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1	Heat transfer properties of metal, metal oxides, and carbon water-based nanofluids in
2	the ethanol condensation process
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12	Abstract
13	This work investigates the convective heat transfer enhancement of water-based nanofluids in
14	pipe heat exchanger used for the ethanol condensation process. The nanofluids were
15	produced with different nature of nanoparticles, Cu, Fe ₃ O ₄ , MWCNT, and graphene, in the
16	volume concentration 0.01-0.1%, using different surfactants. These nanoparticles are
17	characterized by X-ray powder diffraction (XRD), Scanning electron microscopy (SEM),
18	specific surface area (BET), and Dynamic light scattering (DLS). Density, thermal
19	conductivity, and viscosity of base fluids and nanofluids were experimentally determined at a
20	relevant temperature of 20°C. Convective heat transfer enhancement under laminar regime

21 was evaluated from a well-designed experimental setup. As the main results, the thermal conductivity of nanofluids increases up to 3-5% and the viscosity can increase or decrease 22 23 with nanoparticle concentration, showing a lubricating effect of nanoparticles coupled with respective surfactant. It was shown that the heat transfer properties, heat transfer coefficient, 24 25 and Nusselt number, are increased with nanofluids compared to water and base-fluids, up to 20%, in the range of Pe 2000-10000. Experimental heat transfer properties are shown to be 26 27 greater than theoretical ones. Finally, copper nanofluid at low concentration appear to be the best candidate for the application and pipe flow geometry considered. 28

Keywords: Nanofluids; Heat transfer coefficient; Laminar flow; MWCNT; Graphene; Cu; Fe₃O₄

31	Nome	nclature
32	AAD	Absolute average deviation
33	BET	Brunauer, Emmett, and Teller method
34	CH	Chitosan
35	CNT	Carbon Nanotubes
36	Ср	Heat Capacity [kJ.kg ^{-1.} K ⁻¹]
37	D	Diameter (m or mm)
38	DLS	Dynamic Light Scattering
39	EG	ethylene glycol
40	f	friction factor
41	h	heat transfer coefficient [W $m^{-2} K^{-1}$]
42	HTC	Heat Transfer Coefficient [W $m^{-2} K^{-1}$]
43	L	length (m)
44	MWC	NT Multi-Walled Carbon Nanotubes
45	NFs	Nanofluids
46	NPG	Nano Porous Graphene
47	OA	oleic acid
48	PSD	Particle size distribution
49	PVP	Polyvinylpyrrolidone
50	SEM	Scanning Electron Microscope
51	SDS	sodium dodecyl sulfonate
52	Т	temperature [K]
53	vol	volumetric
54	Vp	Volume of pore
55	W	water
56	WEG	water-ethylene glycol mixture
57	wt	weight
58	XRD	X-ray diffraction
59	ASHR	AE American Society of Heating, Refrigerating and Air-Conditioning Engineers handbook
60	Greek	symbols
61	α	thermal diffusivity $[m^2 s^{-1}]$
62	β	nanolayer thickness factor

63	γ	shear rate [s ⁻¹]
64	κ	thermal conductivity [W $m^{-1} K^{-1}$]
65	μ	dynamic viscosity [Pa·s or mPa·s]
66	ρ	density [kg/m ³]
67	τ	shear rate [Pa]
68	f	volume fraction of nanoparticle
69	Subsci	ripts
70	0	reference
71	b	bulk fluid
72	bf	base fluid
73	exp	experimental
74	in	inner
75	h	hydrodynamic
76	LM	Average logarithmic
77	nf	nanofluid
78	np	nanoparticle
79	out	outer
80	th	theoretical
81	wnf	at average temperature of wall
82		
83		

84 **1. Introduction**

85 The process of evaporation and condensation involves the transfer of heat to and from the product stream, and then the change of the alcohol phase from the liquid to the vapor and vice 86 versa. This phase change requires a lot of added/removal heat at a constant temperature [1]. 87 Heat exchangers can perform such a process efficiently. Recently, nanoscience and 88 89 nanotechnology have provided a new solution by introducing nanofluids (NFs) that can ameliorate the heat transfer of thermal systems [2]. Experimental studies have shown that the 90 dispersion of solid metallic (Cu, Ag, Fe, Ni, ...), metallic-oxides (Al₂O₃, CuO, SiC, SiO₂, 91 92 TiO₂, Fe₂O₃, Fe₃O₄, ...) or carbon-based (CNT, MWCNT, graphene, graphene oxide, ...) 93 nanoparticles into the conventional base fluids can significantly enhance heat transfer

94 properties [3-6]. Most previous studies focused on the thermal and hydrodynamic properties of nanofluids, and experimental studies about convectional heat transfer properties and 95 coefficient evaluation are continuously growing. Main studies about the heat transfer 96 coefficients (HTC) evaluation with nanofluids in different flow regimes, laminar and 97 turbulent respectively, are summarized in [7]. The laminar flow regime is much more useful 98 to understand physical phenomena than turbulent or transitional ones, because the fluid flows 99 100 very regularly in the laminar flow, with a simple, smooth flow, and every particle keeps moving along the streamline [7]. In the heat transfer mechanism of nanofluids, thermal 101 102 conductivity and dynamic viscosity of the fluids involved play important roles [8] and must be rightly determined. Some relevant studies in the topic of the present work are introduced 103 hereafter. 104

