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THE EFFECTS OF THE NANOPARTICLE CONCENTRATION AND SURFACE ROUGHNESS ON THE CONTACT ANGLE OF NANOFLUIDS

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Abstract. *The demand for efficient and small-sized thermal systems was intensified during the last decades. One of the alternatives proposed to achieve such goal was the use of nanofluids as primary and secondary working fluid in thermal systems. Safety reasons concerning to nuclear reactors have also attracted the attention of industry and academia to the nanofluid technology. To investigate the benefits of this technology and its applicability is necessary to characterize the thermodynamic and transport properties of nanofluids. During heat transfer processes involving phase-change, the contact angle is an important parameter that affects the performance of nanofluids in heat transfer processes. So, the present paper deals with an experimental investigation on the effects of nanoparticle concentration and surface roughness on the contact angle. Initially, the paper describes a method developed at the Heat Transfer and Micro-fluidics Research Group at EESC-USP to measure the contact angle of a droplet onto a horizontal surface. Then, contact angle experimental results for a nanofluid composed of deionized water and alumina nanoparticles are described and discussed. The contact angle was evaluated for two size of nanoparticles, volumetric concentrations from 0 to 2%, and superficial roughness of 0.02 , 2.34 and 3.24 µm.*

Keywords: *contact angle, wettability, nanofluids, heat transfer enhancement, CHF.*

1. INTRODUCTION

The number of transistors in the microprocessors and, consequently, the amount of heat generated by these devices has exponentially increased since the 60s. Moreover, due to an industrial scenario characterized by progressive increasing of environmental and economic restrictions, new thermal systems are being developed with extremely care and focusing on minimizing their sizes, and maximizing their efficiency. In addition, after the Fukushima Daiichi nuclear disaster, a further incentive was given to researches related to retention of a nuclear accident and minimization of its effects on the surroundings. These scenarios are responsible for intensifying the number of studies concerning heat transfer enhancement and the increase of CHF.

Until mid 90s, most of studies concerning heat transfer enhancement have focused on adding devices capable of increasing mixing effects and developing structured and porous surfaces capable of augmenting the heat transfer rate during evaporating and condensing processes. Until that time little attention was paid to the development of high performance fluids. In 1995, Choi and Eastman (1995) have proposed a new class of fluid named by them as “nanofluids” that consists of a solution of nanosized particles solute in a liquid. Since then, investigations concerning nanofluids have spread through many laboratories around the world, and as pointed out by Taylor et al. 2012, several experiments were conducted in order to evaluate the thermal behavior of these solutions and characterize their transport and thermodynamic properties. In case of heat transfer mechanisms dominated by convective effects, the main contribution of nanofluids is the fact that the addition of a conductive material to a liquid, results in a fluid with higher conductivity than the base fluid as indicated by Wen (2012). In case of heat transfer mechanisms dominated by nucleate boiling effects, Coursey and Kim (2008) and Golubovic *et al* (2009) have pointed out that the addition of nanoparticles increases the wettability of the base fluid . This behavior has been indicated as responsible for the detrimental effect on the heat transfer coefficient by Kim, *et al.*, 2007 and the enhancement of the critical heat flux during pool boiling and flow boiling under low vapor quality conditions by Kwark, *et al.*, 2010. It is important to highlight that some authors as Sharma et al (2013) have proposed that the enhancement of CHF is not directly related to the increase of wettability. Instead, he has proposed that the nanoparticle deposition during the boiling process create a thin porous surface that increases capillarity effects through its matrix postponing the CHF to higher heat fluxes.

According to Carey (1992), the affinity of liquids for solids is referred to as the wettability of the fluid. Generally, the wettability of the liquid is quantified by the contact angle θ, defined as the angle measured from the interface solid-liquid between the solid surface and a plane tangent to the liquid-vapor interface traced from a point at the triple-contact-line (liquid-vapor-solid). The liquid of a sessile droplet will spread more over the surface as the contact angle decreases. Figure 1 illustrates different profile shapes of a liquid droplet and the relationship between the contact angle and the wettability. For θ between 0 and 90°, the liquid is said to be wetting, and for $90^o<\theta<180^o$, the liquid is termed as nonwetting. For $\theta=0^o$, the liquid spreads completely over the surface forming a thin film while for $\theta=180^o$ the liquid is completely nonwetting and touches the surface only on a single point.

