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Thermo-physical properties measurement of water based Fe_3O_4 nanofluids

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Two-step method was used to prepare nanofluids in which nanoparticles were first prepared and then dispersed in basefluid. This paper presents the effect of different volume fractions of magnetic Fe_3O_4 on thermal conductivity and viscosity of water based nanofluid. Thermal conductivity and viscosity of nanofluids having volume fractions in the range of 0.00375 - 0.1 were measured with KD2 Pro based on transient hot-wire method and AR-G2 rheometer respectively. The experimental results show that the thermal conductivity increases with an increase of particle volume fraction and are found that that they have noticeably 29 % higher thermal conductivities than the water. Viscosity of the nanofluid was found to be greater than the base fluid and it increased with the nanoparticle concentration in the base fluid. It was observed that shear rate has no effect on viscosity besides this all nanofluids composition showing Newtonian behavior and shear stress increases with increase in volume fraction.

Introduction: For more than a century, many scientists and engineers have made great efforts to improve various thermo physical properties of traditional heat transfer fluids such as water, oil and ethylene glycol. These fluids play very important role in energy saving systems and which is most important these days. It significantly leads a wide range of application area such as district heating and cooling (DHC) system, energy supplies and production, chemical industries, automobiles, microelectronics, and other micro-sized applications. These fluids have relatively poor thermal conductivity, so several methods have been proposed to enhance their heat transfer performance [1–3]. Since solid materials have higher thermal conductivity compared with those of fluids [4, 5], researchers have tried to improve the thermal conductivity of conventional fluids by suspending solid particles in them. These thermo properties can be enhanced by adding relatively small amount of milli or micro-sized particles into them. The main problem with these milli- or micro-sized particles is that they settle down and they clog the channels of the tube walls. A great deal of work has been done by many researchers to prepare heat transfer fluids with higher thermal conductivities. An innovative way for enhancing the thermal conductivity of fluids is to add particles of nano-size to the fluids known as nanofluids which were first synthesised by Choi at Argonne National Laboratory of USA [7] in 1995.

Nanofluids are solid-liquid composite materials consisting of solid nanoparticles or nanofibers with sizes typically of 1 to 100 nm suspended in liquid. The Nano-fluid is not a simple liquid-solid mixture; the most important criterion of nanofluid is agglomerate-free stable suspension for long duration without causing any chemical changes in base fluid. The advantages of nanofluids are (1) better stability compared to fluids containing micro-or milli-sized particles and (2) higher thermal conductivity than base fluids [6]. A number of recent experiments on nanofluids have indicated dramatic improvements in the effective static thermal conductivity of these fluids compared to their base fluids [7, 9, 11-14]. For example, Xuan and Li [8] directly mixed Cu nanoparticles in about 100 nm diameter with deionized water to form nanofluid. Their studies show that the ratio of the thermal conductivity of the distilled water based Cu nanofluid to that of the base fluid varies from 1.24 to 1.78 when the volume fraction of the nanoparticles increases from 2.5 vol%

to 7.5 vol%. By adding 0.3 vol% Cu nanoparticles with mean diameter smaller than 10 nm into ethylene glycol and after stably suspending, Eastman et al. [9] studied the effective thermal conductivity of ethylene glycol based Cu nanofluids, and got 40% enhancement of thermal conductivity as compared with its base fluid. Many other metallic or non-metallic nanoparticle formed nanofluids also show a significant increase in thermal conductivity compared with their base fluids [10].Also nanofluids containing small amounts of nanoparticles such as SiC, Al_2O_3, TiO_2 and CuO have shown enhanced thermal conductivity [15-24]. Lee et al. [13] reported an increase in thermal conductivity ratio of 14% compared to pure water with 3.5 vol. % CuO particles, while Eastman et al. [25] obtained a 40% increase at the same volume fraction. Duangthongsuk and Wongwises [24] experimentally found that the TiO_2 nanoparticles dispersed in water gave 3–7% greater thermal conductivity than water with volume concentration ranging between 0.2% and 2.0%.

