Research Article

Effect of the Freeze-Thaw on the Suspension Stability and Thermal Conductivity of EG/Water-Based Al$_2$O$_3$ Nanofluids

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This paper reports the effect of the freeze-thaw on the suspension stability, particle size distribution, and thermal conductivity of EG/water-based nano fluids containing Al$_2$O$_3$ nanoparticles that can be used as improved working fluid for cooling systems. The EG/water-based Al$_2$O$_3$ nanofluids were prepared using a two-step method with a nanodisperser and decanting processes. To investigate the effect of freeze-thaw on the suspension stability and thermal conductivity of nanofluids, the prepared nanofluids were frozen at -32°C for 24 hours using a refrigerating chamber, and then they were completely thawed at room temperature for 24 hours. The suspension stability of the thawed nanofluids was quantitatively analysed for over a day using a Turbiscan. In addition, the particle size distributions and deformation of nanoparticles dispersed in the nanofluids were measured using a particle size analyzer (PSA) and TEM. Also, the thermal conductivity of the nanofluids was measured using a transient hot wire (THW) method in temperature from -10 to 70°C. Based on the results, we show that the suspension stability, thermal conductivity, and particle size of EG/water-based Al$_2$O$_3$ nanofluids were not affected by low temperature.

1. Introduction

The efficiency improvement of thermal management system in many industrial fields is one of the most challenging issues [1–6]. Especially, the efficiency of cooling system inherently has limitation because the working fluids such as EG and PG have relatively low thermal properties [7]. To overcome this problem, many researchers have put a lot of effort into enhancing the thermal properties and the convective heat transfer characteristics of working fluids. One of the solutions is nanofluids which is to disperse the nanoparticles into the basefluid with well suspension stability [8–30]. Since Choi [31], it has been reported that the thermal conductivity of working fluids can be enhanced by dispersing the nanoparticles and nanofluids’ thermal conductivity can be controlled with geometry of nanoparticles. Recently, Kim et al. [26] experimentally showed the thermal conductivity of nanofluids strongly depends on the suspension stability. They prepared three kinds of Al$_2$O$_3$ nanofluids with different particle shapes to make a difference of nanofluids’ suspension stability, and then suspension stability and thermal conductivity of manufactured nanofluids were measured. Based on the results, they showed that even though nanofluids have the same volume fraction, the thermal conductivity of nanofluids is more increased in accordance with suspension stability. Furthermore, the convective heat transfer characteristics of nanofluids have been one of the popular topics. Many researchers [11, 19, 20, 23–25, 28] experimentally presented that the convective heat transfer coefficient is enhanced by nanoparticles. Their results showed the engineering parameters which strongly affect the convective heat transfer characteristics of nanofluids such as volume fraction of nanoparticles, nanoparticles’ size, motion of particles, and suspension stability. Especially, the convective heat transfer coefficient of
nano fluids is higher than that of thermal conductivity enhancement because of nanoparticle’s motion [12, 17]. However, when the aggregation and sedimentation of nanoparticles are occurred due to low suspension stability, the convective heat transfer characteristics of nano fluids cannot be improved. From the previous results, it is well known that the key factor of nano fluids with improved thermal characteristics is the suspension stability [21, 22, 26].

For this reason, many researchers have tried to maintain the suspension stability of nano fluids using a variety of methods such as bath sonication, tip sonication, surface treatment, and use of surfactants [26, 32–35]. Despite these efforts, the suspension stability of nano fluids still has problem according to the temperature. Choi et al. [35] reported the effect of temperature on suspension stability of nano fluids. Water-based CNT nano fluids are prepared with four kinds of surfactants, and they reported that the water-based CNT nano fluids with Triton X-100 dramatically decreased suspension stability at high temperature (85°C). Also, the nano fluids with SDS and CTAB formed precipitates in the bottom of the bottle at low temperature (10°C). Wen and Ding [36] also reported that the water-based CNT nano fluids have poor suspension stability at 69.7°C because of strong aggregation, so they did not evaluate the thermal conductivity of nano fluids due to low suspension stability. Based on their results [26, 32–36], several researchers [35–39] explained the reason why temperature affects the stability of nano fluids in high temperature and low temperature, respectively. At the high temperature region, the suspension stability of nano fluids depends on the aggregation of nanoparticles which is occurred by the particle collision due to the intense movement of individual nanoparticles [37–39]. They reported that the viscosity of base fluid is decreased with temperature and collision between fluid molecules and nanoparticles increases. However, at the low temperature region, surfactants used for dispersion process strongly affect the suspension
stability because the surfactants such as CTAB and SDS formed precipitates in the nanofluids because of reduced solubility [35].

Moreover, when the thermal management devices such as automobile and chiller using nanofluids are exposed to temperatures below the freezing point due to winter season or unexpected situations, it is very important to observe the behavior of suspension stability of nanofluids. To the author’s knowledge, there are no experimental data of the effects of freeze-thaw on the suspension stability and thermal properties of nanofluids. Therefore, it is necessary to investigate the effect of freeze-thaw on the suspension stability of nanofluids.