105 Wen and Ding [9] studied the convective heat transfer of Al₂O₃ water-based nanofluids, flowing through a copper tube under laminar conditions. They evidenced that heat transfer 106 107 rising increases with Reynolds number, particle content and mainly produces in the tube entry. Heris et al. [10] investigated the convective heat transfer of CuO and Al₂O₃ water-108 109 based nanofluids in a pipe in the laminar regime and fixed wall temperature. Heat transfer coefficient raised with nanoparticle content and Peclet number, and be higher with Al₂O₃ 110 water nanofluids. He et al. [11] studied the forced convective heat transfer of TiO_2 water-111 112 based nanofluids in a vertical pipe. Heat transfer is significantly improved and shown to be sensitive to nanoparticle content and flow regime. Heris et al. [12] studied heat transfer of 113 Al₂O₃ water-based nanofluid under forced convection and laminar regime in a pipe. 114 Experimentally and analytically, the results showed the convection of nanofluids is linked to 115 thermal conductivity improvement. Heat transfer enhancement reached 16% for 0.2 wt% 116 117 Al₂O₃ nanofluids. Garg et al. [13] examined viscosity, thermal conductivity, and laminar convective heat transfer of MWCNT water-based nanofluids. It was indicated that heat 118 transfer enhancement increased by 32% and was higher than thermal conductivity 119 enhancement reaching 20%. Askari et al. [14] studied the thermal efficiency of a mechanical 120 wet cooling tower with counterflow and showed that carbon-based nanofluids are more 121 122 efficient than pure water. Graphene-Fe₃O₄ hybrid nanofluids with kerosene were investigated in [15]. Thermal and rheological properties were studied as well as convective heat transfer 123 124 under constant heat flux. As the main outcome, the convective heat transfer coefficient 125 reached 66% at 0.3 wt.% and Re~ 4500. Estellé et al. [16] reported thermophysical properties 126 and heat transfer efficiency of CNT water- and WEG-based nanofluids at 45°C under a 127 laminar regime. The maximum heat transfer enhancement for WEG-based nanofluids is around 18.5% at 0.05 wt%, while for water-based nanofluids it is around 11.8% with the 128 same concentration. Colla et al. [17] studied the convective heat transfer of water-based TiO2 129 nanofluids along uniformly heated pipe under laminar forced and mixed flow conditions. The 130 development of the Nusselt number along the pipe and the heat transfer with nanofluids were 131 found to be significantly different from the case of pure water. Barzegarian et al. [18] 132 investigated the thermal performance of Al₂O₃ water-based nanofluid through shell and tube 133 heat exchanger with segmental baffles. The results show a significant improvement of 134 135 Nusselt number as well as the total heat transfer coefficient by increasing the number of Reynolds. The amount of Nusselt number of nanofluids at 0.03, 0.14, and 0.3 vol% compared 136 to base fluid is raised, by 9.7, 20.9, and 29.8%, respectively. Sadeghinezhad et al. [19] 137 reviewed the experimental results regarding graphene nanofluids' thermo-physical properties 138 and heat transfer efficiency, evidencing the relevance of these nanofluids' family as heat 139 transfer enhancers. Guzei et al. [20] investigated the laminar forced convection of Al₂O₃, 140 TiO₂, ZrO₂, and diamond nanofluids at different concentrations from 0.25 to 6 vol%. It has 141 been shown that the local and average heat transfer coefficients in the Reynold number range 142 from 10 to 1500 increase with nanoparticles content. They also illustrated that the heat 143 144 transfer coefficient of nanofluids in the laminar regime is dependent on the thermal-physical properties of nanofluids. The effect of graphene nanofluid on a CO₂ heat pump device in a 145 146 heat interchanger has been studied by Wang et al. [21]. Exergy transfer in the heat exchanger for graphene nanofluids at 0.01~0.1 wt.% was higher than those with 0.5~1 wt.%. Recent 147 numerical simulations of nanofluid heat transfer in laminar flow [22, 23] have demonstrated 148 that the Nusselt number, drag coefficient, pressure drop, and heat transfer coefficient increase 149 150 with increasing nanoparticle concentration at a specified Reynolds number. Gholami et al. [24] as well as Gravndyan et al. [25] reported that the geometry of the heat transfer 151 configuration can have a significant effect on eat transfer increase with MWCNT oil and 152 TiO₂ water-based nanofluids. They investigated in particular how rib shapes affect the heat 153 transfer of nanofluid flow in a rectangular microchannel. 154

The previously cited works evidenced that heat transfer enhancement with nanofluids depends on nanoparticle nature, content, and dispersion state, range of Re number, temperature, thermophysical properties of nanofluids, heat exchanger configuration, ... Among these parameters, low content of nanoparticles in the nanofluids is required in terms of cost and operation, such as preventing blocking, instability, pump power reduction, and other negative factors [26]. Because of its proneness to coagulation, nanofluids can lose their ability for heat transfer. In addition, the key problem when using nanofluids is also dispersion state that can change the thermo-physical properties of nanofluids and their suitability in various applications. This can be improved by considering different stabilization techniques such as the use of surfactants or surface functionalization of the nanoparticles before dispersing them in the base fluid or other methods [27]. Consequently, a challenge is the determination of surfactants' role, which is not always considered.

Finally, our aim is to investigate the thermal efficiency of different water-based nanofluids in 167 a heat exchanger used for the ethanol condensation process. With this goal, several 168 169 nanoparticles that differ in nature were considered, copper (Cu) as metallic nanoparticles, Fe₃O₄ as metal oxides, and MWCNT and NPG as carbon nanoparticles respectively. This was 170 done to clearly compare the impact of nanoparticle nature on heat transfer efficiency. These 171 nanoparticles were fully characterized by XRD, SEM, BET surface area and DLS analysis. 172 173 These nanoparticles are used to prepare water-based nanofluids with low content of 0.01, 0.05, and 0.10 vol.% in concentration using some surfactants. The thermophysical properties 174 of these nanofluids and their base fluids are also evaluated at 20°C. In addition, heat transfer 175 experiments are reported and discussed, showing the impact of surfactants, nanoparticle 176 content, and nature on the heat transfer coefficient and Nusselt number which are compared 177 178 to theoretical correlations.

179

180 **2. Experimental Methods**

181

2.1. Nanoparticles and Nanofluid Preparation

182

In this work, commercial copper nanoparticles (Cu) with an average size of 40 nm were 183 purchased from VWR (Alfa Aesar, ROTI®nanoMETIC), while magnetite nanoparticles 184 (Fe₃O₄), nanoporous graphene (NPG), multi-walled carbon nanotubes (MWCNT) were 185 synthesized by our group in the framework of this investigation as described below, prior to 186 specify the method for producing nanofluids. Materials and chemicals used for the synthesis 187 and production of the different nanoparticles and nanofluids were purchased from 188 commercial sources (Sigma-Aldrich and Merck). In all experiments dedicated to the 189 nanoparticles synthesis, deionized water was used. 190

With regards to Fe_3O_4 , the process was yet fully described in [25][28] and used in the similar way. The reader is referred to these works for a full explanation of the synthesis process.

