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Observation on Cetyl Trimethyl Ammonium Bromide Addition as Cationic Surfactant on Water-based Carbon Microfluid Performance for Quench Medium in Heat Treatment Process

C.A Ramadhani[1*], W.N. Putra[1], D. Rakhman[1], L. Oktavio[1], S. Harjanto[1]**
[1]Material and Metallurgy Department, Faculty of Engineering, Universitas Indonesia, Depok, Indonesia

*caesaria.ayu@ui.ac.id
**wahyuaji@metal.ui.ac.id

Abstract- Quenching takes an important part in the heat treatment process that controls the microstructure, thus enhance its mechanical properties. The heat treatment process starts with heating at an elevated temperature, holding time then rapid cooling to room temperature. It requires a medium with a good thermal conductivity that can be achieved by the addition of nanoparticles to the quench medium, referred to as nanofluids. In this research, carbon particles were prepared by the top-down method, where the reduction of carbon particle was done by planetary ball-mill for 15 hours at 500 rpm. Cetyl Trimethyl Ammonium Bromide is utilized as a cationic surfactant in order to reduce agglomeration at suspended particles thus increase quenching efficiency. Field-Emission Scanning Electron Microscope (FE-SEM), and Energy Dispersive X-Ray Spectroscopy (EDX) were used to observe the composition of material, particle size and particle morphology, and the change of the surface. Initial characterization by FE-SEM showed that the particle size after milling was averaged roughly at 15 μm, therefore, it was still not in the nanometer range. However, EDS result confirmed that the powder used in this research were 99% carbon. Carbon microparticles were added as the particle to distilled water as the microfluid base. Water-based carbon microfluid with a volume of 100 ml was produced by the two-step method, by mixing carbon microparticles at 0.1 wt%, and 0.5 wt% in various concentration of cationic surfactant of 1 wt%, 3 wt%, and 5wt % respectively. Samples of AISI 1045 or JIS S45C carbon steels were heat treated by austenizing at 1000^{o}C in a heating furnace, followed by rapid quenching in microfluid as the medium quench resulting on cooling rate diagram. Mechanical properties and microstructures of the quenched samples will be observed by conducting hardness examination and metallography observation to analyze the effect of various carbon and surfactant concentration used in the water-based carbon microfluid quench medium.

Keywords: Nanofluid, Microfluid, Surfactant, Carbon, Quenching

1. Introduction

Mechanical properties of material play an important and vital role in various kind of industry. It is often achieved by heat-treating due to its effectiveness and economic factor. The principle of heat-treatment in order to enhance properties is to trap martensitic structure in the steel matrix by rapid cooling from heating at above austenitic temperature for it has a needle-like structure to hinder dislocation movements thus higher hardness value can be achieved [1]. Quench

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medium determine the cooling rate of heat treatment and becomes a critical factor for the material's properties and failure [2].

With around 100nm size, nanoparticles can be dispersed to quench medium to increase heat transfer efficiency and capability called nanofluid. In previous researches, nanofluid has been proved to increase the thermal conductivity of the quench medium [3-4]. Titanium dioxide nanoparticles have put an impact when added to water-based nanofluid for enhancing thermal conductivity by the addition of particle concentration [5]. Carbon as nanoparticle has hydrophobic properties and affect directly when dispersed to liquid such as water or oil, to cause agglomeration thus the performance regressed [6]. Stability and good dispersion are desired and it can be acquired by the use of surfactant. Recent studies have proved the use of surfactant to graphene nanofluid have greater stability to graphene nanofluid without surfactant due to the reduction of the contact angle between the nanoparticle and the base fluid, surface tension also increasing the wettability of nanoparticles thus interfacial tension is reduced and heat transfer rate and cooling rate can be improved[7-9]. Recent studies have proved that the greater amount of surfactant concentration compared to nanoparticle concentration used in nanofluid will improve dispersion of nanoparticle in the fluid base [10]. The use of different type of surfactant can affect different leverage to nanofluid, it is classified by the composition of the polarity of the head group which is nonionic, anionic, cationic and amphoteric [11-12]. The adsorption of amphoteric surfactant is better than anionic surfactant due to electrostatic association affecting on the better controlling ability of lubrication and wetting of nanofluid, non-ionic surfactant play a positive effect on carbon nanotubes nanofluid and cationic surfactant has the ability to reduce the viscosity of crude oil improving the clean flow of the oil [13-18].

