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EFFECTS OF SEQUENCE PREPARATION OF TITANIUM DIOXIDE–WATER NANOFLUID USING CETYLTRIMETHYLAMMONIUM BROMIDE SURFACTANT AND TiO_2 NANOPARTICLES FOR ENHANCEMENT OF THERMAL CONDUCTIVITY

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ABSTRACT

To maintain the stability of nanofluid from precipitation and agglomeration, some methods such as ultrasonic vibration, adding surfactant, and controlling the pH value of the system have been studied. Herein, the preparation of titanium dioxide (TiO_2)–water nanofluid, by using TiO_2 nanoparticles (TiO_2 NPs) and the cationic surfactant cetyltrimethylammonium bromide (CTAB), was investigated to determine the effects of the sequence method on the preparation of TiO_2–water nanofluid, its thermal conductivity, its stability, and its temperature distribution. NPs can improve the efficiency of heat transfer fluids and improving the stability of colloidal systems. Some parameters were varied, including sonication times of 5, 10, and 30 minutes, variations of TiO_2 loading in 1–8% volumetric loading, concentrations of CTAB (0.005–0.035 wt%), and pH at 8–12. The procedure sequences of 2 and 5 showed the distribution particle size of TiO_2 nanoparticles in nanofluid had a narrow range (190.3–208.7 nm) compared to other sequence methods (611 nm–5.35 μm). The procedure sequence of 2 is following demineralized water (100 mL), 8% volumetric loading of TiO_2 NPs, ultrasonication time of 10 min and CTAB of 3.2×10^{-3} M, while the procedure sequence of 5 is in the respective order of demineralized water (100 mL), 8% volumetric loading of TiO_2 NPs, ultrasonication time of 10 min and pH at 8. The CTAB surfactant (0.029 wt%) had a greater influence on particle distribution in the nanofluid than the pH. The thermal conductivities of the nanofluid were characterized with TiO_2 nanofluid as the working fluid. The experimental results showed a maximum of 21% thermal conductivity enhancement for 8% volumetric loading of TiO_2 NPs at pH 8 and fourfold increase in critical micelle concentration (0.029 wt%) from CTAB. These findings offer the potential for preparing a stable TiO_2–water nanofluid with a short ultrasonic time of 10 minutes. This process is a desirable and very useful to obtain a stable TiO_2–water nanofluid with a short ultrasonic time for efficient process and low-cost nanofluid with high

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thermal conductivity and stability.

Keywords: Cationic surfactant; Concentration of CTAB; Thermal conductivity; TiO$_2$-water nanofluid; Ultrasonic time

1. INTRODUCTION

The development of high-performance heat transfer fluids using nanoparticles (NPs) has been interesting to investigate in detail (Ahlatli et al., 2016). It is well known that nanofluid, a colloidal mixture produced from a base fluid and a nanoparticle, has valuable heat transfer applications and is among a new generation of heat transfer fluids that enhance thermal conductivity. Studies of various parameters (such as particle size, concentration, temperature, material type and base fluid type) to enhance heat transference have been performed (Putra et al., 2012; Yiamsawasd et al., 2012; Saleh et al., 2013; Ismay et al., 2013). NPs can improve the efficiency of heat transfer fluids (Zhou et al., 2012; Saleh et al., 2013), as their high surface energy makes them easy to coagulate and difficult to disperse in base fluids, improving the stability of colloidal systems (Zhou et al., 2012). The stability of colloidal suspensions of micro and nanosized particles is dependent on the surface force between particles (namely, the zeta potential) that is influenced by pH value (Ismay et al., 2013). Das et al. (2018) experimentally investigated the stability measurement of anatase- sodium dodecyl sulfate (SDS) and anatase-cetyltrimethylammonium bromide (CTAB) nanofluids, which had observable zeta potentials of -17.8 mV and -21.1 mV, respectively. Therefore, anatase-CTAB nanofluid was found to have marginally better stability than the anatase-SDS nanofluid.