Nanoporous graphene was synthesized by chemical vapor deposition (CVD) including 193 194 methane as the carbon source and hydrogen as the carrier gas in a ratio of 4:1, within a quartz tube (diameter 5 cm, length 120 cm) into an electrical horizontal furnace at temperatures of 195 900-1100°C for 30-45 min. After the growth and cooling process, the product was stirred in 196 18% HCl solution for about 16 h at ambient temperature to obtain pure nanoporous graphene 197 and remove the metal nanocatalysts [29]. Then, the sample was washed repeatedly with 198 deionized water until the neutral product was obtained. Finally, the product was dried in an 199 oven at 100 °C for 12 h. 200

The MWCNTs were produced by CVD consisting of 20% methane in hydrogen over Co-201 202 Mo/MgO catalysts [30]. The catalysts were reduced first with the mixture of hydrogen and nitrogen under a flow rate of 200 ml/min. The methane (20% in hydrogen) was used into the 203 reactor as a feed of the reaction. The quartz reactor size is 140 cm in height and 2 cm in inner 204 205 diameter. A porous quartz disc distributor was used to hold the nanocatalysts. For producing MWCNTs, the reaction temperature was increased from 850°C to 1050°C. The growth of 206 carbon nanotubes was obtained using methane in hydrogen with a flow of 250 ml/min. The 207 synthesized nanoparticles and HCl (18%) mixture were stirred at ambient temperature for 16 208 hours to purify the nanoparticles. Once produced, the MWCNTs were washed with distilled 209 water and dried at 120°C for 8 h. 210

For the dispersion and the stabilization of nanoparticles within distilled water considered as the base fluid, different commercial stabilizers and surfactants have been used. Their nature and content are summarized in Figure 1 with the associated nanoparticles.



216

Figure 1. Overview of nanomaterials and stabilizers used for producing nanofluids

217 Nanofluids were produced adding the required mass of nanoparticles into the base fluid with the corresponding surfactants, see figure 1, using ultrasonic mixing for 15 min. A probe 218 sonicator (Qsonica, USA, LLC 60Hz, Q700W, Sonication Pulse Rate: 1s ON, 1s OFF) was 219 used and the temperature sample was controlled from a cooling jacket during nanofluid 220 preparation. A series of nanofluids with different volume concentrations of nanoparticles 221 were accordingly prepared, 0.01, 0.05, & 0.1% respectively, for each type of nanoparticles, 222 e.g. Cu, Fe₃O₄, MWCNT, and NPG. Such concentrations were obtained from the densities 223 values of nanoparticles reported later in Table 1. 224

225 226

2.2. Characterization of Nanomaterials

As in our previous work [25], X-ray diffraction (XRD) patterns of nanopowders were obtained with X' Pert Pro diffractometer (Philips, PW 1800 X-ray, Netherlands) with Cu Ka (l = 1.5406 Å) radiation in the angular domain of $10 < 20 < 80^{\circ}$ to identify the crystal structure of nanoparticles. SEM (Philips, XL-30ESEM, Netherlands) was used for direct examination of nanomaterial morphological structure. The size and morphological characterization of the nanoparticles were investigated at a 15 kV operating voltage. The surface area of the molecular sieve and the porosity of nanopowders were evaluated at 77 K using physisorption of nitrogen with ELSORP-mini II (BEL Japan Inc., Japan). The associated Data Analysis
Software based on the adsorption isotherms was used for determining the BET surface areas
of the nanoparticles.

237 238

2.3. Thermo-physical properties measurements

The thermophysical properties of nanofluids were experimentally evaluated at 20°C. The density of nanofluids was evaluated from the pycnometer method according to ASTM D153. The measurement uncertainty, evaluated with distilled water, is reported to be less than \pm 0.2%.

Dynamic light scattering (DLS) measurement using a Zetasizer Nano S (Malvern Instruments, UK) was performed to evaluate the size of the nanoparticles at 20°C. The samples were prepared using few drops of each nanofluid dispersed in the excess base fluid to achieve an acceptable degree of dilution (approximately 40 times, detection by instrument showing optimum level) for optical clarity, followed by sonication. As previously reported in the literature [17], DLS-size results obtained for low-concentrated dispersions can be representative of dispersion state at slightly higher concentrations.

Rheological measurements of the nanofluids were performed with a stress-controlled 250 rheometer (Malvern Instruments Ltd, Malvern Kinexus Pro, UK) with a 60 mm diameter 251 cone-plate configuration, a 1° cone angle, and a 0.03 mm in gap and controlled temperature 252 by a Peltier system implemented into the lower plate. Experiments were conducted at 20°C 253 for base fluids and nanofluids. Rheological flow curves were obtained imposing shear 254 stresses for covering the shear rate range from 10 to 1000 s⁻¹ with at least 10 points per 255 decade. Shear rate and dynamic viscosities were collected under steady-state conditions. 256 Additional details about rheological experiments can be found in Cabaleiro et al. [31]. The 257 viscosity values of water, which behaves in a Newtonian manner as expected for all 258 259 temperatures, were favorably compared to ASHRAE data [32] with an Absolute Average 260 Deviation (AAD) less than 3%. Experiments were done in 3 replicates for each sample.

A THW-L2 Portable thermal conductivity meter (Thermtest Inc., Canada) was used to measure base fluid and nanofluid thermal conductivity at 20°C. This device is based on a quick hot-wire technique according to the ASTM D7896 standard. In the temperature range 5/20°C, an AAD less than 1.5% was obtained with distilled water compared to ASHRAE data [32, 33]. Experiments were done according to [28] and the reader can refer to this work for more details. Briefly, a power input varying between 90-110 mW was applied, and a short time of 1.5s was used for thermal conductivity measurement avoiding convection. The experiments were repeated 5 times for each sample, the reported values being an average of these data. Each sample was stayed at the required temperature for at least 1 h before being tested.