While these thermal properties are important for heat transfer applications, the viscosity is also important in designing nanofluids for flow and heat transfer applications because the pressure drop and the resulting pumping power depend on the viscosity. Compared to the works on thermal conductivity of nanofluids, only a few studies have been reported on the rheological behavior of nanofluids. Kang et al. [26] measured the viscosities of UDD (ultra dispersed diamond)/ethylene glycol, silver/water, and silica/water nanofluids. They found that the viscosity increase was 50% for UDD/EG nanofluid, 30% increase for silver/water and 20% increase for silica/water nanofluids at volume concentrations of 1%, 2% and 3%, respectively. Prasher et al. [27] demonstrated the viscosity of alumina/propylene glycol (PG) nanofluids was independent of shear rate, proving that the nanofluids are Newtonian in nature and increases as nanoparticle volume concentration increases. They found a 30% increase in viscosity at 3% volume concentration and attributed this increase to aggregation of the nanoparticles in the nanofluid with the size of the aggregates around three times the size of the individual nanoparticles. Murshed et al. [28] measured relative viscosity data for TiO_2 and Al_2O_3/water-based nanofluids, and reported a maximum enhancement of 80% at 4% and 5%, respectively. In the present paper, we prepared nanofluids containing Fe_3O_4 nanoparticles. The thermal transport properties including thermal conductivity and viscosity were measured. The effects of the particle volume fraction on the thermal conductivity and also on the viscosity were further investigated.

Experimental : $FeCl_2{\cdot}4H_2O$, anhydrous $FeCl_3$, urea $((NH_2)2CO)$, ethylene glycol (HO) CH_2–H_2C (OH) are used for the preparation of nanoparticles as received from suppliers and double distilled water as a basefluid is used for the preparation of nanofluid. Double distilled water is prepared via lab based double distilled water unit.

Preparation of nanoparticles : Fe_3O_4 nanoparticles were synthesized by ethylene glycol route presented in S. S. Umare [29]. 100 ml mixed salt solution of $FeCl_2{\cdot}4H_2O$ (0.15M) and anhydrous $FeCl_3$ (0.3M) were taken in three necked round bottom (RB) flask fitted with air condenser. 16 g of urea and 400 ml of ethylene glycol was added. The reaction mixture was mixed properly and then refluxed at 160^0C with continuous magnetic stirring for 6 h. The colour of solution turns black after one and half-hour and then the precipitation starts. The reaction mixture was cooled to room temperature and the colloidal black precipitate was centrifuged at 2000 rpm. Precipitate was washed with methanol and acetone and then dried at 60^0C for 6 hours under vacuum.

Characterization : X-ray diffraction (XRD) analysis was conducted on 'X'Pert PRO, PANalytical X-ray Diffractometer using CuKα radiation (λ=1.5406^{o}A) at 45kV and 40mA. Measurements were performed in the 2θ range from 10 to 100. Average crystallite size (t) of Fe_3O_4 particles has been calculated from the line broadening using Scherer's formula. $t=0.9\lambda/\beta\cos\theta$, where β is the full-width at half maximum of the strongest peak, λ is the X-ray wavelength (λ=1.5406^{o}A) and θ is the angle of diffraction.

The surface morphology was examined from scanning electron microscopy (SEM) on JEOL JSA-840A equipped with an electron probe-micro-analyzer system. Energy dispersive X-ray (EDAX) measurement was also performed on the same equipment to determine chemical composition.

Preparation of nanofluids : The nanoparticles are weighed on a Uni Bloc AUW220D (Shimadzu). The weighed nanoparticles are suspended in double distilled water. The volume of water taken is 80ml and various volume fractions are prepared by taking different weights of Fe_3O_4 nanoparticles. A ChromTech sonicator (Taiwan) with 40 kHz and 1200W with variable intensities was used to ensure that the nanoparticles were well dispersed in the water. After 180 min. of intense sonication nanofluids appeared completely homogeneous.

Thermal conductivity measurement of nanofluids : Experimental setup for measuring thermal conductivity consists of KD2 Pro digital recorder, handheld controller and nanofluids container of 30 mm diameter. The thermal conductivity of nanofluids was measured by using a KD2 Pro thermal property analyser (Decagon Devices, Inc., USA). It consists of a handheld microcontroller and sensor needles. The complete description of instrument has been published elsewhere. All the measurements were performed at an ambient temperature of 26 °C. The accuracy of instrument was checked by measuring thermal conductivity of glycerine provided by suppliers and comparing with the instrument reading of standard samples.