The agglomeration of TiO$_2$ NPs settles and clogs microchannels as well as decreasing the thermal conductivity of nanofluid (Yu & Xie, 2012; Ismay et al., 2013). Many investigations have been conducted to produce a more stable colloidal and suspension of nanofluid. To maintain the stability of nanofluid from precipitation and agglomeration, some methods (such as ultrasonic vibration, adding surfactant, and controlling the pH value of the system) have been reported (Duangthongsuk & Wongwises, 2009). A well-known and effective method to homogenize dispersed NPs in base fluids is adding surfactant (Jiang et al., 2003; Xie et al., 2003; Murshed et al. 2005; Zhou et al., 2012).

The effects of surfactants (such as CTAB, acetic acid (AA), oleic acid (OA), and SDS) on TiO$_2$–water nanofluid have been studied and findings have shown that CTAB and AA provided stable suspensions (Das et al., 2016; Adiwibowo et al., 2018). The TiO$_2$ solid fraction has been varied between 0.1–2.0% while temperatures have ranged from 20 to 60°C (Das et al., 2016). Murshed et al. (2005) reported enhanced thermal conductivity (up to 33%) for deionized water by adding TiO$_2$ NPs (5% volume fraction). That being said, TiO$_2$ NPs have also been reported as causing dye degradation (Rahman et al., 2018; Zulmajdi et al., 2019).

In a previous study, the cationic surfactant CTAB was investigated for its ability to break down particle agglomeration in a suspension and was found more effective than oleic acid (Murshed et al., 2005). However, the TiO$_2$ nanofluid by ultrasonic process for 8 to 10 hours (Murshed et al., 2005). On the other hand, Duangthongsuk and Wongwises (2009) investigated the preparation of TiO$_2$ nanofluid by using ultrasonic within 2 hours. As noted here that the time for preparation of TiO$_2$ nanofluid is too long in the range 2 to 10 hours.

Therefore, to reduce the ultrasonic times in preparation of TiO$_2$–water nanofluid during the ultrasound process, the proper mixing, stabilization and also dispersion of TiO$_2$ NPs in water are very important to investigate for producing the stable of TiO$_2$–water nanofluid. In this study, improving and increasing the thermal conductivity of TiO$_2$–water nanofluid and the optimum thermal conductivity from TiO$_2$–water nanofluid with short time sonication were studied. The effect of CTAB in TiO_2–water nanofluid formulation and the effect of sequence preparation methods in growth of particle size in TiO_2–water nanofluid were also studied in detail. The experimental results and theoretical predictions from the Maxwell (1873) and Bhattacharya et al. (2004) models were also compared.

2. EXPERIMENTAL

2.1. Materials

The TiO_2 NPs (Degussa P25) used had a particle size of 21 nm and a density of 130 g/L. NaOH was purchased from Merck (Germany) and was used for pH adjustment. CTAB ($C_{19}H_{42}BrN$) with a molecular weight of 364.45 g/mol and a critical micelles concentration (CMC) of about 8×10^{-4} M was used to stabilize the TiO_2 nanofluid.

2.2. Characterization of Thermal Conductivity of TiO_2–water Nanofluid

The thermal conductivity of TiO_2–water nanofluid was measured with a KD2 portable thermal analyzer (hot wire type) with a 2-minute measurement speed, a 60-mm long sensor with a diameter of 1.28 mm, and a 72 cm length of wire. Characterization and its measurement for thermal conductivity were according to the Saleh et al. (2014). The sensor contained an integrated heating element and thermo-resistor and was linked to a microprocessor to control and conduct measurements. The thermal conductivity sensor had to be dipped into the nanofluid system, which was placed in a thermostatic circulating bath (TCB) to maintain a constant temperature of 25ºC for all measurements. Threefold measurements were conducted for each nanofluid to ensure the reliability of the thermal conductivity measurements.

2.3. Preparation of TiO_2-water Nanofluid with Various Concentrations

TiO_2–water nanofluids were synthesized with an identical mixing method using starting materials at desired concentration levels prior to being sonicated using ultrasonic instrument. TiO_2 NPs at concentrations from 1 to 8% volumetric loading were added to 100 mL of demineralized water before being mixed using an ultrasonic method for times of 5, 10, and 30 minutes.

The best and optimum ultrasonic time for preparation of TiO_2-water nanofluid was 10 minutes. Thus, we used the optimum time for further preparation of TiO_2 nanofluid. Thermal conductivity of nanofluid was then measured, followed by our selection of the highest thermal conductivity from the samples for further study. The best concentration of TiO_2 NPs with the highest thermal conductivity was obtained at an 8% volume fraction.