Finally, the heat capacity of nanoparticles was measured at 20°C by an adiabatic calorimetry device (AK-9.02-BCT-21 calorimeter, TERMAX, Russia) with an uncertainty of 0.2%. The results are the average of three repeated tests.

274 275

2.4. Heat transfer experiments

As mentioned in the introduction, the process of evaporation and condensation involves the transfer of heat to and from the product stream, and then the change of the alcohol phase from the liquid to the vapor and vice versa. This phase change requires a lot of added/removal heat at a constant temperature [1]. Such a process can be efficiently realized with heat exchangers. Consequently, the present research reports an experimental study and set-up about the heat transfer evaluation during the ethanol condensation process, as described in Figure 2, and discussed in detail further.



Figure 2: Diagram for the HTC experimental set-up for heat transfer experiments.

285 Ethanol is used to test the condensation process in the heat transfer experiments. At first, the ethanol is evaporated in the heater and later it is condensed in the heat exchanger. Water-286 based nanofluids are considered as heat transfer fluids. A double-shell heat exchanger is used 287 and was covered with several distinct layers of sponge rubber insulation foam with a high 288 thermal resistance value (thermal resistance 1.25 K.W⁻¹). K-type thermocouples are 289 positioned along with the flow for the inlet and outlet of the test section for measuring the 290 bulk temperatures of nanofluids as well as wall temperature. To ensure a constant temperature 291 at the inlet of the test section, the heated fluids return to the collection tank passing through a 292 293 cooling unit, which is a shell and tube heat exchanger. The temperatures during experimental runs are recorded with a data acquisition system. Seven different flow rates of fluid into the 294 cold side were tested in the range 6-36 $g.s^{-1}$ varying pump level of the circulator bath. The 295 flow rate on the hot side (ethanol condensation) is 0.2 g.s⁻¹. The temperature in the cold side 296 started at 20°C for water-based nanofluids and reaches a maximum of 26°C for the lowest 297 flow rate of 6 g.s⁻¹ because of the ethanol condensing process on the other side of the heat 298 exchanger. All the data were recorded in the steady-state condition. To ensure consistency, 299 the tests were replicated twice, and the results were repeated with equal precision. A 300 301 manometer was used to measure the liquid pressure drop in the tubing. The pressure drop 302 uncertainty was evaluated around 0.1 %. As it is explained later, different type of water-based nanofluids were considered, varying also their concentration in nanoparticles, as well as the 303 304 flow rate in the heat exchanger to evaluate their performance from heat transfer coefficient evaluation. 305

306 **2.5. Data Analysis**

307 During the experiments, the tube wall temperature, the inlet and outlet temperature of the 308 sample nanofluid, the mass flow rate as well as the static pressure difference were measured. 309 Due to the passage of nanofluids through a circular tube, the convective heat transfer 310 coefficient h and the Nusselt number can be evaluated under laminar flow condition as 311 follows [34]:

312
$$\overline{h_{nf}}(exp) = \frac{Q(exp)}{\pi D_h L(T_w - T_b)_{LM}}$$
 (1)

313
$$Q(exp) = \dot{m}_{nf}C_{Pnf}(T_{b1} - T_{b2})$$
 (2)

314
$$T_{w} = T_{win} - \frac{Q(exp)}{2\pi L_{in}k_{glass}} ln \left(\frac{D_{out}}{D_{in}}\right)$$
(3)

315
$$\overline{\mathrm{Nu}_{\mathrm{nf}}}(\mathrm{exp}) = \frac{\overline{\mathrm{h}_{\mathrm{nf}}}(\mathrm{exp})\mathrm{D}_{\mathrm{h}}}{\mathrm{k}_{\mathrm{nf}}}$$
 (4)

Where $(T_w-T_b)_{LM}$ is the average logarithmic temperature difference. D_{in} (45 mm) and D_{out} (65 mm) are inner and outer diameters of the inner tube which has a thermal conductivity of 0.8 W.m⁻¹.K⁻¹ (K_{glass}) and L_{in} (1.35 m) is its length. The length of the heat exchanger is L=0.295 m and its hydrodynamic diameter is D_h=31.6 mm leading to a L/D ratio of around 9.5. T_{win} and T_w are inner and outer temperature of the wall in the heat exchanger respectively.

In order to evaluate the relevance of the experiments and validate the set-up, first, the measurements were performed using pure water as heat transfer fluid for which correlations under laminar forced convection in a pipe are well-known. Also, the Nusselt number predicted by the Seider-Tate equation was compared with the calculated Nusselt equation using experimental data. The Nusselt number was computed from the following correlation [35].

327
$$\overline{Nu_{nf}}(th) = 1.86 (Re_{nf} Pr_{nf} \frac{D_h}{L})^{1/3} (\frac{\mu_{nf}}{\mu_{wnf}})^{0.14}$$
 (5)

328
$$\overline{h_{nf}}(th) = \frac{\overline{Nu_{nf}}(th) k_{nf}}{D_{h}}$$
 (6)

This correlation is valid for 100 < Re < 2100, 0.6 < Pr < 100, and L/D < 0.1 Re Pr.

330 μ_{wnf} is the viscosity of nanofluids at wall temperature. In addition, the following 331 dimensionless numbers are used for determining the heat transfer coefficient under the 332 laminar flow of nanofluids through the circular tube.

$$333 \qquad \operatorname{Re}_{\mathrm{nf}} = \frac{\rho_{\mathrm{nf}} \mathrm{UD}_{\mathrm{h}}}{\mu_{\mathrm{nf}}} \tag{7}$$

$$334 \qquad \Pr_{\rm nf} = \frac{C_{\rm P_{\rm nf}}\mu_{\rm nf}}{k_{\rm nf}} \tag{8}$$

$$335 \quad Pe_{nf} = \frac{UD_h}{\alpha_{nf}} \tag{9}$$

$$336 \qquad \alpha_{\rm nf} = \frac{k_{\rm nf}}{\rho_{\rm nf} C_{\rm P_{\rm nf}}} \tag{10}$$

337

The thermophysical properties of nanofluids in the previous equations were experimentally determined as described in section 2.3. With regards to specific heat capacity Cp evaluation, the following widely used correlation has been considered, at the average bulk temperature of nanoparticles and water, e.g 20°C [34].