Viscosity measurement of nanofluids : Experimental setup for measuring viscosity consists of AR-G2 Rheometer (TA instruments, USA). The AR-G2 is an advanced controlled stress, direct strain and controlled rate rheometer with a magnetic-levitation thrust bearing and a drag cup motor to allow nanotorque control. The device is suitable for testing of melts, fluids and interfaces. A wide range of cones and plates often called as geometry (diameters ranging from 20 to 60 mm and angles 1, 2 and 4°) is available for use with a Peltier setup or oven. A double wall couette cell is also available. Temperature control can be achieved using a Peltier plate with upper heated plate setup (UHP), a fluid bath or an oven (ETC – N2 atmosphere possible), allowing temperatures from -20°C to 600°C with typical ramp rates of 30°C/min and a temperature resolution of 0.02°C. The magnetic thrust bearing provides stiff, « frictionless » axial support to allow control of ultra-low torques from 0.03 μN.m-200 mN.m with a resolution of 1 nN.m. The advanced drag cup motor further reduces system friction and delivers a faster transient response and an extended angular velocity control range up to 300 rad/s with a displacement resolution of 25nrad. The dynamic frequency range is between 7.5×10^{-7} and 628 rad/s. The device is controlled by the Rheology Advantage software.

Nanofluid sample of 350μl was measured using a FinnPipetteF2 (Thermo Scientific) and for observing effect of volume fraction and shear rate on viscosity 40mm 1^0 geometry was used in rheometer. The temperature was maintained constant and it is 298.2K. Steady state flow step conditions are maintained and shear rate is in the range of 10-250 s^{-1} for obtaining corresponding values of shear stress and viscosity.

Results and discussion :

XRD study : XRD pattern of Fe_3O_4 is shown in Figure 1. The XRD pattern of as prepared Fe_3O_4 matches with JCPDS file no.77-1545 and the particles shows cubic crystal system. The lattice parameter of Fe_3O_4 extracted from XRD data is a = 8.365 A, which is in agreement with the literature value a = 8.396A [JCPDS file no.19-0629], this indicates that the product consists of crystalline single-phase Fe_3O_4. The average crystallite size of Fe_3O_4 using Scherer's relation was found to be 16 nm.

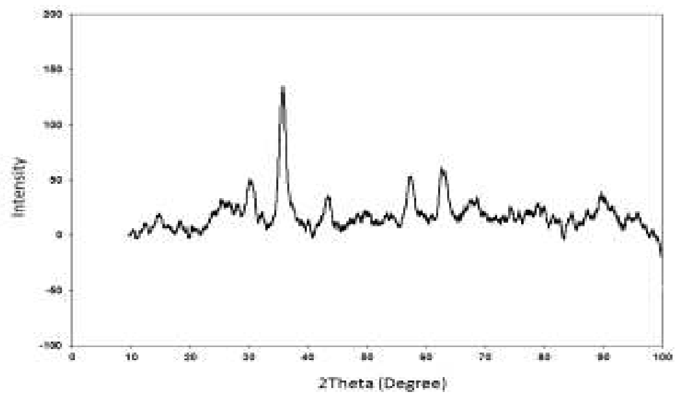

Figure (1) : X-ray diffraction pattern of Fe_3O_4

Morphological studies : Figure 2 shows SEM images of the Fe_3O_4 and it was found that Fe_3O_4 nanoparticles shows nearly spherical morphology.

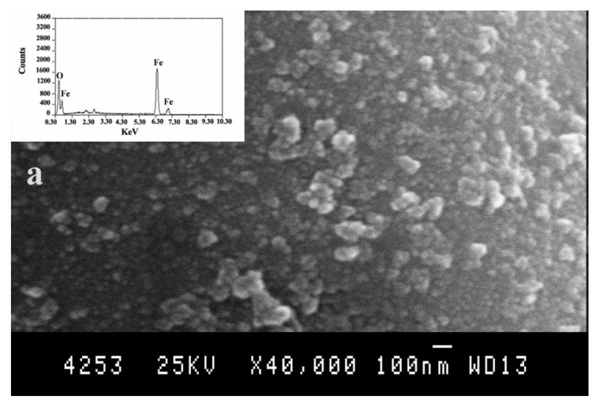

Figure (2) : SEM image of Fe_3O_4 nanoparticles.

Thermal conductivity: In general practice, the experiments showed that, upon addition of Fe_3O_4 nanoparticles into the base fluids, an enhancement in thermal conductivity occurred. This effect is shown in Fig. 3. In this plots the effects of nanoparticles concentration on the thermal conductivity is shown. It is important to give attention to the particle concentration expressed in a volume fraction of nanoparticles in the base fluids.

As shown in Fig. 3 the effective thermal conductivity of the nanofluids increases as the nanoparticles concentration increases. The best possible explanation for the effect is that the distance between nanoparticles decreases as the nanoparticles concentration increases. At the higher concentration particle to particle interaction increases which results into enhancement of thermal conductivity, and this is one of the main factors for this effect. Moreover various modes of heat transfer are also responsible for this [30].

