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An experimental investigation on the effects of ultrasonication time on stability and thermal conductivity of MWCNT-water nanofluid: Finding the optimum ultrasonication time

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ABSTRACT

The primary objective of the present study is to investigate the possible effects of ultrasonication time on stability and thermal conductivity of MWCNT-water nanofluid. The samples have been prepared in three different solid concentrations of 0.1, 0.3, and 0.5 vol.% applying different ultrasonication times, ranging from 10 to 80 min. The stability of the samples has been investigated over 30 days after preparation by conducting zeta potential analysis and visual observation. It is found that increasing the ultrasonication time until 60 min results in enhancing the stability of the samples in all the solid concentrations while prolonging the ultrasonication time leads to deteriorating the stability. The thermal conductivity of the samples has been experimentally measured over different temperatures ranging from 25 to 60 °C, and it is found that increasing the solid concentration and temperature results in enhancing the thermal conductivity. Moreover, the effects of ultrasonication time on thermal conductivity have also been studied, and it is found that increasing the ultrasonication time leads to a gentle enhancement in thermal conductivity. The maximum conductivity was achieved by applying 60 min ultrasonication. Thus, it is concluded that 60 min ultrasonication is the optimum time in which the thermal conductivity and stability reached their highest point.

1. Introduction

The idea of mixing nanoparticle (NPs) into the conventional working fluids (i.e., water, ethylene glycol, oil, and glycerol) to improve the thermophysical properties and heat transfer performance of the working fluids has been proposed for the first time by Choi and Eastman [1]. Following this pioneering study, many researchers investigated different aspects of nanofluids (NFs), as a new class of working fluids,; thermal conductivity [2–6], dynamic viscosity [7–11], employing different neural networks to predict the thermophysical properties [12–16], and heat transfer performance [17,18]. In recent decade, many researchers also tried to review the recent advances and introducing the knowledge gap in preparation methods [19],viscosity [20,21], thermal conductivity [22], heat transfer of NFs and their applications [23,24], and modeling and simulation of NFs [25,26].

There are numerous merits in dispersing NPs compared to millimeter-sized particles into working fluids such as better stability of NPs into base fluids, lower viscosity increase, higher thermal conductivity enhancement, lower corrosion, and so forth [27]. All the mentioned merits will be achieved in the case that the NFs possess good stability, which means that the particles uniformly distributed in the base fluid with the minimum agglomeration and sedimentation. Thus it is understood that the sample preparation with good stability is the most crucial step in conducting experimental studies on thermophysical properties and heat transfer of NFs.

There are two common methods in preparing the NFs' samples; single-step methods and two-step methods. In both of the methods, applying the ultrasonication is the crucial factor in breaking down the large clusters of the NPs into the smaller ones to achieve a long-time stable NFs. Generally, the ultrasonication process utilizes for the following purposes:

A. De-agglomeration of particles
B. Particle size reduction

Abbreviations: SC, solid concentration; NF, nanofluid; NP, nanoparticle; GA, gum arabic

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Table 1
A summary of the available literature on the effects of ultrasonication time on thermophysical properties and stability of different NFs.

Reference	NF	Ultrasonication time	Studied parameter
Amrollahi et al. [35]	CNT-EG	15 min–24 h	Thermal conductivity and colloidal dispersion
Adio et al. [32]	Al_2O_3-Glycerol	1–8 h	Viscosity
Ghadimi and Metselaar [36]	TiO_2-water	3 h ultrasonic horn/probe and 15 min ultrasonic bath	Stability, particle size, thermal conductivity, and viscosity
Nasiri et al. [37]	Different CNTs-water	45 min ultrasonic horn/probe and ultrasonic bath	Thermal conductivity
Mahbubul et al. [38]	Al_2O_3-water	0–180 min	Colloidal structure and viscosity
Shahsavar et al. [39]	Fe_3O_4-water and Fe_3O_4/MWCNT-water	2.5, 5, 7.5, and 10 min	Stability and thermal conductivity
Mahbubul et al. [40]	Al_2O_3-water	0–5 h ultrasonic horn/probe	Rheological behavior; shear stress, yield stress, consistency index, and a flow behavior index
Mahbubul et al. [31]	Al_2O_3-water	0–5 h ultrasonic horn/probe	Rheological behavior; shear stress, consistency index, and a flow behavior index
Asadi et al. [34]	$Mg(OH)_2$-water	10–160 min	Stability and thermal conductivity
Gangadevi et al. [41]	Cuo-water	1–4 h	Thermal conductivity, viscosity, and thermal and electrical efficiency of a PVT solar collector
Li et al. [42]	Cu-EG	0–75 min	Stability and viscosity

Table 2
Detailed information of MWCNT particles.