2.4. Preparation of TiO_2-water Nanofluid with Various pH

The best TiO_2 NPs concentration of 8% volume fraction was selected for further study at various pH from 8–12. The pH was adjusted using a NaOH solution and measured using a pH meter. All thermal conductivities of TiO_2-water nanofluid were recorded using a TCB. The best pH value of nanofluid with the highest thermal conductivity was 8.

2.5. Preparation of TiO_2-water Nanofluid with Addition of Cationic Surfactants

Nanofluids at various CMCs (1–6) were synthesized by adding CTAB. The CMC of CTAB was 8×10^{-4} M. Thermal conductivities of nanofluid prepared were measured using the TCB. The highest thermal conductivity of nanofluid with a CMC of CTAB was fourfold. Considering the results, a fourfold CMC, a TiO_2 NPs concentration of 8% volumetric loading, and a pH value of 8 were chosen for nanofluid preparation analysis under a combination of sequence methods. The CMC of CTAB was calculated as follows:

$$\text{CMC} = (\text{amount of surfactant}/\text{molecular weight}) \times (1000/\text{volume of solvent}) \qquad (1)$$

2.6. Characterizations

Particle size distributions of samples were measured by using the Particle Size Analyzer (PSA), Zetasizer Nano series (Malvern ZEN 1600). The ultrasound process was conducted with a Sonicator Ultrasonic Processor (Brand Chrome Tech, type UP-800, size Tip 13 mm, 800 watt). The thermal conductivity of TiO_2–water nanofluid was measured with a KD2 portable thermal analyzer (hot wire type).

3. RESULTS AND DISCUSSION

3.1. Thermal Conductivity of the Base Fluids and TiO_2-Water Nanofluid with Various Concentrations and Ultrasonic Time

The thermal conductivity of demineralized water as a base for the nanofluid under the experimental conditions is listed in Table 1. At 25–40ºC, the thermal conductivity of demineralized water was found in the range of 0.57–0.63. A TCB was used to measure the thermal conductivity during measurement samples while maintaining a temperature of 25ºC. When measuring pH from 8 to 14, the thermal conductivity remained constant (0.57). Adding the CTAB surfactant with a CMC from 4 to 10 in base fluids, the thermal conductivity also remained constant. Increasing the CMC of CTAB only decreased the pH, because the concentration of CTAB cationic surfactant caused an increase in positive charge thus the fluid is more acid.

Table 1 Thermal conductivity of demineralized water as base fluid in nanofluids in several experimental conditions

No	T (ºC)	K	pH	K	CMC (x)	pH water + CTAB	K water + CTAB
1	25	0.57	8	0.57	4	5.80	0.57
2	30	0.59	10	0.57	6	5.68	0.57
3	35	0.61	12	0.57	8	5.56	0.57
4	40	0.63	14	0.57	10	5.52	0.57

where K: thermal conductivity, CMC: critical micelles concentration

Sonawane et al. (2015) investigated TiO_2–water nanofluid with optimum sonication times of 60 minutes with a 6% volume fraction of TiO_2 NPs, confirming a 22% enhancement in thermal conductivity. In this study, a pH of 8, a fourfold CMC, a constant temperature of 25ºC, and sonication for 10 minutes (see Figure 1) were used as control parameter conditions. TiO_2 NPs were used to increase the base thermal conductivity of demineralized water. The best performance of TiO_2–water nanofluid was synthesized at an 8% volume fraction of NPs, a sonication process of 10 minutes, and an addition of CTAB (0.029 wt%). Sonication was used to improve the dispersion (Prasher et al., 2006) of TiO_2 NPs. The thermal conductivity enhancement was due to the increase in ultrasonic time from 0 minutes to 10 minutes. The Brownian motion of NPs and the intermolecular interaction between particles and fluids in the nanofluids increased after the optimum time increased from 10 to 30 minutes. The clustering of NPs started, which accounted for the decrement in primary NPs for heat transport and thermal conductivity. It is believed that heat transfer is a surface phenomenon and that the surface of NPs is used for thermal energy interaction (Sonawane et al., 2015). When the particles started to agglomerate, the effective surface area to volume ratio decreased, resulting in a reduction of the effective transfer area of particles and causing a decrease in the thermal conductivity of the fluid (Sonawane et al., 2015).

