

## MEASUREMENTS OF NANOFLUIDS' PHYSICAL PROPERTIES

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**Abstract.** *Nanofluids are suspensions of nanoparticles in pure (base) fluids. This work deals with the characterization of nanofluids through the measurement of their physical properties. Different techniques are used for this purpose, including ASTM standard ones, like: Flash method, for the measurement of thermal diffusivity; Line-heat-source method, for the measurement of thermal conductivity; Disc-Rheometer, for the measurement of viscosity; and densimeter, for density. Results are presented for the physical properties of water-based alumina nanofluids. The results obtained for the different properties of nanofluids are compared to those of the respective base fluids.*

**Keywords:** *Nanofluids, Physical properties*

### 1. INTRODUCTION

Thermal conductivity of fluids plays a vital role in the development of energy-efficient heat transfer equipments. However, traditional fluids used in those equipments, such as water or oil, usually have low thermal conductivity (Choi, 1998). Metals in the solid form have thermal conductivity larger by orders-of-magnitude than of those fluids. For example, the thermal conductivity of copper at room temperature is about 700 times greater than that of water and around 3000 times greater than that of engine oil.

Therefore, fluids containing suspended solid metallic particles are expected to display significantly enhanced thermal conductivities relative to conventional heat transfer fluids. Numerous theoretical and experimental studies of the effective thermal conductivity of dispersions containing particles have been conducted since Maxwell's theoretical work was published more than 100 years ago (Maxwell, 1873 apud Choi, 1998). However, many previous studies of the thermal conductivity of suspensions have been confined to those containing mm- or micron-sized particles. Conventional micron-sized particles cannot be used in practical heat-transfer equipment because of severe clogging and sedimentation problems, as well as the impossibility to use these particles in micro systems (Choi, 1998).

Modern nanotechnology provides great opportunities to process and produce materials with average sizes below 50 nm (Choi, 1998). Recognizing an opportunity to apply this emerging nanotechnology to established thermal energy engineering, Choi proposed in 1993 that nanometer-sized metallic particles be suspended in industrial heat transfer fluids, such as water, ethylene glycol, or engine oil, to produce a new class of engineered fluids with high thermal conductivity (Choi, 1998). The author coined the term nanofluids for this new class of heat transfer fluids (Choi, 1995 apud Choi, 1998). Maxwell's concept of enhancing the thermal conductivity of fluids by dispersing solid particles is old. However, the new and innovative concept of nanofluids brings the idea of using the nanometersized particles that have become available to investigators only recently. Recent experiments on nanofluids have indicated significant increases in thermal conductivity compared with liquids without nanoparticles or larger particles and strong temperature dependence of thermal conductivity (Kebinski et al., 2005).

The objective of this paper is to present the results obtained for different physical properties of a nanofluid produced with nanoparticles of Alumina ( $Al_2O_3$ ) dispersed in water, and compare such results with those obtained with the pure-base fluid. The properties measured in this work are the thermal conductivity, thermal diffusivity, viscosity and density. Standard ASTM methods are used in order to measure these properties, such as the Flash method, line heat-source probe and disc-rheometer, as described below. Due to the particular relevance of thermal conductivity values in heat transfer enhancement studies, the two different approaches here adopted for identification of this thermophysical property were critically compared. Emphasis is also placed in the inspection of the temperature effect on the measured thermophysical properties, which shall eventually provide the required correlations for simulations in convective heat transfer with nanofluids.

### 2. MATERIALS AND METHODS

#### 2.1. Nanofluids

The nanofluid used in this work was a colloidal suspension of 1% wt. of  $\gamma$  aluminum oxide ( $Al_2O_3$ - $\gamma$ ) nanoparticles in pure water (Mili-Q). The Aluminum nanoparticles were acquired from Nanostructured & Amorphous Materials. The base liquid used in the fabrication of the nanofluid was the ultra-pure water (Mili-Q), obtained after 18 distillations followed by 18 deionizations, in a proper equipment. The nanofluid used in this work was prepared by mixing nanoparticles to the base liquid in an ultrasonic bath for homogenization and dispersion.

## 2.2. Flash Method

In the method developed by Parker *et al.* (1961), a small and thin specimen is subjected to a high-intensity short-duration radiant energy pulse. The energy of the pulse is absorbed on the front surface of the specimen and the resulting rear surface temperature rise is recorded, as illustrated in Fig. 1. The thermal diffusivity value was calculated by Parker *et al.* (1961) from the specimen thickness and the time required for the rear surface temperature rise to reach 50% of its maximum value. Generally, the temperature rise in the specimen is small, so that the physical properties can be assumed as constant during the test. Therefore, if the thermal diffusivity of the specimen is to be determined over a temperature range, the test procedure must be repeated at each temperature of interest.