Outside diameter	< 7 nm
Inside diameter	2–5 nm
Length	10–30 um
SSA	> 500 m^2/g
Electrical conductivity	> 100 s/cm
True density	2.1 g/cm3
Purity	> 95 wt%

C. Particles synthesis and precipitation
D. Dispersing the particles into the working fluids

There are various parameters in applying the ultrasonication, which affect the stability of the NFs. The most important parameters are as follows:

A. The type of ultrasonic device; Bath or probe
B. Ultrasonication time
C. Ultrasonication power

In this regard, researchers have conducted different experimental studies on the effects of ultrasonication time, power, and type of ultrasonic devices on the stability and thermophysical properties of different NFs [28–30]. Mahbubul et al. [31] studied the effects of ultrasonication time on the stability and rheological properties of Al_2O_3-water NF. They prepared the sample containing 0.5 vol.% spherical Al_2O_3 NPs with a mean diameter of 13 nm. They applied different ultrasonication times of 1, 2, 3, 4, and 5 h using an ultrasonic probe device with the power of 500 W on the pulse mode (2 s ON, 2 s OFF). They observed that prolonging the ultrasonication time leads to enhancing the quality of the colloidal dispersion. They also declared that increasing the ultrasonication time results in decreasing the dynamic viscosity of the NF. The effects of ultrasonication energy on the stability and viscosity of the Al_2O_3-glycol NF has been experimentally studied by Adio et al. [32]. They used the ultrasonic probe device with the power of 200 W to disperse the NPs in different solid concentrations (SCs) up to 5 vol.%. They observed that increasing the sonication time leads to decreasing the viscosity and increasing the stability of the NF. In another experimental investigation, the effects of ultrasonication on stability, dynamic viscosity, and thermal conductivity of a NF containing multi-walled carbon nanotubes (MWCNT) have been studied by Kumar

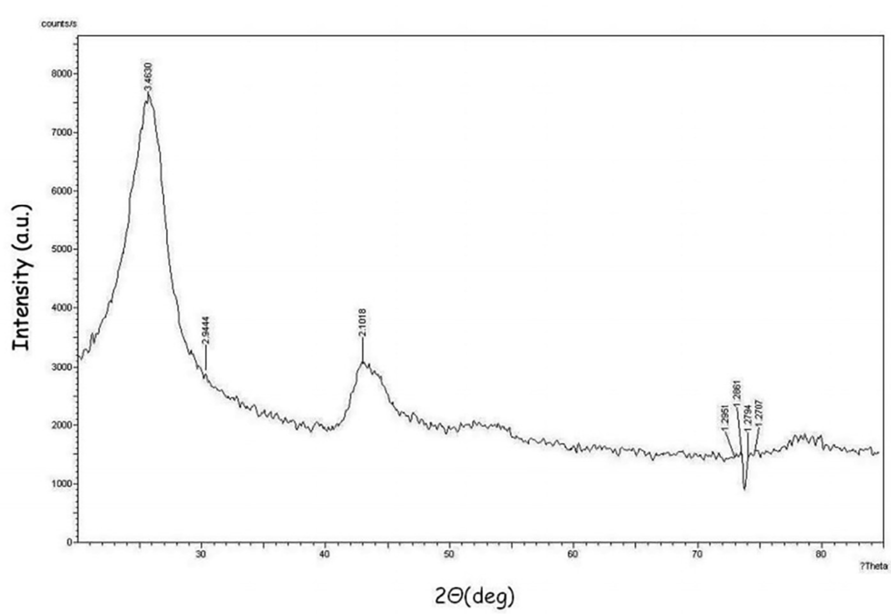

Fig. 1. XRD graph of MWCNT particles.

