Green Synthesis of Al₂O₃ Nanoparticles for Heat Transfer Nanofluid Utilizing Lemon Extract

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Abstract. To develop new cooling fluids, Al₂O₃ nanoparticles have been synthesized by environmentally friendly methods (green synthesis). The precursor used was Al(OH)₃ extracted from local bauxite. The lemon extract was used as a chelating agent. Al(OH)₃ powder was dissolved in water, and then lemon extract was added to it. The mixture solution was heated at 100°C until the solution forms a gel. The gel was calcined at 1100°C for 1 hour. The calcined product was then crushed to obtain Al₂O₃ nanoparticles. The powder of Al₂O₃ nanoparticles was analyzed using X-ray diffraction (XRD) and surface area meter. Nanofluids were prepared from these Al₂O₃ nanoparticles with a concentration of 1-5g/L. The nanofluid heat transfer coefficient was measured using a heat transfer circuit utilizing a radiator as its main component. The results of XRD analysis showed that the resulting Al₂O₃ nanoparticles consisted of two phases namely alpha and gamma. Crystallite size of alpha phase was 37 nm and that of gamma phase was 8 nm. The surface area of Al₂O₃ nanoparticles was 94.4 m²/g. As calculated from the surface area data, particle size of the nanoparticles was 16 nm. Zeta potential of the nanofluids was -26 to -33 mV. From the results of heat transfer coefficients measurements, it was known that the heat transfer coefficients of the nanofluids were greater than the water one. The increase of the heat transfer coefficient were 17%, 23%, and 27 % for concentrations of 1g / L, 3g / L, and 5g / L, respectively.

Keywords: Heat Transfer, Nanoparticles, Nanofluids, Al₂O₃, Lemon Extract, Green Synthesis

1 Introduction

Currently, nanotechnology is developing so fast. Nano sized particles are very promising because they have different properties with larger particles even though they come from the same base material [1]. The effort to increase heat transfer is by adding nanometer sized particles to base fluids such as water, oil, and ethylene glycol [2]. This product is called nanofluid. Nanofluid is a stable suspension made by dispersing 1-100 nm sized nanoparticles into basic fluids such as water, ethylene glycol, and oil [3]. Nanofluids are expected to have higher thermal

performance compared with conventional heat transfer fluid [4]. Nuclear reactors require a cooling system in operation. The cooling system is very important in safety during reactor operation. The cooling system is operated using coolant. Some reactors especially LWR (Light Water Reactor) use water (H₂O) as the coolant [5]. Parts of nuclear reactors that require coolant are primary cooling systems that maintain reactor core temperature, secondary cooling systems, emergency core cooling systems (ECCS), and Reactor Vessel Cooling Systems (RVCS) [6], [7]. Nanoparticles can be made using various methods such as precipitation, coprecipitation, sol-gel, pyrolysis spray, and hydrothermal [8]. Research on nanofluid Al₂O₃ water was carried out by several researchers [9], [10], [11] but none used Al₂O₃ nanoparticles synthesized using lemon extract (green synthesis) and Al(OH)₃ precursors extracted from local bauxite. Utilization of bauxite sand in this study is intended as an attempt to increase the added value of local materials, and the use of lemon is intended to obtain an environmentally friendly method of synthesis. Nanofluid was prepared by a two step method, using water as a base fluid. Characteristics of nanoparticles and nanofluids was discussed and compared to the previous studies.

2 Experimental Methods

1.1. Synthesis of Al₂O₃ Nanoparticles and Characterization

Bauxite sand was mixed with NaOH. The ratio of bauxite and NaOH sand was 1: 1.8. The mixture was then stirred until homogeneous. The mixture was put into 1000 ml of distilled water and heated at 275°C for 90 minutes in a pressurezed pan. The solution resulting from the digestion process was filtered so that the sodium aluminate filtrate was obtained. The filtrate was precipitated by adding 5 M hydrochloric acid to achieve a pH neutral and formed Al(OH)₃. The Al(OH)₃ precipitate was washed with distilled water to remove salt. After the precipitate was free of salt then it was dried at 100°C to obtain Al(OH). Lemon fruit was then cut and squeezed to get the extract, lemon extract is then acidimetric titration to get a total acid concentration contained in a lemon. AlO(OH) powder was then mixed with lemon extract, The mole ratio of AlO(OH) and citric acid was one. Sol is heated at 120°C to produce the gel and then calcined at 1100°C for one hour to produce Al₂O₃ nanoparticle powder. The Al₂O₃ nanoparticle powder was then analyzed by X-Ray Diffraction (XRD), The crystal size was measured using a Debye Scherrer equation [8] and then the surface area measurements were carried out with the Surface Area Analyzer (SAA).

2.2 Water-Al₂O₃ nanofluid preparation and characterization

Nanofluid was made by mixing Al₂O₃ nanoparticles with distilled water and stirring it with ultrasonic waves (sonication). As much as 1 g, 3 g, and 5 g of Al₂O₃ nanoparticles are mixed with 1 L of distilled water and the pH of each nanofluid was adjusted to pH 10 with the addition of NH₄OH then stirred and sonicated for 1 hour and zeta potential of nanofluids was measured using Malvern Zetasizer. Nanofluid then visually observed every day to take pictures. Characterization of heat transfer nanofluid is carried out by flowing heated nanofluid with an inlet temperature of 50°C, then the outlet temperature of the nanofluid from the radiator, air temperature released by the radiator fan, and radiator wall temperature measured as shown in figure 1. The convective heat transfer coefficient is calculated using the heat transfer equation of Equation 1 [13].