342
$$C_{p_{nf}} = \frac{\phi \rho_{np} C_{p_{np}} + (1-\phi) \rho_{bf} C_{p_{bf}}}{\rho_{nf}}$$
 (11)

343 All these thermophysical properties will be used in the following to compare theoretical and experimental heat transfer properties in heat exchanger involved in the ethanol condensation 344 process described previously. The data in Table 1 provides the needed properties of 345 nanoparticles and surfactants for all calculations. 346

347

Table 1: Properties of nanoparticles and surfactants at 20°C. 348

Materials/Properties	Density Kg.m ⁻³	Thermal Conductivity $W.m^{-1}-K^{-1}$	Heat Capacity kJ.kg ^{-1.} K ⁻¹
Water	998.2 *	0.596 *	4.186 ^[32]
Cu	8920 **	401 [36]	0.463 *
Fe ₃ O ₄	5180 [28]	17.7 [37]	0.679 *
MWCNT	2100 [14]	3000 [38]	0.857 *
NPG	2200 [39]	5000 [39]	1.211^{*}
Chitosan	725 **	0.12 [40]	1.04 [40]
Oleic Acid	895 **	0.17 [41]	2.05 [41]
SDS	1010 [42]	0.212 [42]	0.706 ^[42]
PVP	1200 **	0.105 ^[43]	1.54 ^[43]

349 350

* Experimental value *Given by producer

351

2.6. **Uncertainty evaluation** 352

The uncertainty of the variables and the calculated parameters were obtained using the 353 354 following equation [44]:

355
$$\frac{\Delta R}{R} = \frac{1}{R} \sqrt{\sum_{n=1}^{N} \left(\frac{\partial R}{\partial x_i} \Delta x_i\right)^2}$$
(12)

356 The uncertainty of equipment and devices is reported in Table 2. The uncertainty of the heat transfer coefficient, Re number, Nu number for water-based nanofluids were evaluated to 357 ±3.00%, ±3.61%, and ±3.35%, respectively. 358

359 Table 2: Accuracy of the measuring instruments.

Description	Model	Accuracy / Relative accuracy
Temperature	Type K, Thermocouple	$\pm 0.01^{\circ}$ C
Thermal Conductivity	THW-L2, Thermtest Inc.	± 1.5%

Dynamic Viscosity	Rheometer, Malvern Kinexus Pro	± 3%
Fluid flow rate	Circulator Bath	<u>±</u> 2%
Length (L,D _h)	Vernier Caliper	\pm 0.02 mm
Density	Pycnometer	± 0.2%
Pressure	manometer	<u>±</u> 0.1%
Weight	Measuring balance	<u>± 0.0001 g</u>

361 **3. Results and Discussion**

362 **3.1.** Characterizations of nanoparticles

XRD characterization of nanoparticles used for nanofluids production are reported in Figure 363 3. The average nanoparticles' crystallite sizes were calculated with the Scherrer formula [45]. 364 The diffraction peaks for Cu appears at $2\theta = 43.3^{\circ}$, 50.5° and 74.1° and correspond to 365 crystallographic planes of (111), (200), and (220), respectively (indexed to face-centred cubic 366 crystals with JCPDS card No. 4-836) [46, 47]. Crystallite sizes of Cu nanoparticles possess 367 an average size of 30 nm. Diffraction peaks of the Fe₃O₄ (JCPDS file No. 75-0449) take place 368 at 30.3°, 35.7°, 43.3°, 53.7°, 57.3°, and 62.9°, in agreement with crystallographic planes 369 (220), (311), (400), (422), (511), and (440), respectively, with no indication of the possible 370 presence of α -Fe₂O₃ [12]. The average calculated size for Fe3O4 crystal is 10 nm. For NPG 371 XRD patterns, the peaks at $2\theta = 29^{\circ}$ (corresponding to a d-spacing of 0.30 nm), and 48°, can 372 373 be observed corresponding to the diffraction of the crystal planes of graphene which could be indexed to the (002) and (100) planes, respectively (JCPDS No. 01-0646) [29, 48]. With 374 MWCNTs, crystallographic (002) and (100) planes are indexed to the main diffraction peaks 375 376 for MWCNT at $2\theta = 26^{\circ}$, and 43.5° , respectively [49].

377





380

Figure 3: XRD of nanoparticles.

The morphologies of nanoparticles, evaluated from SEM characterization are shown in Figure 3. Fe₃O₄ nanoparticles appear in form of aggregated spheres with a size from 20 to 60 nm. SEM picture of Cu shows that nanoparticles are almost in a spherical shape and are rather in aggregated form with the average size in the range 80-160nm. The SEM image also illustrates the highly porous morphology of the graphene sheets with pore sizes ranging from 0.1 to 1 μ m. For MWCNT, a tubular, filamentary, and multi-walled structure of nanotubes via highly porous network morphology is observed.



389

390

Figure 4. FESEM images of nanoparticles.

391 The BET surface area, pore volume (BJH), and the average pore size of Fe₃O₄, Cu, MWCNT,

and NPG are gathered in Table 3.

393	Table 3:	Surface area,	pores'	size and	volume an	d sizes	of the nano	oparticles.
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Sample	surface area	pore size pore volume		Average Size (nm)
	m ² .g ⁻¹	\mathbf{A}°	cm ³ .g ⁻¹	
Fe ₃ O ₄	114	221	0.77	10.1
Cu	15	n.c.	n.c.	45
MWCNT	994	88	1.41	n.c.
NPG	850	99.5	2.11	n.c.

n.c.: not calculable

The average nanoparticle size d_{BET} approximate as spherical is calculated from the following equation $d_{BET} = 6000/(\rho \times S_{BET})$. Here, S_{BET} is the surface area (m²/g) and ρ is the skeletal

density (g/cm³) [45]. As shown in Table 3, an average size of 10.1 and 56 nm was evaluated

for Fe_3O_4 and Cu nanoparticles respectively, which agrees with crystallite sizes previously reported.

