Synthesis of Aluminium Nanoparticles Using Electrochemical Method

S D Anggraeni[1] **and F Kurniawan**[1,2]

[1] Laboratory of Instrumentation and Analytical Sciences, Chemistry Department, Faculty of Natural Sciences, Institut Teknologi Sepuluh Nopember, Arief Rahman Hakim, Surabaya 60111, Indonesia
[2] ITS Halal Center, Institute of Research and Community Service, Institut Teknologi Sepuluh Nopember, Surabaya 60111, Indonesia

fredy@chem.its.ac.id

Abstract. Aluminium nanoparticles (Al NPs) have been synthesized using electrochemical method. The synthesis was conducted by electrolysis of aluminium metal in a sodium citrate solution as an electrolyte solution. Formation of Al NPs was investigated on the variation of potential (5-50 V), concentration of electrolyte solution (0.02-0.42 M), and electrolysis time (15-75 minutes). The obtained product was characterized by X-ray Diffraction (XRD), Transmission Electron Microscopy (TEM), Energy Dispersive X-Ray (EDX), and UV-Vis Spectrophotometer. XRD pattern indicate that the obtained product was aluminium nanoparticles. The result show that the size of Al NPs were 56-63 nm with the Al compositions of 7.08%. The optimum condition was reached at 40 V with 0.18 M sodium citrate solution during 45 minutes electrolysis process. The plasmon band peak was found at 234 nm. In this condition Al Nps is more stable.

1. Introduction

Nano size and nano dimensional material has attracted many scientist in various fields. The structure of nanomaterials can significantly improve physical, chemical and biological properties [1]. It is due to the main characteristics of nanoparticles, e.g. the small size, surface, quantum size and quanta tunnel effect [2]. Metallic nanoparticles also have potential application in sensing [3,4], photonics catalysis and device fabrication. Aluminium (Al) is one of the metals that can be used as a based material for nanoparticles synthesis. Al has an unusual properties, e.g. light weight, high strength, corrosion resistance and high electrical and thermal conductivity [5]. Al nanoparticles (Al NPs) are considered as an innovative material, which can be used in the application of advanced energetic materials. In addition, Al NPs can be used in optoelectronic applications [6]. Al NPs can be synthesized using solid-phase techniques, liquid-phase techniques and gas-phase processes [2,7]. Electrochemical method included in liquid-phase technique has more advantageous in simplicity, not time consuming, relatively low temperature and cheaper [8,9]. This method has been used to synthesize gold [9], nickel hydroxide [10,11], aluminium oxide [12], silver [13] and tin dioxide nanoparticles [14]. In previous work, the aluminium oxide nanoparticles has been fabricated by Kolo via electrolysis using NaCl as an electrolyte solution. Unfortunately, the nanoparticles obtained has a relatively large size, which were around 96-211 nm [12]. In this study Al NPs were synthesized by electrolysis using sodium citrate as

an electrolyte solution. It is because sodium citrate can be used an a reducing agent and stabilizer to prevent aggregate formation [13]. The effect of potential, electrolyte concentration and electrolysis time were also investigated. The obtained products were characterized using X-ray Diffraction (XRD), Transmission Electron Microscopy (TEM), Energy Dispersive X-ray Analysis (EDX) and UV-Vis Spectrophotometer.

2. Experimental

2.1. Materials

Aluminium (Al) metal sheet (95.96%) with 1 mm thickness was bought from PT. Panca Logam, Indonesia. The metal sheet then cut into a dimension of 90 × 4 mm. The surface were abraded using emery paper. Furthermore, the metal was cleaned using deionized water and acetone consecutively. The metal is wrapped with heat shrink tube. Sodium citrate was purchased from MERCK which is used directly without any purification. The deionized water was obtained from PT. Brataco Chemicals, Indonesia.

2.2. Methods

Al NPs was synthesized by electolysis method as from our previous study [12,14]. Al metals were prepared as described in section 2.1 that used as both cathode and anode. Sodium citrate solution was used as an electrolyte. The electrolysis cell used can be found at previous work [12,14]. The effect of potential, electrolyte concentration and electrolysis time on Al NPs formation was investigated. Potential effect was performed at 5-50 V with an electrolyte concentration of 0.02 M for 15 minutes. Electrolyte concentration effect was carried out at variation of 0.02-0.042 M with potential of 40 V for 15 minutes. The effect of electrolysis time was studied under 15-75 minutes in electrolyte concentration of 0.18 M at 40 V. All measurement was conducted at 95°C accompanied by 50 rpm stirring process. The Al NPs crystalline structure was observed using Philips X'Pert MPD (Multi-Purpose Diffractometer) XRD with Cu $K\alpha_1$ (λ = 1.540598 nm) and $K\alpha_2$ (λ = 1.544426 nm). The morphoplogy and size of Al NPs was monitored by HITACHI H–9500 TEM. The element composition was measured by AMETEK EDAX EDX. The plasmon band peak was observed using GENESYS 10S UV-Vis Spectrophotometer.

