



Synthesis and Thermo-physical Properties of Fe₃O₄ Nanofluid

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Abstract

The purpose of this study is to investigate synthesis and thermo-physical properties including thermal conductivity and viscosity of Fe₃O₄ Nanofluid. In this study, Fe₃O₄ nanoparticles with the size of 20 nm were prepared. The nanoparticles were characterized by X-ray powder diffraction (XRD) analysis and transmission electron microscopy (TEM). Additionally, the effect of many parameters on the Fe₃O₄ nanoparticles was studied. The thermal conductivity and viscosity of nanofluids are measured and it is found that the viscosity increase is substantially higher than the increase in thermal conductivity. All of the property thermal conductivity, electrical conductivity and viscosity of nanofluids increase with the nanoparticle volume concentration. Theoretical models are developed to predict thermal conductivity and viscosity of nanofluids without resorting to the well established Maxwell and Einstein models, respectively. The proposed models show reasonably good agreement with our experimental results.

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Introduction

Several methods have been proposed to enhance the thermal performance of the heat transfer devices. The most common technique has been using nanofluids in heat transfer devices. Nanofluids are suspensions of nano-scale solid particles (1–100 nm) in liquids. These nanofluids may be used in different applications, for example, engine cooling, engine transmission oil, cooling electronics, refrigeration, drilling, lubrications, thermal storage, and so on [1]. Recently, several papers have been published on the synthesis and characterization of these nanofluids, but they are often not coherent and the results are inconsistent. This could be due to the difference in their preparation methods and, consequently, on their stability. There are two main procedures to prepare nanofluids, one is the one-step method in which the particles are directly synthesized into the fluid, and the second is the two-step method involving dispersion of nanopowders in the based fluid [2]. These methods influence the stability and properties of these fluids [3]. Nanoparticles have high-energy surface and their surface properties and chemistry control their behaviour [4]. In aqueous environments, there is a tendency for nanoparticles to aggregate, to reduce the particle surface energy. It depends on a number of factors including surface functionalization, pH, and ionic strength. For this reason, a pH optimization is fundamental [5], and often different dispersants and surfactants are added to the nanofluids [6]. Moreover, different methods of nanoparticles dispersion into the base fluid can result in a different stability [7, 8]. For this reason, before studying thermophysical properties, a stability investigation must be done. In literature, lots of works have been done on the thermal conductivity improvement of nanofluids [9–11], but some results are controversial and the mechanisms behind exceptional

conductivity enhancement is still not well understood. Contrarily, viscosity data in literature are still scarce although this property is fundamental as well as thermal conductivity. Too high viscosity increase can nullify the positive impacts of thermal conductivity enhancement. Oxide nanoparticles are less expensive than other nanoparticles and their synthesis are easy. Among different nanoparticles, Fe₃O₄ is already used to produce stable and commercially available water nanofluids, but, at our knowledge, no thermal conductivity data and only few experimental viscosity data are available in the literature. Therefore, in this investigation, we study the effect of temperature and nanoparticles concentration on thermal conductivity and dynamic viscosity of Fe₃O₄ water-based nanofluids. We compare our results with some literature models, and we propose an experimental correlation for nanofluids viscosity.

Experimental

Preparation of Fe₃O₄ nanoparticles

The process for preparing Fe₃O₄ nanoparticles was controlled chemical coprecipitation. First, 1.99 g (0.01 mol) FeCl₂ · 4H₂O, 5.41 g (0.02 mol) FeCl₃ · 6H₂O was dissolved in 50 mL distilled water, aqueous ammonium hydroxide (25–28%, w/w) solution (1.5 mol/L) was also obtained as this. Then, a certain surfactant (sodium oleate or PEG-6000) was added to the former solutions to obtain Precursor solution II and Precursor solution I. Second, Precursor solution I was added into Precursor solution II dropwise with strong stirring under the protection of dry nitrogen at the desired temperature. Just after mixing the solutions, the color of the solution changed from light brown to black, indicating the forming of Fe₃O₄ nanoparticles.