400 The particle size distribution (PSD) of nanoparticles measured by Dynamic Light Scattering (DLS) at 20°C is shown in Figure 5. The average hydrodynamic diameters of Cu, Fe₃O₄, 401 MWCNT, and NPG nanoparticles in water are 373, 164, 296, and 353 nm respectively. PSDs 402 403 are in agreement with the length and size distribution reported from SEM analysis, but also 404 suggest that nanoparticles can be in the form of small aggregates within water. Figure 5 also shows the presence of double peaks that represent some of the overall fine particles of NPG 405 406 nanoparticles. It should be pointed out that dynamic light scattering (DLS) measurements are based on the assumption that particles are spherical while the studied NPG nanoadditive are 407 408 sheet-like shaped and their orientation can play a role. Two-peak and three-peak DLS size distributions were previously found in the literature for other non-spherical nanoparticles [50-409 410 52]. In addition, in order to evaluate dispersion ageing throughout time, samples were placed in vials and left at rest under ambient conditions. As evidenced by the Figure 6, the visual 411 inspection of the samples did not show any noticeable sedimentation within several days. 412

413





Figure 5. Dynamic light scattering analysis data of diluted nanofluids.



417 Figure 6. Visual aspect of nanofluid sample after 7 days at rest and ambient condition.

3.2. Thermophysical properties of nanofluids



431
$$\rho_{nf} = \phi \rho_{np} + (1 - \phi) \rho_{bf}$$
 (13)

432 Thus, Figure 7 shows that a good agreement is achieved with Cu and Fe₃O₄ nanofluids between experimental and theoretical data. However, the density of NPG and MWCNT 433 deviates from equation (12). Such a gap comes from the fact that part of the base fluid can be 434 adsorbed and trapped within the NPG and MWNCT nanoparticle porosity [53]. The apparent 435 436 volume of the fluid can be reduced, and consequently, the density of nanofluid is increased. Based on this, a theoretical model considering the porosity of nanoparticles is proposed. The 437 total volume of pore (Vp) obtained from BET characterization (see Table 3) is considered to 438 refine the density correlation for MWCNT and NPG nanofluids as follows: 439

$$440 \quad \theta = V_{\rm p} \,\rho_{\rm np} \tag{13}$$

441
$$\rho_{\rm nf} = \left(\frac{1}{1+\theta}\right) \emptyset \ \rho_{\rm np} + (1 - \emptyset \ (1 - \theta)) \ \rho_{\rm bf} \tag{14}$$

A comparison of this theoretical equation and experimental data is also shown in Figure 6. In
that case, a good agreement is achieved, showing the influence of nanoparticle porosity on
the density of carbon nanofluids.

445



Figure 7. Comparison of experimental data of nanofluid density with theoretical
 models

449 Figure 8 shows the dynamic viscosity against shear rate for all tested fluids at 20°C. The figure evidences the difference in rheological behaviour and dynamic viscosity enhancement 450 following the base fluid used and the type of nanoparticles and their content. With Fe₃O₄, 451 both base fluid and nanofluids behave in a Newtonian manner, with no significant change of 452 dynamic viscosity with shear rate (within the experimental uncertainty). The presence of 453 surfactant slightly increases the viscosity of water. Viscosity of nanofluids is increased with 454 nanoparticle content with no significant difference for the higher concentrations. For Cu 455 nanofluids, the trend is different. While the fluids are mainly Newtonian, the viscosity is 456 457 increased by 50% with the surfactant. When nanoparticles are added to the base fluid, the viscosity is decreased with the content until to reach the viscosity of base fluid with the 458 higher content in nanoparticles. With regards to MWCNT, base fluid and nanofluids are also 459 Newtonian. For low concentrations, dynamic viscosity appears to be lower than the one of 460 base fluid, while it is higher for the higher concentration. Finally, NPG nanofluids present 461 more complex behaviour with slight shear-thinning for the more concentrated nanofluids and 462 the base fluid. The dynamic viscosity of nanofluids is lower than the one of base fluid for the 463 two lower concentrations. It is higher by 0.1%. Interestingly, some of the nanofluids have 464 reduced viscosity showing a lubricating effect of the nanoparticles coupled to respective 465 466 surfactant. Some studies [28, 57, 58] also reported that some nanofluids have this lubricating effect, resulting in a decrease in viscosity. 467





469

470 471

Figure 8. Dynamic viscosity versus shear rate of water, base fluid and nanofluids at 20°C – Impact of nanoparticle type and content

472 Figure 9. presents the thermal conductivity of base fluids and water-based nanofluids for different concentrations at 20°C. As a general trend, it is observed that the presence of 473 474 surfactant/stabilizers, that are used to disperse and stabilize nanoparticles into water, tends to 475 slightly decrease the thermal conductivity of water. This behaviour was also previously reported with different other surfactants [59]. However, a recent study using molecular 476 dynamics simulations evaluated the effect of surfactant volume ratio on the thermal 477 performance of the base fluid, as well as the effect of base liquid and surfactant interactions 478 on the thermal efficiency of the nanofluid. As a result, the surfactant-containing nanofluid's 479 thermal conductivity was greater than the primary nanofluid's [60]. Also, it is shown that the 480 thermal conductivity of nanofluids is increased with nanoparticle content, as generally 481 expected. At the highest value of nanoparticle concentration, the increases in thermal 482 conductivity reach 2%, 3%, 2%, and 5% for Cu, Fe₃O₄, MWCNT, NPG nanofluids, 483 respectively. While nanoparticles have different thermal conductivity values (see Table 1) 484 485 following their nature, it appears that the thermal conductivity of nanofluids does not vary a lot with the concentrations considered. With the same type of nanoparticles, previous studies 486 487 have shown an increase in thermal conductivity of water-based nanofluids, typically for Cu, an enhancement of 11% at 0.1 vol.% [61], and 15% at 0.5 wt.% [62] was reported. With 488 489 Fe_3O_4 , previous works showed 7% in thermal conductivity enhancement at 0.1 vol.% [63], and 3% at 0.1 vol.% [64]. For MWCNT, a thermal conductivity increase about 4% at 0.25 490 491 vol.% [65], and 6% at 1 vol.% [66] was demonstrated. With graphene, the thermal conductivity increased by 1.4% at 0.1 wt.% [67] and 3.5% at 0.07 wt.% [68]. However, one 492 493 can noticed that the enhancement we reported here are in the same order of magnitude, while 494 the concentrations considered are low.