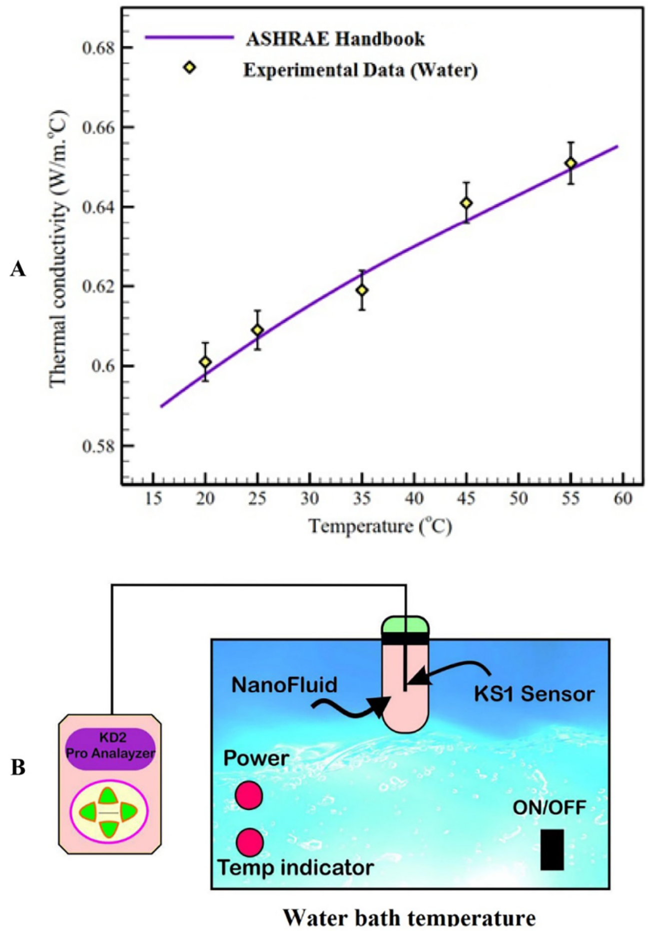

Fig. 2. A) The results of the calibration test by comparing the measured thermal conductivity of water with those available in ASHRAE handbook [55], B) thermal conductivity set-up [56] (Reprinted with the permission from Elsevier with the license number of 4584830863599).

et al. [33]. They also used gum Arabic (GA) surfactant to improve the stability of the samples. Moreover, they applied ultrasonication for different times varied from 30 to 120 min. Investigating the stability of the samples, they used UV–Vis spectrophotometer as a function of sedimentation time. They found that prolonging the ultrasonication time and adding the GA surfactant leads to enhancing the stability of the samples. Moreover, they observed that at the beginning of the ultrasonication process, the dynamic viscosity started to increase, but prolonging the ultrasonication time leads to decreasing the dynamic viscosity. They also observed that adding the GA results in enhancing the thermal conductivity. Asadi et al. [34] studied the effects of ultrasonication time and surfactant addition on stability and thermal conductivity of $Mg(OH)_2$-water NF. They performed the study in different sonication times of 10, 30, 50, 80, and 160 min over different ranges of SCs (0.1–2 vol.%) and temperatures (25–50 °C). They investigated the stability of the NF performing zeta potential analysis over 30 days after the sample preparation. It is observed that in the presence of a surfactant, the optimum sonication time, which leads to the best stability, is 30 min, and after that, the stability of the sample started to deteriorate. They also declared that increasing the sonication time results in decreasing the thermal conductivity of the NF and the maximum thermal conductivity is at the sonication time of 30 min. A summary of the recently published literature on the effects of ultrasonication time on stability and thermophysical properties of different NFs is presented in Table 1.

From what has been discussed, it can be concluded that the ultrasonication process is a crucial and effective step in the preparation of the samples containing sub-micron particles. The literature indicated that ultrasonication leads to breaking down the large clusters of particles into the smaller clusters, which leads to decreasing the sedimentation rate and having the samples with better stability. On the other hand, better stability leads to higher thermal conductivity and lower viscosity of the samples. It is reported that prolonging the ultrasonication process until a specific point results in having a better suspension with minimum sedimentation and maximum thermal conductivity. However, increasing the ultrasonication time (higher than the optimum point) leads to deteriorating the stability and thermophysical properties of the samples.

To the best of the authors' knowledge, there is no comprehensive study of the effects of ultrasonication time on stability and thermal conductivity of nanofluids containing MWCNT particles in low solid concentrations (up to 0.5%), which explains the effects of ultrasonication time, temperature and SC on thermal conductivity. Furthermore, there is no study on the stability of nanofluids containing MWCNT particles over a long period of time (30 days) after the sample preparation. Thus the lack of such a study in the literature is greatly felt. In the present study, the effects of different ultrasonication times on the stability and thermal conductivity of the MWCNT-water NF in three different SCs will be investigated through conducting zeta potential analysis and periodic photography (visual observation) over 30 days after the sample preparation. Then, the thermal conductivity of the samples in three different SCs and over different temperatures will be experimentally measured. Moreover, the effects of ultrasonication time on the thermal conductivity of the samples will be studied and discussed.

2. Materials and methods

2.1. Sample preparation

In the present study, to prepare the samples of NFs, two-step method has been utilized to disperse the MWCNT (US Research Nanomaterials, Inc) particles. The detail information of the NPs has been presented in Table 2, and the XRD graph has been presented in Fig. 1. The average particle size can be calculated based on the Scherrer equation as follows:

$$d = \frac{0.9\lambda}{\beta\cos\theta} \tag{1}$$

where $\boldsymbol{d}$, $\boldsymbol{\lambda}$, $\boldsymbol{\theta}$, and $\boldsymbol{\beta}$ represent the average crystalline size (nm), the wavelength of the incident X-ray, the angle at the maximum peak, and the full width at the half maximum intensity, respectively.

