

Experimental study on ultrasonication, thermal conductivity and dynamic viscosity of Therminol 55-Al₂O₃(1.0wt%)/GNP(0.075wt%) hybrid nanofluid

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Abstract— Hybrid nanofluids, compared to mono-nanofluids, are a new class of heat transfer fluids that provide more control over the characteristics of the base fluid. In this study, an experimental investigation has been conducted on the effect of ultrasonication on the thermal conductivity and viscosity of the Therminol 55 (TH55) oil-based hybrid nanofluid containing alumina nanoparticles (Al₂O₃) and graphene nanoplatelets (GNP). The oleic acid stabilized TH55-Al₂O₃(1.0%)/GNP(0.075%) hybrid nanofluid has been prepared using the two-step method. The optimum bath ultrasonication duration has been determined by measuring thermal conductivity and viscosity as a function of ultrasonication time. The thermal conductivity of the hybrid nanofluid increases with temperature, reaching 15.52% at 65°C when compared to the base fluid, while pure TH55 oil exhibits a decreasing trend in contradiction. The increase in temperature resulted in the dynamic viscosity of the hybrid nanofluid decreasing by 84.5%, while the viscosity increased with the nanoparticle dispersion into the base fluid. Higher thermal conductivity with a promising particle dispersion stability has been obtained at three hours of ultrasonication duration with a 4000 Hz sonication frequency. Therminol 55 based hybrid nanofluids could be a potential candidate for medium temperature heat transfer applications based on the desirable properties over mono-nanofluids and conventional working fluids.

Keywords— Nanofluid, heat transfer fluid, ultrasonication, thermal conductivity, viscosity Introduction (*Heading 1*)

I Introduction

The fast growth of nanotechnology has opened up a new way for typical heat transfer fluids (HTF) to develop nanofluids, which can significantly enhance the performance of thermal systems. As a result, there is a lot of research going on in this sector to improve the thermophysical properties of the HTFs. Among the various conventional HTFs used in medium temperature solar thermal collectors and process industries, Therminol 55 (TH55) is one of the most popular [1]. TH55 possesses a low freezing point and a high boiling point with a maximum operating temperature of 305°C and a low pouring point [2]. The main limitation of TH55 oil is its poor thermal conductivity. The addition of nanoparticles in TH55 oil can change its properties [2-4].

Nanoparticles play a vital role in this scenario. Dispersion of nanoparticles having a size less than 100nm in the conventional HTF imparts significant enhancement in the performance of heat exchangers [5]. Alumina (Al₂O₃) is a high potential nanomaterial among the many choices for nanofluids. They are relatively cheap and more chemically stable than some metal particles. Furthermore, Al₂O₃ is regarded as a promising nanoparticle for heat transfer applications due to its excellent thermophysical characteristics compared to other metal oxide nanoparticles. The thermal conductivity of Al₂O₃ at ambient temperature is about 36 W/m.K [6], which is more than 60 times that of water [7]. Heris et al. [8] carried out studies with Al₂O₃ and CuO nanofluids, confirming that the heat transfer improvement of the base fluid with the addition of Al₂O₃ is more than the addition of CuO nanoparticles.

Likewise, many investigators have experimentally evaluated carbon derivatives such as nanotubes [9,10], GO [11,12], and graphene platelets (GNP) [13,14]. GNPs have an outstanding thermal conductivity of around 5000 Wm⁻¹K⁻¹ [15,16], higher than the thermal conductivity of carbon nanotubes. Further, since GNP is a 2D material, the heat transfer properties are distinct from the 0-dimensional nanoparticles and 1-dimensional carbon nanotube. Various researchers' findings suggested that GNP could be a promising candidate for thermal energy conversion applications [17].