In this study, we investigate the influence of a variety of cationic surfactant composition, Cetyl Trimethyl Ammonium Bromide (CTAB) on carbon microfluid by observation of the microstructures of S45C steel and a hardness value of the material before and after heat-treated by quenching.

2. Materials and Methods

2.1. Material and Heat Treatment Experiments

The material used for this study was S45C steel rod with carbon composition ranging from 0.42 - 0.48 wt.% in 15mm x 10mm x 10mm dimension cut by hand saw. Heat treatment was conducted in a furnace from room temperature around 25°C, then pre-heated to 600°C with 10°C/minute rate, followed by holding for 15 minutes. Pre-heat was done to avoid cracking of the material [19]. S45C steel then carried out to heat at 1000°C for one hour at an austenizing temperature to acquire austenite structure. Samples of steel then quench and immersed in carbon water-based microfluid in pure distilled water to achieve martensite structure. Quench medium used were pure distilled water to observe microstructures and hardness without microparticles, 0.1 wt% and 0.5 wt% carbon micro-sized particles composition with the addition of CTAB in 1 wt%, 3 wt%, and 5 wt% composition.

2.2.*Preparation of Carbon Water-based Microfluid*

Carbon microparticles were done by the two-step method in laboratory grade obtained from Sigma-Aldrich. The preparation of carbon microparticles was carried out by milling of carbon powders for 15 hours at 500 rpm using planetary ball mill in Department of Metallurgy and Material Faculty of Engineering Universitas Indonesia, Depok. This process was originally aimed to decrease carbon powder to nano-sized. The next method was the dispersion of microparticles to distilled water with the addition of CTAB surfactant in 1 wt%, 3 wt%, and 5 wt% weight composition. Sonification was done using an ultrasonic cleaner for 280 seconds in room temperature to achieve greater dispersion and stability of carbon microparticles and surfactant in the fluid before followed by quenching [20].

2.3.Experimental Methods

After immersed in quench medium, samples of steel were washed by water then prepared by metallographic procedure then followed by etching in Nital containing 3ml nitric acid and 97 ml ethyl alcohol for five seconds after polishing. Observation of microstructures was carried out using an optical microscope at 100x magnification and hardness was obtained using a Vickers hardness test using 300gf diamond indenter load for 10 seconds. The result of the indentor size in samples was observed with a microscope then the calculation is done to the obtained final hardness value. Carbon powder characterization was done at Centre for Material Processing and Failure Analysis (CMPFA) Universitas Indonesia using Field Emission Scanning Electron Microscope (FE-SEM) and Energy Dispersive X-ray (EDX).

3. Results and Discussion

3.1. Carbon Microparticles Characterization

Carbon microparticles characterization is done using FE-SEM and EDX Test. Figure 1 shows the imaging result of FE-SEM examination of carbon microparticles in (A) 500x magnification and (B) 1000x magnification. The objective of this examination is to observe the particle size of the laboratory-grade carbon powder used in this study. The measurement of average size of carbon microparticles resulting in 15.08 µm.

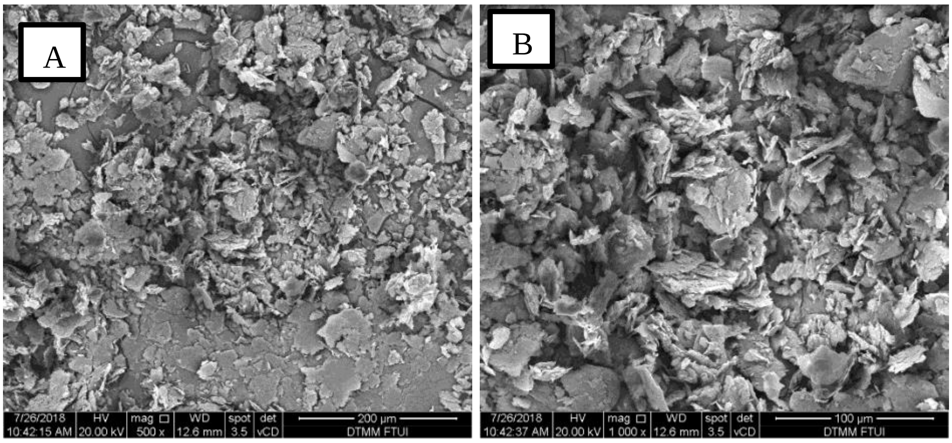

Figure 1. The result of FE-SEM for laboratory-grade carbon microparticles at (A) 500x and (B) 1000x magnification.