Thermal Conductivity Measurement Apparatus

A TPS 2500 S was used for thermal conductivity measurements. The instrument is based on the hot-disk technique and can measure thermal conductivity and thermal diffusivity of several materials. The hot disk sensor consists of a double spiral of thin nickel wire and works as a continuous plane heat source. During the experiment, a small constant current is supplied to the sensor, which also serves as a temperature sensor, so that the temperature increase in the sensor is accurately calculated through resistance measurement. The temperature increase is registered over a short period of time after the start of the experiment. A theoretical description of this method is provided by [12]. A proper box containing the sensor and the fluid was put in a water thermostatic bath to reach the test temperature. The power supplied for each measurement was 30mW, and the time of the power input was 4 s. The declared instrument uncertainty is 5%.

Dynamic Viscosity Measurement Apparatus

The dynamic viscosity data were measured at ambient pressure and in a temperature range between 20-60°C, with steps of 5°C, by means of an AR-G2 rheometer (TA Instruments). It is a rotational rheometer with magnetic bearing for ultra-low nanotorque control. The suitable used geometry is a plate-cone type, with a 1°C one and diameter of 40 mm. In order to stabilize the measurement temperature, a proper device (Upper Heated Plate) was used. A critical point in this measurement is the sample loading. After some trials with water, a constant quantity of about 0.34mL was considered optimal for the analysis. The sample was deposited using a pipette, taking care no air bubbles were inside. Before the measurements, the rheometer was carefully calibrated at each temperature, as fully explained in [13].

Data Reduction

In the present study, the Fe₃O₄ nanoparticles dispersed in water with volume concentrations of 0.2, 0.4, 0.6 and 0.8v/v% were used. During the test, cold water absorbed heat from hot nanofluid. The heat transfer rate from the heating fluid was calculated from the following equation:

$$\rho_{\text{nanofluid}} = (1 - \varphi)\rho_{\text{water}} + \varphi\rho_{\text{Fe}_3\text{O}_4} \quad (1)$$

$$C_{\text{nanofluid}} = (1 - \varphi)C_{\text{water}} + \varphi C_{\text{Fe}_3\text{O}_4} \quad (2)$$

Timofeeva et al.[14] suggested the effective medium theory to calculate thermal conductivity:

$$k_{\text{nanofluid}} = (1 + 3\varphi)k_{\text{water}} \quad (3)$$

The Wasp model [15] predicts results similar to those of Hamilton– Crosser under spherical particles.

$$k_{\text{nanofluid}} = \left[\frac{k_{\text{Fe}_3\text{O}_4} + 2k_{\text{water}} - 2\varphi(k_{\text{water}} - k_{\text{Fe}_3\text{O}_4})}{k_{\text{Fe}_3\text{O}_4} + 2k_{\text{water}} + \varphi(k_{\text{water}} - k_{\text{Fe}_3\text{O}_4})} \right] k_{\text{water}} \quad (4)$$

$$K_{\text{nanofluid}} = K_{\text{water}} \left[\frac{1 + 2\varphi \left(\frac{1 - \frac{K_{\text{water}}}{K_{\text{Fe}_3\text{O}_4}}}{2 \frac{K_{\text{water}}}{K_{\text{Fe}_3\text{O}_4}} + 1} \right)}{1 - \varphi \left(\frac{1 - \frac{K_{\text{water}}}{K_{\text{Fe}_3\text{O}_4}}}{\frac{K_{\text{water}}}{K_{\text{Fe}_3\text{O}_4}} + 1} \right)} \right] \quad (5)$$

