



Qualitative Stability and Thermal Properties Investigation of TiO₂-EG/W Nanofluids Through Experimental Validation

Investigasi Stabilitas Kualitatif dan Sifat Termal Nanofluida TiO₂-EG/W Melalui Validasi Eksperimental

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Abstract

Over the past two decades, researchers have been extremely interested in developing TiO₂ nanofluids for heat exchanger applications. Therefore, this study evaluates the performance of employing TiO₂ nanofluids, which were prepared using ethylene glycol (EG) and distilled water as the base fluid, then called TiO₂-EG/W. The qualitative stability and thermal conductivity properties were measured through the experimental investigation. XRD and SEM analyses were also carried out to investigate the structures of TiO₂ nanoparticles used in terms of their crystalline and morphological structures. The results showed a positive impact of stability even for 15 days, and after that, the nanoparticles dropped to the sedimentation by about 58%. Then, the highest thermal conductivity at the temperature of 80 °C was increased by about 17.08% compared to the base fluid. Supported by the results of XRD and SEM analysis, respectively, highlight that TiO₂ nanoparticles have a rutile phase with an average crystallite size of 20.23 nm and are small spherical in morphology. This paper also provided the challenge and future perspective of TiO₂ nanofluid to appear as an innovation for the development of TiO₂ nanofluid in the further studies of heat exchanger applications.

Keywords: stability, thermal conductivity, nanofluids, TiO₂-EG/W.

SDGs:



Abstrak

Selama dua dekade terakhir, para peneliti sangat tertarik untuk mengembangkan nanofluida TiO₂ untuk aplikasi penukar panas. Oleh karena itu, penelitian ini mengevaluasi kinerja penggunaan nanofluida TiO₂ yang dibuat menggunakan etilen glikol (EG) dan air suling sebagai cairan dasar, yang kemudian disebut TiO₂-EG/W. Stabilitas kualitatif dan sifat konduktivitas termal diukur melalui penyelidikan eksperimental. Analisis XRD dan SEM juga dilakukan untuk menyelidiki struktur nanopartikel TiO₂ yang digunakan ditinjau dari struktur kristal dan morfologinya. Hasilnya menunjukkan dampak positif stabilitas bahkan selama 15 hari, dan setelah itu nanopartikel turun ke sedimentasi sekitar 58%. Kemudian konduktivitas termal tertinggi pada suhu 80 °C meningkat sekitar 17,08% dibandingkan dengan fluida dasar. Didukung oleh hasil analisis XRD dan SEM, masing-masing menyoroti bahwa nanopartikel TiO₂ memiliki fase rutil dengan ukuran kristal rata-rata 20,23 nm dan morfologinya berbentuk bola kecil. Tulisan ini juga memberikan tantangan dan perspektif masa depan nanofluida TiO₂ untuk tampil sebagai inovasi pengembangan nanofluida TiO₂ pada penelitian lebih lanjut pada aplikasi penukar panas di masa depan.

Kata Kunci: stabilitas, termal konduktivitas, nanofluida, TiO₂-EG/W.

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1. INTRODUCTION

All the industrial sectors in the world use heat exchanger tools for their operation, such as the automotive industry, petroleum industry, electronics, renewable energy, and so on (Gupta, Verma and Yadav, 2022). Therefore, heat exchanger plays an important role in the industrial sector to run an operating system and influence the effectiveness of its conversion energy systems (Tuncer *et al.*, 2023). Heat exchangers have utilized the heat transfer concept in their principal work to provide the thermal energy flow using the fluid at a certain range of temperatures (Pordanjani *et al.*, 2019). However, the evolution of technology caused the performance of heat exchangers to decrease due to the need for devices that can work more optimally. Some recent findings confirmed that the heat exchanger device is undesirable due to its limited thermal properties. Thermal properties is play an important role for heat transfer performance (Kristiawan, Kamal and Yanuar, 2016). For now, the researchers have tried to develop this study to obtain a higher coefficient heat transfer to improve performance (Zhong, Zhong and Wen, 2020). Muneeshwaran *et al.*, introduced an alternative solution to this problem by improving the thermal properties of fluids in the heat exchanger system and no doubt conducted due to its simple, low cost, and easy preparation (Muneeshwaran *et al.*, 2021). Then, in 2022, Louis *et al.* stated that nanofluids are the best fluids for improving the heat exchanger (Louis *et al.*, 2022).