The thermophysical properties of a nanofluid are determined mainly by the key step, synthesis. Nanofluids can be synthesized either by the one-step or two-step process. The one-step technique uses wet chemistry procedures including plasma arc, spraying or sputtering, laser ablation, or electric ablation [5] and the fabrication and dispersion of the nanoparticles in the base fluid is carried out concurrently. The two-step approach requires two distinct procedures: fabrication followed by dispersion using various stabilizing methods. Even though the dispersion stability of nanoparticles in one-step synthesized nanofluid is superior, the process is complex and expensive [18]. Most researchers use two-step synthesis because of its more effortless production procedure and can better improve thermophysical properties than one-step synthesis [5,19,20]. Mohammadpoor

et al. [21] developed Cu based nanofluids using both one-step and two-step techniques and investigated the fluids' thermophysical characteristics and dispersion stability. They reported that nanofluids developed in the one-step process were more stable than fluids developed in the two-step process. Still, the nanofluids fabricated in the two-step technique have better heat transport properties.

The dispersion stability of nanoparticles plays a vital role in the resulting thermophysical properties of nanofluids. Because of their larger surface energy, nanoparticles agglomerate gradually over time. Brownian motions of the nanoparticles lead to Vander Walls forces of attraction between the particles, causing clustering and agglomeration. The aggregation of nanoparticles leads to sedimentation, which reduces the thermal conductivity of nanofluids [22]. The primary limitation of two-step synthesized nanofluids is the deterioration of expected thermal properties due to their short time dispersion stability. Various methods for enhancing the stability of nanofluids include mechanical agitation, magnetic stirring, ultrasonication, pH regulation, use of dispersants, functionalization of nanoparticles, and so on [23].

Ultrasonication is a popular approach used by researchers to stabilize nanofluids. High frequency (>20kHz) sound waves induce nanoparticles to disperse evenly in the base fluid with no modification in nanoparticle surface properties. Krishnakumar et al. [24] followed the ultrasonication method while synthesizing Al_2O_3 nanofluid with different concentrations of Al_2O_3 nanoparticles in ethylene glycol, fabricated by two-step technique. Sanukrishna and Jose Prakash [25] prepared TiO_2 -PAG nanolubricant by the same method. Increased ultrasonication duration gives a more homogeneous nanofluid up to a certain point, after which more ultrasonication degrades dispersion stability due to sedimentation at excessive ultrasonication [5]. Adding a surfactant or dispersant to the base fluid is another simple and popular method to improve nanofluid stability. Suspensions of Al_2O_3 particles have limited stability in TH55 due to the low dynamic viscosity of the base fluid, which causes them to settle in a short duration of hours despite the addition of surfactants. The typical ultrasound sonication cannot shatter the Al_2O_3 agglomeration by itself [26]. Because of its similar density, miscibility, and thermal stability to TH55, Oleic acid (OA) is the most usually applied surfactant in TH55-based nanofluids [2, 27].

Ultrasonication has already been reported to help enhance the dispersion stability of nanofluids. It is essential to determine the appropriate ultrasonication parameters for a particular nanofluid. This work aims to report the suitable ultrasonication parameters for the dispersion of GNP and Al_2O_3 nanoparticles within TH55 using a two-step synthesizing process. The developed hybrid nanofluid samples have been ultrasonicated for one to five hours at 4000 Hz using an ultrasonic agitator. The transmission electron microscopic technique, thermal conductivity, dynamic viscosity, visual image photographic monitoring, and UV-vis spectroscopy techniques have been conducted to investigate the nanofluid characteristics. Furthermore, the thermal conductivity and viscosity of the hybrid nanofluid have been experimentally determined and compared with that of the TH55 oil over a wide range of temperatures.

