

Thermophysical Characterization of Al_2O_3 and ZrO_2 Nanofluids as Emergency Cooling Fluids of Future Generations of Nuclear Reactors

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Abstract – Among the countless applications presently proposed for the nanofluids, the applications in energy have special attention by academic and industrial interest. Studies demonstrate that nanofluids based on metal oxide nanoparticles have physical properties that characterize them as promising working fluids, mainly, in industrial systems in which high heat flux want to be removed. Nuclear reactors for power production are examples of industry where such an application has been proposed. However, there are no concrete results about the ionizing radiation effects on nanofluids properties. This work aims to present the initial results of the current study carried out with the objective to check the effects caused by that ionizing radiation on nanofluids based on Al_2O_3 and ZrO_2 nanoparticles. Results from thermophysical analyses demonstrate that particular behavior on thermal conductivity, and density of such nanofluids can be observed as a function of temperature under no ionizing radiation effect. New investigations will analyze the application potentiality of some nanofluids in nuclear systems for heat transfer enhancement under ionizing radiation influence.

I. INTRODUCTION

The challenge on developing new generations of the most safe, costless, and efficient nuclear reactors wraps the development of researches on new materials and processes, its characteristics and economical-technical viability. In special, implies in the breakaway of paradigms and investment in development of new technologies applied in all the wrapped sciences.

Concerning increase of power density of future generations of nuclear reactors (Generation IV), new materials, fuel, cooling fluids, and more efficient heat transfer processes, are in constant development to attend such challenge. Furthermore, due to energy economic aspects, the development of new generation of nuclear reactors implies on costs-reducing design, manufacture, plant construction and operation, without losing sight of safety factors increasingly restrictive due to accident occurrence like recently Fukushima earthquake and

consequent nuclear power plant accident [1]. In this context, nanofluids are a promising technology for application in nuclear reactor systems for high heat flux transport.

Nowadays, there are three principal technologies of nuclear reactors classified in accordance with the working fluid: Light Water Reactors (H_2O) and Heavy Water Reactors (D_2O), Gas Cooled Reactors (CO_2 and H_2), and Liquid Metal Reactors (NaK, Hg) [2,3].

In accordance to the criteria of choice of the working fluid responsible for the heat flux transport of a nuclear reactor, requirements related to thermal quality (heat transfer capacity), physical-chemistry properties, nuclear and economics of the fluid (Bouré, 1964) [4] must be considered. The criterion of thermal quality regards the pumping power, the heat flux removing capacity and the function of thermal transport of this fluid, being a commitment between these two characteristics. The

criterion of physical-chemistry properties regards the thermodynamic properties and stability due to temperature and to the ionizing radiation (chemical dissociation and ionization phenomena), and to the chemical activity (corrosion and toxicity) of the fluid. The nuclear criterion regards the capacity of absorption and thermal moderation of neutrons and to its activation in the presence of ionizing radiation. The third criterion, but not less important, is the economic criterion, which regards production, storage and treatment costs.

As demonstrated by recent researches, nanofluids have very interesting physical properties with respect to its ability to remove and transport of heat. There is, currently, research groups in the world conducting investigations on the influence of ionizing radiation on nanofluids and the possibility of its use as working fluid or cooling of nuclear reactors core in cases of accidents [5-12]. These studies are still focused on more precise knowledge about the thermophysical properties without and with the action of ionizing radiation. In addition, other researchers have investigated the nanofluids heat transfer capacity [13-20].

State-of-the-art research on nanofluids and [13-20], one can set the nanofluids are as colloidal fluids composed of nanoparticles chemically stable and dispersed in a fluid (water, aqueous solutions, refrigerant, and others) at concentrations ranging from 0.01% to 50% by volume. According to literature, the more used and researched nanoparticles are the metals (Cu, Ag, Au, Fe, Ti, and others), the oxides (Al_2O_3 , CuO, FeO, ZrO_2 , SiO_2 , MgO, TiO_2 , ZnO, and others) and the carbon compounds (graphite, diamond, carbon nanotubes).

Concerning nanofluids application in heat transfer processes, thermal-hydraulic properties are very important to classify them a usable fluid. In literature, most of researches focus are heat transfer capacity, thermal conductivity and pressure drop characteristics of nanofluids in systems. Next section describes the main results obtained by researches about those physical properties giving a perspective of nanofluids applications.