A long time ago, Choi and Eastman (1995) introduced nanofluid as a promising fluid for heat exchanger application (Pavía *et al.*, 2021). Nanofluid is a homogeneous mixture of nanoparticles and conventional fluid (base fluid). These conventional fluids include water, oil, or ethylene glycol (EG) (Okonkwo *et al.*, 2021). The nanofluid offers more advantages, such as a higher heat transfer rate and greater stability, to enhance the performance of heat exchanger devices (Kumar and Sarviya, 2021). Nanofluids also have a large potential to applied in electronic devices, automotive, and other sectors which coexist with the human life (Kristiawan *et al.*, 2022). For that reason, the study of nanofluids

under different conditions and modifications has become a new trend among researchers. Those studies are focused on determining the right compilers of nanofluids that have a positive impact on the performances of heat exchanger devices. In other words, the nanoparticles and base fluid used in the nanofluids fabrication should be selected appropriately. Nfawa *et al.*, in 2021., claim that metal oxide is a promising nanoparticle for nanofluid due to the low prices and their abundance (Nfawa *et al.*, 2021). Another advantage of metal oxide nanoparticles for nanofluid applications is that there is no clogging in the heat exchanger and better homogeneity (Dharmakkan *et al.*, 2023). Several metal oxides that have been fabricated to the nanofluid are MgO (Dehaj and Mohiabadi, 2019), CuO (Shang *et al.*, 2022), ZrO₂ (Yushuang Huang *et al.*, 2022), ZnO (Sharma *et al.*, 2022), Al₂O₃ (Bai *et al.*, 2023), SiO₂ (Zou *et al.*, 2022), and TiO₂ (Arifin *et al.*, 2022). Among these nanoparticles, TiO₂ has attracted more attention due to its characteristics. TiO₂ has good thermal properties and is easily dissolved. It is difficult to descend to the sedimentation (Arsana *et al.*, 2022). Up to now, several studies about the utilization of TiO₂ nanofluid for heat exchanger applications have been reported. In 2022, Thianpong *et al.* revealed that the TiO₂ working fluid significantly improved the heat transfer properties and their device's overall performance by up to 5.0% (Thianpong *et al.*, 2022). Then, the right choice of base fluid also plays an important role in the nanofluid performance. According to Dosodia *et al.*, base fluids used in the fabrication of nanofluid should be controlled to obtain high performance, e.g., a mixture of several base fluids such as EG-water (Dosodia *et al.*, 2022). A mixture of EG-water is believed to increase the performance of the heat exchanger device by up to 70% compared to the other base fluids (Zheng *et al.*, 2020). Rashidi *et al.* also reported that study in 2023. their work varied the sample of EG-water base fluid in several ratios. Then, it was found that all EG-water ratios positively impacted the heat transfer properties and proposed a great potential to study further the significant increase in the performance of heat exchanger applications (Rashidi *et al.*, 2023). The similar studies have

reported by Thiyana that found EG-water mixture has significantly improved the heat transfer performance (Thiyana et al., 2023).