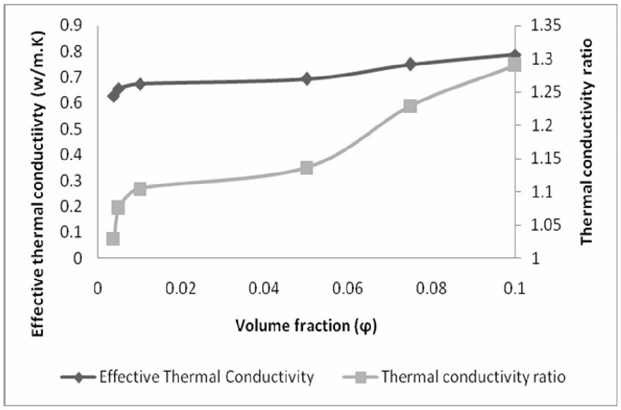

Figure (3) : Effective thermal conductivity and thermal conductivity ratio as function of volume fraction for Fe_3O_4 nanoparticles – water nanofluids.

Rheological behaviour analysis : The experimental observations showed an enhancement in the rheological behaviour upon addition of Fe_3O_4 nanoparticles into base fluids. These nanofluids of various volume fractions were analysed on a rheometer and the effect of shear rate on shear stress and viscosity was found out. Shear stress was found to increase with increasing particle volume fractions.

Figure 4 shows the experimental findings of dynamic viscosity as function of shear rate for different volume fraction of nanoparticles. It is observed that for higher volume concentration of nanoparticles nanofluids shows shear thinning behaviour and at lower concentration no effect of shear rate on viscosity.

Nanofluid behavior is always Newtonian, as can be observed in Figure 5, where shear stress is represented as a function of shear rate for the different volume fraction of nanoparticles nanofluid. All plots are linear and converge to the origin of the diagram.

Conclusion : Stable homogeneous dispersion of Fe_3O_4 nanoparticles is a crucial issue for both scientific research and practical application. In this work, Fe_3O_4 nanoparticles were synthesized by ethylene glycol route, structure and crystal size was confirmed by XRD and surface morphology using SEM studies. Synthesized nanoparticles were crystalline 16 nm spherical shape in nature. Fe_3O_4 – water based nanofluids have been synthesized at varying volume fractions of Fe_3O_4 nanoparticles. KD2 Pro was used to measure the thermal conductivity of nanofluids using transient hot wire method and maximum of 29 % enhancement observed. Another parameter i.e. viscosity was measured on AR-G2 rheometer under variable shear rate condition. It is observed that viscosity of nanofluids increases with addition of nanoparticles. All the compositions showing Newtonian flow behavior.

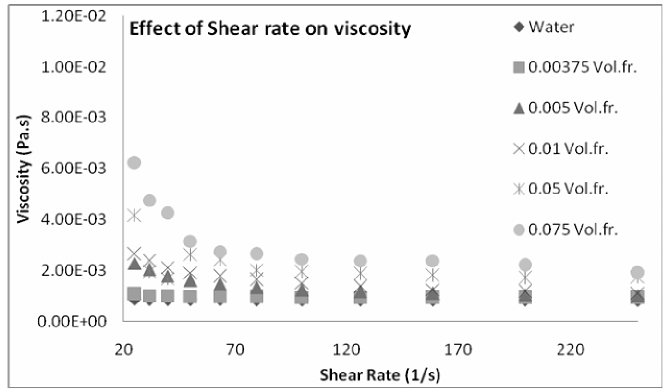

Figure (4) : Viscosity as function of shear rate for water - Fe_3O_4 nanofluids.

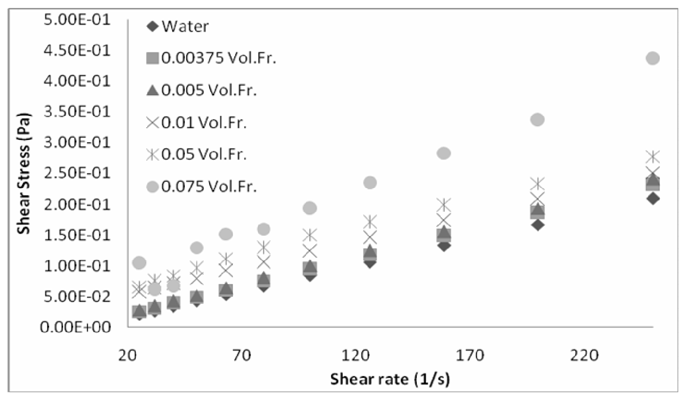

Figure (5) : A Shear stress as function of shear rate for water- Fe_3O_4 nanofluids.

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