498

Figure 9. Thermal conductivity values of base fluids and nanofluids as a function of concentration and type of nanoparticles at 20°C. 499

3.3. Heat transfer properties of nanofluids 500

First, as a validation of the experimental set-up and theoretical analysis, we report in Figure 501 10 the results for distilled water. This Figure gives the evolution of Nu against Pe and friction 502 factor against Re, comparing theoretical and experimental data. The results of the Nu 503 504 numbers obtained from equation (4) and the experimental values shown in Figure 10 follow the same trend and they are close, with a standard deviation of $\pm 11\%$. Finally, a quite good 505 agreement is achieved between the current study and the relevant predictions defined 506 previously. Moreover, the Figure also demonstrates the good agreement between the wall 507 friction factor f calculated by the Darcy–Weisbach equation $f = 2D\Delta P/\rho u^2 L$ for Newtonian 508 liquids [69] and the classical correlation, f = 64/Re for liquids. Presently, the Reynolds 509 number resulting from pumping power ranges from 300 to 1500. 510

495



The convective transfer coefficient h with respect to the Peclet number Pe for water, base fluids, and nanofluids at 20°C is reported in Figure 11. This Figure shows that the heat transfer coefficient is increased with Pe and some differences can be noticed between base fluids and nanofluids, as well as with the nature of nanoparticle and their content.



Figure 11. Base fluids and nanofluids heat transfer coefficient as a function of Peclet
number.

523 To get a clear picture of the different impacts of surfactant and nanoparticle nature and 524 content on heat transfer coefficient compared to pure distilled water, Figure 12 shows the 525 heat transfer coefficient ratio of both base fluids and nanofluids to water.



526

Figure 12. Ratio of base fluids and nanofluids' heat transfer coefficient to water heat
transfer coefficient vs Peclet number

529 First, it is observed that surfactants alone do not induce similar results. The presence of some 530 surfactants can penalize the heat transfer coefficient of water, as the ratio is lower than 1. That is mainly the case for the mixture of PVP SDS and OA SDS. With SDS alone, in the 531 range of Pe number, the ratio is lower than 1 for low Pe number, while it is higher than 1 for 532 high Pe number. For CH, the ratio is always higher than one. Then, nanoparticles addition to 533 base fluids improves the heat transfer coefficient as the ratio is higher than 1, and can reach 534 up to 20% in enhancement. Hence, all nanofluids have a higher coefficient of heat transfer 535 than distilled water while the thermal conductivity of nanofluids was not greatly improved 536 with nanoparticles. The results show that the higher the percentage of nanoparticles in the 537 fluid, the higher is the heat transfer ratio. From previous findings, it is known that the heat 538 539 transfer coefficient increases with an increase in the concentration of nanoparticles [3, 70]. Actually, Cu, Fe₃O₄, MWCNT, and NPG nanofluids have higher heat transfer coefficients 540

than water. The enhancements compared to water are 13, 7, 4, and 4.5% for Cu, Fe₃O₄, 541 MWCNT, and NPG nanofluids at 0.05 vol.% respectively, at a low Peclet number of 2300. 542 This enhancement reaches 18, 12, 10, and 8.5% at high Peclet number (8000), respectively 543 for Cu, Fe₃O₄, MWCNT, and NPG nanofluids at 0.05 vol.%. Such an enhancement can be 544 545 attributed to the coupled effect, at these low concentrations, of the slight thermal conductivity increase while the viscosity is moderately increased or even decreased compared to water. 546 Finally, among all nanofluids and concentrations, Cu nanofluids provide the best 547 enhancement in heat transfer compare to water in the entire range of Pe number. High 548 enhancement is also obtained for Fe₃O₄ and MWCNT but only at higher Pe numbers. These 549 results are consistent with previous studies. Actually, although the heat transfer geometry, 550 nanomaterials, and experimental conditions are not fully identical, our results can be 551 552 compared with some studies. For example, it has been reported that the local heat transfer coefficient at Re = 1600 has increased by 8% for Al_2O_3 water-based nanofluids at 0.6 vol. %, 553 the thermal conductivity of this nanofluid being around 3.5 % higher than the base fluid [9]. 554 Other works with water-based nanofluids reported a heat transfer enhancement of 5% for 555 556 Al₂O₃ nanofluids (0.2 vol.%) and 6% with CuO nanofluids (0.2 vol.%) at Peclet number of 5000 [10]. Also, changing the Pe number from 2500 to 6000 leads to an heat transfer increase 557 from 8 to 16% for Al_2O_3 nanofluids (0.2 vol.%) [12]. Similarly, a heat transfer enhancement 558 559 about 6 % was obtained with MWNTs nanofluid (0.1 wt.%) and NPG nanofluid (0.1 wt.%) at 33 °C [15]. Such an enhancement reaches 11.8 % for CNT nanofluids (0.055 wt.%) in [16] 560 and 5.4, 10.3 and 19.1% with Al₂O₃ nanofluids with the following concentrations 0.03, 0.14 561 and 0.3 vol.%, respectively [18]. In addition, an increase in Re number from 200 to 1000 562 rises the heat transfer coefficient from 6 to 20% for Al₂O₃ nanofluids (4 vol.%) and 8 to 16% 563 for TiO_2 nanofluids (4 vol.%) respectively [20]. Finally, it is observed that the heat transfer 564 enhancement presently reported can reach higher value while the concentration in 565 566 nanoparticles is lower.