The Flash Method is an ASTM standard (ASTM 1461-01, 2001). The essential components of the apparatus are the flash source, sample holder, environmental enclosure (optional), temperature response detector, and recording device. The flash source may be a pulse laser, a flash or Xenon lamp, or other device capable to generate a short duration pulse of substantial energy. The duration of the energy flash should be less than 2 % of the time required for the rear face temperature rise to reach one half of its maximum value. The pulse hitting the specimen's surface must be spatially uniform in intensity (ASTM 1461-01, 2001).

An environmental control chamber is required for measurements above and below room temperature. The specimen support shall be loosely coupled thermally to the specimen. (ASTM 1461-01, 2001).

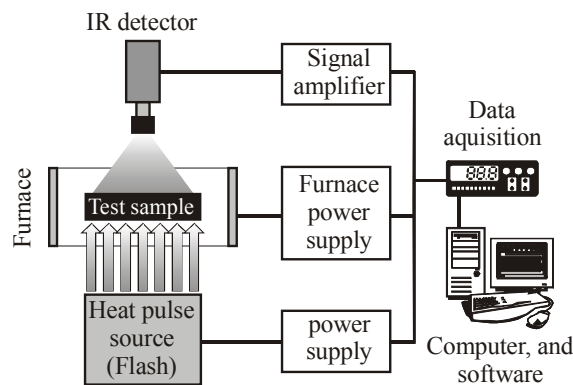


Figure 1. Flash Method

The detector can be a thermocouple, infrared detector, optical pyrometer, or any other means that can provide a linear electrical output proportional to a small temperature rise. It shall be capable of detecting a 0.05 K change above the specimen's initial temperature. The detector and its associated amplifier must have a response time substantially smaller than 2 % of the half time value (ASTM E-1461-01, 2001). Infrared detectors became popular as a reliable and accurate sensor for small temperature variations, within the time scale required for the Flash Method.

The Flash Method can also be used for the identification of the specific heat of the specimen, by comparing its temperature rise with that of a sample of a reference material (Parker *et al.*, 1961). Although this is a very attractive extension of the method, one must exercise extreme caution in performing it, as the opportunity for errors abounds (ASTM E-1461-01, 2001). In the course of an ordinary thermal diffusivity test, the amount of energy absorbed by the sample is important only to the extent that it will generate a sufficient rear surface signal (Parker *et al.*, 1961). On the other hand, the absorbed energy must be controllable, repeatable and accurately known for the identification of the specific heat. In this case, the temperature rise of the reference material is used to compute the absorbed energy. There are several conditions that must be satisfied for this process to be valid (ASTM E-1461-01, 2001).

After identifying the thermal diffusivity and the specific heat with the Flash Method, the thermal conductivity can be obtained from the definition of thermal diffusivity. In order to do so, the specimen's density can be calculated from its mass and volume measurements (ASTM E-1461-01, 2001).

In order to obtain the thermal diffusivity from the time required for the rear surface temperature to reach 50% of its maximum variation, Parker *et al.* (1961) used a one-dimensional heat conduction model, neglecting heat losses and assuming that the energy input was instantly deposited within a small depth of the specimen. In order to cope with experimental conditions where heat losses cannot be neglected and with a finite pulse duration, other authors proposed alternative mathematical models for the heat conduction problem in the specimen, such as those described in (Cowan, 1961 and 1963; Cape and Lehman, 1963; and Clark and Taylor, 1975), as well as for two or three-layered systems (Hartmann *et al.*, 1993). Recently, more involved models dealing with the coupled conduction-radiation heat transfer within the specimen were proposed for semi-transparent materials (Mehling *et al.*, 1998; and Lazard *et al.*, 2004).

In this work we use the Netzsch Nanoflash LFA 447/1 available in the Laboratory of Heat Transmission and Technology (LTTC/COPPE/UFRJ). The LFA 447/1 is a tabletop instrument that works with a high power Xenon-Flash lamp in the temperature range of room temperature to 200°C, and it has an integrated sample changer for 4 samples. Figure 2a shows a schematic of the instrument systems. A high-power Xenon flash lamp is surrounded by a parabolic mirror (reflector). The Xenon flash lamp is able to supply a pulse energy up to 10 J (up to 5 J/cm<sup>2</sup>) selectable by the measurement software in the wavelength range of 150 nm to 2000 nm. The software also allows the energy pulse length

to be adjusted to 0.1, 0.2 or 0.4 ms. The LFA 447/1 uses an InSb-IR detector in the wavelength range of 2000 to 5000 nm cooled by liquid nitrogen, which allows a non-contact fast measurement of the temperature rise in the back of the sample. The close connection to the preamplifier system permits a fast data acquisition (500 kHz, 12 Bit) and a measurement of 2000 points per shot (detector signal). An integrated furnace (heater) maintains the sample temperature stable during measurement. The heater is integrated in the automatic sample changer, which has a low thermal mass allowing fast heating/cooling rates. A Julabo Chiller is used to cool the surrounding components.