Fig.1. Set of heat transfer experiments

3 Result and Discussion

3.1 X-Ray Difraction (XRD) of Al₂O₃ Nanoparticles

 Al_2O_3 nanoparticles produced from the solgel synthesis with the addition of citric acid contained in the lemon extract as a chelating agent can be seen in figure 2. The diffraction pattern of Al_2O_3 nanoparticles calcined at 1100°C for 1 hour is shown in Figure 3. Al_2O_3 powder consists of two phases namely alpha (α) and Gamma (γ). The crystallite size of the alpha phase nanoparticles is 37 nm and gamma phase nanoparticles is 8 nm which was calculated using the Debye-Scherrer equation [8].



Fig.2. Al₂O₃ nanoparticles



Figure 3. X-ray diffraction pattern of Al₂O₃ nanoparticles synthesized with sol gel method using lemon extract as chelating agent

3.2 Surface Area Meter Al₂O₃ nanoparticles

The surface area of the Al_2O_3 nanoparticles calcined at $1100^{\circ}C$ is $94.44 \text{ m}^2/\text{gram}$. The density of Al_2O_3 is known to be 3.97 g/cm^3 , so the particle size of Al_2O_3 can be calculated using the equation (3.2.1) from the measured data of specific surface area of the sample[8]:

$$D = \frac{6}{\rho S} \tag{3.2.1}$$

Where;

D = particle size

 $\rho = density Al_2O_3 g/cm^3 (3,97 g/cm^3)$

 $S \ = the \ surface \ area \ of \ the \ Al_2O_3 \ (m^2/g)$

From equation 3.2.1, the Al₂O₃ particles diameter obtained is 16.95 nm. The large surface area will produce small particle sizes. From the data of the particle size, the Al₂O₃ obtained in this work can be classified as nanoparticles because it has a particle size in the range of 1-100 nm. Al₂O₃ nanoparticles made can already be used for making water - Al₂O₃ nanofluid. Water - Al₂O₃ nanofluid performance test was carried out using a heat transfer system completed with a radiator.

3.3 Zeta potential water-Al₂O₃ nanofluid

The visual display of the synthesized nanofluid can be seen in Figure 4.



Fig.4. Water – Al_2O_3 nanofluid with a concentration of 1 g / L, 3 g / L, and 5 g / L

The difference in concentration can be seen from the white level of the Al_2O_3 nanofluid and the zeta potential of the nanofluids shown in Table 1.

Concentration (g / L)	zeta potential (mV)
1	-26,3
3	-30,0
5	-33,2

Table. 1 Value zeta potential of water - Al2O3 nanofluid

The zeta potential of water $- Al_2O_3$ nanofluid increases with increasing concentration of Al_2O_3 nanoparticles. The important thing in making water- Al_2O_3 nanofluid is the sonication process using an ultrasonic device for dispersing Al_2O_3 nanoparticles in the base fluid (in this work is water). Sonication was carried out for one hour so that the dispersed nanoparticles were well distributed. Ultrasonic vibrations can reduce the occurrence of agglomeration of nanoparticles. Water- Al_2O_3 nanofluid tends to form an aggregate because it has a large surface area. According to Syarif (2019) stable nanofluid has a potential zeta greater than +30 mV or smaller than -30 mV where when a potential zeta value is large. Water- Al_2O_3 nanofluid produces a reject resistance between large particles. Large stability of nanofluid be easier to settle, the negative value on the potential zeta shows that the nanofluid is in alkaline state and the charge spread on the surface of the nanoparticles comes from OH- ions. This is in line with that expressed by Judenta [3] and Syarif [12] that the effect of citric acid contained in lemon extract as chelating agent makes the Al_2O_3 particles not accumulate.

3.4 Heat Transfer Water - Al₂O₃ Nanofluid

The results of heat transfer testing on radiators using water – Al_2O_3 nanofluid as coolant can be seen in Figure 4. which shows that the Convective Heat Transfer Coefficient (CHTC) nanofluid increases with increasing concentration of nanofluid. The CHTC value of the nanofluid is far greater than the CHTC value of the base fluid (0 g / L). Nanofluids with a concentration of 1 g / L increased about 17% larger than the base fluid, while the value of CHTC of nanofluids in a concentration of 3 g / L increased about 7% from the CHTC of the nanofluid with a concentration below (1 g / L), and nanofluid with a concentration of 5 g / L experienced an increase of about 4% from the CHTC of the nanofluid with a concentration of 3 g / L. The pH of water – Al_2O_3 nanofluid is 10 (alkaline). The alkaline condition avoid corrosion of the radiator used when testing the performance of the radiator.



Fig.5 The chart of Convective Heat Transfer Coefficient of water - Al2O3 nanofluid

This increase in CHTC of water-Al₂O₃ nanofluid is in line with the research conducted by Selvam [13] on the radiator using nanofluid (water-EG)-Graphene. The results of this study indicate that the CHTC value increases with increasing concentration of nanoparticles and it is known that the CHTC value increase by 10% to 30%.

4 Conclution

 Al_2O_3 nanoparticles were successfully synthesized using sol-gel method with calcination temperature of 1100°C using the lemon extract as a chelating agent and local bauxite sand as raw material. The crystallite size of the alpha phase nanoparticles is 37 nm and gamma phase nanoparticles is 8 nm (Measured using the Debye-Scherrer method). The synthesized nanofluids are stable nanofluids whith zeta potential of -26.3 mV for concentrations of 1g / L, -30mV for 3g / L wt and -33.2 mV for concentrations of 5g / L. The CHTC value increases with increasing concentration of nanoparticles and it is known that the CHTC value increase by 17%, 23%, and 27% for each concentration of 1g / L, 3g / L, and 5g / L.

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