IOP Conf. Series: Journal of Physics: Conf. Series **799** (2017) 012014 doi:10.1088/1742-6596/799/1/012014

Synthesis of Al_2O_3 Nanoparticles from Local Bauxite for Water- Al_2O_3 Nanofluids egy

Dani Gustaman Syarif*, Djoko Hadi Prajitno, Efrizon Umar

Center for Applied Nuclear Science and Technology (PSTNT)
National Nuclear Energy Agency (BATAN)
Jl. Tamansari 71, Bandung 40132, Indonesia

*Email: danigus@batan.go.id

Abstract. A study on synthesis and characterization of Al_2O_3 nanoparticles from bauxite for water-Al_2O_3 nanofluids as an alternative coolant for nuclear and non-nuclear applications has been done. The Al_2O_3 nanoparticles were synthesized by heating AlOOH as a precursor derived from local bauxite. The Al_2O_3 nanoparticles were dispersed in water and ultrasonicated to produce nanofluids. XRD data showed that the Al_2O_3 nanoparticles crystallized in gamma alumina with crystallite size of 4.5 nm (Debye Scherrer method). Surface area of the Al_2O_3 nanoparticles was 195 m^2/gram. Data of TEM showed that the particle size was smaller than 10 nm. According to zeta potential data, the nanofluids were stable at neutral pH of 7.3 with zeta potential of 28-51 mV. The height of the nanofluid surface decreased about 13 % after 6 days. In addition, the CHF of the water-Al_2O_3 nanofluids produced in this study increased about 55-161 % compared to that of water.

1. Introduction

There is a need to improve heat transfer capability of conventional fluid such as water, ethylene glycol, and oil as reported by some researchers [1-6]. The improvement of the heat transfer capability of the conventional fluids is to increase the efficiency of some systems based on heat transfer such as automotive and nuclear reactor, and of some equipments and processes such as electronics, and metal forming. Introducing nanofluids, as a product of nanotechnology, to heat transfer system to replace the conventional fluids is intensively studied over decades [1-5]. The nanofluids are stable suspensions consisting dispersed nanoparticles with particle size of 1-100 nm and base fluids such as water, ethylene glycol, and oil [2,6].

Some characteristics are required by the nanofluids before application and one important characteristic of the nanofluids is critical heat flux (CHF) i.e. the maximum heat flux attainable from the nucleate boiling. An enhanced CHF is advantageous for increasing the safety margin of the thermal system, and to design compact and efficient cooling systems. The nanofluid with good CHF is required for some applications such as ECCS (Emergency Core Cooling System) and RVCS

(Reactor Vessel Cooling System) mainly required in nuclear reactor system, quenching in metal heat treatment, metal cutting and machining.

Some works dealing with CHF of the nanofluids have been reported [1,3, 7-11]. It was reported that some nanofluids had good potential as fluid having large CHF namely water-CuO [3], water-ZrO_2 [3], water-Fe_3O_4 [11,12], water-ZnO [13], and water-Al_2O_3 [1, 3, 10,11]. Nanofluid of water-Al_2O_3 is interesting in heat transfer application due to special characteristic of Al_2O_3 namely low density, low neutron absorption coefficient, and chemical inertness that is good to avoid corrosion problem. Although the nanofluid of water-Al_2O_3 has been studied in relation with the CHF, however, the CHF of the nanofluids with neutral pH in the previous studies was not reported and discussed. It was known that some nanofluids had acidic and basic pH. The neutral pH is important to avoid corrosion problem.

In order to increase the added value of mineral abundant in Indonesia especially bauxite, in this work, the water-Al_2O_3 nanofluid was prepared by utilizing Al_2O_3 nanoparticles derived from local bauxite. The nanofluids having neutral pH were prepared and characterized. The characteristics of the nanofluids were discussed in relation with the CHF and heat transfer.

2. Experimental Method

Synthesis and Characterization of Al_2O_3 Nanoparticle

An amount of bauxite and NaOH was mixed and ground. The mixture was suspended in water, and then the suspension was heated in an autoclave at about 175°C for 3 hours. After heating, the suspension was cooled down. Precipitate of red mud was separated from filtrate by filtration. The filtrate was precipitated by adding HCl. The precipitate was separated by filtration, and washed using water. The precipitate was heated at 200°C to form AlOOH.

An amount of AlOOH was heated at 900°C for 3 hours to get Al_2O_3 nanoparticles. The Al_2O_3 nanoparticles were analyzed using XRF to know the chemical composition, and then they were subjected to XRD analysis to know their crystal structure and crystallite size (Debye Scherrer method). Nanoparticle size was measured using a TEM belongs to TEM laboratory of Chemistry Department of UGM, and surface area of the nanoparticles was measured using a surface area meter of quantachrome NOVA 2200.