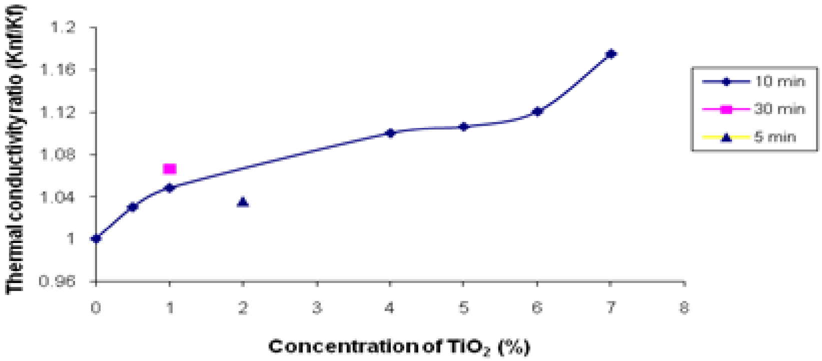


Figure 1 Effect of ultrasonic time on thermal conductivity at room temperature

Figure 2 shows the thermal conductivity ratio of TiO_2–water nanofluid as a function of NPs volume concentration. The addition of TiO_2 NPs to the base fluids enhanced their thermal conductivity from 3 to 21% (Figure 2). The experimental results show a maximum of 21% thermal conductivity enhancement for an 8% volume fraction of TiO_2 NPs. The TiO_2 NPs was well dispersed into the base fluids, making the thermal conductivity of the TiO_2 NPs greater than the base fluids. Thermal conductivity enhancement can be achieved if the Brownian motion present and occurred in the system (Prasher et al., 2006). Brownian motion caused the colloidal particles to be uniformly dispersed in the base fluids and TiO_2 NPs could not be separated from the dispersion when silenced. This observation provides greater value than previous research, confirming that as much as 5.8% thermal conductivity enhancement is achieved with a 2% volume fraction of TiO_2 NPs at 2 hours of sonication time (Sonawane et al., 2015).

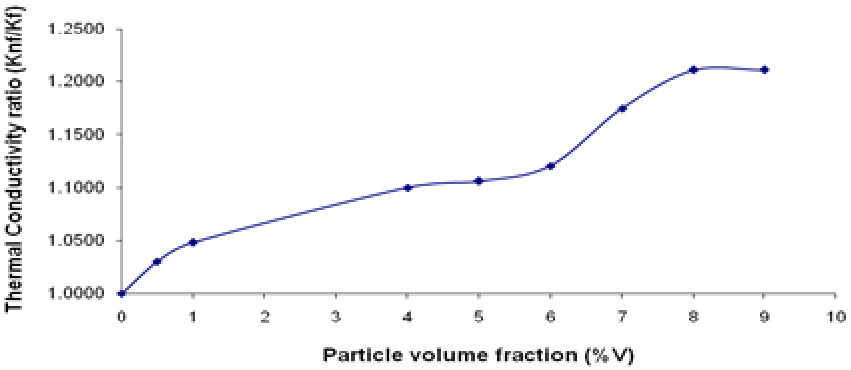


Figure 2 Enhancement of thermal conductivity of TiO_2–water nanofluids with the demineralized water as base fluid

The increased thermal conductivity was influenced by NPs size, shape, and properties as well as the interaction between NPs and the base fluid surface, which caused cluster formation (Xie et al., 2003; Leong et al., 2006). Our study results showed thermal conductivity increasing linearly with increasing amounts of TiO_2 NPs, reaching an optimal thermal conductivity ratio at 8% volume fraction. These results are similar to thermal conductivity increases achieved with Fe nanofluid using a base of ethylene glycol reported by Hong et al. (2005).

Both the experimental data and the theoretical results from the Maxwell (1873) and Bhattacharya et al. (2004) models showed that the thermal conductivity of TiO_2–water nanofluid increased with the volume fraction of TiO_2 NPs. The thermal conductivity predictions for TiO_2–water nanofluid obtained using the Maxwell model were similar to the results from this study. Both differed from predictions proposed under the Bhattacharya model (see Figure 3).