The MWCNT particles have been used in three different SCs of 0.1, 0.3, and 0.5 vol.%. The needed amount of the MWCNT in each solid concentration has been calculated using the following equation:

$$\varphi = \left[\frac{(m/\rho)_{MWCNT}}{(m/\rho)_{MWCNT} + (m/\rho)_{water}}\right] \times 100 \tag{2}$$

where $\boldsymbol{\varphi}$, $\boldsymbol{m}$, and ρ is solid concentration, the mass of particles (kg), and the density (kg/m^3), respectively. After adding the needed amount of the nanoparticles into water, a magnetic stirrer is used for 2 h to disperse the nanoparticles into the base fluid. The effectiveness of magnetic stirrer in the two-step method for preparing the samples of NFs has been proofed and recommended by many researchers [43–45]. Then, the suspension has been subjected to an ultrasonic processor. In the present investigation, the direct ultrasonication device, probe-type device (Hielscher UP400S, 400 W, 24 kHz, Germany), has been employed in seven different times of 10, 20, 40, 60, 70, 75, and 80 min. Mahbubul et al. [28] indicated that the direct ultrasonication device (probe) performs better compared to the indirect ultrasonication (ultrasonic bath device) to disperse dry powders into the base fluids. The

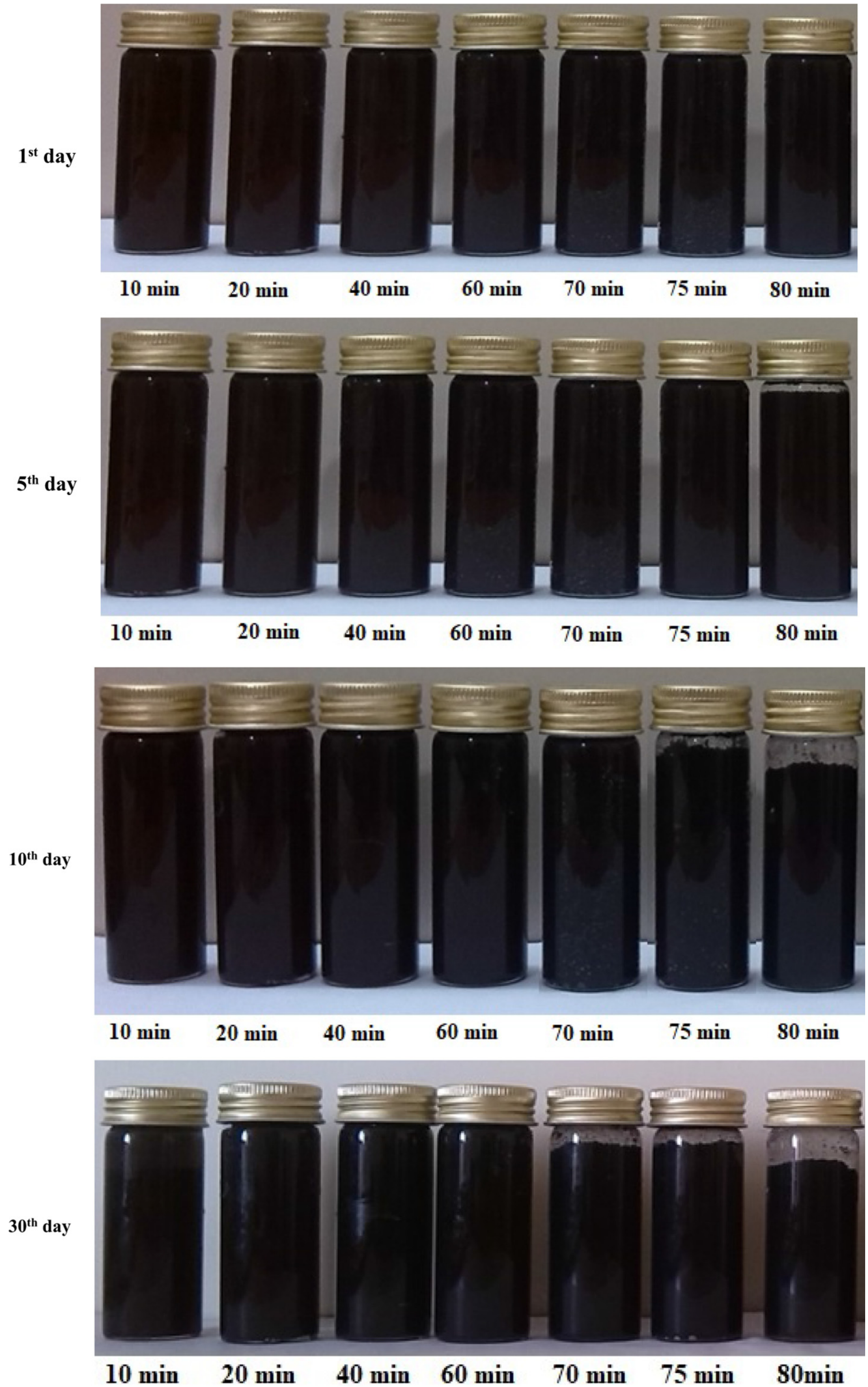

Fig. 3. Stability analysis of the NF samples through visual observation over 30 days after preparation.