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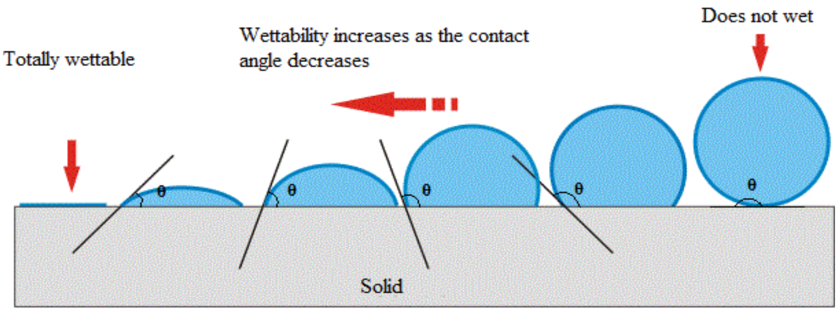

Figure 1. Profile shape of liquid droplets and the relationship between contact angle and wettability.

It should be highlighted that the study of nanofluids is not a simple task. Different methods of preparation and stabilization of solutions are employed and these differences can affect the thermodynamic and transport properties of the nanofluids. So, it is important when investigating nanofluids to provide a detailed description of the experiments, giving detailed information about the preparation process and measurement procedure. Information such as temperature, sonification time, delay time, particle size and stabilization method are essential, as seen in Hasda (2011), in order to guarantee the repeatability of the experiments.

Initially, the present study involves the detailed description of an experimental bench developed in the Heat Transfer and Micro-fluidics Research Group at EESC-USP to measure the contact angle of a sessile droplet onto a surface. Then, the experimental procedures used to prepare the nanofluid and evaluate its contact angle over an aluminum surface having different roughness are carefully described. Finally, experimental results concerning the effect of the alumina nanoparticle concentration, nanoparticle size and surface roughness on the contact angle are described.

2. MATERIALS AND METHODS

2.1 Experimental Method and Apparatus

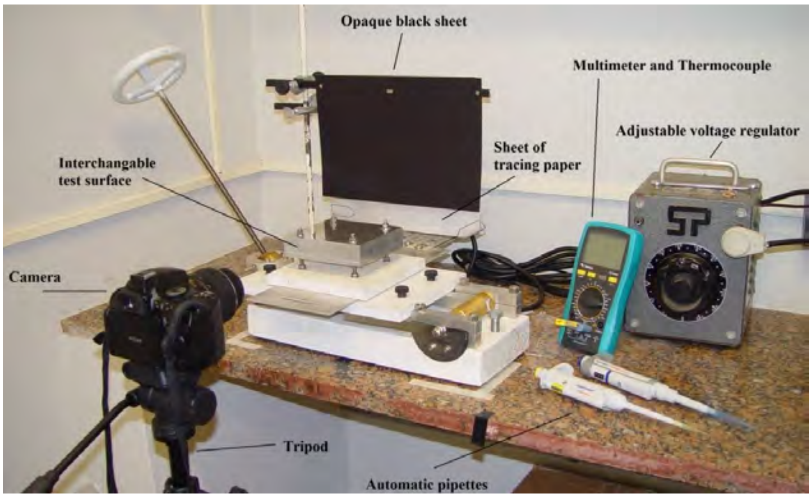

Figure 2. Experimental bench developed to measure contact angle of droplets onto different surfaces and instruments used to acquire the data.

The measurement method is based on analyses of pictures of a sessile droplet on a clean and horizontal surface. The pictures are analyzed through software developed based on Labview that provides the contact angle using a procedure similar to the one used by Konduru (2010). The contact angle measurements were performed using the experimental apparatus shown in Fig. 2. The apparatus consists basically of the test surface, a camera, a high intensity lamp, pipettes, sheet of tracing and opaque black papers, an adjustable voltage regulator and a thick sheet of carbon steel in which was fixed the test surface. In this apparatus, it is possible to interchange the test surface and alter its inclination through a worm screw connected to a flywheel. In the present study, the contact angle was evaluated only for a horizontal surface. The test surfaces consist of aluminum blocks, type 5052-F, with plain surfaces with three levels of roughness given according to Ra standards as 0.02, 2.34 and 3.24 μm and uncertainties, respectively, of 0.004, 0.10 and 0.20 μm. The different surface roughnesses were obtained by blasting of glass´s microsphere from different sizes. The measurements of surface roughness were obtained as the average value of five random measurements of each block by using a roughness meter Talylor Hobson (Talysurf 10). Both before and after each measurement, the test section was carefully cleaned with