3. Result and discussion

3.1. XRD analysis

Diffraction pattern of the Al NPs can be seen in figure 1. The diffractogram showed that several peaks were similar with Al_2O_3 standart according to JCPDS 046-1212 for α-Al_2O_3, JCPDS 023-1009 for θ-Al_2O_3 and JCPDS 046-1131 for δ-Al_2O_3. It can be seen that the typical peak for α-Al_2O_3 was appeared on the data, while the typical peaks for θ-Al_2O_3 and δ-Al_2O_3 were not found. This indicates that the α-Al_2O_3 phase is more dominant in the product than the other two phase. The high peaks on diffractogram may resulted from the phase accumulation with sodium citrate as an impurities (JCPDS 043-1526 for $C_6H_7NaO_7$).

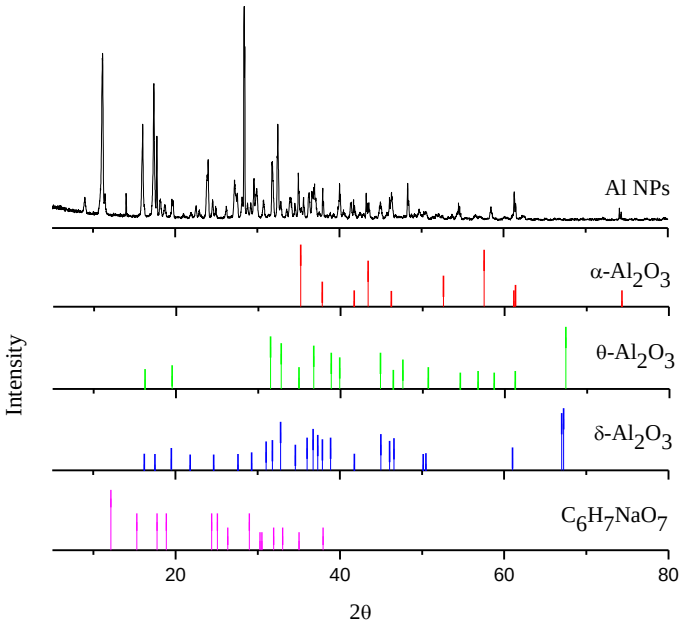

Figure 1. Diffractogram of Al NPs

3.2. TEM-EDX analysis

The result of TEM analysis is shown in figure 2. The size of Al NPs were around of 56.85-63.65 nm with slightly rounded and almost similar shape. The EDX analysis results identify the presence of aluminium (Al) signal from Al NPs, where the Al element is shown in the third peak at 1.47 KeV with composition of 7.08% (figure 3). The first peak at 0.52 KeV signifies the presence of oxigen (O) that involved in the product with composition of 82.65%. The second peak at 1,04 KeV signifies the presence of sodium (Na) from the electrolyte solution with composition of 10.27 %.

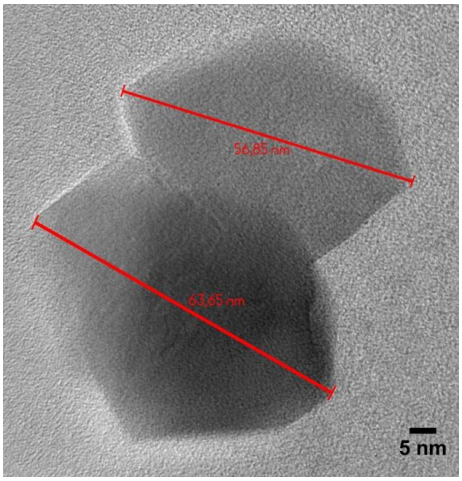

Figure 2. TEM image of Al NPs

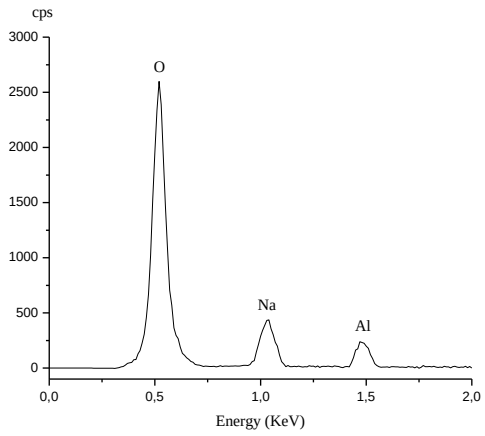

Figure 3. EDX analysis of Al NPs

3.3. UV-Vis Analysis

3.3.1. The Effect of Potential. The optimum condition in this variation was obtained in a potential of 40 V with a maximum absorbance value of 0.290 at 223 nm (figure 4). It can be seen that the potential

did not significantly affected on the shift of wavelength. The wavelength provides information about the particle size. It can be assumed that the size of the Al NPs obtained at this various potential were similar. The absorbance value give information about the number of particles produced [9,10,12–15]. The Al NPs spectrum has shown different absorbance values for each given potential, which mean the higher absorbance value produced more nanoparticles.

