



## Dispersion regimes in alumina/water-based nanofluids: Simultaneous measurements of thermal conductivity and dynamic viscosity



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### ABSTRACT

The present work proposes a detailed experimental analysis of the dispersion regimes within nanofluids. For this purpose, simultaneous measurements of thermal conductivity and dynamic viscosity are performed for alumina/water-based nanofluids at volumetric concentrations  $\varphi$  ranging from 0.2 to 2%. Depending on the pH values, five dispersion regimes have been identified for intermediate values of  $\varphi$ . The well-dispersed regime characterized by a local maximum of the thermal conductivity and an absolute minimum of the dynamic viscosity is not recovered for  $\varphi = 2\%$ , while the chain-like agglomeration regime is not observed for  $\varphi = 0.2\%$ . The results are corroborated by the distribution of the Mouromtseff number, which appears as a reliable parameter to identify the most efficient nanofluids. For an optimized nanofluid, comparable efficiency may be achieved even at low concentration  $\varphi = 0.2\%$ .

### 1. Introduction

Nanofluids (Nfs) are innovative heat transfer fluids with improved thermophysical properties obtained by suspending nanoparticles (Nps) into conventional fluids. A complete, unique and well-grounded definition regrettably does not exist to date. This may be due to the fact that the field is relatively new and some concepts, especially heat transfer enhancement mechanisms, are not yet fully understood. Nevertheless, numerous definitions can be found, most of them derive from the first definition proposed by Choi and Eastman [1] and enriched in a revisited version [2] in which the authors emerge dispersion as key element. Due to their small size ( $< 100$  nm), nanoparticles benefit from a large surface/volume ratio ( $S/V$ ) allowing them to provide much higher thermal conductivity ( $k$ ) than that predicted by the classical theories [1, 3, 4, 5, 6, 7]. However, due to the challenges associated with the preparation of similar nanofluids, the literature results are often contradictory. As an example, Buongiorno et al. [8] proposed an experimental benchmark on the thermal conductivity of various nanofluids. The authors did not observe noticeable enhancement due to the inaccuracy of some measurement techniques [9, 10]. Actually, some methods, originally designed for solid thermal conductivity measurements, are indeed not appropriate for liquids, being very affected by natural convection. The KD2Pro Thermal Property Analyzer is the most significant example. This apparatus is erroneously frequently presented as a measuring system based on the transient hot-wire (THW) method while it is not as reliable.

Studies are constantly carried out to better understand the underlying mechanisms responsible for the improved thermal conductivity of nanofluids [11, 12, 13, 14, 15]. The lack of adequate experimental methods allowing to correlate the macroscopic effects to the nanoscale behavior of the particles makes this task hard. In fact, most of the works are based on theoretical models and when experiments exist, they focus much more on the stability indicators (zeta potential, absorbency, particle size ...) (see in [16, 17, 18]). Unfortunately, these techniques based mainly on the light scattering method are only suitable for very low particle concentrations far from the concentrations used in real applications. It remains a challenge and a subject of intense debate to identify clearly the heat transfer mechanisms in nanofluids [19]: higher specific surface area of nanoparticles, dispersion and electric charge, Brownian motion, interfacial nanolayer, collision between particles and linear nanoclusters like chains that can produce thermal bridges within the nanofluid. On the contrary, the scientific community agrees that nanoparticle aggregation plays a significant role in thermal transport [18]. Clustering is a complex process that depends on the properties of the host fluid and the surface energy of the particles. The electric charges in the solution govern the level of aggregation and consequently the configuration of the particles within the base fluid which can lead to different dispersion regimes, namely: well dispersed (WD), weakly attracted (WA), chain-like agglomeration (CL Agg.), partially agglomerated (P Agg.) and fully agglomerated (F Agg.) [19]. Each of the possible mechanisms behind thermal conductivity enhancement may be either accentuated or attenuated by these different

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dispersion regimes. Controlling the agglomeration becomes then crucial for understanding the physics of nanofluids. Especially, always more studies report that an optimum level of agglomeration may achieve a maximum  $k$  enhancement by nanoclustering effect and that excessive particle clustering is unfavorable [18]. Many observations from scanning electron microscopy (SEM) show the presence of chain-like aggregation [20, 21, 22]. Nevertheless, this technique cannot evaluate clustering in real suspensions since it requires drying the base fluid.