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a piece of cotton soaked in pure acetone and a paper tissue with the aim of remove the nanofluid and impurity that could be present on the surface

A great effort was put in order to obtain pictures of droplets with sharp border lines. To obtain such a goal the following steps were taken: i) backlight was used in order of making the droplet and the surface dark and the background bright; ii) then, a sheet of tracing paper was positioned between the lamp and the droplet in order to minimize obfuscation effects; iii) moreover, an additional opaque black sheet was positioned just in front of the tracing paper; iv) finally, the intensity of the light emitted by the lamp was manually controlled by the operator through an adjustable voltage regulator in order to capture a clear picture with well defined borders. Figure 3 displays a picture of six droplets, obtained in the present study before any treatment. The camera was fixed in a photography tripod that possess a clinometer (bubble level) used to guarantee a horizontal picture, parallel to the leveled test surface.

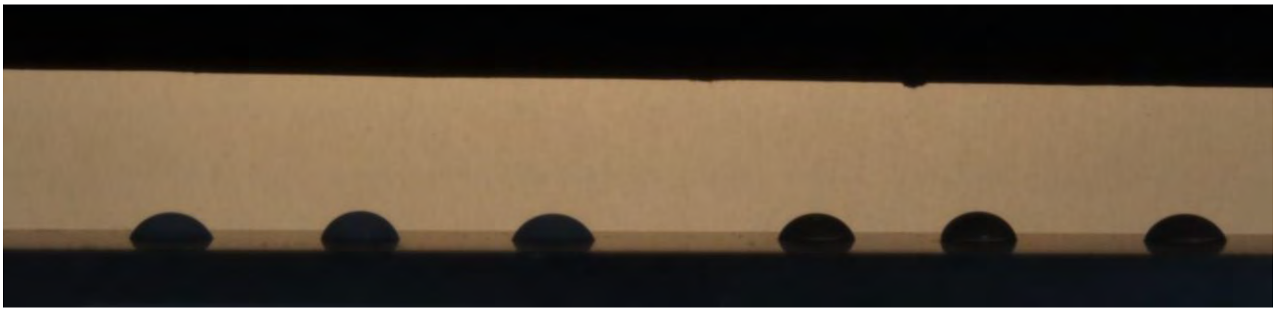

Figure 3: Six droplets of nanofluid over the test surface. Picture taken using the experimental bench developed in the present study.

Tests were also performed in order to evaluate the effect of the residence time on bubble format. It was observed that right after its deposition on the surface, the droplet requires some time to stabilize, until all the superficial forces reach a balance. After several tests, a residence time of 10 minutes was considered enough for the droplet stabilization. Nevertheless, if the droplet stays too long over the surface, over 25 minutes, evaporation effects becomes relevant affecting the droplet original volume and shape. So, all the pictures were obtained for a residence time of 15 minutes after the droplet deposition.