1.1 Physical Properties

Nanofluids have higher viscosity than their base fluids and potentially require greater pumping power to have the same thermal performance. They have flow properties similar to those of a liquid base phase and have little or modest increase in the turbulent pressure loss. The increase in thermal conductivity can be compensated by an increase in viscosity, decrease in effective specific heat or change in wettability [12,13]. This flow behavior is attractive for the applications in engineering. To obtain good results in practical applications processes, heat transfer fluids should be designed to increase the heat transfer coefficient without

penalizing the pressure loss. This requires an accurate selection of particle shape, size, materials and concentrations. In the case of applications in reactor core (as postulated) with the presence of ionizing radiation, they require nanofluids with low activation characteristic in such a way that low radiation doses occurs.

Researches carried out in this specific field show that there is linearity correlating data and behavior of Newtonian fluid for nanofluids analyzed. A particle size is a factor that must also be considered. Results show that, for example, Al_2O_3 -based nanofluids viscosity not only increases in a non-linear way with concentration, but also with the nanoparticles size in the tube sides. There are findings which show zero viscous shear stresses for CuO/ethylene glycol based nanofluids, and which changes abruptly when the volume fraction of particulate becomes greater than 0.2%. Therefore, the volume fraction is regarded as the limit dilution. Substantial improvement in thermal conductivity is achievable only when the concentration of particles is less than the dilution limit. At concentrations above this limit, where both rotation and translational Brownian are restricted, there is no further increase in conductivity predictions beyond the effective medium theory. For some nanofluids the aggregate particles have a strong effect on the viscosity as much on the thermal conductivity of nanofluids.

1.1.1 Nanofluid thermal conductivity: theoretical and experimental models

The main feature of nanofluids is the fact that theirs thermal conductivity is observed tube higher than the base fluid's. The first experimental studies on thermal transport properties of nanofluids were aimed to study the surprisingly changes created by high concentrations of metal oxides nanoparticles in a water based fluid [14,17]. Currently, studies on nanofluids thermal conductivity are focused on fluid behavior due to the increase of that property. There are, for example, studies indicating a nonlinear relationship between the thermal conductivity and the concentration in case of nanofluids containing carbon nanotubes. Furthermore, it is observed that thermal conductivity is strongly temperature dependent and that there is significant increase in the critical heat flux (CHF) at boiling heat transfer processes. There are reports that nanofluids tested exhibit thermal conductivity higher than 50% of the base fluid. The nanoparticles added to a base fluid and also increases significantly the critical heat flux (CHF) during boiling heat transfer processes

Timofeeva et al (2007) [18] present a theoretical and experimental study combining heat conduction and particle agglomeration in nanofluids. In the experimental part, nanofluids Al_2O_3 in water and ethylene glycol are characterized by measurements of thermal conductivity,

viscosity, dynamic light scattering, and other techniques. Results show that the particle agglomeration state evolves in time, even the use of surfactants. The data also show that the thermal conductivity is predicted within the range by the effective medium theory. On the theoretical side, a model was developed for heat conduction through a fluid containing nanoparticles and clusters of different geometries. Calculations show that the elongated and dendritic structures are more efficient in increasing the thermal conductivity than the compact spherical structures with the same volume fraction; and surface tension is the major factor resulting in lower thermal conductivity than the proposed model.

Recent studies have sought to explain how nanofluids thermal conductivity widely varies depending on variables such as nanoparticle concentration and temperature. Some effective theories introduced by Mossotti, Clausius, Maxwell and Lorenz in the late 19th century, firmly established with the work of Bruggeman (1935), have been extensively verified and applied in many fields of science and engineering since then [14-16].

Buongiorno (2006) [6] reported a 40% increase in thermal conductivity of ethylene glycol with 0.3 vol% of copper nanoparticles of 10 nm in average diameter. Das et al. [14] observed increasing of 10-25% in thermal conductivity of water based nanofluid with 4.1 % vol. of Al_2O_3 nanoparticles. Moreover, it appears that the thermal conductivity of nanofluids increasing with increasing temperature, much more than for pure fluids.

The simplest models to explain the effects of increased thermal conductivity composites require that the particles are spherical, where the interface effects are negligible. In other words, at this stage, we do not consider the finite thermal conductance of interface particle/fluid. In the limit of low concentrations of nanoparticles, all versions of the theories presented so far converge to the same solution, but in the limit of high concentrations, there is no consensus among the theories presented yet.

1.1.2 Viscosity

Most of authors referred before present conclusions about cinematic viscosity of nanofluids. It is an important parameter concerning convective heat transfer capacity of nanofluids in hydraulic circuits. According to Motta (2012) [24], in a general way, nanofluids viscosity follow the base pure fluids behavior. For most metal oxide base nanofluids investigated, cinematic viscosity of nanofluids increase with volumetric concentration of nanoparticles; temperature is inversely related to the cinematic viscosity of nanofluids [24].