literature showed that applying ultrasonication leads to breaking down the possible large agglomeration of particles into the smaller ones and results in having a long-time stable sample with the minimum sedimentation [46–48].

2.2. Stability measurement

There are various characterization techniques to determine the stability of NFs, such as *Electron microscopy*, which focuses on the illumination of electrons to produce an image of the nanomaterial. The results of electron microscopy can determine the size, shape, strength, and ductility of the nanoparticles. Scanning electron microscopy (SEM), which can determine the structure and agglomeration size of particles, and transmission electron microscopy (TEM) are two major types of electron microscopy analysis. Another method to evaluate the stability of nanofluids is *sedimentations techniques*. It is a simple technique which measures the stability of nanofluids by recording the sediment heights over time using high-speed cameras to record the settling velocity of nanoparticles. Then the sedimentations ratio, which is the ratio of the sediment height (H_s) to the total height of the nanofluids samples (H_T),

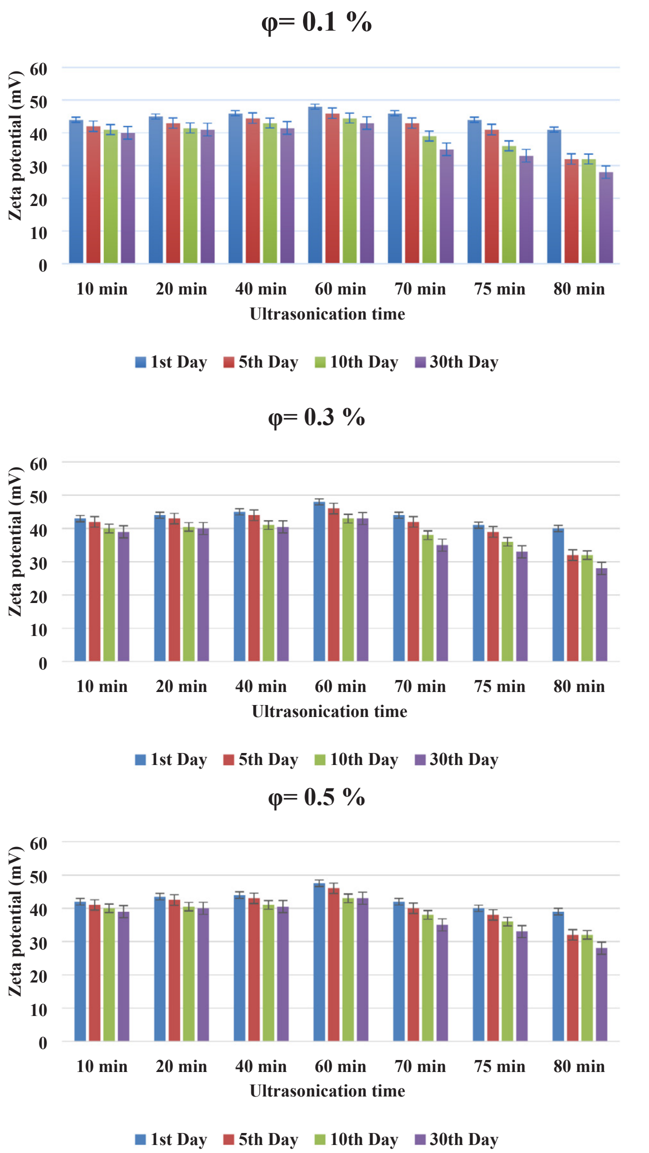

Fig. 4. Variations of the Zeta potential versus ultrasonication time in different days and SCs.

can be determined as follows:

$$SR = \frac{H_s}{H_T} \tag{3}$$

Another method to evaluate the stability of nanofluid is *Ultraviolet (UV)-visible (Vis) spectroscopy,* which operates based on absorption spectroscopy in the UV visible spectral region. There are also other techniques to evaluate the stability of nanofluids. Dynamic light scattering (DLS) is a technique which identifies the average agglomerate size of nanoparticles in nanofluids [49].