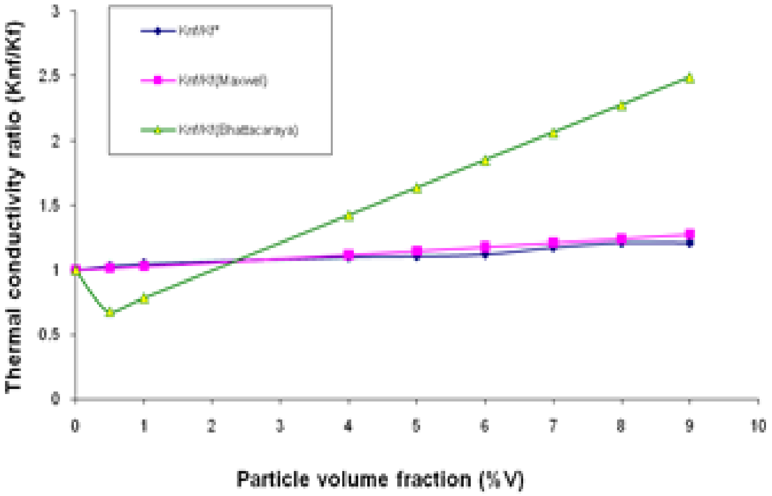


Figure 3 Comparison thermal conductivity between theoretical predictions by the Maxwell and Bhattacharya models as well as resulting experimentation ($K_{NF/}K_{water}^{*}$)

3.2. Thermal Conductivity of TiO$_2$-Water Nanofluid with Various pH

In this study, pH value adjustment was used to modify the base fluid and the surface of the TiO$_2$ NPs, providing electrostatic repulsive force to offset the van der Waals forces between particles, so that NP aggregation did not occur (Li et al., 2008). Increasing the pH value enhanced thermal conductivity due to the increase in electrostatic charge on the particle surface (Li et al., 2008). However, our study showed that increasing pH values did not influence the thermal conductivity of TiO$_2$–water nanofluid. At the optimal TiO$_2$ NPs volume fraction of 8%, we evaluated the effects of pH value on thermal conductivity for the TiO$_2$–water nanofluid. We noted that the changing of pH values did not influence the thermal conductivity when compared with the base fluid. Changing the pH value from 8 to 12 actually reduced the thermal conductivity ratio (K_{nf}/K_f) of the nanofluid from 1.211 to 1.158. The increased pH value meant that a higher concentration of NaOH reduced the electrical double layer, causing decreased electrostatic repulsive force. Consequently, agglomeration would form and reduce thermal conductivity. The effects of pH value on the thermal conductivity of the TiO$_2$–water nanofluid is presented in Figure 4.

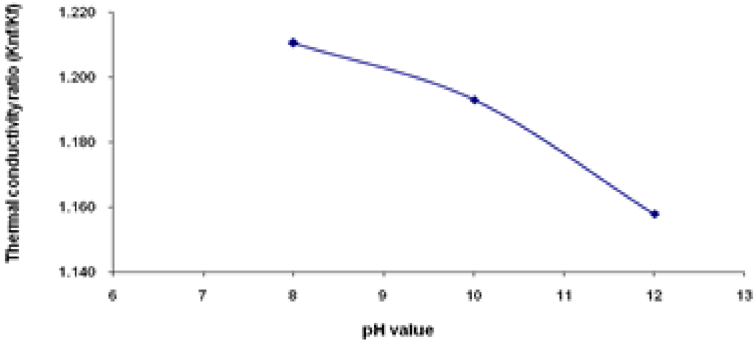
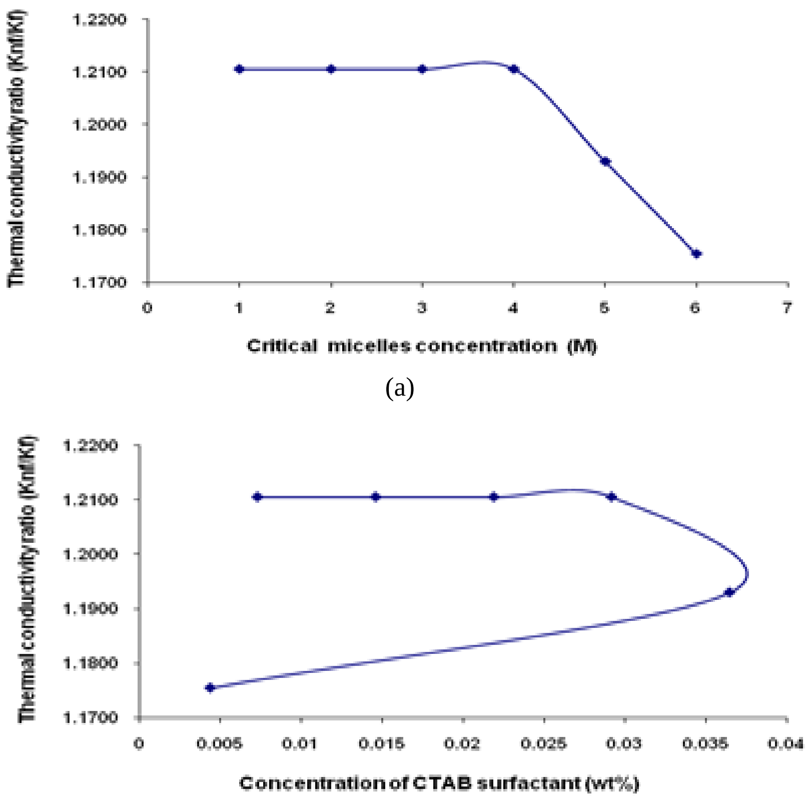