The LFA 447/1 is capable of measuring thermal diffusivity in the range of 0.01 mm<sup>2</sup>/s up to 1000 mm<sup>2</sup>/s, with an accuracy of 3-5% for most materials. The specific heat accuracy is 5-7%. This allows the calculation of the thermal conductivity in the range of 0.1 W/mK 2000 W/mK with an accuracy of 3-7% for most materials.

The analysis of experimental data is performed with a software called Proteus, provided by Netzsch, which includes different models for the data analysis. The models used are: Adiabatic (Parket *et al.*, 1961), Cape-Lehmann (Cape and Lehmann, 1963), Cowan (Cowan, 1961 and 1963), and Radiation (Mehling *et al.*, 1998), as well as two-layer and three-layer models.

Figure 2b shows the equipment operating at COPPE/UFRJ.

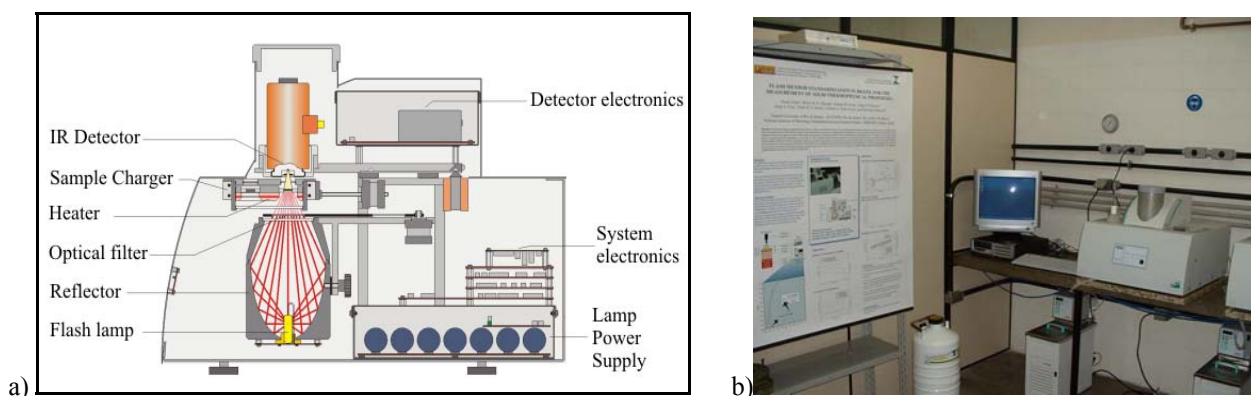


Figure. 2 a) Netzsch Nanoflash LFA 447/1 b) Netzsch Nanoflash operating at COPPE/UFRJ

For liquids, it's necessary to use a special container, as shown in Fig. 3. A three-layer mathematical model is required for the analysis of the data obtained with this container. The three-layer model involves the bottom of the container, the liquid under analysis and the lid of the container. The thermophysical properties of bottom and top layers must be known in order to measure the thermal conductivity and thermal diffusivity of the liquid in the intermediate layer. Such physical properties were provided by Netzsch.



Figure 3. Container for the determination of thermophysical properties of nanofluid.

### 2.3. Line Heat Source Probe

The line heat source method consists of a thin tube of known dimensions and thermal properties (the probe), which contains a source of heat and a temperature sensor. The probe is immersed in the medium whose properties are unknown. With the aid of suitable theoretical relations, these properties are then deduced from a record of the probe temperature *versus* elapsed time (Blackwell, 1954). For large times, the temperature variation of the heat source is shown to be linear with respect to the logarithm of time; the thermal conductivity can be computed from the slope of such variation.

The experimental methodology consists in placing the probe inside the material with unknown thermal properties. An electrical current of known and controlled intensity is passed through the electrical resistance and the temperature response of the thermocouple inside the probe is recorded. The temperature measurements are subsequently used to estimate the thermal properties of the material surrounding the probe.

In this work we use a commercial line heat source probe, the Hukseflux The TP-02, which is shown in Fig. 4a. It consists of a needle of stainless steel with 150 mm length and 1.5 mm external diameter, connected to a stainless steel base with 50 mm length and 10 mm external diameter. Inside the needle there's a heating wire, as well as two K type thermocouples connected in a way to provide the temperature difference between the probe and the medium. Inside the base there's a PT-1000 temperature sensor for the measurement of the temperature of the cold joints of the thermocouples.