II Methodology

A. Preparation of nanofluids

GNP and Al_2O_3 nanoparticles are purchased from Alfa Aesar, UK. GNP had a density of 2 g/cm^3 , a surface area of $500 \text{ m}^2/\text{g}$, and a melting point of 2760°C . Al_2O_3 had labelled with 3.965 g/cm^3 density, $32\text{--}40 \text{ m}^2/\text{g}$ surface area, and 2045°C melting point. Therminol 55 oil (TH55) is acquired from Eastman heat transfer fluids, USA. According to the material property datasheet, at 20°C , TH55 had a density of 872 kg/m^3 , 47.7 cSt viscosity, 351°C boiling point, 0.1284 W/m.K thermal conductivity, and 1.91 kJ/kg.K specific heat. Nanofluids are fabricated by using the two-step method. First, a suspension of Al_2O_3 and GNP in TH55 is stirred for one hour with oleic acid (0.5ml oleic acid for one gram of nanoparticle [4, 27]). Magnetic stirring at 1600 rpm is employed through the mixing of the nanoparticles. After that, the suspension is agitated at 4000 Hz for different time periods using an ultra-sonication agitator (BRANSON – 3800). Mono-nanofluids with weight fractions of 1.0% Al_2O_3 and 0.075% GNP are prepared separately to evaluate the distinct effects of Al_2O_3 and GNP nanomaterials on the base fluid. Afterwards, T55-GNP(0.075wt.%)/ Al_2O_3 (1.0wt.%) hybrid nanofluid is prepared.

B. Transmission electron microscope imaging

The hybrid suspension's morphological characterization is evaluated using the transmission electron microscopy (TEM) technique. The resolution of TEM images is much higher than that of scanning electron microscopy (SEM) images, and the TEM photographs indicate the size and distribution of nanoparticles in the suspension. In this work, the TEM analyses are carried out in a JEOL/JEM 2100 transmission electron microscope at Sophisticated Test and Instrumentation Centre (STIC), Cochin University of Science and Technology, Kochi.

C. Measurement of thermal conductivity

Thermal conductivity measurements using steady-state techniques are not suited for liquids since they need more time. The heat loss during the extended measurement time cannot be estimated, resulting in substantial inaccuracies in results. Furthermore, natural convection may occur during this time, giving an extra uncertainty in the measurements. In this study, the KD2 pro thermal property analyzer, which measures the thermal conductivity using the transient hot-wire principle, is used to measure the thermal conductivity of the nanofluids with an accuracy of 5%. Before conducting experimental measurements, the KD2 Pro analyzer is calibrated using the verification standard, glycerine, supplied by the manufacturer, with an estimated accuracy of $\pm 1.6\%$. In this study, five measurements are taken at each temperature, and the average is reported as the result.

D. Measurement of Dynamic Viscosity

The dynamic viscosity of the samples is measured using a Brookfield LVDV-II+Pro cone-and-plate viscometer with a measurement range between 1.0 to 2000 cSt and spindle speeds 0 to 150 RPM. A constant-temperature water-circulating bath (Julabo F25 HE, Germany) keeps the samples at different constant temperatures during the experiment. Before doing the measurements, the viscometer is calibrated using the standard fluid provided by the manufacturer. The maximum uncertainty is found to be 1.9%.

E. Stability of nanofluid

Uniform dispersion of nanoparticles and long-term stability of the nanofluids are extremely important for their widespread use. To access the stability of the hybrid nanofluid, visual-photographic image monitoring and UV-visible spectroscopy analysis have been done on the nanofluid at two distinct times; one day after the nanofluid fabrication and after 14 days of fabrication. The principle of stability evaluation in UV-visible spectroscopy is that absorbance at a particular wavelength is dependent on the amount of dispersed nanoparticles in the nanofluid [28]. Additionally, over three months, eye monitoring and photo capturing techniques have been utilized to assess the stability of the hybrid nanofluid.

III Results and discussion

A. Effect of sonication time

The optimum time required for ultrasonic agitation is determined by tracking the changes in thermal conductivity and dynamic viscosity of the hybrid nanofluid as a function of ultrasonication time. Fig. 1(a) and (b) depicts the variation in thermal conductivity and viscosity of the TH55- $\text{Al}_2\text{O}_3(1.0\%)/\text{GNP}(0.075\%)$ nanofluid, at 50°C , as a result of different ultrasonication time, respectively.