Synthesis and Characterization of Water-Al_2O_3 Nanofluids

The Al_2O_3 nanoparticles of 0.5, 1, 1.5, and 2 grams were suspended in 100 ml water, and ultrasonicated using an ultrasonic bath for 2 hours to produce nanofluids. After calculation, the concentrations of nanofluids are 0.125, 0.250, 0.375, and 0.5 vol %. Visually, a typical nanofluid was observed time to time. The height of the nanofluid surface was also measured time to time. Zeta potential of the nanofluids was measured using Zetasizer from Malvren. Meanwhile, viscosity of the nanofluids was measured using a vibro viscometer SV-10. CHF of the nanofluids was measured using a method found in works of Lee, et al.[11] and Hiswankar and Kshirsagar [13] by utilizing Cu wires with diameter of 0.2 mm.

3. Results and Discussion

Visual appearance of the Al_2O_3 nanoparticles is depicted in Fig. 1 and its XRD pattern can be seen in Fig. 2. Chemical composition of Al_2O_3 nanoparticles analyzed using XRF is depicted in Table 1. From Fig. 2, it can be seen that the Al_2O_3 nanoparticle crystallizes in gamma alumina. Peaks belong to the pattern of the Fig. 2 are broadening indicating very small crystallite of the powder. Based on a calculation using Debye Scherrer method [14], the average crystallite size of the nanoparticles was 4.5 nm. Specific surface area associated with the nanoparticles is 195 m^2/gram. This surface area is large enough. When particle size of the nanoparticles is estimated from the specific surface area by using equation (1) [15], it is known that particle size is 7.75 nm. This value is in the same range with that calculated from the XRD pattern using Debye Scherrer method. The little difference is caused by agglomeration of the nanoparticles.

$$d = \frac{6000}{\rho.As} \quad \text{.............................(1)}$$

Where, d = particle size (nm), ρ = theoretical density of nanoparticles (g/cm^3), and As = specific surface area (m^2/g).

Figure 1. Appearance of the Al_2O_3 nanoparticles.

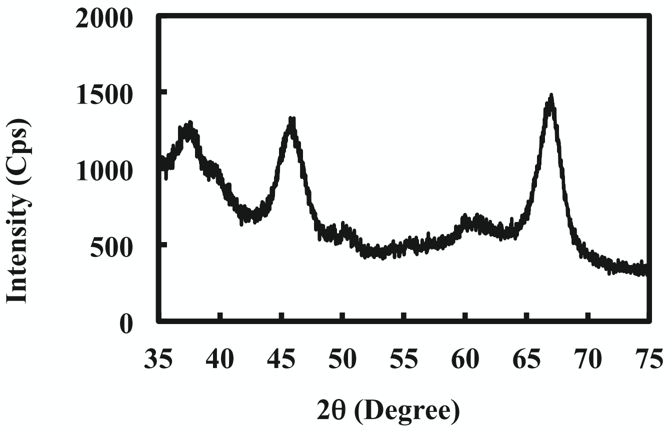

Figure 2. XRD pattern of the Al_2O_3 nanoparticles.

Table 1. Chemical composition of the Al_2O_3 nanoparticles.

No.	Component	Concentration (Weight %)
1	Al_2O_3	99.443
2	SiO_2	0.230
3	TiO_2	0.002
4	Fe_2O_3	0.035
5	CaO	0.061
6	MgO	0.134
7	Na_2O	0.054
8	K_2O	0.026
9	SO_3	0.016

Fig. 3 is a TEM image of the Al_2O_3 nanoparticles. According to Fig. 3, the Al_2O_3 nanoparticles are small with size about 7 nm and tend to form agglomerates. Al_2O_3 powder forming agglomerate with size about 80 nm contains individual small particle was also synthesized by Karim et al. [16] using sol gel utilizing urea as the chelating agent. Surface area of the powder of Karim et al. [16] is only 4.4 m^2/g. This value is much smaller than that of the powder in this work of 195 m^2/g. The difference may be due to different treatment.

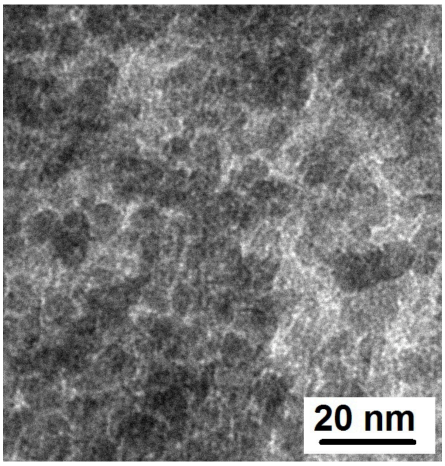

Fig. 3. TEM image of the Al_2O_3 nanoparticles.