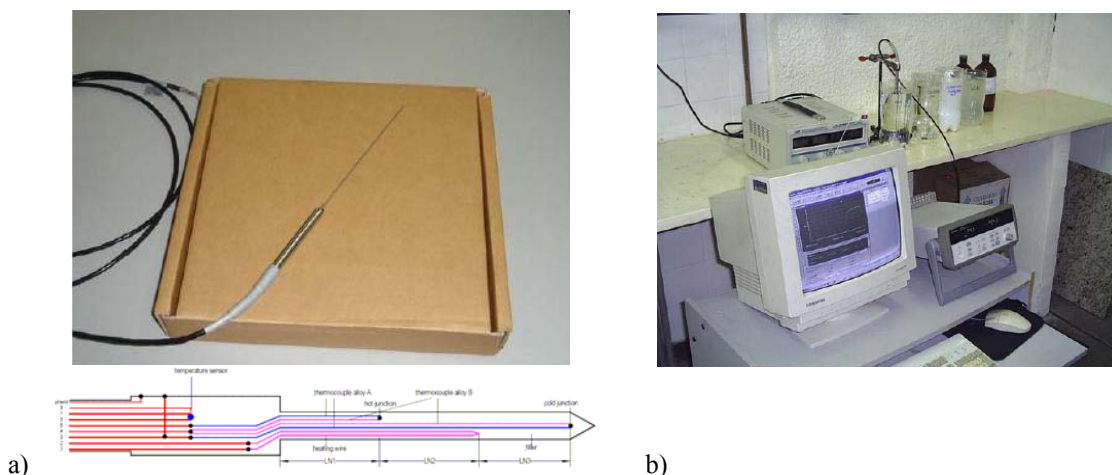


Figure 4. a) COPPE/UFRJ's TP02 line heat source probe from Hukseflux and connections.  
b) Experimental set up for the line heat source probe at COPPE/UFRJ

The heater was connected to a DC source. The current through the heater, the electric potential generated by the junctions of the thermocouple and the resistance of PT-1000 were measured with data acquisition system (Agilent 34970A) as shown in Fig. 4b.

## 2.4. Rheometer

The rheometer used in this work was the LVDV-IIIU from Brookfield, which is a rotational rheometer. It measures parameters of shear stress and viscosity of small samples at given shear rates. The principle of the DVIII-U is to drive a spindle (which is immersed in the test fluid) through a calibrated spring. The viscous drag of the fluid against the spindle is measured by the spring deflection. Spring deflection is measured with a rotary transducer. The viscosity measurement range of the DVIII-U is determined by the rotation speed of the spindle, the size and shape of the spindle, the container the spindle is rotating in, and the full-scale torque of the calibrated spring.

The geometry chosen for tests with nanofluids was the cone/plate, because this configuration requires a small sample (0.5 ml). Besides, this is the geometry recommended for tests with materials that have low viscosity, and allows an excellent temperature control.

To determine the viscosity of the specimen over a temperature range, the test procedure must be repeated at each temperature of interest with the temperature being controlled by a circulating water bath. Figure 5 shows the rheometer operating at COPPE/UFRJ.



Figure 5. LVDV-IIIU rheometer operating at COPPE/UFRJ

Equation (1) is used for the determination of the viscosity. Figure 6 shows the main variables appearing in Eq.(1).

$$\mu = \frac{\tau}{\dot{\gamma}} = \frac{M\theta}{\frac{2}{3}\pi R^3 \omega} \quad (1)$$

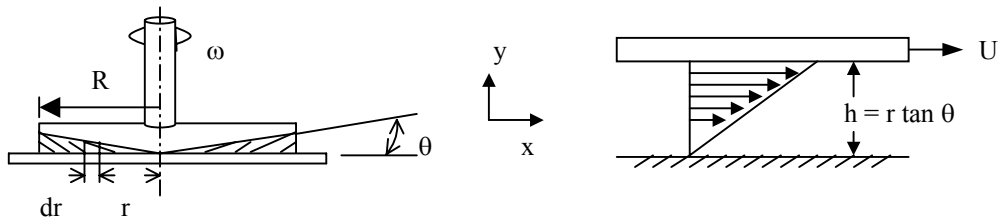


Figure 6. Configuration of the cone/plate scheme.

where  $\tau$  is the shear stress,  $M$  is the applied torque,  $\dot{\gamma}$  is the shear rate,  $R$  is the radius of the spindle and  $\omega$  is the speed of the spindle (Fonseca, 2007).