Brinkman [16] suggested an equation for calculating the viscosity of the suspension, which is defined as follows:

$$\mu_{\text{nanofluid}} = \frac{1}{(1 - \varphi)^{2.5}} \mu_{\text{water}} \quad (6)$$

$$\mu_{\text{nanofluid}} = (1 + 2.5\varphi) \mu_{\text{water}} \quad (7)$$

A correlation for calculating the viscosity of nanofluids with spherical shape nanoparticles is defined as:

$$\mu_{\text{nf}} = (1 + 2.5\varphi + 6.2\varphi^2) \mu_{\text{w}} \quad (8)$$

Results and Discussion

Thermal conductivity

The measurements accuracy was assessed measuring water thermal conductivity at each temperature. Thermal conductivity measurements were performed in the temperature range between 20°C and 60°C, with steps of 5°C. Figure 1 presents the base fluid thermal conductivity as a function of temperature, showing an almost linear enhancement with temperature at all the concentrations. In this figure, experimental data and obtained reference data for thermal conductivity of water are compared also. Results show a good agreement between experimental data and reference data. Figures 2 and 3 represent experimental data and obtained data from Maxwell model for thermal conductivity of Fe₃O₄ nanofluid with different volume concentrations of 0.0%, 0.2%, 0.4%, 0.6% and 0.8% in the temperature range between 20-60°C with steps of 5°C, respectively.

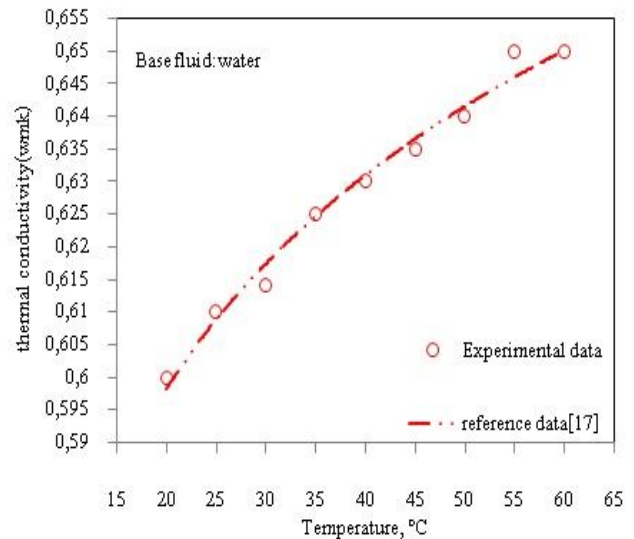


Figure 1: Experimental data and reference data for thermal conductivity of base fluid in the temperature range between 20-60°C with steps of 5°C.[17]

Dynamic Viscosity

To evaluate the rheometer performance, preliminary tests were performed on a well-known fluid as water. Figure 4 shows the experimental data and obtained values from Einstein model for the base fluid viscosity in the temperature range between 20-60°C with steps of 5°C. Figures 5 and 6 represent experimental data and obtained data from Einstein model for viscosity of Fe₃O₄ nanofluid with different volume concentrations of 0.0%, 0.2%, 0.4%, 0.6% and 0.8% in the same temperature range as base fluid, respectively. In general, as found in figures 4-6, for Fe₃O₄ nanofluid and base fluid, experimental data and theoretical values are in good agreement.

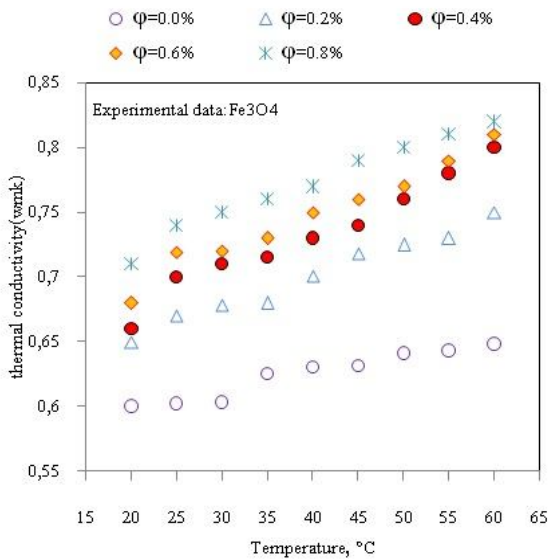


Figure 2: Experimental data for thermal conductivity of Fe₃O₄ nanofluid with different volume concentrations of 0.0%, 0.2%, 0.4%, 0.6% and 0.8% in the temperature range between 20-60°C with steps of 5°C.