A nanofluid with Al_2O_3 concentration of 0.25 vol % that was prepared using the Al_2O_3 nanoparticle synthesized in this study is shown in Fig. 4. The observation was done only to the nanofluid with pH 7.3 because the most applicable nanofluid is that having neutral pH (with no corrosion problem). The height of the surface of the nanofluid decreases time to time. Quantitatively the decrease of the height of the nanofluid surface is shown in Fig. 5. The height of the nanofluid surface decreases about 13 % of the initial height of the nanofluid surface after 6 days observation. Compared to the water-Al_2O_3 nanofluid in the previous study [17], the stability of this nanofluid is better. In the previous study [17], the height of the nanofluid decreased 20% after 6 days observation. The stability of the water-Al_2O_3 nanofluid in this study is also better than that of the same nanofluid found in a literature [18]. The good stability may be due to small particle size and large surface area.

a b c

Fig.4. Water-Al_2O_3 nanofluids at different observation times (a. Initial, b. 2 days, c. 8 days).

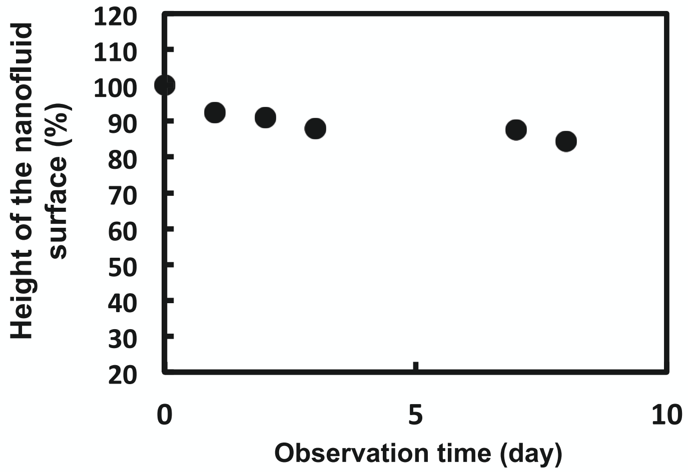

Fig. 5. The relation between the height of the nanofluid surface and observation time. Concentration of Al_2O_3 is 0.25 vol% and pH 7.3.

Fig. 6 shows zeta potential of the water-Al_2O_3 nanofluids. It can be seen that the zeta potential changes is relatively independent on the change of concentration of the nanoparticles. The nanofluid is relatively stable when zeta potential is larger than 25 mV or smaller than -25 mV. According to this ctriterium, from Fig. 6, it can be seen that the nanofluids with different concentrations prepared in this study are stable at pH 7.3.

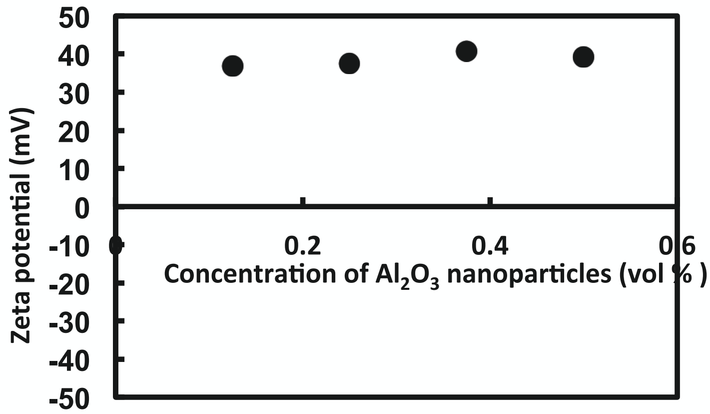

Fig.6. Zeta potential of the water-Al_2O_3 nanofluids.

Viscosity data for the nanofluid with pH 7.3 depicted in Fig. 7. The viscosity of the water-Al_2O_3 nanofluid with pH of 7.3 increases linearly with concentration of Al_2O_3 nanoparticles within the range of concentration. Compared to Einstein prediction, the gradient of increase is larger. It means that the viscosity increase of the nanofluids as the increase of concentration does not obey the Einstein prediction. This difference happens because the nanofluids are more complicated than the suspension used for the Einstein prediction. The nanofluid does not simply refer to a liquid-solid mixture. The characteristics such as pH, zeta potential, and Brownian motion effect that were not considered during formulation of the Einstein prediction are very important for the nanofluids.

