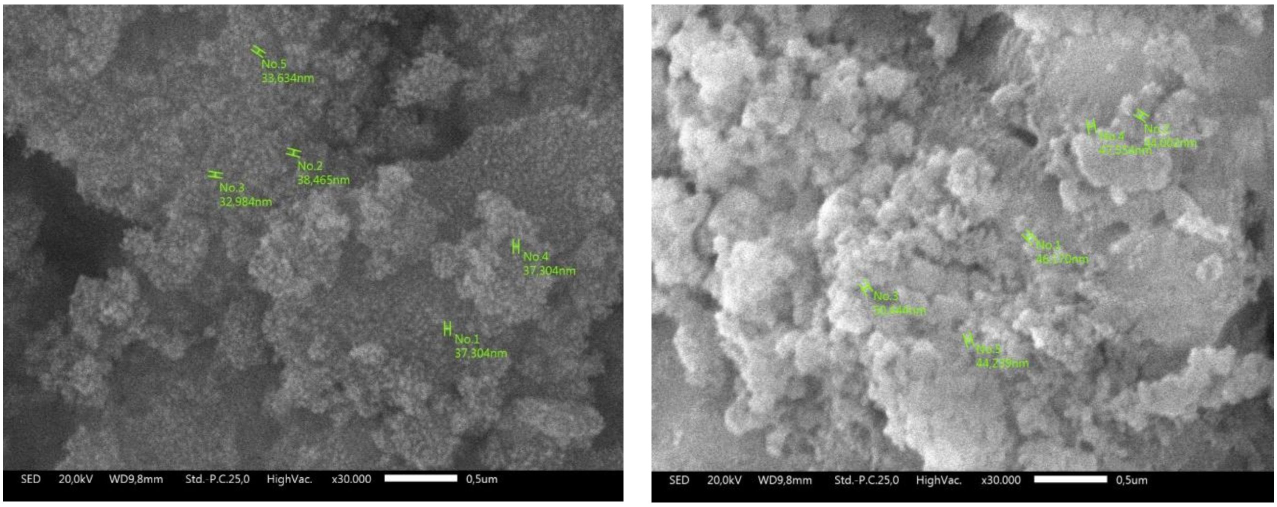
3.1 Characterization of ZrO_2 and SiO_2 Nanoparticles

Nano-Zircon and Nano-Silicate in the form of powder obtained from the synthesis process based on the temperature of 550 °C and 50 °C, respectively, can be seen in Figure 6. Figure 6(a) presented the powder form and colour which are almost the same and visual appearance of Zircon nanoparticles has powder and bright colour. Similarly, the distribution of particles in the form of powder and light colour for silicate nanoparticles is shown in Figure 6(b). Visually, this result illustrates a close match with several studies to obtain Zircon Nanoparticles and Silicate Nanoparticles [35,39,42].

(a) (b)

Fig. 6. Visualisation of (a) Zircon nanoparticles and (b) Silicate nanoparticles

Figure 7 illustrates the microstructure analysis of Nano-Zircon and Nano-Silicate using a scanning electron microscope (SEM). SEM images of Nano-Zircon treated with a temperature of 550 °C and Nano-Silicate at a temperature of 50°C, respectively. It can be seen that the size of the Nano-Zircon particles is regular at a temperature of 550°C with a size variation of 32.984-38.465 nm (Figure 7(a)). However, the size of Silicate nanoparticles looks regular with a variation of 44.002-50.444 nm with the condition of ultrasonification temperature at 50°c, the distribution pattern can be seen in Figure 7(b). Both pictures have shown that the particle size distribution is almost uniform and has formed the expected nanometer size.