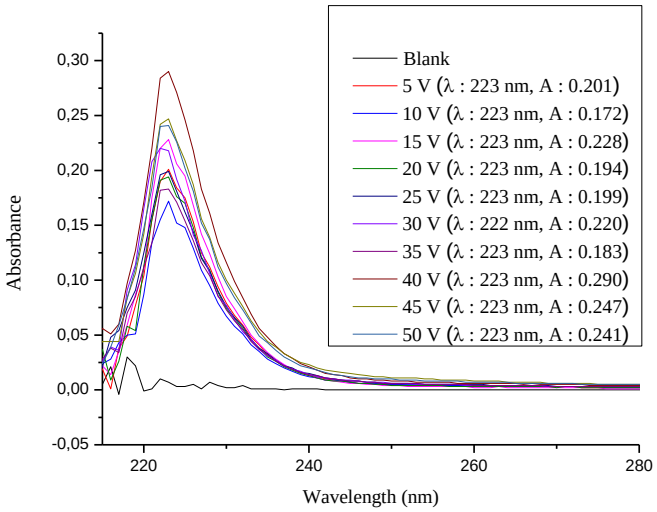

Figure 4. The effect of potential on Al NPs formation

3.3.2. The Effect of Electrolyte Concentration. Best result in this variation was obtained in 0.18 M electrolyte concentration with maximum absorbance value of 0.414 at 233 nm (figure 5). This result shows that the addition of electrolyte concentration causing a wavelength shift. The higher concentration give the greater wavelength. So it can be assumed that the size of the particles increased in the addition of electrolyte concentration. The highest absorbance value indicate the largest number of nanoparticles obtained. Decreasing of absorbance values at subsequent concentrations can be caused by precipitation of big particles [15].

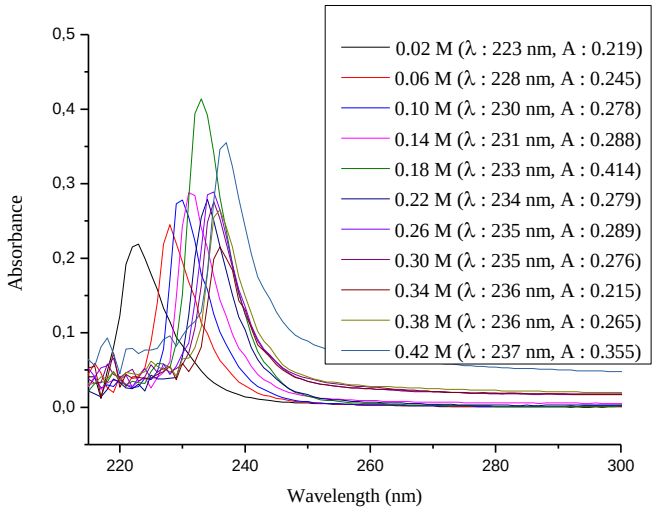

Figure 5. The effect of electrolyte concentration on Al NPs formation

3.3.3. The Effect of Electrolysis Time. Electrolysis time slightly affected in wavelength shift. It can be found at figure 6. The optimum condition in this variation was obtained at 45 minutes with maximum absorbance value of 0.525 at 234 nm. However, the obtained absorbance values also getting higher. It is indicate that the number of the nanoparticles obtained were greater. The optimum condition were determined based on the stability of the product. The electrolysis process more than 45 minutes can caused precipitation and produced white precipitate. The result show that the product has big particle size and unstable. Increasing of absorbance values in the range of 60-75 minutes can be caused by a lot of particles formed, but the distribution of the particle sizes are heterogen. It can be assumed that the precipitation was caused by the bigger particles and the increasing of the absorbance values was caused by smaller particle sizes contribution.