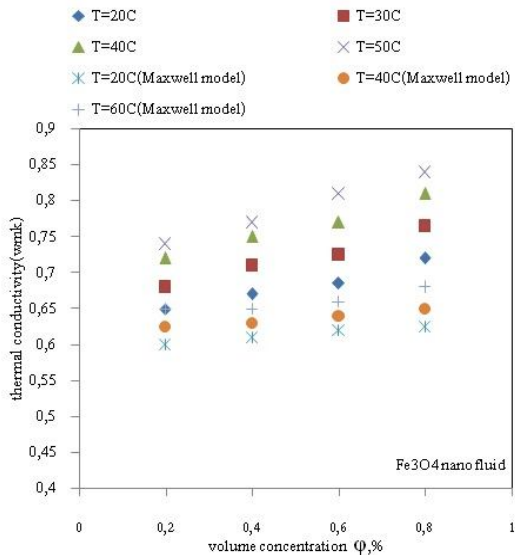


Figure 3: Obtained data from Maxwell model for thermal conductivity of Fe₃O₄ nanofluid with different volume concentrations of 0.0%, 0.2%, 0.4%, 0.6% and 0.8% in the temperature range between 20-60°C with steps of 5°C.

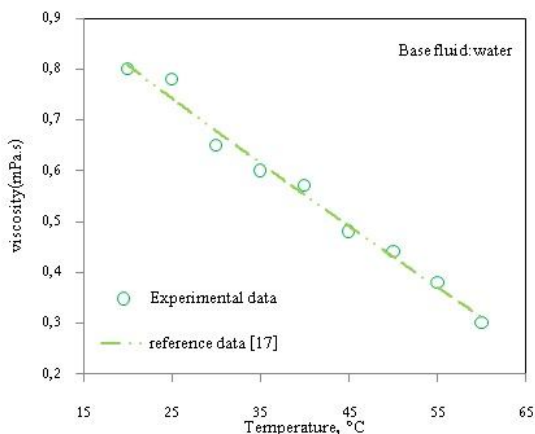


Figure 4: Experimental data and Einstein data for viscosity of base fluid in the temperature range between 20-60°C with steps of 5°C.[17]

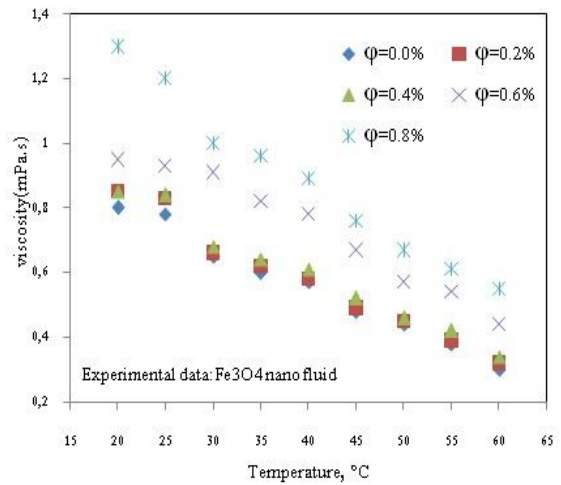


Figure 5: Experimental data for viscosity of Fe₃O₄ nanofluid with different volume concentrations of 0.0%, 0.2%, 0.4%, 0.6% and 0.8% in the temperature range between 20-60°C with steps of 5°C.