1.3 Surface contact angle

The surface contact angle of a cooling fluid has been shown as an important variable concerning heat transfer capacity, mainly in boiling conditions. The critical heat transfer (CHF) of a certain fluid is intrinsically related with surface wettability that is closely influenced by surface contact angle of that fluid. The surface contact angle of measured metal oxide nanofluids varies with particle volumetric concentration.

1.4 Heat transfer capacity

For laminar convection heat transfer process, studies show that the laminar heat transfer coefficient of some nanofluids increases rapidly to values of higher Reynolds number (Re) and may be up to 150%. This coefficient increases with the dimensionless axial distance x/D . Heat transfer coefficient of the laminar nanofluids nearly doubled the upper limit of the Reynolds number range tested, but also decreased with increasing concentration in the range from 1.1 to 4.4 vol%.

For nanofluids turbulent flow heat transfer the Nusselt number (Nu) increases the heat transfer coefficient up to 3-12%. Heat transfer coefficient in turbulent nanofluids based on Cu/water increased by about 40% for a volumetric concentration of 2%. The friction coefficient is not affected by the concentration of nanoparticles for a given for laminar or turbulent flow Reynolds number (Re). Research conducted at the Massachusetts Institute of Technology (MIT) [5-10] showed that, for heat transfer in single-phase convection, heat loss in nanofluids $\text{Al}_2\text{O}_3/\text{H}_2\text{O}$ $\text{ZrO}_2/\text{H}_2\text{O}$ present no resemblance to the behavior of pure fluids. In this case, the temperature dependence and thermal load with respect to thermophysical properties were measured and used for definitions of dimensionless numbers Reynolds (Re), Prandtl (Pr), and Nusselt (Nu).

In addition, with respect to particle size influence on convective heat transfer in single phase flow, it was shown that thermal conductivity increases with decrease in nanoparticle size. The nanoparticles in the range of 95-210 nm have a marginal effect on the coefficient of heat transfer. However, there are recent studies showing the opposite trend. This finding is consistent with the recent results for turbulent flow, showing the heat transfer coefficient during convective flow on the dependence of nanoparticles size. This is a significant conclusion.

In the case of boiling heat transfer, most of the experiments concerning boiling of nanofluids in a pool shows that the nanoparticles deteriorate heat transfer coefficient. However, for diluted nanofluids ratio with ratio of 0.3 % vol. it was observed that Al_2O_3 nanoparticles can improve the boiling heat transfer coefficient related to the based fluid up to 40%. Interestingly, some studies have

also shown that nanofluids heat transfer coefficient increases for small volume fractions to the order of 2%, but decreases for volume fractions larger than this. Therefore, the nanoparticle concentration has been shown to be an important factor in the heat transfer process.

An increase of up to three times of critical heat flux (CHF) for nanofluids $\text{Al}_2\text{O}_3/\text{H}_2\text{O}$ relatively to the base fluid (water) at a concentration of 10 ppm has been observed. The critical heat flux increase has been confirmed also for $\text{SiO}_2/\text{H}_2\text{O}$ particles, besides the fact that nanofluid pH increases the CHF up to 350%. In studies on heat transfer after-critical heat flux (post-CHF), a group of MIT [6-11] first demonstrated that nanofluids may improve significantly post-CHF boiling (film boiling). Other studies also have shown that nanofluids have a higher rate of heat transfer in film boiling. These findings are significant because they could pave the way to make nuclear reactors safe. Furthermore, it was shown that in a mini heat pipe, the heat transfer coefficient and the CHF of nanofluids increases for CuO based nanofluids considerably with decreasing pressure, compared with the water.

II. EXPERIMENTS

2.1 Sample characterization

Commercial nanofluids containing Al_2O_3 and ZrO_2 nanoparticles dissolved in deionized water of 10% and 5% volume fraction, respectively, were supplied by Sigma-Aldrich and studied in this work. The Al_2O_3 nanoparticles were declared to have distribution into a range of 30-60 nm, a mass fraction purity of 0.99 and a declared value of density $\rho = 1.06 \text{ g cm}^{-3}$ (@ 25 °C). The ZrO_2 nanoparticles were declared to have a distribution range < 100 nm. Nanofluids were dispersed into a predetermined water volume to obtain the desired volume concentrations studied in the first part of the project: 0.01%, and 0.1%.

Samples of nanofluids were prepared and visually analyzed in a scanning electron microscope (SEM-FEG) were performed. The samples were diluted and dispersed in deionized water in an ultrasonic bath for 10 minutes before being placed directly on the sample holder (brass or aluminum) and analyzed by scanning electron microscope JEOL, model JSM 6701F, operating at 5 kV at a distance work of 3.0 mm. The nanofluids images are shown in Fig 1.