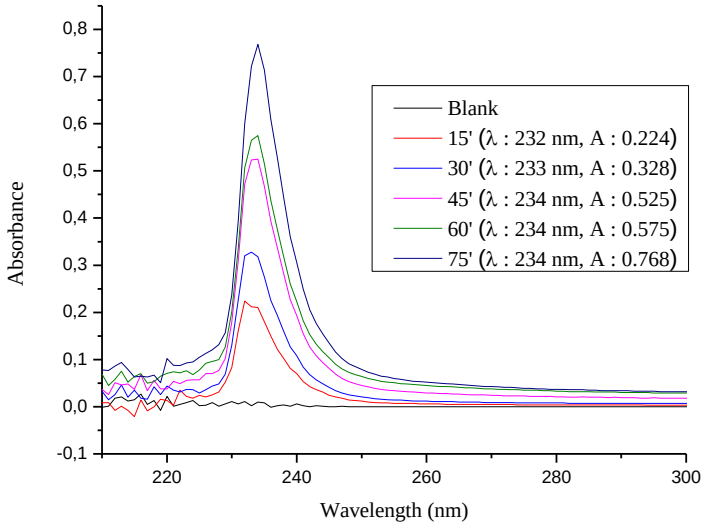

Figure 6. The effect of electrolysis time on Al NPs formation

4. Conclusion

Al NPs can be prepared using electrochemical method in sodium citrate solution. The result show that the product was Al_2O_3 nanoparticles with sizes of 56-63 nm and Al composition of 7.08%. Electrolysis at 40 V in 0.18 M sodium citrate solution during 45 minutes synthesis process can produces a more stable product.

Acknowledgments

The authors would like to acknowledge a RISTEKDIKTI (Ministry of Research, Technology and Higher Education of the Republic of Indonesia) for support a research fund under the project scheme of Penelitian Unggulan Perguruan Tinggi (PUPT).

References

[1] Joshi M, Bhattacharyya A and Ali S W 2008 Characterization techniques for nanotechnology applications in textiles *IJFTR Vol333 Sept. 2008*

[2] Kassaee M Z and Buazar F 2009 Al nanoparticles: Impact of media and current on the arc fabrication *J. Manuf. Process.* **11** 31–7

[3] Fitriyana F and Kurniawan F 2015 Polyaniline-Invertase-Gold Nanoparticles Modified Gold Electrode for Sucrose Detection *Indones. J. Chem.* **15** 226–233

[4] Zulkarnain Z, Suprapto S, Ersam T and Kurniawan F 2016 A Novel Selective and Sensitive Electrochemical Sensor for Insulin Detection *Indones. J. Electr. Eng. Comput. Sci.* **3** 496–502

[5] Mahendiran C, Ganesan R and Gedanken A 2009 Sonoelectrochemical Synthesis of Metallic Aluminum Nanoparticles *Eur. J. Inorg. Chem.* **2009** 2050–3

[6] Kuladeep R, Jyothi L, Prakash P, Mayank Shekhar S, Durga Prasad M and Narayana Rao D 2013 Investigation of optical limiting properties of Aluminium nanoparticles prepared by pulsed laser ablation in different carrier media *J. Appl. Phys.* **114** 243101

[7] Ghorbani H 2014 A Review of Methods for Synthesis of Al Nanoparticles *Orient. J. Chem.* **30** 1941–9

[8] Kurniawan F, Al Kiswiyah N S, Madurani K A and Tominaga M 2017 Single-Walled Carbon Nanotubes-Modified Gold Electrode for Dopamine Detection *ECS J. Solid State Sci. Technol.* **6** M3109–M3112

[9] Husna Q 2011 *Optimasi Konsentrasi Na-sitrat dan Pengaruh Potensial dalam Sintesis Emas Nanopartikel* (Surabaya: Institut Teknologi Sepuluh Nopember)

[10] Budipramana Y, Ersam T and Kurniawan F 2014 Synthesis nickel hydroxide by electrolysis at high voltage *J. Eng. Appl. Sci.*

[11] Budipramana Y, Suprapto, Ersam T and Kurniawan F 2016 Influence of CTAB and Sonication on Nickel Hydroxide Nanoparticles Synthesis by Electrolysis at High Voltage *Proceedings of Second International Conference on Electrical Systems, Technology and Information 2015 (ICESTI 2015)* Lecture Notes in Electrical Engineering (Springer, Singapore) pp 345–51

[12] Kolo M M 2016 *Sintesis Nanopartikel Aluminium Oksida dengan Metode Elektrokimia* (Surabaya: Institut Teknologi Sepuluh Nopember)

[13] Rahmah S and Kurniawan F 2017 Electrochemical Methods for Manufacturing Silver Nanoparticles

[14] Kurniawan F and Rahmi R 2017 Synthesis of SnO2 Nanoparticles by High Potential Electrolysis *Bull. Chem. React. Eng. Catal.* **12** 281–286

[15] Kurniawan F 2009 *New analytical applications of gold nanoparticles*