567

Furthermore, Figure 13 presents the convective heat transfer coefficient ratio of both base fluids and nanofluids to the theoretical value defined previously in section 2.5. The nanofluid's heat transfer coefficients are significantly higher than theoretical values, as seen in Figure 13. Particle size, materials, shape and concentration, type of base fluids, surfactant, boundary condition, and range of Reynolds number are critical considerations for evaluating

their effect on the heat transfer coefficient in the laminar flow in circular tubes [71, 72]. 573 Although nanofluid heat transfer behavior depends on the volume fraction, average size and 574 species of nanoparticles, and flow conditions [73, 74]. The reported difference in heat transfer 575 coefficient is correlated to different factors and mechanisms not included in the theoretical 576 577 development. A better prediction for nanofluids heat transfers could be found by analysing factors such as the interaction flow structure and the collision intensity of nanoparticles, as 578 well as the dispersion and relative movement of these particles throughout the vicinity of the 579 tube wall, the temperature profile, and the thickness of the boundary layer [18, 75]. For 580 example, the temperature near the wall tends to be greater than that used for calculation, and 581 the properties of nanofluids can evolve during heat transfer, as the temperature slighty 582 increases during the condensation process, thereby enhancing the nanofluid's heat transfer. 583 584 The strong effect of particle properties on fluid and the impact of nanofluid composition on flow and heat transfer properties, experimental uncertainty, a key mechanism of nanofluid 585 586 flow, can be the source of the gap between experimental data and theoretical prediction.



587

588 Figure 13. Ratio of base fluids and nanofluids' heat transfer coefficient 589 to theoretical heat transfer coefficient in function of Peclet number

Nusselt numbers are shown in Figure 14 with respect to the Peclet number for all base fluidsand water-based nanofluid at various concentrations. Experimental values are also compared

592 to theoretical ones. Thus, the Nusselt number increases with an increase in the Peclet for all fluids. The increase is greater for nanofluids, compared to base fluids, with an increase in the 593 percentage of nanoparticles, particularly in higher Peclet. Additionally, previous research 594 indicates that increasing the Reynolds number (which results in an increase in the number of 595 596 Peclet) and the volume concentration of particles increases the Nusselt number [12, 20]. For example, Nusselt numbers increased by approximately 9.9, 20.9, and 29.8% for Al₂O₃ 597 nanofluids at the highest tested Reynolds number for volume concentration of 0.03, 0.14, and 598 0.3, respectively [18]. The Nusselt number of all fluids, however, is greater than the 599 theoretical number calculated by Seider-Tate's correlation (Equation 5), but the increasing 600 trend is consistent with the theoretical calculation. Although it was reported in [20] that the 601 number of Nusselt depends to a small extent on the type of nanoparticles, other work [10] 602 603 shows that the increase in Nusselt number for Al₂O₃ water nanofluid is greater than the increase in Nusselt number for CuO water nanofluid. Nevertheless, the results of this study 604 605 show that, in addition to the type of nanomaterials used, type of surfactants can fundamentally change Nusselt numbers by altering thermophysical properties of nanofluids. 606









611

612 613

Figure 14. Nusselt number of base fluids and water-based nanofluids versus Peclet number at 20°C. – Influence of nanoparticle nature and concentration

For comparison purposes between nanofluids, the Nusselt number with respect to the Peclet 614 615 number for all water-based nanofluids with 0.05% in vol. at 20°C are gathered in Figure 15. The pattern of Nusselt number with Peclet number for all nanofluids is consistent with the 616 data obtained from theoretical calculations, as also shown previously in Figure 13. At low 617 Pe=2300, the Nusselt numbers are enhanced by 12%, 4.5%, 2.5%, and 2.5% for Cu, Fe3O4, 618 MWCNT, and NPG nanofluids, respectively, at 0.05 vol%. At high Pe=8000, the Nusselt 619 numbers are enhanced by 22%, 12%, 10%, and 8% for Cu, Fe₃O₄, MWCNT, and NPG 620 nanofluids, respectively at the same concentration. Consequently, as evidenced by heat 621 transfer coefficient evolution, the best nanofluid is based on copper nanoparticles. 622

623



629

Figure 15. Nu number of base fluids and water-based nanofluids at 0.05 vol.% in
 respect Peclet number at 20°C.

628 **4.** Conclusion

630 We reported in this paper the heat transfer properties of water-based nanofluids during the ethanol condensation process. Different types of nanoparticles were considered for nanofluids 631 preparation, including Cu, Fe₃O₄, MWCNT, and NPG in volume fraction from 0.01 to 0.1%. 632 Different surfactants have been used to produce nanofluids. The comprehensive 633 634 characterization of the nanoparticles has been completed including XRD, SEM, BET surface area, and DLS analysis. Then, the density, thermal conductivity, and rheological properties of 635 nanofluids were experimentally evaluated. It was shown that the density of nanofluids 636 increases with nanoparticle content, this trend is correlated to nanoparticle porosity with 637 carbon-based nanoparticles. The thermal conductivity of nanofluid was reported to slightly 638 increase with nanoparticle volume fraction. The nanofluids mainly behave as Newtonian 639 fluids in the shear rate range investigated. Following the type of nanoparticles, the viscosity 640 can increase or decrease with nanoparticle concentration, showing a lubricating effect of 641 nanoparticles coupled with respective surfactants. 642

In addition, it was demonstrated that the heat transfer properties, heat transfer coefficient, and
Nusselt number, are increased with nanofluids compared to water and base-fluids, up to 20%.
The results are slightly dependent on the type of nanoparticles and concentration. The

addition of nanoparticles can significantly enhance heat transfer performance at low and high
Peclet numbers for tested water-based nanofluids. Also, it was shown that these experimental
heat transfer properties are higher than those predicted by theoretical correlations, while
experimental thermophysical were used. Finally, based on our results, copper nanofluids
appear to be the best candidate for the application and pipe flow geometry considered.

651

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