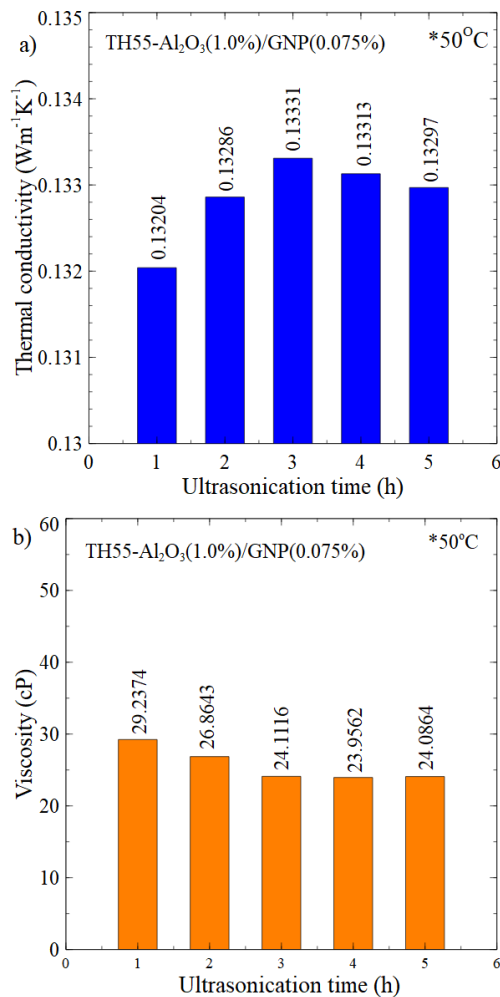


Fig.1 Property variation as a function of ultrasonication time (a) Thermal conductivity (b) dynamic viscosity

As shown in Fig. 1(a), the thermal conductivity for 3 hours sonicated samples exhibited a maximum thermal

conductivity of 0.13331 W/m.K . The dynamic viscosity of the hybrid nanofluid sample is larger at shorter sonication times, as illustrated in Fig. 1(b). In comparison, it decreases and remains almost the same for more extended sonication periods of 3 hours or more.

B. Transmission Electron Microscopy

Transmission electron microscope (TEM) observation is conducted to study the nanoparticles' structure, shape, and distribution in the hybrid suspension. As depicted from Fig. 2, the GNP and Al_2O_3 nanoparticles are evenly distributed in the hybrid suspension. It must be noted from Fig.2 that many GNP nanoplatelets are stucked over the spherical Al_2O_3 nanoparticles. This is because of GNP's smaller particle size distribution than Al_2O_3 nanoparticles. The ultrasonication and addition of surfactant keep the nanoparticles stable and uniformly distributed in base-fluid by electrostatic repulsive force among the particles and steric repulsions provided by the dispersant to counteract the van der Waals attraction force [29].