So, in this paper, we experimentally investigated the effects of freeze-thaw on the suspension stability and thermal properties of nanofluids. For this purpose, we prepared the nanofluids using Al₂O₃ nanoparticles, and antifreeze coolant (ethylene glycol and water at a volume ratio of 1:1) was employed as the basefluid. Especially, we used a two-step method with a nanodisperser and decanting processes to disperse the Al₂O₃ nanoparticles into the basefluid. To observe the effect of freeze-thaw on nanofluids’ suspension stability and thermal conductivity, the prepared nanofluids were frozen at -32°C for 24 hours, and then stored for 24 hours at room temperature in order to thaw it completely. Suspension stability, particle size distribution, deformation of nanoparticles, and thermal conductivity of nanofluids without freeze-thaw and with freeze-thaw were measured by Turbiscan, a particle size analyzer, TEM, and the transient hot wire method, respectively. The comparison results between the nanofluids without freeze-thaw and with freeze-thaw were experimentally presented.
2. Experimental Study

2.1. Manufacturing Processes of EG/Water-Based Al2O3 Nano fluids. Al2O3 nanoparticles were used (D = 40 to 50 nm, Nanophase) to manufacture the EG/water-based Al2O3 nano fluids. The EG/water-based Al2O3 nano fluids were produced using the two-step method with a nanodisperser (NH500-Y100, Ilshinautoclave) and decanting process as shown in Figure 1. The dispersion method using a nanodisperser is a method of passing the fluid and nanoparticles through a micro-orifice nozzle while being pressurized at ultra-high pressure (up to 1500 bar). Base fluid passing through the fine orifice nozzle were atomized and mixed uniformly with the nanoparticles due to the cavitation, turbulence, and high shear stress. Using this method, we obtained 2 liters of nano fluids at 6 volume fractions per unit minute. After that, a three-step decanting method was employed to enhance the suspension stability and thermal properties of nano fluids [40]. Finally, the manufactured nano fluids at 1.43 volume fractions were evaluated for suspension stability, thermal conductivity, and particle size distribution.

2.2. Freeze-Thaw Process of EG/Water-Based Al2O3 Nano fluids. In this study, a thermostatic chamber was used to freeze the nano fluids at -32°C for 24 hours. The freezing temperature of -32°C is based on the US military standard (MIL-STD-810G method 502.5). It provides temperature criteria for evaluating the effect of low temperature conditions on material safety, integrity and performance during storage, operation, and manipulation. Figure 2 shows the frozen EG/water-based Al2O3 nano fluids. To completely thaw the frozen nano fluids, they were stored for 24 hours at room temperature. The nano fluids with freeze-thaw process were compared with the nano fluids without the process. It is difficult to determine whether the suspension stability of nano fluids has changed due to the freeze-thaw process. Because of this, we quantitatively evaluated the effect of the freeze-thaw process on the suspension stability of nano fluids using Turbiscan.

2.3. Suspension Characterization of EG/Water-Based Al2O3 Nano fluids. The suspension stability of EG/water-based Al2O3 nano fluids was quantitatively evaluated by Turbiscan (Turbiscan Lab, Fullbrook Systems Ltd.) which can be widely used to evaluate the suspension stability of nano fluids in previous researches [41–46]. Turbiscan can measure the intensity of the transmitted light through the nano fluids with elapsed time using the emitted wavelength of 880 nm. Based on the data, TSI (Turbiscan stability index) as given by equation (1) was calculated according to the sample’s height with elapsed time.

\[
TSI = \sum_{i} \frac{\sum_{h=T_i(h)-T_{i-1}(h)}}{H},
\]

where \(i, h, T_i, \) and \(H\) are measured time, measured position, transmittance of light, and total sample height, respectively. If the nano fluids have good stability, the TSI is nearly not changed with the elapsed time. Moreover, the large values of TSI indicate lower suspension stability.

2.4. Thermal Conductivity of EG/Water-Based Al2O3 Nano fluids. To investigate the effect of the freeze-thaw on the thermal conductivity of the nano fluids, it is measured by transient hot wire method in temperature range from -10°C to 70°C [47, 48]. To measure the thermal conductivity over a wide temperature range, including high temperatures, two challenge issues must be solved. The first is to maintain the tension of a hot wire because the tension of a hot wire is easily changed due to the temperature variation in measurement device. So, it is very important to maintain constant tension of a hot wire (platinum wire) when...
measuring thermal conductivity by using transient hot wire method. Second, the generation of non-condensable gases inside the fluid should be restrained. Non-condensable gases inside the fluid are generated and attached at the hot wire surface. Bubbles on the surface of the hot wire dramatically decrease the accuracy of thermal conductivity measurement at high temperature (>40°C). So, two challenge issues must be solved to measure the accurate thermal conductivity.

To overcome these challenges for measuring the accurate thermal conductivity of nanofluids, we employed spring tensioner and pressure vessel as shown in Figure 3. A spring tensioner maintains the tension of the hot wire (platinum wire) even under a wide temperature change. Moreover, pressure vessel pressurizes the fluid to 5 bars to suppress bubbles at high temperatures.