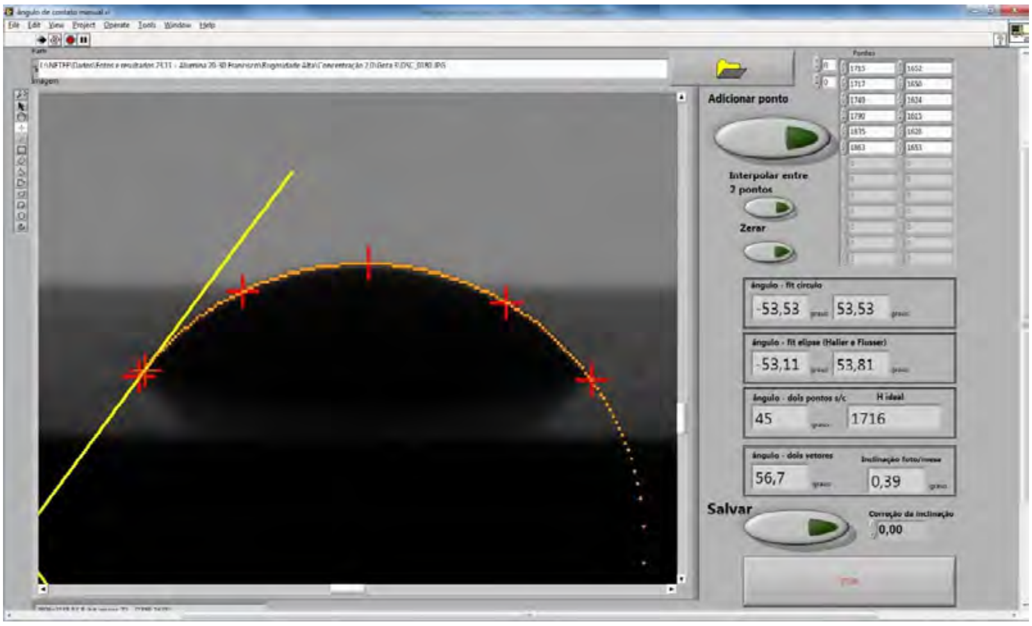

Figure 4. Image of the interface of the program developed in a Labview platform to measure the contact angle.

Software for analyzing and measuring the contact angle from the droplet image was developed based on LabView. The program recognizes the droplet picture and builds a Cartesian space over it. Then, the image is shown in the screen so that the operator can adjust it to have a better view of the droplet and the triple contact point. Then, the operator defines some points at the border of the droplet, starting from the triple contact point. The routine of the program realizes a numerical calculation using the points given by the operator in order to find the best equation of a circle that fits the

ISSN 2176-5480

droplet shape. The circle is then shown on the screen so that the operator can analyze if the equations are fitting properly the surface of the droplet. If the number of given points is appropriate and a good fit of droplet shape is obtained, then the program routine derives the circle equation passing through the triple point (triple line), in order to find the tangent line in that point. Possessing the equation of the tangent line, the angle between this line and the "X" axis (horizontal) is calculated. A picture of the interface of the developed program is shown in Fig. 3.

The following additional methods to obtain the contact angle were also implemented: i) the method of Halir and Flusser (1998) that fit the best ellipse to a droplet using some border points determined by the operator. Although the ellipse method seems more general than the one implemented in the present study, this method provides always the smaller ellipse and consequently most of the contact angle measurements results were near to 90°; ii) two points method, this method considers a point at the contact line and another one closer to it at the droplet border, both given by the operator, to trace a line tangent to the droplet surface. This line and the horizontal axis define the contact angle; iii) two vectors, in this method two vectors are built, one connecting both triple contact points at the opposite sides of the droplet and the second one formed by a point at the contact line and another one closer to it at the droplet border. The angle between the two vectors provides the contact angle. The methods ii and iii are very dependent of the operator and so were also not considered.

The method developed in the present study was validated by comparing its measurements against angles previously known by using proof bodies (semi-cylinder), with shapes similar to the 2D image of the droplet, positioned on the surface. For this purpose, a little shaft with a diameter of 15 mm was machined and polished. Then, this shaft was cut axially in two pieces in such a way that two specimens with different circular shapes were obtained. Measuring the height of the specimen and knowing its previous diameter it was possible to make simple geometric calculations and find the contact angle between the specimens and the test surface. Figure 5 display a picture of the proof bodies.

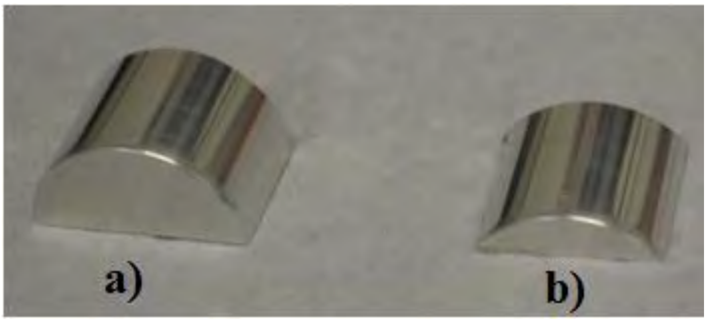

Figure 5: The two proof bodies of aluminum used to validate the experimental bench.