### 2.5 Density determination kit

The density determination kit used for the measurement of density of nanofluids was the Sartorius YDK 01, assembled on a Marte AM 220 precision scale (see Fig. 7a). The components of the kit are (Fig. 7b): 1 beaker (C), 1 bar frame (D), 1 thermometer (E), 1 glass plummet for the determination of density of liquids (F), 1 sample holder (A), 1 metal platform (B) and one adapter (G).

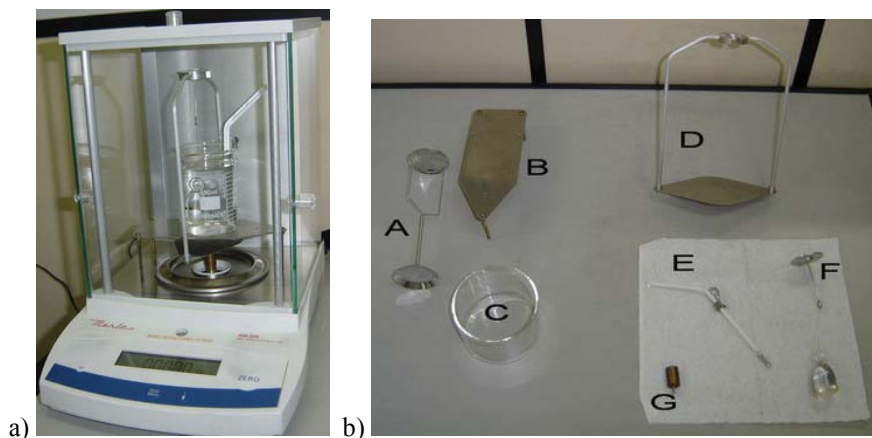


Figure 7. a) Densimeter operating at COPPE/UFRJ  
 b) Components of densimeter

With a hydrostatic balance, which enables to weigh a solid in air as well as in water, it is possible to determine the density of a liquid if the volume of the immersed solid is known. The density is computed as follows:

$$\rho(fl) = \frac{W(a) - W(fl)}{V} \quad (2)$$

where  $\rho(fl)$  is the density of the liquid,  $W(a)$  is the weight of the plummet in the air,  $W(fl)$  is the weight of the plummet in the liquid and  $V$  is the volume of the plummet. The accuracy of this technique is  $\pm 10^{-3} \text{ g/cm}^3$  (Fonseca, 2007).

### 3. RESULTS AND DISCUSSIONS

We now present the results for density, thermal conductivity, thermal diffusivity and viscosity for the nanofluid under investigation. Previous tests were made in fluids with well-known properties for the sake of validation, namely: Water, Ethylene Glycol and Glycerin. The results are in excellent agreement with those available in the literature, validating the experimental methodologies utilized (Fonseca, 2007).

### 3.1. Density

The experimental result obtained in this work for the density of the  $\text{Al}_2\text{O}_3$  nanofluid was  $(1.006 \pm 10^{-3}) \text{ g/cm}^3$ . The theoretical density of the nanofluid was calculated from the mixture formula:

$$\rho_{nf} = (1 - \varphi) \rho_f + \varphi \rho_p \quad (3)$$

where,  $\rho_{nf}$  is the density of the nanofluid,  $\varphi$  is the weight fraction of nanoparticles in water,  $\rho_p$  is the density of the nanoparticle ( $3.97 \text{ g/cm}^3$ ) and  $\rho_f$  is the density of water. The density calculated from the mixture formula was  $1.027 \text{ g/cm}^3$ . Hence, the discrepancy between measured and theoretical values is about 2%.

Figure 8 presents a comparison between the values for the density of water and of the  $\text{Al}_2\text{O}_3$  nanofluid. This figure shows an increase of about 1% of nanofluid's density with respect to water. Such small increase was expected because of the small concentration of solid particles. The uncertainty bars presented in figure 8 are in accordance with the recommendation of the manufacturer of the density kit, that is  $\pm 10^{-3} \text{ g/cm}^3$ .