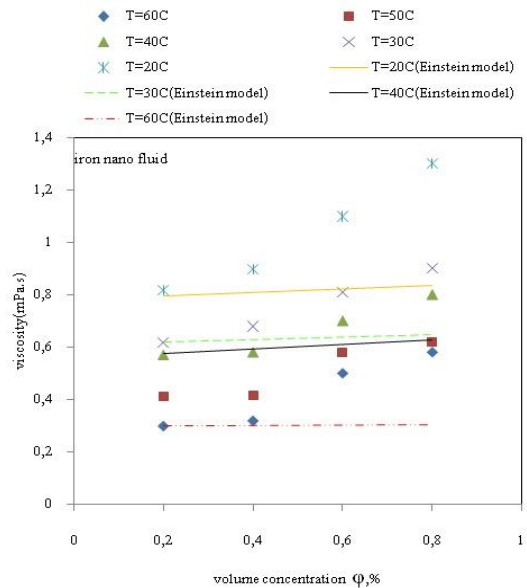


Figure 6: Obtained data from Einstein model for viscosity of Fe₃O₄ nanofluid with different volume concentrations of 0.0%, 0.2%, 0.4%, 0.6% and 0.8% in the temperature range between 20-60°C with steps of 5°C.

Electrical Conductivity

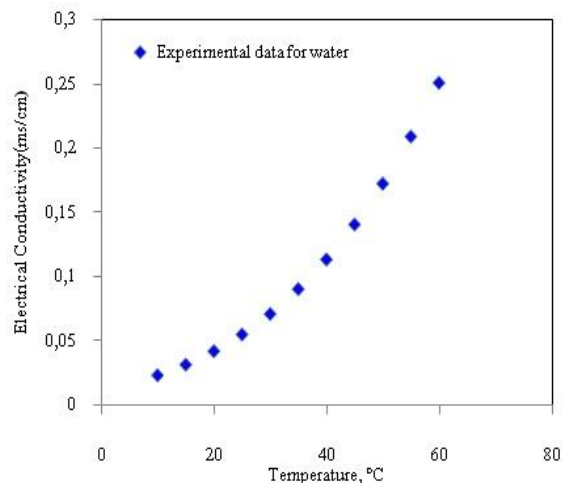


Figure 7: Experimental Electrical Conductivity (ms/cm) for water.

Figure 7 shows the experimental electrical conductivities for base fluid in the temperature range between 20-60°C with steps of 5°C. Electrical conductivity measurements for Fe_3O_4 nanofluid were performed at different volume concentrations of 0.0%, 0.2%, 0.4%, 0.6% and 0.8% in the same temperature range as the base fluid. Figures 8 and 9 shows the experimental data and obtained theoretical values for electrical conductivity of Fe_3O_4 nanofluid.

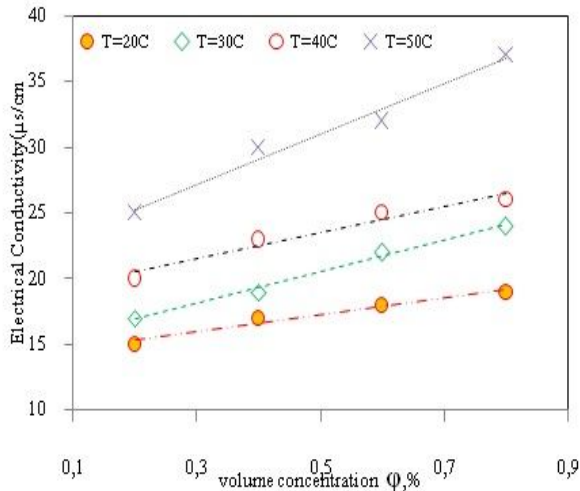


Figure 8: Electrical Conductivity data of nanofluid iron oxide

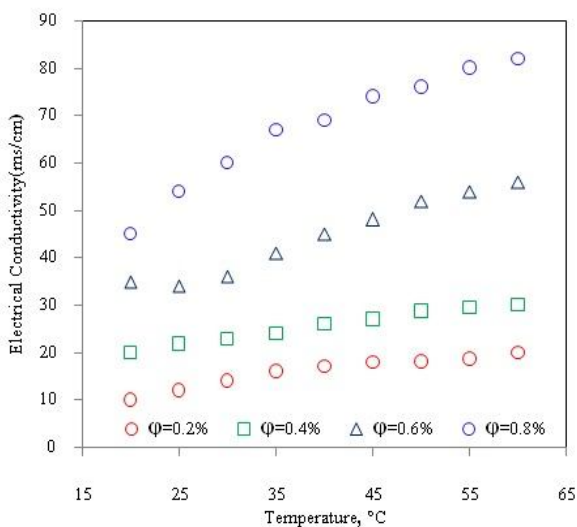


Figure 9: Theoretical Electrical Conductivity data of nano fluid iron oxide in different temperatures.