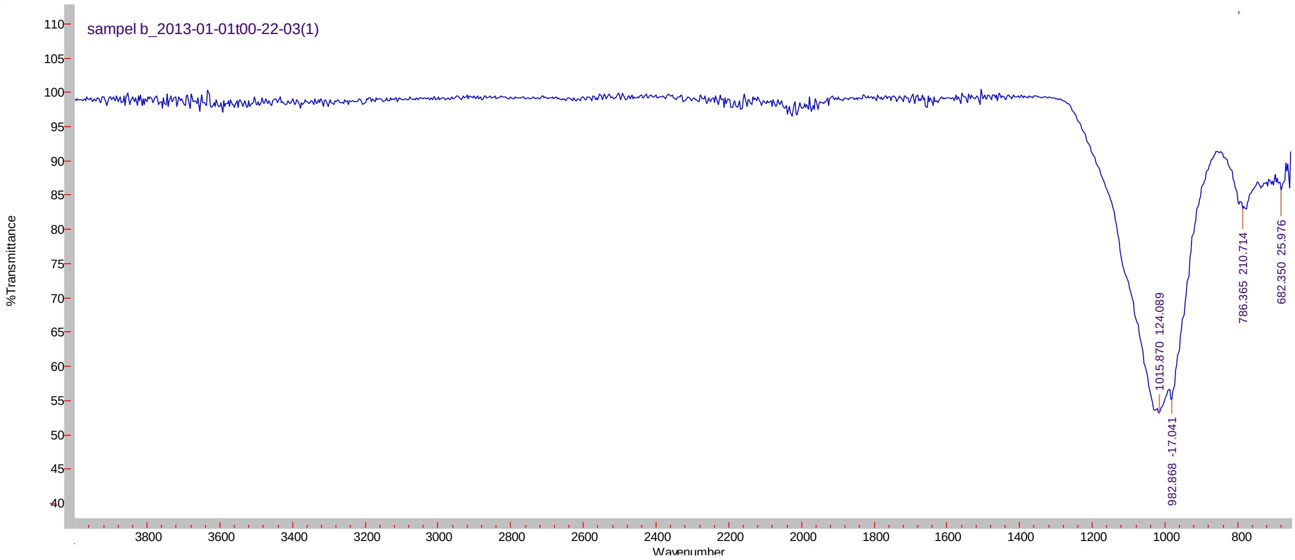


(a) (b)

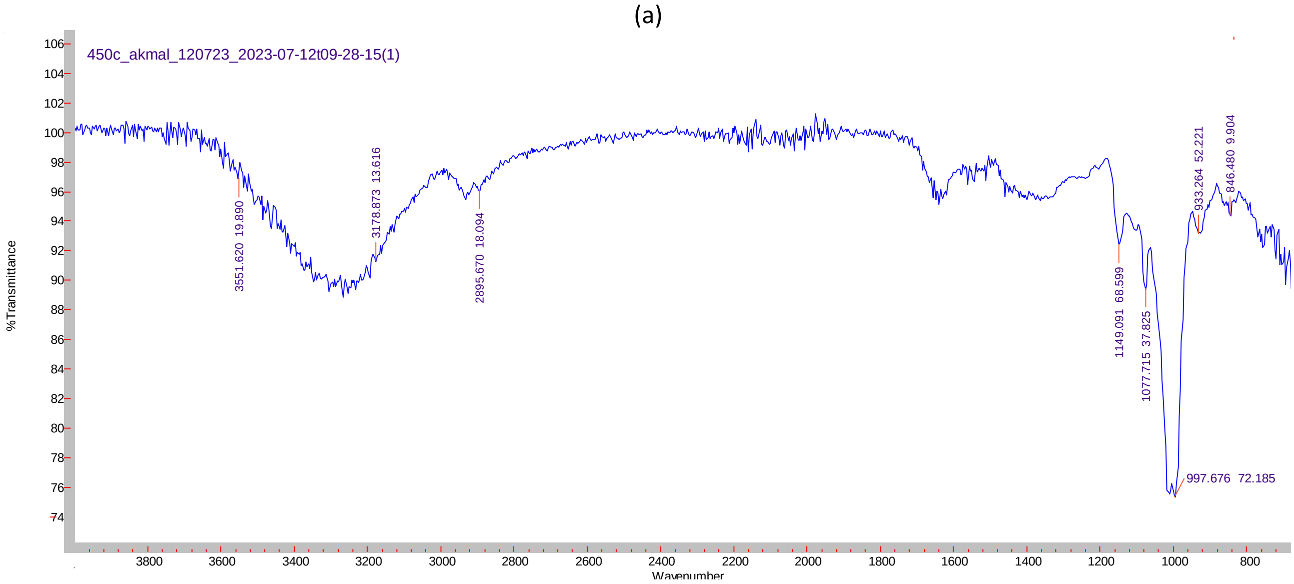
Fig. 7. The SEM image for (a) Nano-Zircon with temperature 550 °C, and (b) Nano-Silicate with temperature 50°C