Among all the techniques Zeta potential, which measures the effective electric charge on the surface of the suspended nano-sized particles in the working fluids, and visual observation is the two methods which are widely used by researchers [36,50,51]. In the present study, to investigate the effects of ultrasonication time on the stability of the samples, the visual observation and Zeta potential analysis have been performed over 30 days after the preparation of samples.

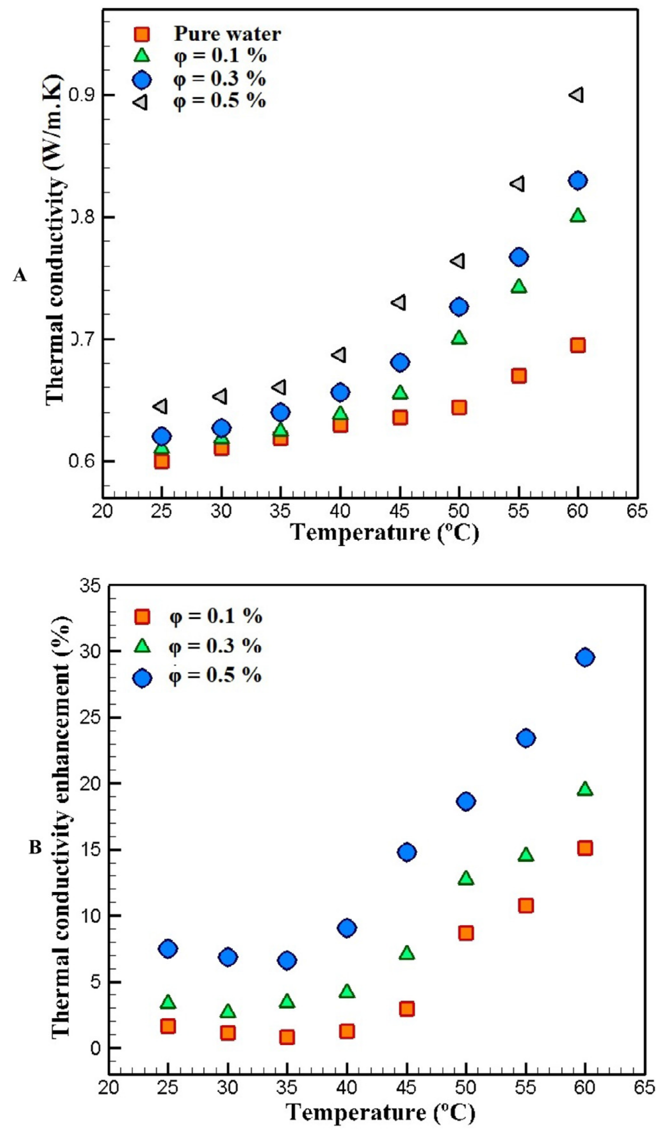

Fig. 5. A) Variations of thermal conductivity and B) the percentage of thermal conductivity enhancement with respect to temperature in different SCs.

2.3. Thermal conductivity measurement

Various techniques and devices have been used thus far to measure the thermal conductivity of NFs. Amongst all the devices and techniques, KD2 Pro thermal analyzer (Decagon device instrument Inc., USA), which works based on the transient hot-wire method, is the widely used devices by researchers [52–54]. It is a handheld device and can be equipped with different sensors. In the present study, the device has been equipped with the KS-1 sensor, which is 6 cm long, and its diameter is 1.3 mm. This sensor has been designed to accurately measures the thermal conductivity of liquid samples and insulating materials with the accuracy of ± 0.01%, and it measures the thermal conductivity in the range of 0.02–2.00 W/m.K. It should be mentioned that the sensor can operate at temperatures ranging from −50 to 150 °C. Making sure the device measures thermal conductivity as accurate as possible, the calibration test has been performed by the glycerol provided by the manufacturer and pure water before starting the experiments. Fig. 2A presents the results of the calibration test by comparing the measured results of the thermal conductivity of pure water and comparing the results with the data available in ASHRAE handbook. It is also very important to keep the samples' temperature as stable as

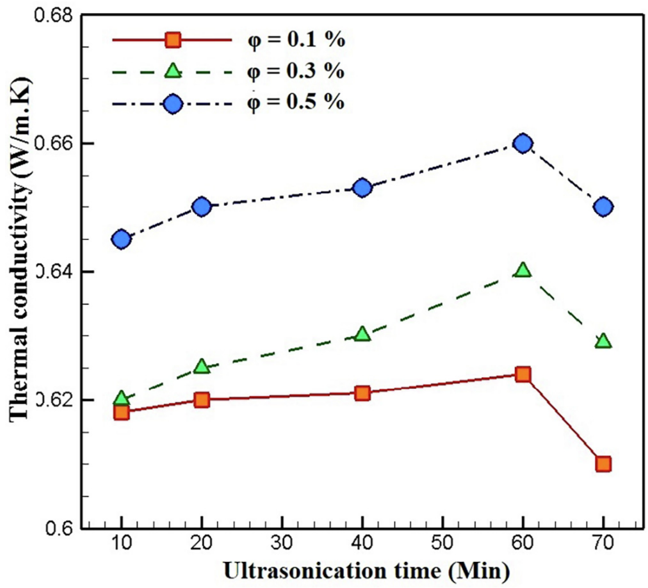

Fig. 6. Variations of the thermal conductivity with respect to ultrasonication time at the temperature of 35 °C and different SCs.