The validation process consisted in taking 40 pictures of each proof body positioned on the test surface. Figure 6 shows a picture taken from the two proof bodies positioned on the test surface and using the experimental resources applied in order to highlight the droplet borders. Then, two different operators analyzed each image of each proof body and individually measured its contact angle by using the software developed in the present study and above described. The standard-deviation of the measurements of each semi-cylinder were estimated according to a t-student distribution for a confidence interval of 95%. Table 1 compares the average contact angle based on the measurements and the respective estimated value from geometrical relations. This table also gives the standard deviation of the measurement. By analyzing the results displayed in Table 1, it can be concluded that the method developed in the present study is reasonably accurate.

Figure 6. This figure shows how the specimens looked like when placed in the experimental bench.

Table 1: Results of the contact angle measured of two solid specimens simulating droplets.

	Semi-cylinder (**a**) (°)	Standard deviation (2σ)	Semi-cylinder (**b**) (°)	Standard Deviation (2σ)
Operator 1	87.5	1.5	55.6	0.7

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Operator 2	87.9	1.7	55.8	0.6
Estimated contact angle (calculated)	88.7	-	55.6	-

2.2 Nanofluid Preparation

The nanofluids were prepared according to the two-step method as described by Ghadimi, *et al.*, 2011 and Yu and Xie (2011). This method was chosen due to its simplicity and precision. The method consists in mixing a fluid and a powder composed of nanoparticles previously prepared. The big issue about this method is to guarantee the stability of the nanofluid once the particles tend to attract themselves forming agglomerates that precipitate. In order to minimize this problem, the solution was submitted to ultrasonic vibration (sonificator Cole Parmer - model CV334), responsible for breaking the bonds between the clustered particles. The agitation process took 1 hour per each concentration of nanofluid and was implemented under controlled temperature in order to avoid the liquid evaporation with the consequent variation of the concentration of the solution. Previous studies have revealed that the nanofluids prepared through this process can be considered stable even after 6 hours of its preparation. So, in the present the properties were measured within this period of time.

In the present study, Mili-Q deionized water was used the base fluid. Care was exercised in order of preparing nanofluid and evaluating its contact angle just few hours after producing water. The nanofluids were prepared with nanoparticles of aluminum oxide of nominal diameter between 20 and 30 (1020MR, NANOAMOR), and between 40 and 80 nanometers (1030HT, NANOAMOR). The nanoparticle volumetric concentration of nanofluid was determined as follows: (i) based on nanoparticle density provided by the producer (3700 $kg.m^{-3}$), the mass of nanoparticles necessary to achieve a specific volumetric concentration of nanoparticle is calculated; (ii) then, the nanoparticle mass is progressively deposited in a reagent bottle and evaluated with an analytical balance (METTLER TOLEDO, AG245), with accuracy of 0.0001 g, until achieve the desired mass; (iii) hereafter, 50 ml of Mili-Q deionized water from a measuring cylinder (accuracy: ±1ml) is added and mixed to the nanoparticles. Nanofluids with nanoparticle volumetric concentrations of 0.1, 0.5, 1.0, 1.5 and 2.0% were prepared for both nanoparticle diameters. The experiments were performed in a room with controlled temperature kept almost constant at $20^{o}C$.

3. EXPERIMENTAL RESULTS

Once the experimental bench for measuring the static contact angle of a droplet was ready and validated, nanofluids with different concentrations were prepared and their contact angle evaluated for 3 different surfaces of aluminum with different roughness. First, nanofluids composed by nanoparticles with diameters between 20 and 30nm and volumetric concentration from 0% (pure deionized water) to 2% were evaluated. Droplets of all the concentrations of nanofluids were deposited and tested in the three different surfaces, but each droplet in a different spot Three droplets of each concentration were deposited on each surface, and three pictures were taken of each droplet. Each picture was analyzed by the software developed and the contact angle measured. Then, a medium contact angle value was calculated based in all the data from one concentration in one surface. These results are displayed in Fig. 7 and 8. The standard deviation was also calculated for each concentration in each surface, and the medium value found was 2 degrees, which is the length of the error bars.

From an analysis of Fig.7, clear behaviors of the contact angle with varying the nanoparticle concentration cannot be identified. This can also be seen in recent work published by Cieśliński and Krygier (2013), where contact angle of nanofluids were also measured for different concentrations and surfaces.