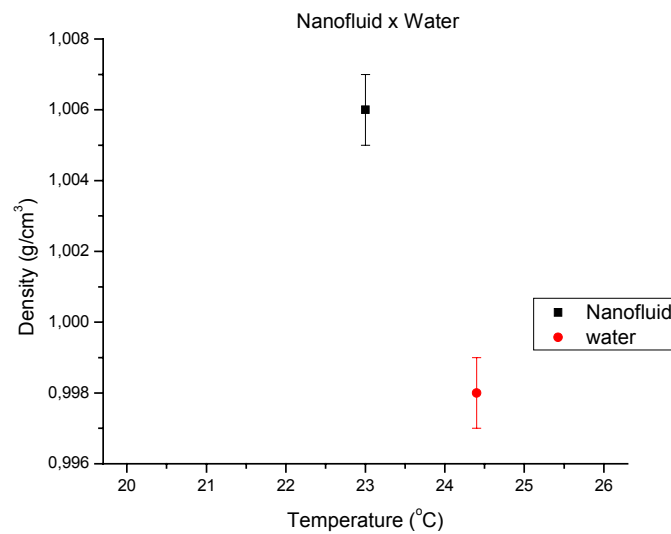


Figure 8. Density of nanofluid and water

### 3.2. Viscosity

Tests were performed at different temperatures with a variation of the shear rate to observe the rheological behavior of the nanofluid. The average viscosity was calculated from the values measured at each shear rate for a given temperature. The uncertainty was calculated in accordance with (INMETRO, 1998), as a function of the uncertainties of the measured viscosity at a given shear rate.

Figures 9 present a comparison between the results obtained for the viscosity of the nanofluid and the viscosity of water at  $20^\circ\text{C}$ ,  $40^\circ\text{C}$  and  $60^\circ\text{C}$ , respectively. Note in this figure that the nanofluid viscosity remains constant with the variation of the shear rate. This fact shows that 1% wt. of nanoparticle did not change the rheological behavior of water, that is, the nanofluid behaved as a Newtonian fluid. At  $20^\circ\text{C}$ ,  $40^\circ\text{C}$  and  $60^\circ\text{C}$ , the measured viscosities were  $(1.06 \pm 0.01) \text{ cP}$ ,  $(0.69 \pm 0.01) \text{ cP}$  and  $(0.54 \pm 0.01) \text{ cP}$ , respectively; that is, 4.7%, 5.8% and 13% higher than those of water, respectively.