Based on these explanations, this study brings up the motivation, innovation, and aim to contribute to increasing the performance of the heat exchanger device through the preliminary study on the development of nanofluids. The main focus of this present study is to evaluate the characteristics of TiO₂ nanofluid with a volume fraction of 3.0%. The higher concentration than 3.0% is not studied due to the disadvantage to obtain the high viscosity of nanofluid (Kristiawan et al., 2020). Then, these nanofluid was successfully fabricated with the base fluid prepared from the mixture of EG and water in the 25:75 (1:3) ratio. The characteristics that evaluated are stability and thermal conductivity. These two are the fundamental characteristics that the nanofluids should achieve. For heat exchanger device performance, the nanofluid should have long-term stability and high thermal conductivity (Adun, Kavaz and Dagbasi, 2021). The crystal and morphological structure are also analyzed using X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM) to support the characterization results. Then, the review of the heat exchanger application using the TiO₂ nanofluid is also provided to show the challenge and opportunity of this study in future work. This research will have a significant impact on the development of heat exchanger devices and can be applied in a variety of industries.

2. METHODOLOGY

2.1. Chemicals and Equipment's

This work uses several chemicals consisting of titanium oxide (TiO₂) with the white powder in visual characteristics as illustrated in Figure 1 and the other properties shown in Table 1, ethylene glycol (EG), and distilled water. Then, the equipment consists of digital scales, beaker glass, magnetic stirrer, and ultrasonication, respectively, for measuring the chemical with accurate composition, measuring the volume of based fluid, making the homogeneous nanofluid, and dispersing nanofluids. The thermal properties

validation uses a Portable KS-3 Tempos Thermal Properties Analyzer Instrument (METER Group, Inc., USA). All chemicals used in this research were purchase from Brataco Chemical, its analytical grade without any purification process.



Figure 1. White powder visualization of TiO₂ nanoparticles.

Table 1. The properties of TiO₂ nanoparticles.

Properties	Specifications
Molecular formula	TiO ₂
Shape	Spherical
Average size (nm)	30
Purity (%)	>99.95
Appearance	White
density	4.23 g/cm ³

2.2. Preparation of Nanofluids

This stage begins with a chemical preparation consisting of 63.5 g of TiO₂, 25 ml of EG, and 75 ml of distilled water in a 25:75 (1:3) ratio for EG-distilled water. This mixture is a 3.0% volume fraction of TiO₂ nanofluids with 25% EG on the base fluid (TiO₂-EG/W). The number of volume fractions is calculated using equation (1) (Krishnakumar et al., 2019).

$$\phi = \frac{\left(\frac{m}{\rho}\right)_{TiO_2}}{\left(\frac{m}{\rho}\right)_{TiO_2} + \left(\frac{m}{\rho}\right)_{Base\ fluid}} \times 100\% \quad (1)$$

where, ϕ represents the percentage volume fraction of the nanofluid, m refers to the mass of the TiO₂ and base fluid. Then, ρ is the density for TiO₂ and base fluid, respectively.

The nanofluid is then prepared using a two-step method, as shown in Figure 2. This method is preferred for dispersing nanoparticles in different base fluids (Elsaid et al., 2021).

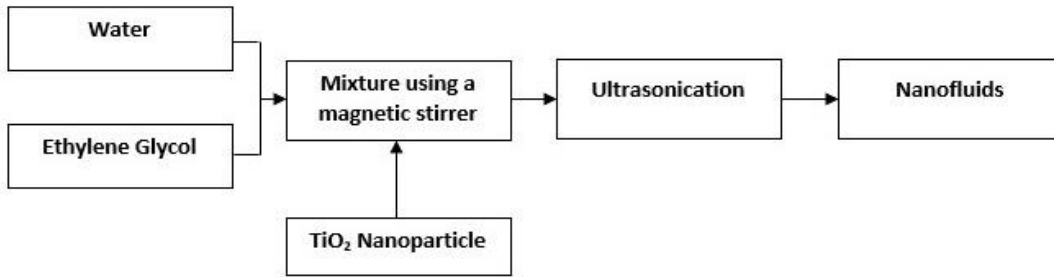


Figure 2. Illustration of TiO₂-EG/W preparation.