On the other hand, it seems that the roughest surface provides always the lowest contact angle, what is quite different from the results observed in the article mentioned above. Although their preparation method of nanofluids is very similar to what was done in this study, the nanoparticles used and the surface roughness where the droplets were put are not the same; fact that can explain such variations of data. Negligible effects of the nanoparticle concentration on the contact angle have been already observed by Kim et al (2007) when evaluating the contact angle on stainless steel surfaces of solutions of Al_2O_3 , ZrO_2 and SiO_2 nanoparticles in water .

Through an analysis of the contact angle measured based on the pictures of a single droplet, small deviations among the measurements are observed. However, when comparing contact angles measurements of three droplets of the same fluid in the same surface, the deviations are reasonable, achieving values up to 4 degrees. Such a result indicates that the contact angle is strongly dependent of the local conditions at the triple contact line. In this sense, it is important to highlight that the roughness measurement method gives average values over a certain length instead of local values. So, it can be speculated that inhomogeneity of surface roughness is responsible for such difference.

Contact angles for nanofluids composed by deionized water and alumina nanoparticles with diameters between 40 and 80 nanometers are illustrated in Fig. 8. According to this figure, it seems that, first the contact angle increases with increasing the nanoparticle concentration, passes through a peak at a concentration of almost 1% and then decreases with further increment of nanoparticle concentration. Similar results have been already presented in the literature by Cieśliński and Krygier (2013) when evaluating the contact angle for solutions of alumina and TiO_2 nanoparticles in water.

As already shown in Fig. 7, according to Fig. 8 the roughest surface provided always the lowest contact angle.

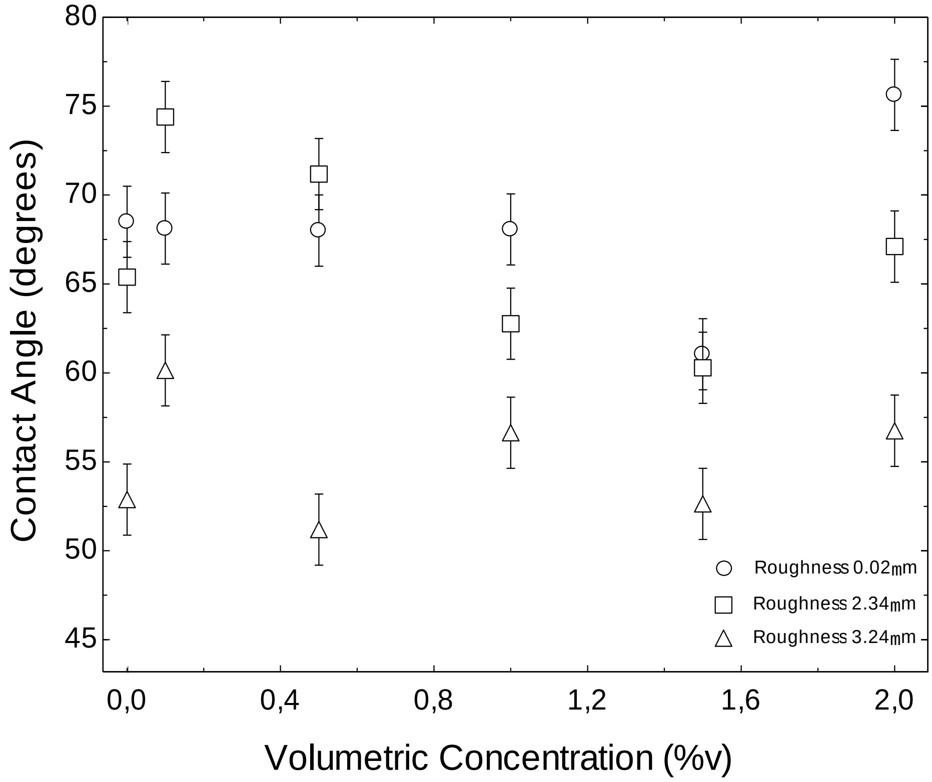

Figure 7. Effect of nanoparticle volumetric concentration and surface roughness on the contact angle of alumina with diameter of 20-30nm.