In Fig. 1, one can observe the agglomeration of nanoparticles of Al_2O_3 forming flat large particles. It was observed that the dispersion of nanoparticles was not sufficient with ultrasonic bath. New tests were carried out with dispersion of nanoparticles into an ultrasonic disruptor, obtaining better results.

The same visual analysis was carried out for the ZrO_2 nanofluid. Figure 2 shows the results of visualization of ZrO_2 nanofluids samples in volume concentration of 5% in a scanning electron microscope (SEM-FEG). It shows a good dispersion in which small spherical nanoparticles can be observed. Both results are important for the qualitative analysis.

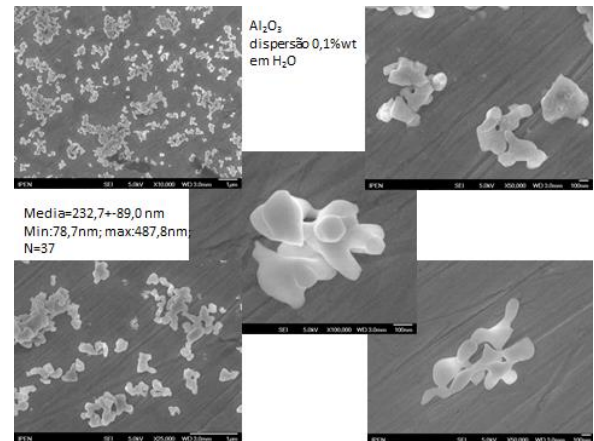


Figure 1. SEM images Visualization analysis of Al_2O_3 nanofluid.

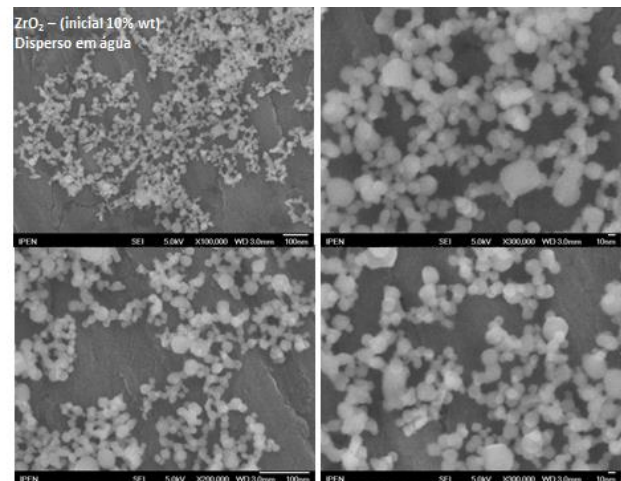


Figure 2. SEM images of ZrO_2 nanofluid.

Figure 3 shows the cumulative counts of Al_2O_3 and ZrO_2 nanoparticles diameters analyzed from the SEM images. It is possible to see that nanoparticles sizes are in accordance to the specified by the supplier

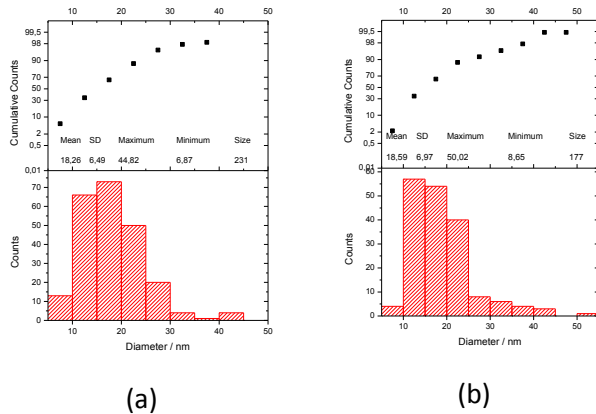


Figure 3. Cumulative counts of the nanoparticles (a) Al_2O_3 and (b) ZrO_2 .

2.2 Experimental tests without irradiation

Preliminary tests for determining the thermophysical properties of nanofluids based on Al_2O_3 and ZrO_2 without the effect of ionizing radiation were performed. These tests were necessary and arose from initial studies made by the working team members to establish a preliminary analysis of the compatibility of nanofluids concentrations into the nuclear reactor environment in which they would be tested. Preliminary tests for determining the thermophysical properties of nanofluids were:

a) Preliminary tests to measure the density of nanofluids: in this step consists on measuring densities of the nanofluids samples of Al_2O_3 and ZrO_2 in the volume concentration of 1%. The densities were measured with the aid of precision scales by the volumetric flask method [26]. The results are shown in Table 1;

Table 1 - nanofluids density measures with volumetric concentration of 1% (@ 20°C; average of 3 measurements).