Adding nanoparticles affect the other properties of the base fluid, particularly the dynamic viscosity ( $\mu$ ) [23]. Most of experimental studies deal with the thermal conductivity of colloids and few works examine their viscosity, and when they exist, these data are rarely coupled. The nanofluid viscosity is also very sensitive to agglomeration. Clustering has indeed an effect on the organization of the nanoparticles, by changing their average starting sizes, the particle size distribution, their shape and aspect ratio. As for  $k$ , each of these parameters has a direct influence on  $\mu$  [24]. As an example, Bhanushali et al. [25] investigated the effect of particle shape on the properties of nanofluids using a range of distinct filler particle shapes. They concluded that higher aspect ratio favors the thermal conductivity and is detrimental for the viscosity. Simultaneous measurements of  $k$  and  $\mu$  could then inform us about the state of dispersion and bring response elements on the heat transport mechanisms in nanofluids, which is the main objective of the present paper. Such experimental data, made simultaneously on the same sample with accurate equipments, can also be exploited to establish trustworthy global energy performance criteria allowing to select the proper nanofluids for use in energy systems.

## 2. Experimental methods

The two-step method has demonstrated to be suitable to prepare oxide Nfs [2]. Alumina/water-based Nfs have been selected here for their experimental flexibility unlike metallic Nfs which must absolutely be prepared with the one-step method. The  $\text{Al}_2\text{O}_3$  nanoparticles are purchased from US Research Nanomaterials and exhibit the following properties: purity of 99.9%, averaged diameter of 50 nm.

Water-based nanofluids allow a direct control on the degree of interaction between nanoparticles by regulating the pH. The preparation step requires indeed to use the appropriate surfactant, to control the pH of the solution and to disperse the Nps by ultrasound techniques. In a former study [26], a careful experimental investigation on the stability of  $\text{Al}_2\text{O}_3$ -water based nanofluids has identified the pH as a key parameter for dispersing and stabilizing suspensions. The pH is controlled here using hydrochloric acid (HCl) and sodium hydroxide (NaOH) in analytical grade. Being temperature dependent, it is measured with LabQuest 2 from Vernier (Beaverton, USA) coupled to a temperature probe. One will mainly focus, in the following section, on the optimal pH range [4.5–6.5] identified by Bouguerra et al. [26]. The pH of the mother solution is set to 5.5 and pH is later adjusted to the desired value during the primary magnetic stirring.

The thermal conductivity of suspensions is measured with the THW-L1 Liquid Thermal Conductivity System from Thermtest Instruments. This device offers an accurate and fast measure avoiding the error due to natural convection. The measuring cell is integrated into a system controlling the temperature with an accuracy of 0.1 °C (heat exchanger + thermostat bath circulator). The principle, description and advantage of THW-method are detailed in [26]. Before each series of measurements, calibration and validation steps have been performed using distilled water and glycerin test fluids. The repeatability and the uncertainty of the measurements remain better than 0.5%.

The rheological properties of nanofluids are performed on a stress controlled rheometer (TA HR-2) using a DIN coaxial cylinder geometry. A Peltier cylinder regulates temperature with an accuracy of 0.1 °C. For low viscosity fluids, the rheometer may have a maximum beyond which it is no longer able to measure the angular velocity accurately. Then, one first delimits the validity range of the shear rate ( $\dot{\gamma}$ ) relative to the

present gap (5917.1  $\mu\text{m}$ ) by carrying out preliminary experiments on distilled water. The results compare very well with the literature values (uncertainty < 3%, repeatability < 0.5%). During the experiments on nanofluids, ones evaluate  $\mu$  of all samples as a function of  $\dot{\gamma}$ . Whatever the operating conditions, the present nanofluids exhibit a Newtonian behavior over the range [0.01–100]  $\text{s}^{-1}$ .