EDS test result is shown in Table 1. The objective of EDS is to obtain the composition of carbon microparticles and the purity of the powders. It is shown that the carbon composition is 94.16 wt.% and the rest of the powder is oxygen in 5.84 wt%. No impurities are detected from the result of EDS examination.

Table 1. The composition of laboratory-grade carbon microparticles from EDS. (%wt.)

Laboratory Grade Carbon Powder	C	O
	94.16	5.84

3.2. Microstructures Observation

Microstructures of S45C steel is shown in Figure 2. Figure 2A shows the microstructures of steel immersed in the pure distilled water. Figure 2B shows the microstructures in 0.1 wt.% carbon, then followed by the addition of CTAB 1% in 2C, CTAB 3% in 2D and CTAB 5% in

2E. Figure 2F shows the microstructure in 0.5 wt% carbon, with the addition of 1% CTAB in 2G, 3% CTAB in 2H and 5% CTAB in 2I.

Martensitic structures can be observed explicitly 2C-E and 2G-I marked by the darker area in brown color [21]. The greatest area of martensitic structure can be observed at 2C in 0.1wt% carbon and 1% CTAB. The comparison can be observed clearly to 2A and 2B which fewer brownish/darker area appear. The martensitic structure also has a fewer amount at 2B with 0.1 wt,% carbon than in 2F in 0.5wt% composition. It can be 0concluded that the addition of carbon can affect the greater amount of martensite achieved. Black spot captured in microstructure images can be explained with carbon sputtering out of the steel containing medium carbon composition by decarburization in high temperature due to the reaction of carbon reacts with oxygen or hydrogen in the furnace when heat treated thus the loss of carbon atoms in the surface happened to result on a black spot in microstructure images [22-23].