The first step involves mixing EG and distilled water with a magnetic stirrer for about one hour in 600 rpm. This solution is then known as the base fluid.

TiO₂ was then gradually introduced into the base fluid while stirring. Once all mixed, the stirring was subsequently continued until a homogeneous nanofluid was obtained. Afterwards, the homogeneous TiO₂ nanofluid is dispersed by ultrasonication for approximately one hour in deionized water. According to Poloju et al., ultrasonication caused the breaking of nanofluid aggregation and its change in the thermophysical properties with a long-term nanofluid stability (Poloju et al., 2022). Several characterizations are then carried out to investigate the properties of the nanofluids, i.e. crystal structure using an XRD Benchtop Miniflex 600 and morphological structure using an SEM SU3500. The XRD characterization operates in scan mode between 0 to 90°. The characterization is carried out using Cu-K α radiation at a wavelength of 1.5046 Å. Furthermore, this characterization also estimated the crystallite size of the nanoparticle using the Debye-Scherrer equation, as shown in equation 2 (Sundar, 2023). Meanwhile, SEM operates at 4.0 kV to produce a high-quality image. XRD and SEM characterizations are carried out on the dry of TiO₂-EG/W.

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (2)$$

where, D is the crystallite size (nm), λ is the wavelength of the X-ray source used for characterization, β is the full width at half maximum intensity peak, and θ is the angle of the diffraction process.

2.3. Qualitative Stability Analysis and Thermal Conductivity Measurement

The qualitative stability is investigated by observing TiO₂-EG/W visualization day by day until the nanofluid's stability is down. This observation confirms the stability level of the nanofluid. The stability is observed by the sedimentation that formed from nanofluid. The fastest sedimentation formed indicates a low stability of nanofluids. Then, the thermal conductivity of TiO₂-EG/W nanofluid was measured using a Portable KS-3 Tempos Thermal Properties Analyzer Instrument (METER Group, Inc., USA) with a schematic illustrated in Figure 3.

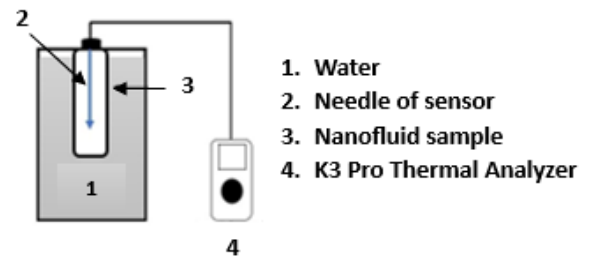


Figure 3. Illustration of thermal conductivity measurement.

The KS-3 type employed is the needle with an embedded temperature sensor. This instrument is claimed to be the best tool for measuring the thermal conductivity of fluids and has been used by several researchers, including Sundaram (Sundaram et al., 2023). In this work, the measurement is performed in the temperature range between 30° to 80° with the data collected at each 5° increase in temperature. These range of temperature was chosen due to the average of optimal working

temperature of heat transfer devices (Yicheng Huang et al., 2022). Initially, the nanofluid sample was stored in a heat-resistant tube. The KS-3 needle was then inserted into the tube vertically without touching the tube's wall surface. This condition was maintained to ensure the accuracy of the measuring results. Measurements with repeatability are performed to improve the precision with which the thermal conductivity of nanofluids is determined. In addition, the results are validated by comparison to the ASHRAE standard.

3. RESULTS AND DISCUSSION

XRD and SEM characterization are carried out to validate the structure of TiO₂-EG/W for crystal and morphological structures, respectively. The XRD analysis is presented in a graphical intensity pattern to Bragg's angle as shown in Figure 4. The Bragg angles observed at 27.60° (101), 36.24° (200), 39.37° (111), 41.42° (120), 44.21° (211), 54.49° (220), 56.77° (002), 63.23° (130), 64.19° (221), 69.16° (301), 69.97° (112), 82.53° (202) belong to the rutile phase of TiO₂.