Fourier Transform Infra-Red (FTIR) characterization using FTIR Carry 630, one type of FTIR device. This characterization aims to determine the functional group of the material. The information obtained from this characterization is the transmittance spectrum and wave number, so that from the FTIR results it can be known the bond or functional group of Nano-Zircon and Nano-Silicate obtained from the synthesis process. Nano-Zircon functional group with a temperature of 550°C is shown in Figure 8(a). FTIR characterization showed the presence of an absorption peak from the sample. These peaks show absorption bands that are characteristic of molecular vibrations at wave numbers at 2258.504 and 680 cm^{-1}. Figure 8(b) shows the absorption cluster peak for molecular in

Nano-Silicate with molecular vibration characteristics at wave number (1027.339 and 781.538) cm^{-1}. In principle, FTIR is used to determine the functional group found in a compound, so it can be used to determine a compound whose content is not yet known. FTIR analysis of nanoparticle samples has similarities with several researchers [27,28,31].



(a)



(b)

Fig. 8. Fourier Transform Infra-Red (FTIR) characterization for (a) Nano-Zircon and (b) Nano-Silicate

3.2 Stability of Green Nanofluids

Absorbance observations for volume concentrations of 0.1, 0.2 and 0.3% are shown in Figure 9. ZrO_2-SiO_2 Nanofluids absorbance increases linearly with increasing volume concentration. This tendency is in accordance with the Beer-Lambert Law, that is, the absorbance value is equal to the concentration. The stability of ZrO_2-SiO_2 nanofluids is further confirmed by the absorbance ratio, A_r and presented in Figure 10.

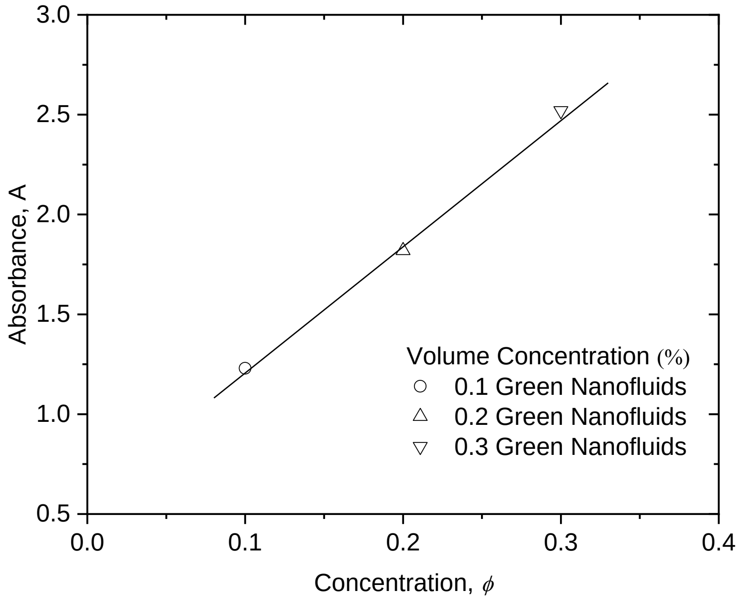


Fig. 9. UV-Vis spectrophotometer linear relation graph between absorbance and green nanofluids concentration