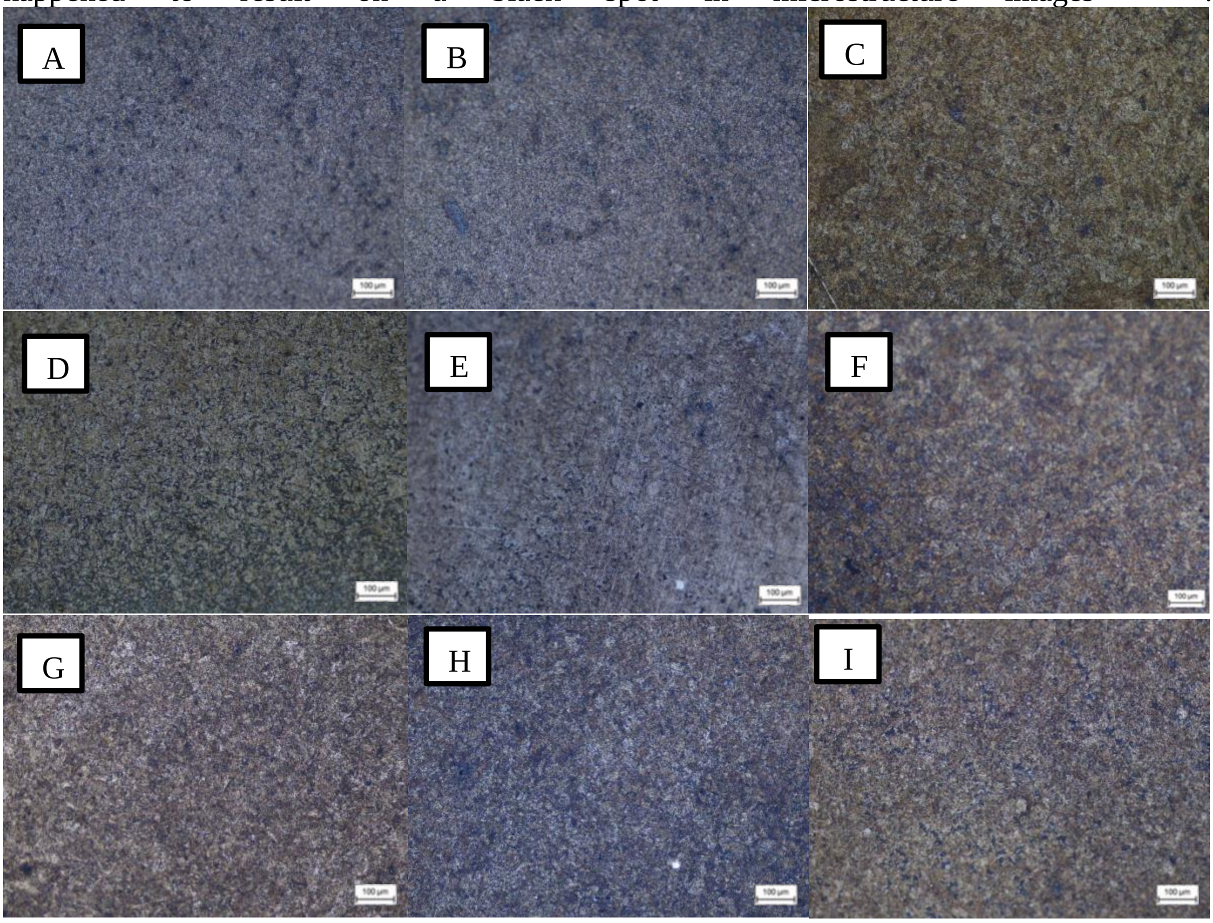

Figure 2. Microstructures observation of S45C steel in (A) pure distilled water, (B) 0.1wt% carbon, 0.1 wt% carbon with CTAB addition of (C) 1%, (D) 3% (E) 5%, (F) 0.5 wt% carbon 0.5 wt% carbon with CTAB addition of (G) 1%, (H) 3% (I) 5% in 100x magnification.

3.3.Hardness Values

The result of the hardness value is shown in Table 2. The hardness of the material in carbon 0.1wt% and 0.5wt% unexpectedly to be the same value which is 457.4 HV. The highest hardness value is 665.3 HV in 0.1wt% carbon composition with 1% CTAB addition, followed by 614.7 HV in carbon 0.5wt% with 3% addition. It can be stated that the optimum addition of the surfactant is 1% in 0.1wt% carbon microparticles and 3% in 0.5wt% carbon microparticles. The addition of surfactant in higher concentration seem to improve hardness value. This might due to the ability to reduce agglomeration. While the addition of surfactant more than optimum concentration can reduce hardness value.

Table 2. The hardness value of S45C steel in various components of microfluid.

CTAB Composition		Hardness (HV)
Carbon 0.1%	0%	457.4
	1%	665.3
	3%	569.7
	5%	578.9
Carbon 0.5%	0%	457.4
	1%	508.3
	3%	614.7
	5%	554.8

Figure 3 shows the graph of hardness value in various concentration of carbon and the addition of CTAB surfactant. The optimum CTAB concentration in 0.1wt% carbon is 1 wt% and 3 wt% in 0.5 wt % carbon microparticles. There is no close correlation between the carbon 0.1wt% and carbon 0.5 wt% that can be concluded. Both of the composition of microparticles yield a different result of hardness value depending on the addition of CTAB. The optimum concentration of CTAB and the highest hardness value obtained differ from one another. Yet it can be concluded that the addition of surfactant improved the hardness value compared to 0% use of surfactant.

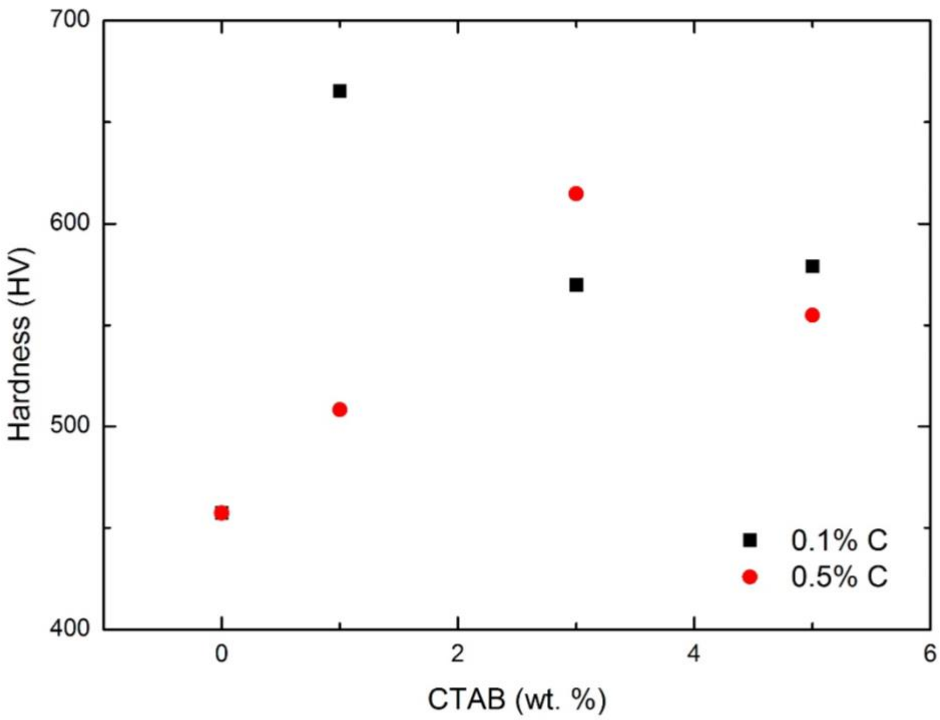

Figure 3. The visual graph of hardness value S45C steel immersed in various components of microfluid.