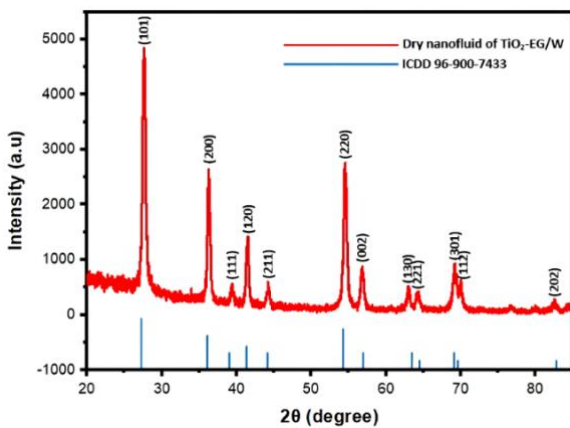


Figure 4. XRD pattern for dry nanofluid of TiO₂ nanoparticle.

According to Ali in 2018, the rutile phase of TiO₂ shows a good performance for nanofluids (Ali et al., 2018). Then, the pattern is matched to the ICDD 96-900-7433 in Match! 3 software, this analysis indicates that the nanofluid is pure TiO₂ without any impurity. The XRD analysis confirms that the TiO₂ nanoparticle has an average crystallite size of 20.23 nm. This number is obtained from the analysis using the Debye

Scherrer formula, as shown in equation 2. In addition, SEM analysis was performed to confirm the structure of TiO₂ nanoparticles in nanofluid. It was observed that the morphological structure of the TiO₂ nanoparticle appeared to be a spherical shape, as illustrated in Figure 5. The SEM analysis also confirms that TiO₂ nanoparticles are predicted to be easily agglomerated. This finding will be a drawback for nanofluid applications. However, this condition needs further observation in the stability of TiO₂-EG/W nanofluid. The stability of the TiO₂ nanofluid was then evaluated. In this work, the evaluation was carried out by qualitative experiment. This method was reported by Shi et al. in 2023 (Shi et al., 2023).

Qualitative stability is investigated using observation day by day. This evaluation produces the stability information of nanofluid from the visual image of nanofluid taken at different time intervals. In this work, the TiO₂-EG/W is observed at 0 days (after preparation), 15 days, and 30 days, as shown in Figure 6a. From that information, it was found that the decreasing of TiO₂-EG/W nanofluid stability is 58% for 15 days as shown in Figure 6b. It informed that sedimentation occurred on this condition; however, it cannot predict the reaction behind the sedimentation process. It might occur due to the strong van der Waals forces and gravity of TiO₂ nanoparticles (Rehman et al., 2023). Nevertheless, several studies report the same condition regarding the sedimentation formed in TiO₂ nanoparticles, as shown in Table 2. It concludes that the sedimentation formed on the 15th day from the preparation process is still better than others.

Table 2. Several studies about the sedimentation of TiO₂ nanofluid investigation.

Materials	Sedimentation investigation	Ref.
TiO ₂ /Water	7 days	(Zhang et al., 2022)
CuO/TiO ₂ /SiO ₂	10 days	(Muzaidi et al., 2021)
TiO ₂ -EG/W	15 days	This work

Figure 7a shows the plot of the thermal conductivity of TiO₂-EG/W nanofluid prepared by a two-step method at a temperature range of