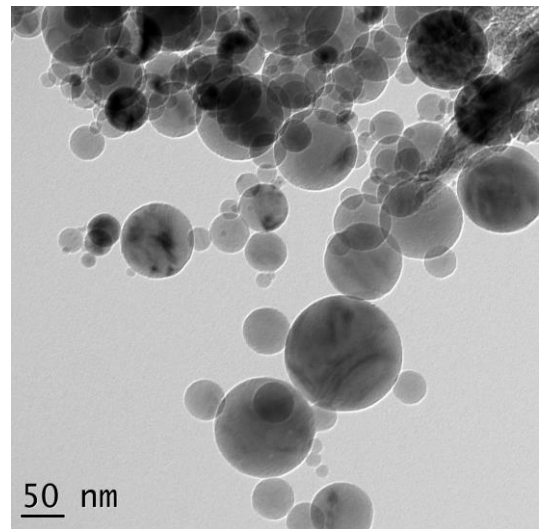


Fig. 2 TEM image of hybrid suspension

C. Dispersion stability of the hybrid nanofluid

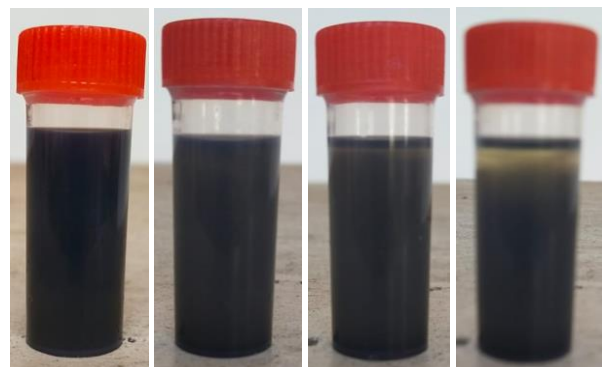


Fig. 3 The prepared hybrid nanofluid (a) 1 day (b) 14 days (c) 30 days (d) 90 days after the preparation

The stability of the hybrid nanofluid has been assessed by visualization of static nanofluid photographs in terms of the

time required for nanoparticle sedimentation. Many earlier studies employed sedimentation observation to characterize the stability of nanofluids [31, 32]. The visual appearance of the hybrid nanofluid over 90 days after preparation is shown in Fig. 3. As the static storage period increases, the top portion of the fluid column becomes more and more transparent, which is related to the stability of the hybrid nanofluid. No noticeable sedimentation can be observed in the sample after one day and fourteen days of preparation (Fig.3(a) and (b)). After 30 days of preparation (Fig. 3(c)), the sample exhibits little evidence of sedimentation due to the agglomeration process of the nanoparticles. A clear separation between the nanoparticles and the base fluid can be seen in the sample kept for ninety days of static storage, as shown in Fig. 3(d).

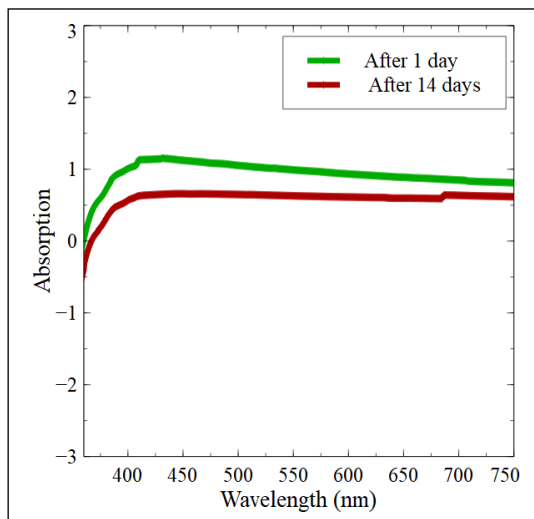


Fig.4 UV-vis spectroscopy of the hybrid nanofluid

In addition to the visual photograph method, UV- vis spectroscopy is carried over fourteen days to verify the dispersion stability of the hybrid nanofluid. It utilizes Beer-Lambert's law, which relates the nanofluid's absorbance and concentration of dispersed nanoparticles. Figure 4 shows the absorbance of TH55-Al₂O₃/GNP hybrid nanofluid after one day and fourteen days of preparation. The peak absorbance of the hybrid nanofluid occurs at a 420 nm wavelength. After one day and fourteen days of preparation, the peak absorbance of the sample is 1.15 and 0.658, respectively. The absorbance of 0.658 after fourteen days of the nanofluid preparation in the nanofluid with surfactant is a promising indication of the nanofluid stability.

Furthermore, the nanofluid does not have a long idle time in medium temperature applications like solar thermal systems, further lowering the possibility of nanoparticle settling. Moreover, it should be noted that this reduction in nanoparticle dispersion caused by agglomeration and sedimentation could not be observed using the visual digital photograph technique. As a result, it is inferred that the visual-photographic approach alone cannot clearly indicate the stability of nanofluids.