4. Conclusions

Cetyl trimethyl ammonium bromide (CTAB) was used as a cationic surfactant along with carbon microparticles in a water-based fluid as microfluid quenchants in order to investigate the effect of the surfactant on S45C microstructures and hardness. Carbon microparticles were used in 0.1wt% and 0.5wt% composition along with the addition of 1 wt%, 3 wt% and 5 wt% CTAB. Heat treatment by quenching produced martensitic structure marked by brown-colored area. Steel immersed in 0.1wt% carbon microparticles and 1% CTAB surfactant showed the greatest area of martensite. It also proved by the highest hardness value obtained which is 665.3 HV, followed by immersion in 0.5wt% carbon microparticles composition and 3% CTAB resulting in 614.7 HV. The addition of CTAB surfactant seem to improve the hardness of the material respectively. This improvment was probably due to the CTAB surfactant effect on the agglomeration reduction of carbon microparticles in a water-based microfluid.

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6. References

[1] Babu, K., Arularasan, R., & Srinath Ramkumar, S. (2017). Quenching performance of AISI 1010 in CNT nanofluids. *Materials Today: Proceedings*, *4*(10), 11044–11049. https://doi.org/10.1016/j.matpr.2017.08.065

[2] Gu, X., Zhang, F., Li, Y., Zhang, J., Chen, S., Qu, C., & Chen, G. (2018). Investigation of cationic surfactants as clean flow improvers for crude oil and a mechanism study. *Journal of Petroleum Science and Engineering*, *164*(September 2017), 87–90. https://doi.org/10.1016/j.petrol.2018.01.045

[3] Salamah, A., Phan, C. M., & Pham, H. G. (2015). Dynamic adsorption of cetyl trimethyl ammonium bromide at decane/water interface. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, *484*, 313–317. https://doi.org/10.1016/j.colsurfa.2015.08.010

[4] Das, P. K., Mallik, A. K., Ganguly, R., & Santra, A. K. (2018). Stability and thermophysical measurements of TiO2(anatase) nanofluids with different surfactants. *Journal of Molecular Liquids*, *254*, 98–107. https://doi.org/10.1016/j.molliq.2018.01.075

[5] Bílková, K., & Gulbrandsen, E. (2008). Kinetic and mechanistic study of CO2corrosion inhibition by cetyl trimethyl ammonium bromide. *Electrochimica Acta*, *53*(16), 5423–5433. https://doi.org/10.1016/j.electacta.2008.02.096

[6] Chen, L., & Xie, H. (2010). Properties of carbon nanotube nanofluids stabilized by cationic gemini surfactant. *Thermochimica Acta*, *506*(1–2), 62–66. https://doi.org/10.1016/j.tca.2010.04.016

[7] Soror, T. Y., & El-Ziady, M. A. (2003). Effect of cetyl trimethyl ammonium bromide on the corrosion of carbon steel in acids. *Materials Chemistry and Physics*, *77*(3), 697–703. https://doi.org/10.1016/S0254-0584(02)00129-3

[8] Kim, S., Tserengombo, B., Choi, S. H., Noh, J., Huh, S., Choi, B., … Jeong, H. (2018). Experimental investigation of dispersion characteristics and thermal conductivity of various surfactants on carbon based nanomaterial. *International Communications in Heat*

and Mass Transfer, *91*(December 2017), 95 102. https://doi.org/10.1016/j.icheatmasstransfer.2017.12.011

[9] Ueki, Y., Aoki, T., Ueda, K., & Shibahara, M. (2017). Thermophysical properties of carbon-based material nanofluid. *International Journal of Heat and Mass Transfer*, *113*, 1130–1134. https://doi.org/10.1016/j.ijheatmasstransfer.2017.06.008

[10] Choi, T. J., Jang, S. P., & Kedzierski, M. A. (2018). Effect of surfactants on the stability and solar thermal absorption characteristics of water-based nanofluids with multi-walled carbon nanotubes. *International Journal of Heat and Mass Transfer*, *122*, 483–490. https://doi.org/10.1016/j.ijheatmasstransfer.2018.01.141