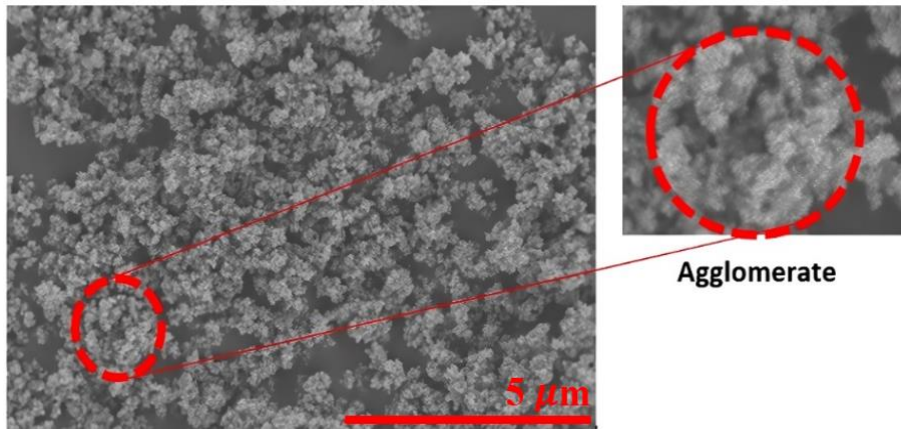


Figure 5. Nanoparticle morphological investigation using SEM.

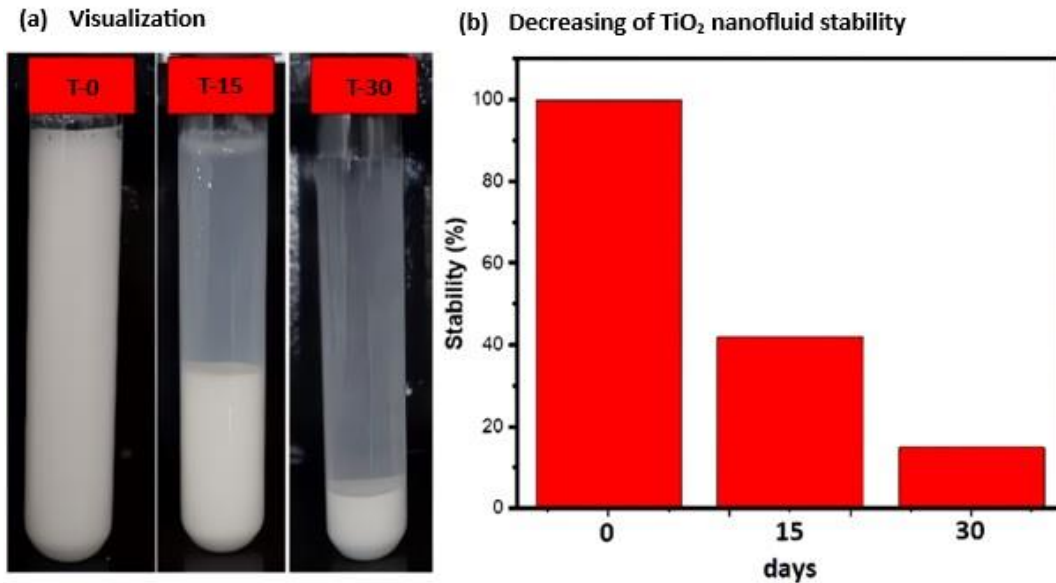


Figure 6. (a) Qualitative stability observation of TiO₂-EG/W nanofluid at 0 day/after preparation (T-0), 15 days (T-15), and 30 days (T-30), (b) Decreasing of TiO₂-EG/W nanofluid stability.