Figure 4 Effects of pH value on the thermal conductivity of TiO$_2$-water nanofluid

3.3. Thermal Conductivity of TiO$_2$-Water Nanofluid with the Addition of Cationic Surfactant

Surfactant was added to the base fluid to reduce particle agglomeration and aggregation (Zhou et al., 2012), as well as clusters, increasing stability and enhancing the dispersion of TiO$_2$ NPs into the base fluid. The optimum amount of CTAB surfactant added to the base fluid is presented in Figure 5a. It was determined that adding cationic surfactant (CTAB) at rates higher than fourfold of the CMC would reduce the thermal conductivity of TiO_2–water nanofluid. We found that the CMC of CTAB surfactant was about 8×10^{-4} M, a smaller value than that found in determining CTAB surfactant through the fiber refractive index sensor principle, which was about 9.11×10^{-4} M (Zhen-Jian et al., 2010).

At a fourfold CMC, CTAB surfactant formed micellar, which attached to the TiO_2 NPs. This layer may have offset the Van der Waals force, which prevented the deposition and maintained the thermal conductivity (Yu & Xie, 2012). The thermal conductivity decreased when we used CTAB surfactant at a greater than fourfold CMC (0.029 wt%). This occurred because of the nanofluid containing more cationic nitrogen (N^+), causing a shift in the pH value from TiO_2–anatase (pH 6.4) toward the point of zero charge (PZC). When the pH value approached PZC, the particle charge and steric repulsive force would be lost, causing particles to stick together and decreasing thermal conductivity (Zhu et al., 2008). These results are similar to those reported by Wang et al. (2009), who indicated that increased SDBS (sodium dodecylbenzene sulfonate) surfactant into an Al_2O_3-deionised water nanosuspension also produced swiftly decreasing thermal conductivity ratios. The SDBS concentrations of 0.1 and 0.07% were deemed optimal concentrations for 0.1% Al_2O_3 and 0.1% Cu nanosuspensions, respectively (Wang et al., 2009).



(a)

(b)

Figure 5 (a) Effects of CTAB surfactant on the thermal conductivity of TiO_2–water nanofluids vs. critical micelles concentration; (b) thermal conductivity ratio vs. CTAB concentrations

Murshed et al. (2005) reported that CTAB surfactant could improve stability and provide good dispersion in fluids without influencing the thermo-physical properties of a nanofluid while also providing heat transfer performance if a very low concentration of CTAB (0.01–0.02 wt%) was used. We observed that the effects of surfactant concentration on the thermal conductivity ratio of the surfactant solution at room temperature was similar to concentrations ranging from 0.0073 to 0.029 wt% and that the ratio started to decrease after CTAB surfactant concentrations reached 0.0365 wt% (Figure 5b). At low concentrations of CTAB surfactant, the thermal conductivity ratio remained stable. The cationic surfactant linked to itself in the base fluids while the water molecules bonded to the CTAB surfactant chain influence, networking the water molecules (Zhou et al., 2012). We noted that thermal conductivity decreased due to the dynamic cross-linking and united adjacent chains (Jayasree et al., 2006).