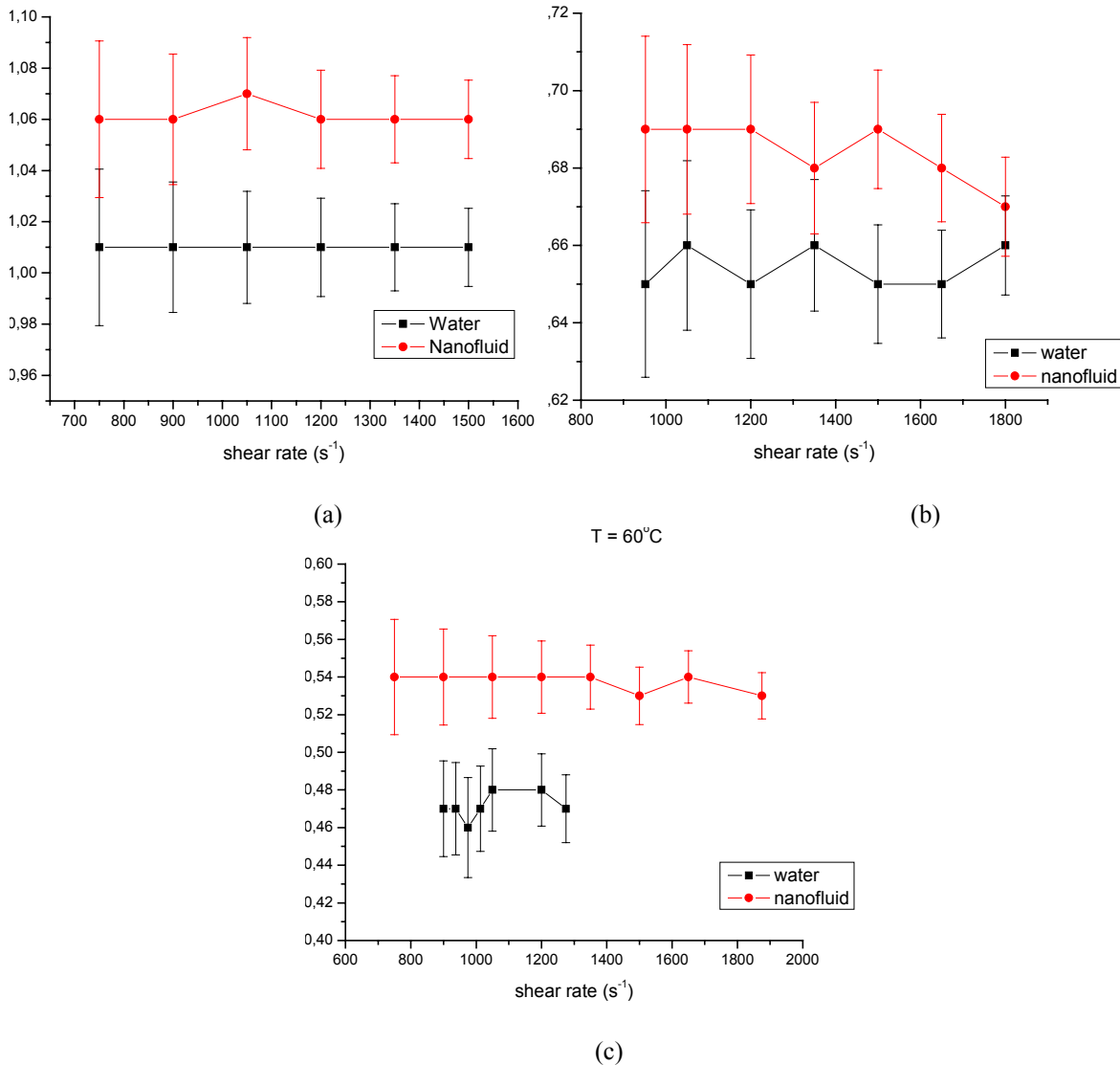


Figure 9. Comparison between the viscosity of water and nanofluid at (a) 20 °C, (b) 40 °C and (c) 60 °C.

### 3.3. Thermophysical Properties

Figure 10 shows the results for thermal conductivity measured with the line heat source probe and with the flash method. All experiments with the line heat source probe were carried out at room temperature and the voltage applied to the probe was about 2.4V. The heat dissipated per unit length of the probe was about 4.15 W/m. The heating time was controlled so that the temperature rise was not higher than 4 °C for water and for the nanofluid, in order to reduce convective effects in the fluid.

After each experiment, the curve of the temperature rise versus the logarithm of time was plotted. Short time measurements were disregarded and only the linear region was used for a linear fit of data, and the slope was used for the calculation of the thermal conductivity (Blackwell, 1954). Figure 10a shows a comparison between the thermal conductivity of the nanofluid measured with the flash method and that measured with the line heat source. It can be seen in Fig. 10a an excellent agreement between the obtained results. Thermal conductivity measured with the flash method was  $(0.598 \pm 0.004)Wm^{-1}K^{-1}$ , and with the line heat source was  $(0.605 \pm 0.012)Wm^{-1}K^{-1}$ .

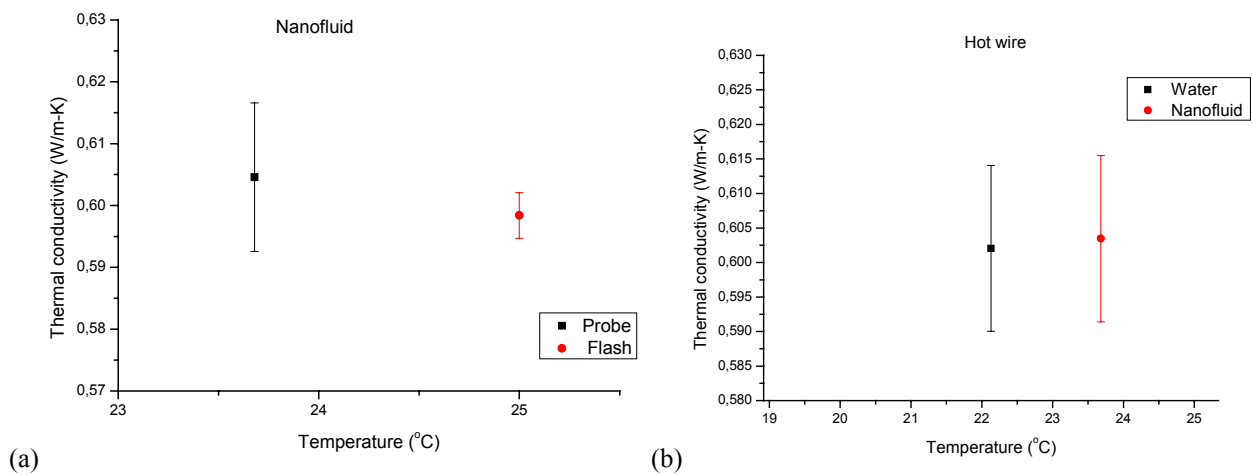
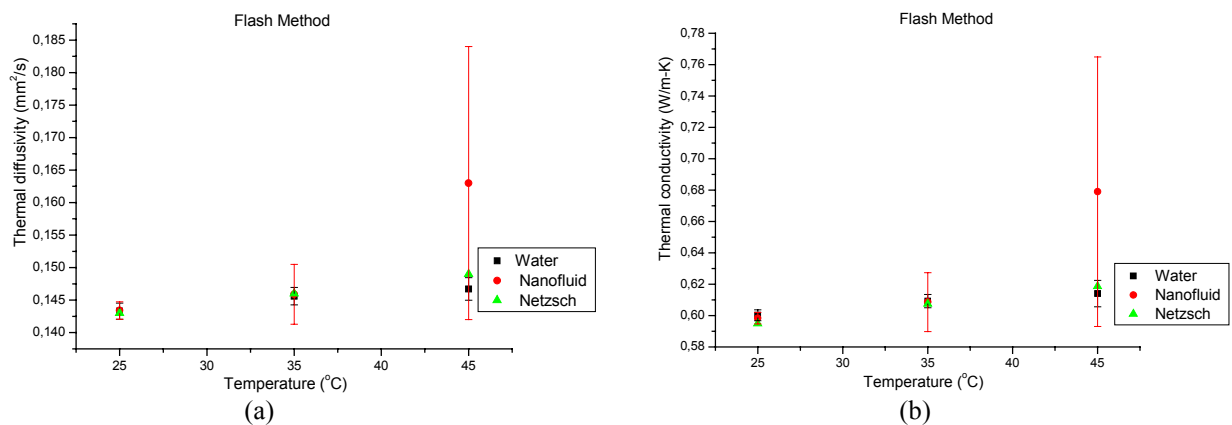