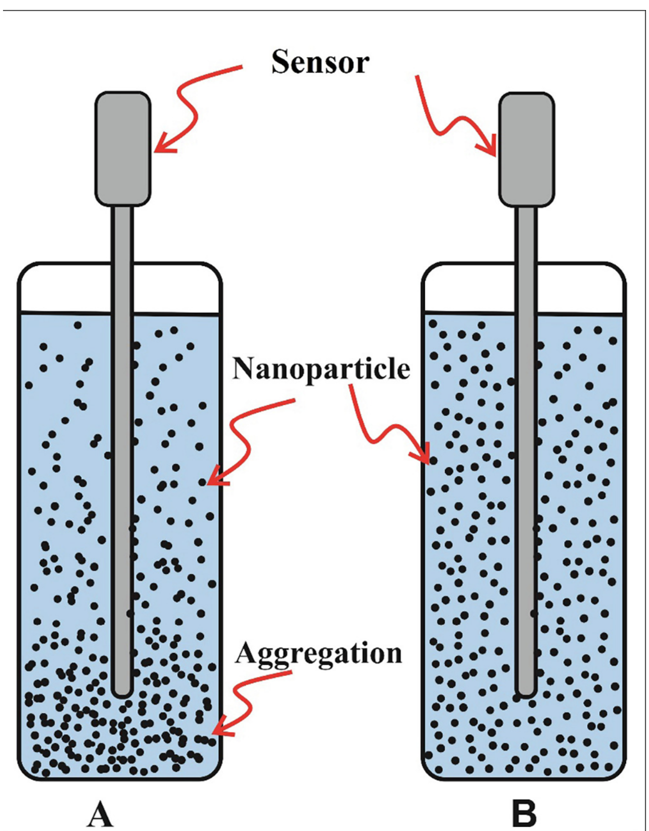

Fig. 7. The schematic view of the responsible mechanism influenced by ultrasonication on thermal conductivity; A) Without or having low ultrasonication time; strong aggregation and larger clusters, B) Higher ultrasonication time; no aggregation [60].

possible during the experiments. To do that, a hot-water bath temperature was employed to control and stabilize the temperature during the experiments. Fig. 2B shows a schematic view of the thermal conductivity set-up. It should also be mentioned that each experiment was repeated three times to investigate the repeatability of the results and the mean values have been recorded.

3. Results and discussion

3.1. Effects of ultrasonication time on the stability of NF

In order to study the effects of ultrasonication time on the stability of MWCNT-water NF, the samples have been prepared without using any surfactant by applying different ultrasonication times of 10, 20, 40, 60, 70, 75, and 80 min. Two methods have been selected to evaluate the stability of the samples over 30 days after preparation; visual observation and Zeta potential analysis. These two methods have been widely used by researchers to investigate the stability of different NFs, [36,50,51,46,47]. Fig. 3 presents the results of the visual observation of the samples' stability in different times after preparation; 1st, 5th, 10th, and 30th day after preparation. As can be seen, all the samples showed good stability on the first day after preparation in all the ultrasonication times. On the 5th day, the MWCNT particles in the sample, which is subjected to 80 min ultrasonication, started to sediment. On the 10th day, the sample subjected to 75 min ultrasonication showed some sedimentation and the samples subjected to 80 min ultrasonication showed more sedimentation rate compared to the 5th day. Finally, after the 30th day, the samples subjected to 10, 20, 40, and 60 min ultrasonication showed good stability without any sedimentation, which can be seen by the naked eyes, while the samples subjected to 70, 75, and 80 min ultrasonication showed a tremendous amount of sedimentation. It should be mentioned that the presented results in Fig. 3 shows the stability analysis of the samples containing 0.3 vol.% MWCNT while for the other SCs, the results showed the same trend as the 0.3 vol.% concentration. It is previously reported by researchers that applying ultrasonication longer than the optimum time leads to having agglomerated particles, which results in increasing the sedimentation rate [36,58,59].