[11] Xing, M., Yu, J., & Wang, R. (2016). Experimental investigation and modeling on the thermal conductivity of CNTs based nanofluids. *International Journal of Thermal Sciences*, *104*, 404–411. https://doi.org/10.1016/j.ijthermalsci.2016.01.024

[12] Das, P. K., Islam, N., Santra, A. K., & Ganguly, R. (2017). Experimental investigation of thermophysical properties of Al2O3–water nanofluid: Role of surfactants. *Journal of Molecular Liquids*, *237*, 304–312. https://doi.org/10.1016/j.molliq.2017.04.099

[13] Sánchez-Coronilla, A., Martín, E. I., Navas, J., Aguilar, T., Gómez-Villarejo, R., Alcántara, R., … Fernández-Lorenzo, C. (2018). Experimental and theoretical analysis of NiO nanofluids in presence of surfactants. *Journal of Molecular Liquids*, *252*, 211–217. https://doi.org/10.1016/j.molliq.2017.12.140

[14] Khaleduzzaman, S. S., Mahbubul, I. M., Shahrul, I. M., & Saidur, R. (2013). Effect of particle concentration, temperature, and surfactant on surface tension of nanofluids. *International Communications in Heat and Mass Transfer*, *49*, 110–114. https://doi.org/10.1016/j.icheatmasstransfer.2013.10.010

[15] Xuan, Y., Li, Q., & Tie, P. (2013). The effect of surfactants on heat transfer feature of nanofluids. *Experimental Thermal and Fluid Science*, *46*, 259–262. https://doi.org/10.1016/j.expthermflusci.2012.12.004

[16] Tiara, A. M., Chakraborty, S., Sarkar, I., Ashok, A., Pal, S. K., & Chakraborty, S. (2017). Heat transfer enhancement using surfactant based alumina nanofluid jet from a hot steel plate. *Experimental Thermal and Fluid Science*, *89*(March), 295–303. https://doi.org/10.1016/j.expthermflusci.2017.08.023

[17] Almahfood, M., & Bai, B. (2018). The synergistic effects of nanoparticle-surfactant nanofluids in EOR applications. *Journal of Petroleum Science and Engineering*, *171*(May), 196–210. https://doi.org/10.1016/j.petrol.2018.07.030

[18] Gnyusov, S. F., Rotshtein, V. P., Mayer, A. E., Astafurova, E. G., Rostov, V. V., Gunin, A. V., & Maier, G. G. (2017). Comparative study of shock-wave hardening and substructure evolution of 304L and Hadfield steels irradiated with a nanosecond relativistic high-current electron beam. *Journal of Alloys and Compounds*, *714*, 232–244. https://doi.org/10.1016/j.jallcom.2017.04.219

[19] Ulset, E. T., Kosinski, P., & Balakin, B. V. (2018). Solar steam in an aqueous carbon black nanofluid. *Applied Thermal Engineering*, *137*(December 2017), 62–65. https://doi.org/10.1016/j.applthermaleng.2018.03.038

[20] Estellé, P., Cabaleiro, D., Żyła, G., Lugo, L., & Murshed, S. M. S. (2018). Current trends in surface tension and wetting behavior of nanofluids. *Renewable and Sustainable Energy Reviews*, *94*(November 2017), 931–944. https://doi.org/10.1016/j.rser.2018.07.006

[21] Esmaeilzadeh, P., Bahramian, A., & Fakhroueian, Z. (2011). Adsorption of anionic, cationic and nonionic surfactants on carbonate rock in presence of ZrO2nanoparticles. *Physics Procedia*, *22*, 63–67. https://doi.org/10.1016/j.phpro.2011.11.009

[22] Soleimani, H., Baig, M. K., Yahya, N., Khodapanah, L., Sabet, M., Demiral, B. M. R., & Burda, M. (2018). Impact of carbon nanotubes based nanofluid on oil recovery efficiency using core flooding. *Results in Physics*, *9*, 39–48. https://doi.org/10.1016/j.rinp.2018.01.072

[23] Gangadevi, R., Vinayagam, B. K., & Senthilraja, S. (2018). Effects of sonication time and temperature on thermal conductivity of CuO/water and Al2O3/water nanofluids with and without surfactant. *Materials Today: Proceedings*, *5*(2), 9004–9011. https://doi.org/10.1016/j.matpr.2017.12.347