As explained above, one presents an experimental approach including a careful preparation step and the most appropriate measurement techniques for alumina/water based nanofluids. Particular attention is paid to the synchronization of the thermal and rheological tests. The first step in the experimental procedure is the weighing of the nanoparticles. Colloids with an ultra-precise concentration are prepared by homogenizing particles in water using a magnetic stirrer. The effective dispersion is achieved out by applying ultrasonic vibrations. Using this method, an initial solution of 800 ml of nanofluid is sonicated for 12 h. Mahbubul et al. [27] showed that the sonification time has no longer influence on the viscosity of alumina/water-based nanofluids for temperatures between 10 °C and 50 °C after 2 h. The required samples volumes for THW and rheological measurements are 45 ml and 25 ml, respectively. Therefore, the initial solution is divided into samples of 80 ml, the pH of each sample is adjusted to the desired value and a sonication of 15 mn is carried out before each test. The homogenization of the samples is performed with the Q700-Sonicator (Qsonica), while the initial solution is sonicated using the UIP1000hdT (1000 W, Hielscher Ultrasonics) which is more suited for large volumes. The next step is the calibration of the measuring instruments and the setting of the test temperature (25 °C). Once the test cell of each device is filled, simultaneous measurements of  $k$  and  $\mu$  from the same sample can begin. The above procedure is repeated for different pH values and volume concentrations of nanoparticles (vol%). Temperature is fixed to 25 °C for all measurements and controlled by incorporating a jacketed glass beaker connected to a thermostat bath circulator at all stages of preparation.

## 3. Results and discussion

Figs. 1–4 show the effective thermal conductivity ( $k_{nf}/k_{bf}$ ) and dynamic viscosity ( $\mu_{nf}/\mu_{bf}$ ) of the nanofluids as a function of pH for different volume percent concentration (from 0.2% to 2%). Here,  $k_{nf}$  and  $\mu_{nf}$  are the thermal conductivity and dynamic viscosity, respectively, of the nanofluid whereas  $k_{bf}$  and  $\mu_{bf}$  are that of the base fluid. Examination of the results represented by all of these measuring points makes it possible to correlate the consequences of the agglomeration on  $k$  and  $\mu$ . Indeed, changes in the thermal and rheological behaviors occur simultaneously in the same pH ranges. This can be explained by the structural modifications and the variation of the nanoparticle's arrangement in the base fluid. The pH of the solutions affects the surface charge of the particles, which cause their repulsion or attraction, resulting in many states of colloidal dispersion. A high surface charge results in well-dispersed particles, while a fully developed agglomeration occurs when the surface charges vanish (point of zero charge). Between these two limit states, different levels of charge can lead to other dispersions regimes. The WD regime is characterized by the smallest particle size, which results in a decrease of  $\mu$  and is accompanied by an increase in  $k$ . An inverse variation of these two quantities is observed during the F.Agg. regime, which leads to a larger particle size. Consequently, the combined analysis of  $k$  and  $\mu$  curves can provide reliable information on the dispersion state.

Given the opposite variations of  $k$  and  $\mu$ , it is not clear whether a nanofluid is efficient or not. It is then necessary to find a global parameter able to quantify its global quality for heat exchanges. The Mouromtseff number ( $Mo$ ) [28] is a factor of merit based on four properties of the fluid: density ( $\rho$ ), dynamic viscosity ( $\mu$ ), thermal conductivity ( $k$ ) and heat capacity ( $C_p$ ). Higher values of  $Mo$  indicate higher heat transfer capabilities.

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