D. Measurement of thermal conductivity

The thermal conductivity of the TH55-Al₂O₃(1.0%)/GNP(0.075) hybrid nanofluid and the base fluid is measured for temperatures ranging from 0°C to 65°C. As

depicted in Fig. 5(a), the thermal conductivity of the base fluid decreased by increasing temperature. This reduction in thermal conductivity of the pure TH55 oil with the increasing temperature is also evident from the manufacturers' property datasheet [33]. However, the thermal conductivity of the hybrid nanofluid is observed to increase with an increase in temperature. An enhancement of 1.14% at 25°C and 15.52% at 65°C are observed compared with TH55 for the same temperatures. Based on the measurements, "thermal conductivity ratio", the ratio of the thermal conductivity of TH55-Al₂O₃/GNP hybrid nanofluid to the thermal conductivity of TH55 is depicted in Fig. 5(b). It is more convenient to present the relative enhancement of the thermal conductivity of the nanofluids over the thermal conductivity of the base fluid in terms of thermal conductivity ratio. Higher thermal conductivity ratios (>1) are desirable for heat transfer applications. It can be observed that the thermal conductivity ratio is always greater than one, over the temperature range of 25°C to 65°C.

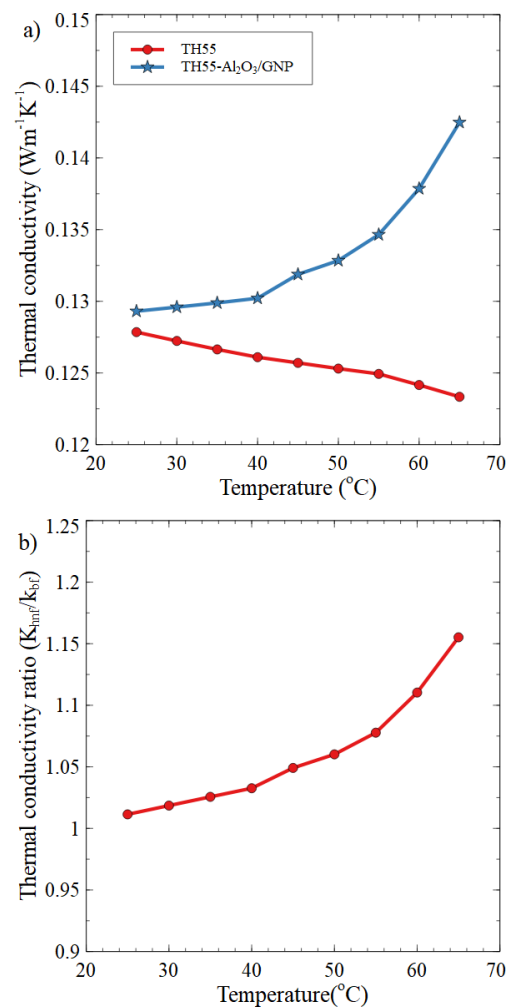


Fig. 5 Effect of temperature on (a) thermal conductivity (b) thermal conductivity ratio

The improvement of thermal conductivity is significant at higher temperatures because the nanoparticles possess high kinetic energy, which leads to the increased Brownian motion effect, as explained by Manikandan and Rajan [27].

E. Measurement of viscosity

The dynamic viscosity of TH55- $\text{Al}_2\text{O}_3(1.0\%)/\text{GNP}(0.075\%)$ hybrid nanofluid and that of TH55 HTF as a function of temperature is shown in Fig. 6(a). It can be seen that the temperature has a strong effect on the viscosity of both fluids. The value of the viscosity of hybrid nanofluid at 20°C is 63.29 cP, which is higher than by 56.52% compared with that of the base fluid. However, the hybrid nanofluid's viscosity is reduced by 86.69% as the temperature rises from 20°C to 90°C . At 20°C , the difference in viscosities between the hybrid nanofluid and TH55 oil is 22.85cP, while reduced to 4.85cP at 90°C . These anomalous nanofluid experimental viscosity trends have been reported previously, the possible reasons that might explain it remain controversial. The suspended nanoparticles in the base fluid increase its viscosity because of the collisions between nanoparticles and the base fluid. Increasing the nanoparticles in hybrid suspension compared to mono-nanofluids will result in greater nano-racemes due to Van der Waals forces [34]. The particles agglomeration within the suspension increases internal shear stress in hybrid nanofluid, increasing viscosity. Figure 6(b) shows the variations in relative viscosity, the ratio of the viscosity of the hybrid nanofluid with temperature to that of the TH55 oil. As shown in the diagram, at 90°C , the viscosity of the developed hybrid nanofluid is 2.36 times that TH55. Minia et al. [35] reported 2.3 times to increase in viscosity for a $\text{Cu}/\text{Al}_2\text{O}_3$ -water hybrid nanofluid at 1% volume concentration.