3.4. Sequence Method Analysis for Preparing of TiO_2-Water Nanofluid

To obtain the optimum parameters when preparing of TiO_2–water nanofluid by varying pH values and CTAB surfactant, combinations of preparation methods were investigated to determine the effects of nanofluid thermal conductivity and stability. A summary of the experimental sequence methods for preparing TiO_2–water nanofluid with demineralized water as a base fluid and an 8% volume fraction of TiO_2 NPs is given in Table 2.

To observe the effect of sequences and typical preparation methods in the growth of TiO_2-water nanofluid particle size, estimations were made using a PSA after two weeks the TiO_2-water nanofluids were prepared. Among the seven of sequence procedures (see Table 2), the procedure sequences numbered 2 and 5 showed particle size distributions for the NPs in the TiO_2–water nanofluid system within a narrow range when compared to the other methods. The procedure sequence of 2 is following demineralized water (100 mL), 8% volumetric loading of TiO_2 NPs, ultrasonic time of 10 min and CTAB of 3.2×10^{-3} M, while the procedure sequence of 5 is in the respective order of demineralized water (100 mL), 8% volumetric loading of TiO_2 NPs, ultrasonic time of 10 min and pH at 8.

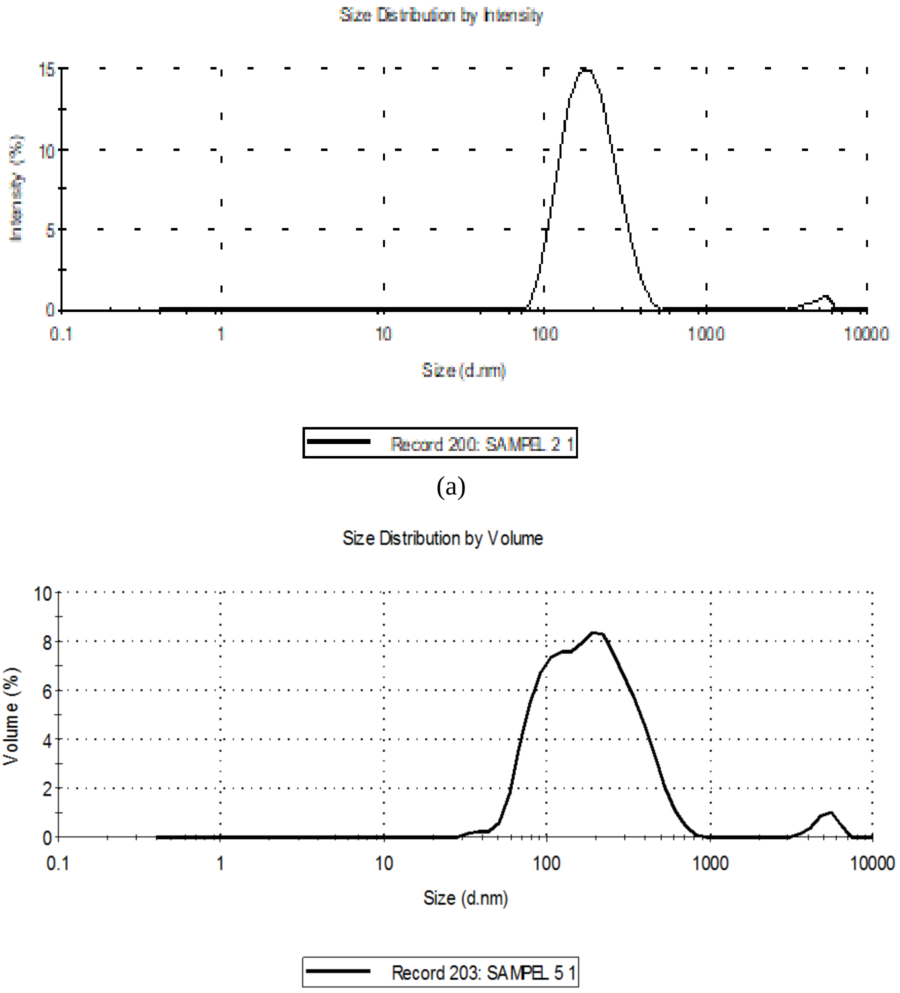
Table 2 The procedure sequence to prepare of TiO_2-water nanofluids