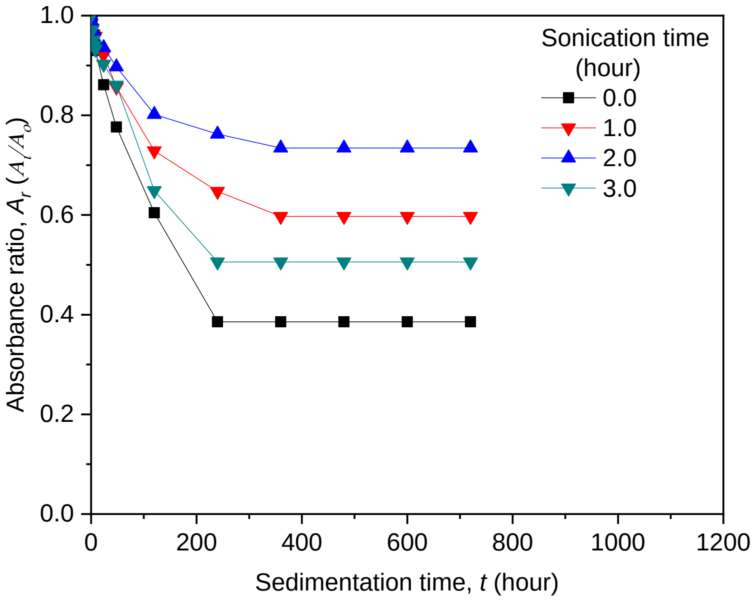


Fig. 10. The absorbance ratio of green nanofluids for different sonication time within 30 day

The absorbance ratio at 0.1% volume concentration of ZrO_2-SiO_2 Nanofluids for 4 different sonication variations. The ideal absorbance ratio is one (100%) which indicates ideal fluid stability. The variation of conditions with sonication for 3 hours started to decrease after 50 hours by 70% and continued to decrease until stable at 30 days (720 hours) by 50%. While the sonication time is 2 hours, it remains at a good concentration ratio value of around 70-80% until 30 days later. The condition of sonication for 1 hour reduced the absorbance ratio by 60%. For the variation of sonication 0 hours or after preparation there is a decrease of 40%. From the picture it can be seen that the sonication time of 2 hours shows the best absorbance ratio compared to other sonication times. Thus, the manufacturing of ZrO_2-SiO_2 Nanofluids in this research uses 2 hours for the sonication process. The absorbance ratio was found to be 70-80% for 30 days.

The absorbance ratio of ZrO_2-SiO_2 Nanofluids in this research is compared with Hwang *et al.,* [36] and shown in Figure 11. Investigation by Hwang *et al.,* [36], using multi-walled carbon nanotubes (MWCNT) in an aqueous alkaline liquid. As shown in the graph, MWCNT nanofluids have poor stability. In this research, Nano-Zircon and Nano-Silicate are used in the EG/Water mixture and use a sonication time of 2 hours. The absorbance ratio was found to be 70-80% for 30 days. This condition is stable.