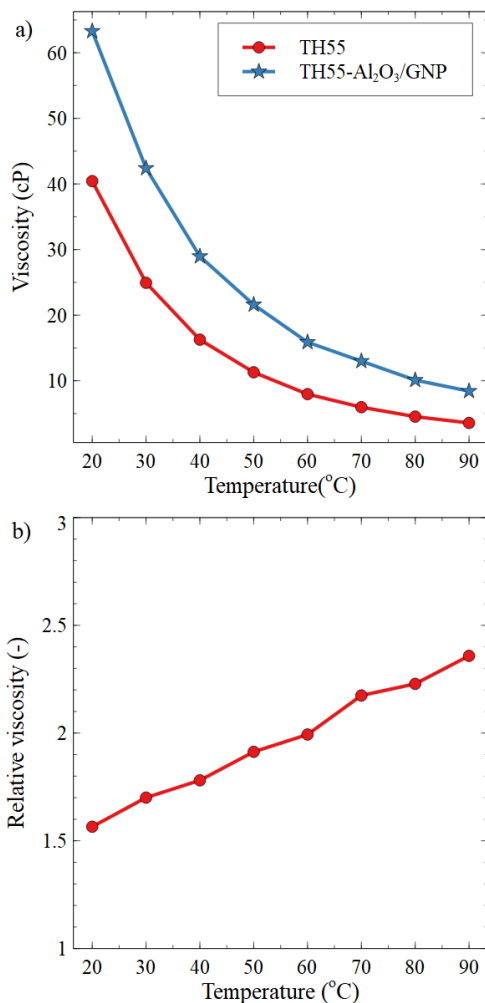


Fig. 6 Effect of temperature on (a) viscosity (b) relative viscosity

The decrease in nanofluid viscosity with increased temperature is expected due to the weakening of the inter-particle and inter-molecular adhesion forces. Similar trends have also been observed in almost all other varieties of nanofluids [36, 37]. The increased viscosity of the hybrid nanofluid leads to a pumping power penalty. However, this is minor compared to the favourable benefits of heat gain and absorption in solar thermal applications.

F. Effect of Hybridisation

To study the effect of hybridization over the mono-nanofluids, TH55- $\text{Al}_2\text{O}_3(1.0\%)$ and TH55- $\text{GNP}(0.075\%)$ mono-nanofluids are prepared, and their thermal conductivity and viscosity are compared with that of hybrid TH55- $\text{Al}_2\text{O}_3(1.0\%)/\text{GNP}(0.075\%)$ nanofluid (Fig. 7). All samples are prepared by the two-step synthesis method with 3 hours of ultrasonication.