Sequence	Procedure					
	Demineralized water	TiO_2 NPs (8%)	Ultrasonic time (10 min)	CTAB (4× CMC)	pH 8	Particle Size Distribution after 2 weeks (nm)
1	ν (i)	ν (ii)	ν (iii)	-	-	611.6
2	**ν (i)**	**ν (ii)**	**ν (iii)**	**ν (iv)**	**-**	**190.3**
3	ν (i)	ν (ii)	ν (iv)	ν (iii)	-	5,346
4	ν (i)	ν (ii)	ν (iii)	-	ν (iv)	869; 188.1
5	**ν (i)**	**ν (ii)**	**ν (iv)**	**-**	**ν (iii)**	**208.7**
6	ν (i)	ν (ii)	ν (iii)	ν (iv)	ν (v)	1,181
7	ν (i)	ν (ii)	ν (v)	ν (iv)	ν (iii)	2,113

Note: Numbers (i), (ii, iii), (iv), and (v) are to show the sequences of procedure. The bold type indicated the best sequence method in preparing of TiO_2-water nanofluid

Sequence procedure 2 exhibited a particle size distribution of NPs in TiO_2-water nanofluid of 190.3 nm (94.8%; Figure 6a). Sequence procedure 5 showed a particle size distribution of 208.7 nm (96.7%; Figure 6b). The other methods showed the particle sizes distribution in the range of 611 nm to 5.35 μm. We observed that sequence procedure 1 and 4 gave better particle sizes distribution compared to sequence procedures 3 and 7. In sequence procedure 1, we prepared the TiO_2–water nanofluid without adding CTAB surfactant and found that the particle sizes distribution of TiO_2 NPs was 611.6 nm. Here, the pH value was affected more by the particle sizes distribution than the CTAB surfactant. In sequence procedure 4, the particle size distribution had a large range from 188.1 to 869 nm. In sequence methods 4 and 5, we only observed the effect of ultrasonic time and pH without adding CTAB surfactant. Had we used CTAB surfactant and pH value simultaneously, the particle size distribution of TiO_2 NPs would have been higher when compared to the preparation of TiO_2–water nanofluid that only had CTAB surfactant added and/or an adjusted pH value. The deposition and precipitation process was accelerated, causing damage to the micelle system due to the heat from the sonication process (Assael et al., 2005, Murshed et al., 2008). The double layer that formed from the steric stability gave greater force than the double layer formed from electrostatic stability when the pH was adjusted, causing steric stability to form micelles between NPs (Yu & Xie, 2012).

Due to the PZC (Jin et al., 2009) of TiO_2 NPs being an acidic pH position, if we use an acidic condition, TiO_2 NPs immediately precipitate because the surface of the TiO_2 has no charge. Thus, we adjusted the pH value to a base and the surface of TiO_2 NPs absorbed a number of similar anions. This produced a repulsive force among TiO_2 NPs, keeping the TiO_2 NPs away from each other and preventing agglomeration. However, the application of a heat exchange system is not recommended under an acidic pH condition, which can damage the heat pipe.



(a)

(b)

Figure 6 Particle sizes distribution of TiO_2-water nanofluids synthesized by sequence procedures of (a) 2 and (b) 5

4. CONCLUSION

This study was carried out to find a new method for preparing TiO_2–water nanofluid with high thermal conductivity. The optimal conditions for preparing TiO_2–water nanofluid were found to be an addition of 8% volume fraction of TiO_2 NPs, an adjusted pH of 8, and a cationic surfactant of 0.0073–0.029 wt%. The best sequencing methods were obtained for sequence procedure 2 using demineralized water as a base fluid and an 8% volume fraction of TiO_2 NPs, followed by a 10-minute sonication process and the addition of CTAB (0.029 wt%). These results have a great potential for improving the thermal conductivity of TiO_2–water nanofluid, which can be applied as a heat transfer in heat pipes. In conclusion, the addition of CTAB surfactant into the nanofluid can be used to minimize NP aggregation and improve the dispersion behavior of nanofluid.

5. ACKNOWLEDGEMENT

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