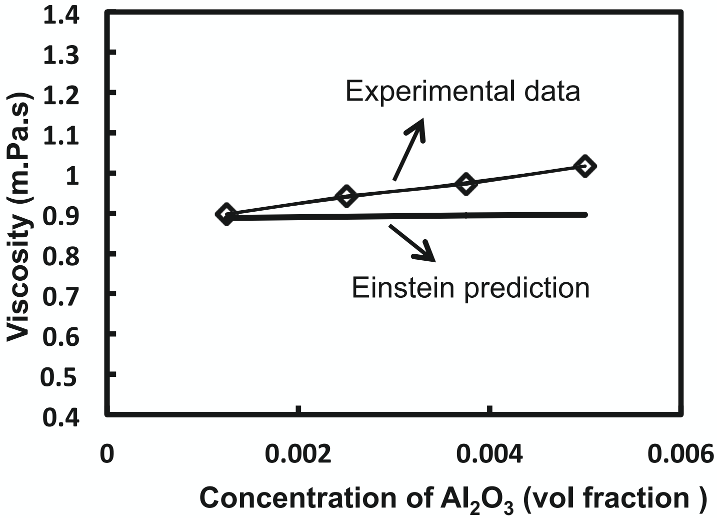

Fig.7. Viscosity of the water-Al_2O_3 nanofluid with pH 7.3.

Fig. 8 shows the increase of CHF as function of nanoparticles concentration. The increase of CHF increases almost linearly from 55 to 161 % with nanoparticles concentration. This means that heat transfer from Cu wire to the nanofluids goes well when the nanoparticles concentration increases. The mechanism of the increase of CHF in the nanofluid is due to nanoparticles coating on Cu wire surface [8]. The heated surfaces are changed by the deposition of nanoparticles. The deposited nanoparticles generated a porous layer on the Cu wire, which then improve the wettability of the wire. The heat transfer becomes better when the nanoparticles concentration increases. The degree of CHF enhancement increases as the amount of deposited nanoparticles increases. So why the CHF is more enhanced at higher concentrations of the nanoparticles. By considering the characteristics of the nanofluids prepared in this study, the nanofluids have possibility to be applied in nuclear reactor as fluid or coolant for ECCS (Emergency Core Cooling System) and RVCS (Reactor Vessel Cooling System), and in automotive, electronics, and machining and cutting process as also the coolant.

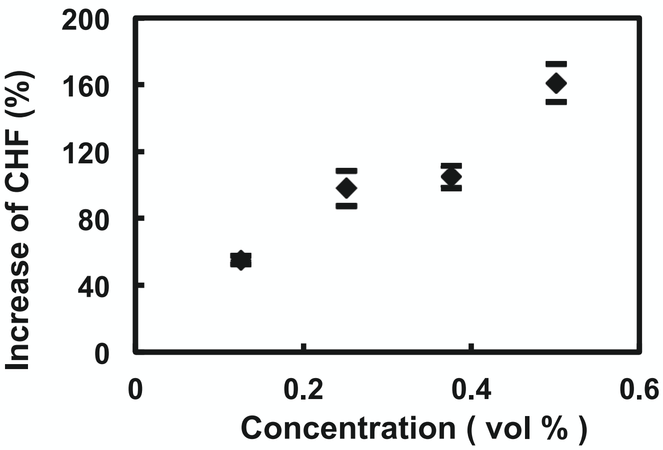

Fig. 8. Increase of CHF of the water-Al_2O_3 nanofluids as function of Al_2O_3 concentration.

4. Conclusion

Al_2O_3 nanoparticle with crystallite size of 4.5 nm has been well synthesized from AlOOH as precursor derived from local bauxite by calcination. Although the nanoparticle forms agglomerate, the nanofluid made of this nanoparticle has relatively good stability at neutral pH (about 7), the pH that is required to avoid corrosion problem. The CHF of the nanofluids is larger than that of water, and increases from 55 to 161 % with Al_2O_3 concentration from 0.125 to 0.5 vol %. The nanofluids have possibility to be applied in nuclear application as fluid or coolant for ECCS (Emergency Core Cooling System) and RVCS (Reactor Vessel Cooling System), and in non-nuclear applications as coolant for automotive, electronics, metal heat treatment (for quenching), and machining and cutting process.

Acknowledgments

Authors would like to acknowledge the financial support from BATAN through DIPA program year 2014 and 2015. Special thanks to Mr. M. Yamin for his help during synthesis of Al_2O_3.

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