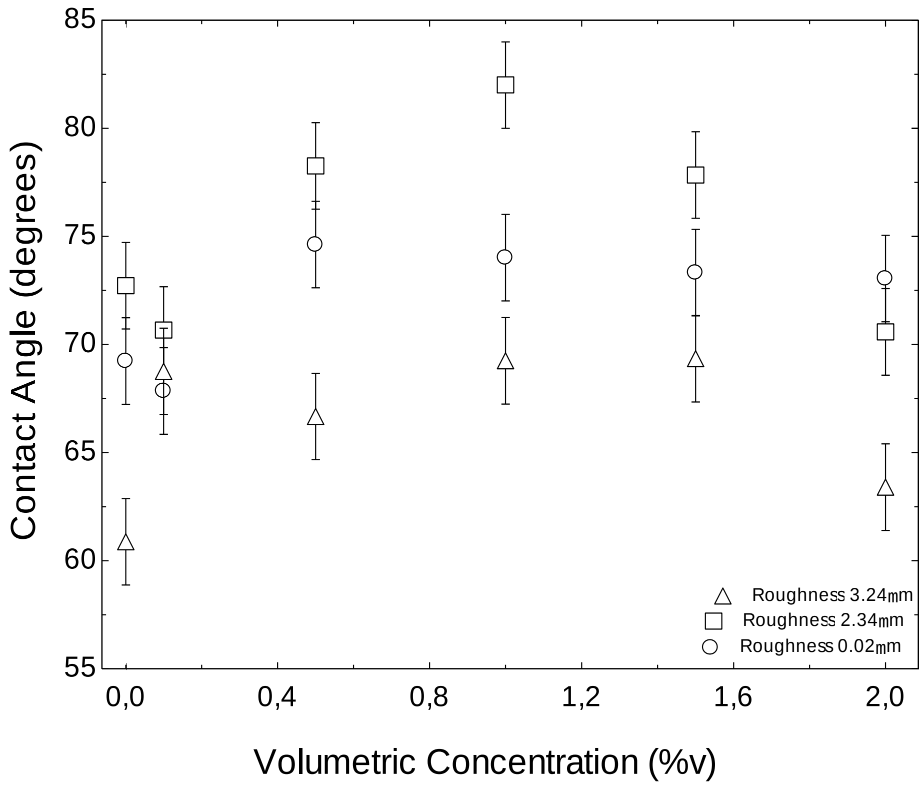

Figure 8. Effect of nanoparticle volumetric concentration and surface roughness on the contact angle of alumina with diameter of 40-80nm.

4. CONCLUSIONS

An experimental apparatus and procedure were developed in order of measuring the contact angle of sessile droplets onto solid surfaces. It was shown that the apparatus and the experimental method work reasonably well, providing accurate measurements. The wettability of nanofluids composed by alumina nanoparticles mixed with deionized water was evaluated based on contact angle measurements. For nanoparticles with smaller diameter, the results were not conclusive about the influence of the volumetric concentration of nanoparticles on the wettability. Although all the experiments have been conducted with rigid control of the volumetric concentrations, constant temperature and surface cleanness, the data obtained did not show any pattern solid enough to conclude what is the difference between nanofluids and the base fluid concerning wettability. Here, it is important to emphasize two variables that could possibly explain these results: surface heterogeneity and dropping method. The first one was a parameter taken into consideration when the surfaces were treated with a blasting of sand, which is the more homogeneous method found to change superficial roughness, but maybe it had not been enough. The second variable is the fact that although care has been taken by the operators, the dropping height of the droplets presents small variations. Even a trained operator using good pipettes cannot drop the fluid always from the same height, and that could cause variations in the shape of the droplets, and consequently in the contact angle measured. To take control over these two variables is the next step in order to enhance the accuracy of the contact angles measured and obtain more reliable data concerning wettability of nanofluids.
In case of the nanoparticles with diameters from 40 to 80nm, it seems that, first the contact angle increases with increasing the nanoparticle concentration, passes through a peak at a concentration of almost 1% and then decreases with further increment of nanoparticle concentration. Although, this average behavior was found, the effect of the local roughness on the contact angle was also observed as in the case of the nanoparticles with smaller diameters. It was also observed that the roughest surface provides the smallest contact angle.

5. ACKNOWLEDGMENTS

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