Nanofluid	Volume (ml)	Density (kg/m^3)	Uncertainty
H_2O	100	99.9107	+/- 1%
Al_2O_3	1.5	100.02	+/- 1%
ZrO_2	1.5	100.49	+/- 1%

b) Preliminary tests for measurement of the thermal conductivity of nanofluids: this step consists on measuring the thermal conductivities of nanofluids of Al_2O_3 and ZrO_2 in a concentration of 0.01 and 0.1% by volume at 20 °C were measured. The conductivities were measured with the

hot wire method by KD2-PRO conductivity meter. The results are shown in Table 2;

Table 2 - Colocar o nome da tabela

Temperature (°C)	Thermal Conductivity γ_K (W/m.K)	Thermal Resistivity ρ (°C.cm/W)	Uncertainty (%)
23-27 ^a	2.6 -3.06	32.6-52.2	3-5
23-27 ^b	2.2 - 2.6	38 – 45	3 - 5

^a ZrO_2 nanofluids

^b Al_2O_3 nanofluids

II. FUTURE AND EXPECTED RESULTS

Research proposal concerning the study of nanofluids physical properties study and investigating their cooling capacity for applications in special systems like nuclear reactors should be encouraged. The choice of the best nanofluids is function of two fundamental aspects, the increase of thermal conductivity and critical heat flux, as demonstrated by important works. According to Buongiorno & Hu (2009) [7], to improve the thermal performance on nuclear reactors, the requirements for nanofluids applications the nanofluids should attend the following requirement: (i) minimal impact of the nanoparticles on the neutronic core behavior, maintaining operational safety and cost-effectiveness. Low nanoparticle concentrations appear to be of negligible impact on neutron transport in the core; (ii) minimal activation of the nanoparticles under ionizing radiation avoiding excessive coolant radioactivity. The nanoparticle material selection is important to avoid nanofluid long time activation (e.g., silica, alumina); (iii) to have compatibility with the reactor's chemical and radiation environment (stabilization, pH). In this case, the use of surfactants is probably incompatible (complex molecules). The production in large volumes may have relatively low cost (Al_2O_3 @ 0.01% vol., runs at about \$50 per gallon) if compared to benefit of having higher power density in the reactor core.

The improvement of the efficiency and security of future generations of nuclear reactors can be achieved through the following aspects: (i) understanding of: the changing in the nanofluids physical properties compared to their base fluid (water), especially those related to the high cooling capacity; the relationship between the thermal conductivity and other physical properties against the nanoparticles concentrations the effects that ionizing radiation can generate about the nanofluids studied; the generation of activation products in samples and / or changes in the structure of the nanofluids; the benefits of nanofluids use concerned the improvement of the

efficiency and security of future nuclear reactors generations. These are some of desired results for the new research line which has been showing to be one of the most promising on nuclear engineering and material application.

Currently, samples of Al_2O_3 and ZrO_2 based nanofluids in 0.1%, 0.05% and 0.01% volumetric concentrations are being irradiated in gamma source of Cesium (Cs) Dosimetry Laboratory high doses (LDA) center of radiation. The nanofluids were prepared and placed in 1.5 ml samples and irradiated in a calibrated source of Cs in Dosimetry Laboratory high doses (LDA) metrologies at the radiation center at Nuclear and Energy Research Institute-IPEN during 40 min time interval, with a 3.0 Gy total dose. The samples were stored in a shielded compartment shielded for approximately 48 hours after irradiation. Subsequently the samples were sent for viewing analysis in a scanning electron microscope. The samples are still being analyzed at the moment. The next step of the investigation will be the nanofluids samples irradiation in a neutron source (Nuclear Research Reactor IEA-R1 at IPEN-CNEN/SP, 5 MW_{th}, 10^{11} ncm⁻²s⁻¹).

III. CONCLUSIONS AND REMARKS

An extended literature review on nanofluids properties and applications, mainly for new generations of nuclear reactors was carried out aiming to give an overview on the actual status of such research line worldwide, and to be a conduction line for the new research area in development at IPEN-CNEN-SP in the Nuclear Engineering Center-CEN group.

Concerning density, thermal conductivity and thermal resistivity of Al_2O_3 and ZrO_2 nanofluids that were investigated without influence of ionizing radiation, it is possible to conclude that volumetric concentration, it is possible to conclude that volumetric concentration, particle size/shape and temperature are important variables. Investigations need to be performed to characterize the effects of ionizing radiation on nanofluids and its application proving its ability as a working fluid in nuclear reactors emergency conditions.

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