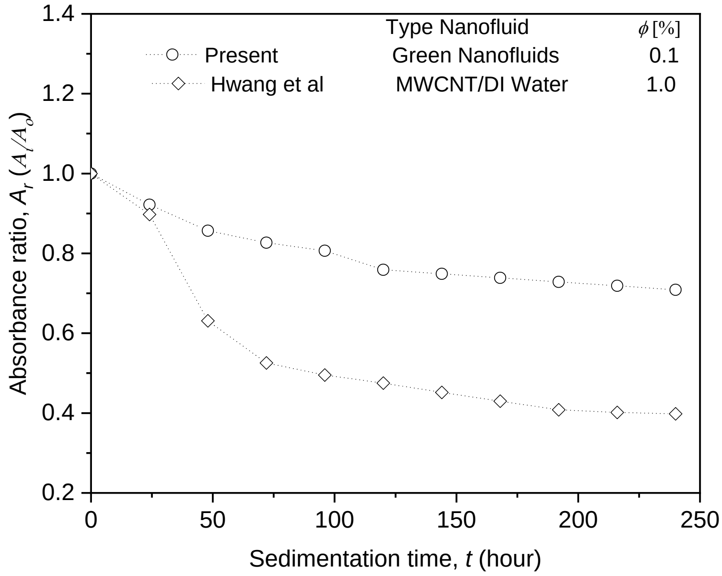


Fig. 11. Comparison of absorbance ratio with sedimentation ratio from literature

The measurement of zeta potential is one of the most important tests to validate the stability quality of nanofluids through the study of electrophoresis behavior [37,56-59]. Zeta potential evaluation is a standard quantitative method for stability evaluation [22,35,60-62]. Usually, the received zeta potential values are summarized in Table 2. Generally, suspensions with measured zeta potential above 30 mV (absolute value) are considered to have good stability [63]. The absolute value of the zeta potential is used to show the agglomeration of SiO_2 nanoparticles in EG/W. The higher the absolute value, the better the particle dispersion, was resulting in better stability.

Table 2
Zeta potential and classification stability [63]

Zeta potential (mV)	Stability
0	Little or no stability
15	Some stability
30	Moderate stability
45	Good stability
60	Very good stability

Zeta potential measurements for ZrO_2-SiO_2 Nanofluids were recorded up to 45.37 mV. The results were then compared with the classification of nanofluid stability based on zeta potential with moderate stability values compiled by Ghadimi *et al.,* [63] as shown in Figure 12. Absolute values above 30 mV are desired for good stability of the ZrO_2-SiO_2 Nanofluids. It is proved that the nanofluid is outside the stable limit of 30 mV. Therefore, evaluation of the zeta potential has confirmed the stability of the ZrO_2-SiO_2 Nanofluids.

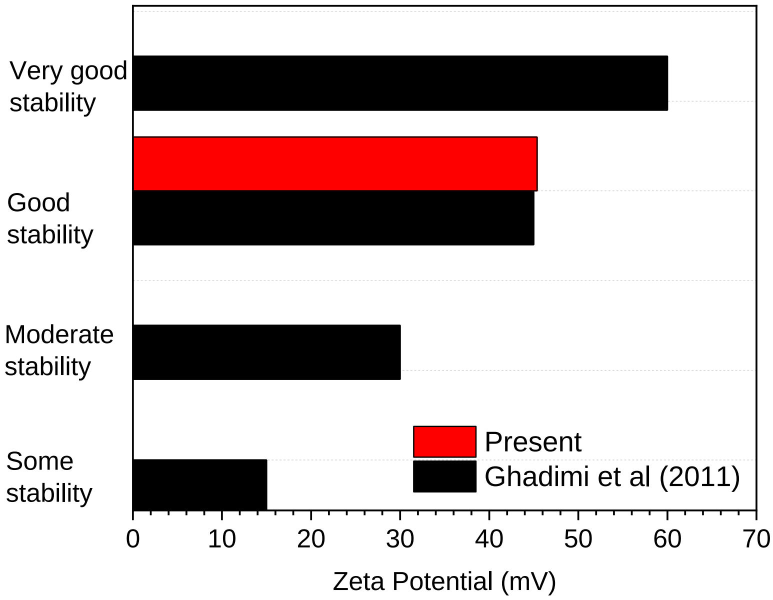


Fig. 12. Zeta potential of ZrO_2-SiO_2 Nanofluids-W/EG

Figure 13 illustrated ZrO_2-SiO_2 Nanofluids-W/EG for volume concentration of 0.1. 0.2, and 0.3%. Images of ZrO_2-SiO_2 Nanofluids-W/EG was taken only after preparation and after 30 days. Based on Figure 13(a) to Figure 13(d), no particle sedimentation was observed after the nanofluid was prepared after 21 days. Agglomeration did not occur in the sample up to 30 days in Figure 13(d).