TEM image of Fe_3O_4 nanoparticles

Figure 10 shows the typical transmission electron microscope (TEM) image of Fe_3O_4 nanoparticles, from which we can see that the sizes of Fe_3O_4 nanoparticles are almost uniform and most of Fe_3O_4 nanoparticles are approximately spherical with the mean diameters (D_v) of 20 nm.

X-ray power diffraction

Figure 11 shows the XRD pattern of the sample, indicating that the sample has a cubic crystal system. Also, we can see that no characteristic peaks of impurities were observed. The mean particle diameters were also calculated from the XRD pattern.

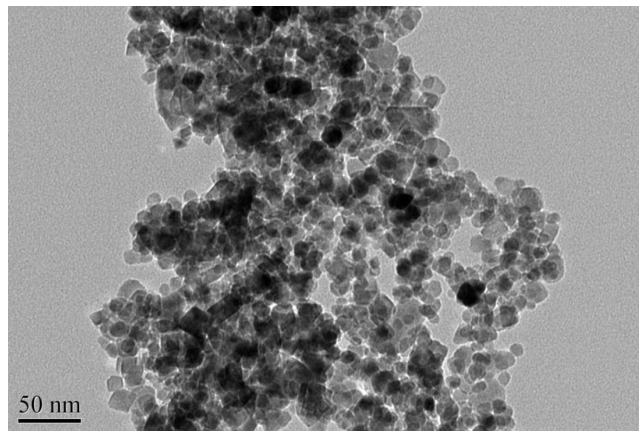


Figure 10. TEM image of Fe_3O_4 nanoparticles

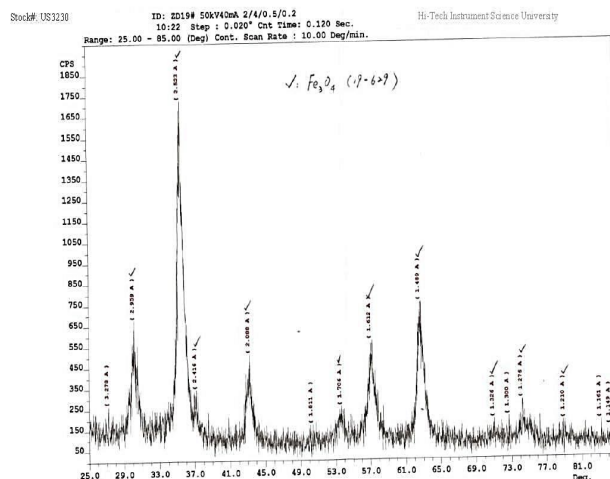


Figure 11. XRD pattern of Fe_3O_4 nanoparticles

Conclusions

Ultrafine, uniform, nearly spherical, and high purity Fe_3O_4 nanoparticles could be prepared by the Controlled chemical coprecipitation method from the solution of ferrous/ferric mixed salt-solution in aqueous ammonium hydroxide ($NH_3 \cdot H_2O$) solution when sodium oleate was chosen as the apt surfactant. The Fe_3O_4 nanoparticles have a perfect biocompatibility and can also be well dispersed in an aqueous solution as the presence of COO₋ at the surface of magnetite nanoparticles. The results show that Fe_3O_4 nanoparticles can be produced in the sizes range from 20 nm by changing the operational parameters. Fe_3O_4 water-based nanofluids have long time stability also at high concentration as 0.8v/v%. Thermal conductivity increases with mass fraction and with temperature. Thermal conductivity ratio is greater at the highest concentrations. The rheological behaviour of the nanofluids is Newtonian, and the dynamic viscosity increases considerably in respect of water, mainly at volume fraction of 0.8v/v%. Hence, the increment in thermal conductivity is combined with a rising in dynamic viscosity.

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