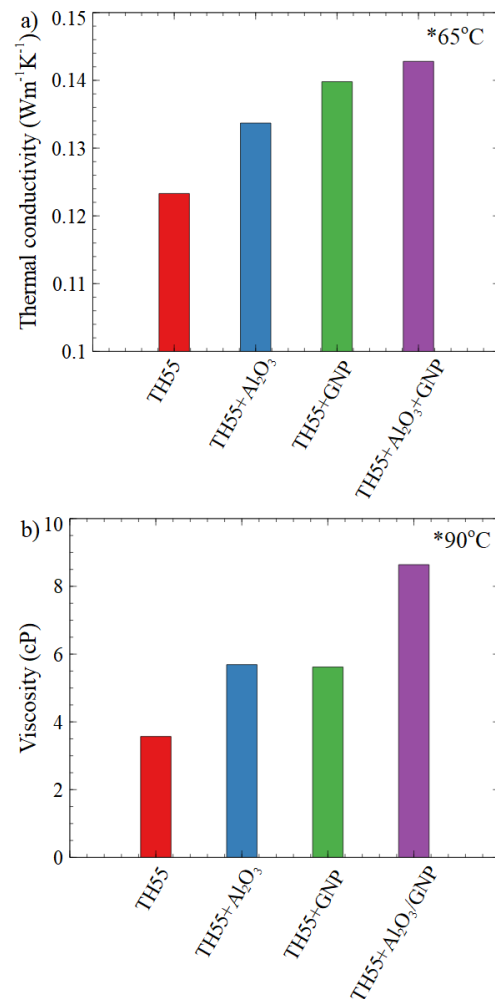


Fig.7. Comparison of mono and hybrid nanofluids with the base fluid (a) Thermal conductivity (b) dynamic viscosity

At 65°C , the thermal conductivity of pure TH55 oil, Al_2O_3 mono-nanofluid, GNP mono-nanofluid and $\text{Al}_2\text{O}_3/\text{GNP}$ hybrid nanofluid are 0.1233, 0.1337, 0.1398, and 0.1428 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ respectively. The thermal conductivity enhancement for the Al_2O_3 mono-nanofluid, GNP mono-nanofluid, and $\text{Al}_2\text{O}_3/\text{GNP}$ hybrid nanofluid relative to the TH55 oil are 8.44%, 13.41%, and 15.52%, at 65°C . The results reveal that GNP has a greater influence on thermal conductivity than Al_2O_3 nanoparticles. At 90°C , the dynamic viscosity of the base fluid, Al_2O_3 mono-nanofluid, GNP

mono-nanofluid and Al₂O₃/GNP hybrid nanofluid are 3.57, 5.69, 5.62, 8.64 cP, respectively. The increase of viscosity at 90°C for Al₂O₃ mono-nanofluid, GNP mono-nanofluid and Al₂O₃/GNP hybrid nanofluid relative to TH55 oil are 59.45%, 57.44%, and 142.06%. It should be noted that despite the very low concentration of GNP (0.075%) compared to the Al₂O₃ concentration (1.0%), its influence on the viscosity of the base fluid is considerable. This indicates that a higher concentration of GNP in nanofluids leads to a pumping power penalty, which is not desirable in process heating applications involving HTF circulation.

Conclusion

The thermal conductivity and dynamic viscosity of the hybrid TH55-Al₂O₃(1.0%)/GNP(0.075%) nanofluid is determined for different ultrasonication times at 4000 Hz. Magnetic stirring of one hour at 1600RPM followed by three hours of ultrasonication is sufficient to develop a promisingly stable hybrid nanofluid with the addition of OA. In comparison to TH55, the thermal conductivity of the mono Al₂O₃ and GNP nanofluids are 8.43% and 13.38% higher at 65°C. The thermal conductivity of the hybrid nanofluid increased by 15.52% at 65°C compared to TH55 oil. At 90°C, the viscosity of the Al₂O₃ and GNP mono-nanofluids and that of the hybrid nanofluid are 1.59, 1.54, and 2.42 times higher than that of the TH55 oil. The viscosity of the hybrid nanofluid showed about 22.85 cP of increase at 20°C, while it is only 4.85 cP at 90°C compared to the base fluid. After 14 days of static storage, the hybrid nanofluid exhibited an absorbance of 0.658 at 420 nm wavelength. With the enhanced thermal conductivity and the good dispersion stability by adding nanoparticles, this hybrid nanofluid could be used as an alternative HTF in medium temperature heat transfer applications such as solar thermal collectors.

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