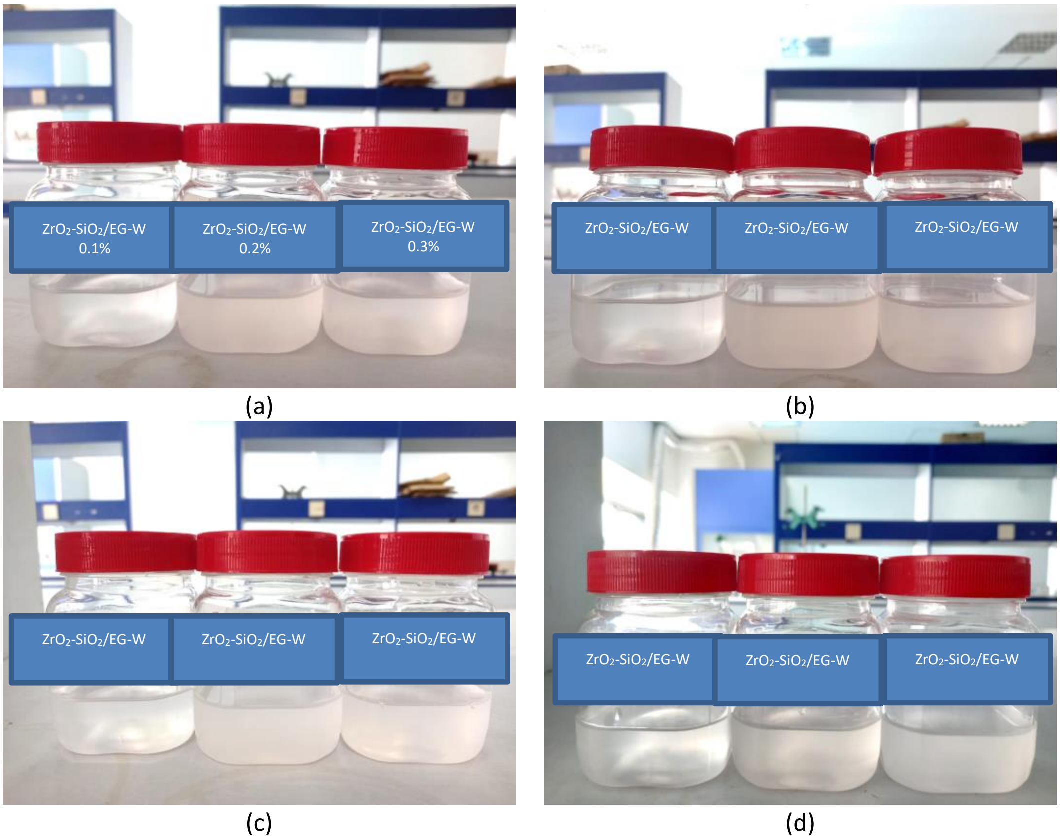


Fig. 13. Sedimentation observation of ZrO_2-SiO_2 Nanofluids: (a) 0 day, (b) 7 days, (c) 21 days, and (d) 30 days

4. Conclusions

In this research, the manufacture of ZrO_2-SiO_2 Nanofluids-W/EG from the results of the synthesis process of Nano-Zircon derived from zircon sand and Nano-Silicate can be made from empty palm oil shell (EPS). Both materials available from local natural resources in Indonesia. Visualization of Nano-Zircon and Nano-Silicate in powder form has a powder and bright colour. The characterization of Nano-Silicate can be seen using SEM (Scanning Electron Microscopy) which is investigated for the Caustic Fusion method at 550°C (Nano-Zircon) and an ultra-sonication temperature of 50 °C for Nano-Silicate. The size of the Nano-Zircon particles of 32.984-38.465 nm and Silicate nanoparticles with a variation of 44.002-50.444 nm. Analysis of the stability of ZrO_2-SiO_2 Nanofluids-W/EG made with the UV-Vis method is stable up to 30 days after preparation with a sonication time of 2 hours. Data comparison of the ratio of concentration to sedimentation for ZrO_2-SiO_2 Nanofluids-W/EG shows that it remains stable with a value of 70-80%. The zeta potential evaluation performed for green ZrO_2-SiO_2 Nanofluids-W/EG obtained a value of 45.37 mV with good stability classification. Sedimentation from this visual observation obtained the absence of agglomeration after 30 days.

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