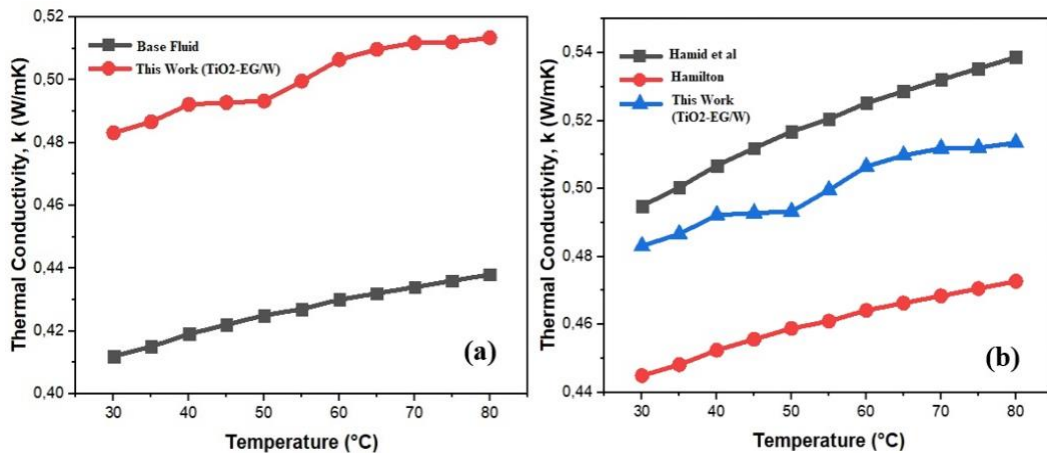


Figure 7. (a) Thermal conductivity of TiO₂-EG/W compared to base fluid, (b) compared to other studies (Hamid et al., 2015).

30 °C to 80 °C compared to the base fluid of ASHRAE. It informs that the highest thermal conductivity is reached at 80 °C, about 17.08% higher than the base fluid. This finding is an advantage for TiO₂-EG/W that is applied to heat exchanger application. Then, thermal conductivity was also compared to the Hamid et al. (Hamid et al., 2016) and Hamilton model Figure 7b shows that the thermal conductivity of TiO₂-EG/W in this present study is higher than Hamilton and lower than Hamid. However, overall characteristics found in this work are still within the normal limits of TiO₂ for heat exchanger application.

This study then findings several characteristics of TiO₂ nanofluid properties that might be useful and helpful for the innovation of upcoming work. To obtain improved performance of TiO₂-EG/W, it is suggested to vary several factors, i.e., volume fraction or EG-water ratio. Another suggestion is to develop a calculation model to determine the thermal conductivity. In addition, TiO₂ nanofluid still has great potential to be developed in future work due to their abundance in nature. Then, it can be developed to apply the TiO₂ nanoparticles from nature as nanofluid in heat exchanger applications and compare their performances with the analytical grade of TiO₂ nanoparticles. However, there are several challenges to developing the TiO₂ nanofluid; one is challenging to control the heat transfer properties due to the technological increase. It follows the fulfilment of nanofluid properties from time to time, customized to heat exchanger devices.

4. CONCLUSION

This study reported the investigation of nanofluid-based TiO₂-EG/W with the ratio of EG: water is 40:60 (2:3) and the volume fraction of nanofluid equal to 3.0%. The nanofluid-based TiO₂-EG/W was successfully prepared by a two-step method. It was confirmed by an XRD analysis, which showed the rutile phase of TiO₂ and, following the aim of this study, indicated good properties for nanofluid application. It is supported by SEM analysis that confirms the morphological structure of TiO₂ in nanofluid is

approached to be spherical shape. However, the disadvantage was that TiO₂ nanoparticles easily agglomerate in a nanofluid. These findings follow the stability characteristics, which showed the decrease of TiO₂ nanoparticles in nanofluid and formed the sedimentations after 15 days.

The stability is observed using qualitative method and informed the stability decrease of about 58% from the initial or after the preparation. This investigation confirmed that TiO₂-EG/W nanofluid in this present study has good stability even for 15 days, and then the evaluation of TiO₂-EG/W nanofluid properties is ready to be conducted for further investigation. The thermal conductivity of TiO₂-EG/W was investigated at the range temperatures of 30 °C to 80 °C. The maximum value of thermal conductivity was obtained at 80 °C of temperature. That value has enhanced up to 17.08% relative to the base fluid. These findings also confirmed the good characteristics of TiO₂-EG/W nanofluid for heat transfer or other applications related to this topic.

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