Figure 10. a) Thermal conductivity of nanofluid measured with the flash method and the line heat source probe. b) Thermal conductivity of water and nanofluid measured with the line heat source probe.

Figure 10b shows a comparison between thermal conductivity of water and nanofluid, measured with the line heat source probe at room temperature. It can be seen that the thermal conductivity of the nanofluid remains the same as that of water at this temperature. Such is probably the case due to the small volume of nanoparticles added to the base liquid in the nanofluid produced.

Figures 11 a,b show the thermal diffusivity and the thermal conductivity measured with the flash method for nanofluid and for water, in a temperature range from 25 to 45°C, respectively. In addition, these figures present the thermal conductivity and thermal diffusivity for water, measured by the manufacturer of the equipment (Netzsch). The agreement between the values of thermal diffusivity and thermal conductivity of water measured by COPPE/UFRJ and by Netzsch is excellent. It can also be seen that thermal conductivity and thermal diffusivity of nanofluid are very close to that of water, except at 45 °C, where it can be noticed an increase of about 10% in the average measured value.

Similarly to the results shown in Fig. 10b that were obtained with the line heat source probe, Fig. 11b shows no increase in thermal conductivity at room temperature when compared with water, in the results obtained with the Flash method.



Figures 11. (a) Comparison between thermal diffusivity of water and nanofluid measured by the flash method (b) Comparison between thermal conductivity of water and nanofluid measured by the flash method.

All results for the line heat source probe and for the flash method were the average of 10 experiments. The uncertainties of the line heat source probe were calculated in accordance with the manufacturer instructions ( $\pm 3\%+0.02$ )  $\text{Wm}^{-1}\text{K}^{-1}$  for each measurement. For the average resultant value, the uncertainties were calculated in accordance with (INMETRO, 1998), as a function of the uncertainties obtained for the thermal conductivity obtained with each test. For the flash method, the uncertainties were obtained for a 99% confidence interval ( $\varepsilon = \pm 2.576 \sigma$ ), where the standard-deviation  $\sigma$  was based on the spread of the measured values.

The experimental results in this work are in excellent agreement with those presented in literature for Alumina nanofluids. Das *et al* (2003) and Chon *et al* (2005) described the enhancement of thermal conductivity with increasing volume fraction and increasing temperature. Although in the present work the nanofluid was fabricated with 1%wt. (about 0,25%vol), while generally in the literature the volume fraction ranges from 1% to 4%, there exists a consistent behavior regarding the increase in thermal conductivity with temperature.



#### 4. CONCLUSIONS

Nanofluids present a great potential of improving heat transfer processes. This is because the suspension of superfine particles increases the thermal conductivity of the base fluid. The mass fraction and the temperature have a great influence in the thermal conductivity of nanofluids. Thermal conductivity and thermal diffusivity were obtained with classical methods for measuring thermophysical properties.

For the nanofluid tested in this work, with 1%wt. of  $Al_2O_3$  dispersed in water, density was 1% higher than that of pure water at room temperature, while measured viscosities were 4.7% higher than that of pure water at 20°C, 5.8% higher at 40 °C and 13% higher at 60°C. In addition, the nanofluid under study presented a Newtonian behavior. The thermal conductivity and thermal diffusivity were 10% higher for the nanofluid than for water, at 45°C. On the other hand, thermal conductivity and thermal diffusivity for the nanofluid and for water were practically identical for smaller temperatures.

The results obtained with the line heat source probe and with the flash method were in excellent agreement for the nanofluid and water.

#### 5. ACKNOWLEDGEMENTS

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