Fig. 4 presents the results of the Zeta potential analysis with respect to ultrasonication time in different SCs. It is stated in the literature that the samples with the Zeta potential lower than 30 mV assumes as limited stability (unacceptable stability), between 30 and 60 assumes as physical stability (good/moderately acceptable stability), and the samples with the zeta potential higher than 60 assumes as excellent stability [28,50]. As can be seen in Fig. 4, the stability of the samples started to enhance as the ultrasonication time applied, and all the samples showed acceptable stability on the 1st and 5th day after preparation expect the samples subjected to 80 min ultrasonication on the 5th day. The best stability achieved by applying 60 min ultrasonication and after that, prolonging the ultrasonication time deteriorated the stability of the samples. After 30th day, it is seen that the samples subjected to 60 min ultrasonication possesses the best stability among the other samples. Thus it can be concluded that the 60 min ultrasonication is the optimum time in which the NF possesses the best stability over the 30 days after preparation. It is also evident that increasing the SC results in having lower Zeta potential values.

3.2. Effects of ultrasonication time on thermal conductivity

Fig. 5A shows the variations of thermal conductivity with respect to temperature in different SCs. As can be seen, the thermal conductivity showed an increasing trend as the temperature increased. The increase is more noticeable in temperatures higher than 45 °C compared to those lowers (> 45). The main reason would be that increasing the temperature results in increasing the Brownian motion of the particles, and, as a result, the thermal conductivity increases. Moreover, adding MWCNT nanoparticles into the water leads to enhancing the thermal conductivity of the NF in all the studied SCs. The maximum enhancement was evidenced at the SC of 0.5% and the temperature of 60 °C by well under 30% (Fig. 5B). This noticeable increase would be because of the high thermal conductivity of MWCNT nanoparticles, which leads to enhancing the thermal conductivity of water-based NF.

Investigating the effects of ultrasonication time on thermal conductivity, the thermal conductivity of the samples subjected to different ultrasonication times has been measured over the 30 days after preparation. Fig. 6 presents the results of the measured thermal conductivity with respect to ultrasonication time at the temperature of 35 °C and different SCs. As can be seen, increasing the ultrasonication time until 60 min results in a gentle increase in the thermal conductivity of the samples, and after that, it started to decrease. This trend has been similar in all the temperatures. Increasing the thermal conductivity by increasing the ultrasonication time has been previously reported in the literature since ultrasonication leads to breaking down the large agglomeration of particles to the less dense agglomeration which can be suspended in the solution [36,57]. Since the samples subjected to 75 and 80 min ultrasonication showed poor stability after 10 days of preparation, as can be seen in Figs. 3 and 4, the measurements have not been done to these samples. The most important reason for enhancing the thermal conductivity by prolonging the ultrasonication time would be the fact that prolonging the ultrasonication leads to breaking down the large clusters of particles into the smaller ones. It would be better understood by the aid of Fig. 7. As can be seen, prolonging the ultrasonication period results in having more homogenous dispersion with the minimum agglomeration of NPs, which leads to enhancing the thermal conductivity [60].

4. Concluding remarks

In the present study, the possible effects of ultrasonication time on the stability and thermal conductivity of MWCNT-water NF has been experimentally investigated. The samples have been prepared in three different SCs of 0.1, 0.3, and 0.5 vol.% subjecting the samples to different ultrasonication times of 10, 20, 40, 60, 70, 75, and 80 min. Furthermore, the thermal conductivity measurements have been done over different temperatures ranging from 25 to 60 °C employing KD2 Pro thermal analyzer. The stability of the samples has been evaluated by conducting the visual observation and Zeta potential analysis in different time steps; 1st day, 5th day, 10th day, and 30th day after the preparation. It is observed that increasing the ultrasonication time until 60 min results in enhancing the stability quality of the samples while prolonging the ultrasonication leads to deteriorating the stability of the samples. Moreover, the thermal conductivity measurements showed that adding MWCNT nanoparticles to the base fluid leads to enhancing the thermal conductivity of the samples. It is also observed that increasing the temperature leads to increasing the thermal conductivity of the samples in all the studied SCs. The thermal conductivity of the samples subjected to different ultrasonication times revealed that the samples with 60 min ultrasonication possesses the highest thermal conductivity. Increasing the ultrasonication time until 60 min results in a gentle increase in thermal conductivity and after that point, it started to decrease. Thus, it is concluded that the 60 min ultrasonication is the optimum time in which the samples possess the highest stability and thermal conductivity.

Based on the achieved results of thermal conductivity, which shows good enhancement by adding a low amount of particle, it is recommended for future investigations to study the effects of ultrasonication time on rheological properties and heat transfer performance in order to present a clear view of the potential of the nanofluid as a heat transfer fluid in practical applications.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ultsonch.2019.104639.

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