The validation of the transient hot wire system was conducted using EG as well as DI water, and it had 1% uncertainty as shown in Figure 4 [49, 50].

3. Results and Discussion

3.1. Suspension Characterization of EG/Water-Based Al2O3 Nano fluids. Figure 5 shows the measured transmittances for the two kinds of nanofluids (without freeze-thaw and with freeze-thaw) according to the sample’s height at room temperature (approximately 25°C). The transmittances of two types of nanofluids were not significantly different according with the sample’s height. It means that the freeze-thaw process does not have a sharp influence on the suspension stability of the nanofluids.

Moreover, we measured transmittances of two kinds of nanofluids with elapsed time to evaluate the long-term suspension stability of nanofluids. With the data, TSI values are shown in Figure 6. Both nanofluids have a TSI variation of less than 1% and their suspension stability is well maintained for 24 hours. Based on the result, the freeze-thaw process does not affect the suspension stability of nanofluids.

3.2. Particle Size Distribution and Particle Deformation of EG/Water-Based Al2O3 Nano fluids. Many researchers reported that suspension stability of nanofluids and particle agglomeration are closely related [35, 36, 51–53]. Moreover, they noted that particle agglomeration can affect the properties of nanofluids such as thermal conductivity, viscosity, and optical properties. Because of this, we also measured the particle size distribution suspended in the nanofluids with particle size analyzer (Zetasizer Nano S90, Malvern). Figure 7 shows the particle size distributions of two types of the nanofluids, respectively. The average particle size of EG/water-based Al2O3 nano fluids before freeze-thaw process was 67.63 nm and the size of nanofluids after freeze-thaw was 68.07 nm. Moreover, the particle size distribution is similar between the two nanofluids. This result indicates that the freeze-thaw process of nanofluids did not affect the particle agglomeration nor the suspension stability.

Moreover, TEM images were employed to observe the deformation of particles. We prepared TEM samples using special method. First, the nanofluids were diluted with base-fluid, and then moisture of diluted nanofluids were eliminated by paper towel. The remaining moisture was completely removed through natural drying at room temperature. This process can minimize the agglomeration of nanoparticles during the drying process. As shown in Figure 8, any particle deformation between the two types of nanofluids was not observed.

3.3. Thermal Conductivity of EG/Water-Based Al2O3 Nano fluids. To evaluate the effect of freeze-thaw on thermal conductivity of nanofluids (without freeze-thaw and with freeze-thaw), we measured the thermal conductivity of the two types of nanofluids using transient hot wire method. As shown in Figure 9, the thermal conductivity of both nanofluids nearly is the same and within the error range of the transient hot wire system. It clearly shows that the freeze-thaw process does not affect the thermal conductivity of the EG/water-based Al2O3 nano fluids. In addition, it is reasonable because the particle size distribution as well as suspension stability was not affected by the freeze-thaw process of nanofluids.
4. Conclusions

This investigation experimentally presents the effect of freeze-thaw on the suspension stability, the particle size distribution, and the thermal conductivity of EG/water-based nanofluids containing Al$_2$O$_3$ nanoparticles that have been used as a working fluid for various cooling systems. The EG/water-based Al$_2$O$_3$ nanofluids were prepared using a two-step method with a nanodisperser and decanting processes. To evaluate the suspension stability of the nanofluids with freeze-thaw process and without the process quantitatively, Turbiscan was employed to measure the intensity of the transmitted light through the nanofluids with elapsed time and TSI value was used to analyze it. It is shown that both nanofluids with and without the freeze-thaw process have well maintained their suspension stability for 24 hours. Also, particle size distributions and particle deformation in both nanofluids are experimentally presented with PSA and TEM. We observed that the particle deformation in both nanofluids were almost similar regardless of the freeze-thaw process. Moreover, particle deformation also was not observed. The thermal conductivity of the two types of nanofluids were measured using the transient hot wire method at the temperature range -10 to 70°C, respectively. The thermal conductivities of both nanofluids also did not changed by freeze-thaw process. With the experimental results, we clearly show that the freeze-thaw process did not affect the particle size dispersed in nanofluids, the suspension stability, nor the thermal conductivity of nanofluids.

Data Availability

The data in Figures 4–9 used to support the findings of this study are included within the supplementary information files as follows: Supplementary 1 (data in Figure 4); Supplementary 2 (data in Figure 5); Supplementary 3 (data in Figure 6); Supplementary 4 (data in Figure 7); and Supplementary 5 (data in Figure 9).

Conflicts of Interest

The authors of this work declare that there are no conflicts of interest regarding the publication of this paper.

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Supplementary Materials

Supplementary 1. It includes experimental data for thermal conductivities measured by the transient hot wire method as shown in Figure 4.

Supplementary 2. It includes experimental data for transmittance measured by the Turbiscan as shown in Figure 5.

Supplementary 3. It includes experimental data for TSI values measured by the Turbiscan as shown in Figure 6.

Supplementary 4. It includes experimental data for PSA results measured by the particle size analyzer as shown in Figure 7.

Supplementary 5. It includes experimental data for thermal conductivities measured by the transient hot wire method as shown in Figure 9.

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