Electrolysis Synthesis and Characterization Properties of Nickel Oxide Nanoparticle

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***Abstract*—Synthesis nickel oxide nanoparticles based on electrolysis method at high voltage (55 V), with concentration natrium citrate optional condition at 0.3 M. The nickel oxide nanoparticles sample were characterized using, X-Ray Diffraction (XRD), Fourier Transform Infrared spectrometer (FTIR), Thermo Gravimetric Analysis (TGA), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). XRD result show diffractogram from the product have the same lattice with standard ICDD, Thermogram shows at a temperature above 400 ºC already formed nickel oxide nanoparticles. Spherical nanoparticles were obtained by this method.**

***Index Terms*—NiO; $Ni(OH)_2$; Electrolysis Synthesis.**

I. INTRODUCTION

Nanoparticles are in great demand because of their application in the field of science of technology, information technology, environment, new materials and energy. Experiments on the manufacture of nanoparticles continue to be performed to produce a cheap and easy to scale up. One of the interesting transition-metal hydroxides in mineral materials, is nickel hydroxide. $Ni(OH)_2$ was also applied in industries such as sensors and batteries. Nickel oxide (NiO) nanoparticles were also applied in modern industries such as electrochromic films, electrochromic windows, super capacitors, and battery cathodes [1]. Various of morphologies nickel oxide have also been fabricated such as nanofiber, nanospherical, nanocluster, and nanorods. There are many methods have been used to obtain nickel oxide nanoparticles [2]. Niasari et al., (2009) synthesis nickel oxide (NiO) nanoclusters from $[Ni(O_4C_2)(H_2O)_4]$ as precursor and oleylamine ($C_{18}H_{37}N$) using Thermal decomposition process [3]. Ananda et al., (2011) synthesis NiO via solvothermal method using nickel acetate ($Ni(CH_3COO)_2.4H_2O$) as precursor [4]. Allagui et al., (2011) reported that synthesis nickel oxide used ultrasonic method in the aqueous solution H_2SO_4 and ethanol [5]. Gondal et al., (2012) used Pulsed Laser Ablation to synthesize nickel oxide from nickel metal and H_2O_2 was used as precipitating agent [6]. Tahmasian et al., (2012) that synthesis nickel oxide used sonication from $NiNO_3(6H_2O)$, H_3L and NaOH [7]. Barani et al., (2014) synthesis NiO nanoparticles using via cathodic electrodeposition followed by a heat-treatment method using $Ni(NO_3)_2.6H_2O$ as a precursor and KOH as reagen [8]. On previous work we reported synthesis nickel hydroxide nanoparticles with electrolysis and sonication process with adding *Cetyl Trimethyl Amonium Bromide* as a surfactant to prevent nanoparticles from agglomeration [9]. In this article we demonstrated, synthesis method to get nickel oxide nanoparticles. The advantages of this method are fast, easy, and environmentally friendly.

II. EXPERIMENT

The synthesis of nickel oxide nanoparticles by electrolysis on this experiment was following the previous method done by Budipramana et al. [10]. The nickel oxide nanoparticles were prepared by one-step electrolysis method using pure nickel metal sheet with impurities 99% was bought from PT. INCO, Indonesia with the size of 1 mm × 1 cm × 7.5 cm as the cathode and anode, natrium citrate with concentration 0.3 M as starting material. 400 ml of H_2O and 10 ml of natrium citrate 0.3 M were placed in 500 ml beaker glass, are mixed together at room temperature, the solution was heated at 100 ºC and the power supply unit was operated at constant potential 55 V, stirred 30 minutes was applied the electrolysis process.

The crystal structure of nickel oxide nanoparticles obtained from electrolysis process was used to study phase structure composition from the sample was analyzed by powder X-Ray Diffraction (Philips PW 1800) using Cu K_α radiation (λ = 0.1540598 nm), as the source with a scan step of 0,02º (2θ). A Fourier Transform Infrared spectroscope (FTIR Thermo Nicolet) was used for recorded absorption spectrum from 4000 to 400 cm^{-1}. Thermal Gravimetric Analysis measurement was conducted using Shimadzu TGA-50H with a flow rate of 20.0 ml min^{-1} and heating rate 10 C min^{-1}. The morphology of the nickel oxide nanoparticles obtained was observed using Scanning Electron Microscope (nano SEM 230 machine) and Transmission electron microscopy (TEM Philips EM 2085). The specific surface area analysis was calculated based on the adsorption curve Brunauer, Emmet and Teller (BET) analyzer (Nova Quantachrome, USA).

III. RESULT AND DISCUSSIONS

In this experiment the electrolysis was performed at high potential (55V) because at low potential the reduction-oxidation reaction cannot produce nickel hydroxide nanoparticles. In the experiment known to have formed nanoparticles from the color change from colorless change to light green in the solution [11]. The reactions in the cathode and anode as Equation (1) and (2):

$$\text{Cathode} : 2\,H_2O_{(l)} + 2e^- \longrightarrow 2OH^- + H_2(g) \quad (1)$$

$$\text{Anode} : Ni_{(s)} \longrightarrow Ni^{2+}_{(l)} + 2e^- \quad (2)$$

The Ni^{2+} obtained at the anode diffused in the solution and

then mixed together to form $Ni(OH)_2$ nanoparticles as Equation (3).

$$Ni_{(s)} + 2OH^-_{(aq)} \longrightarrow Ni(OH)_{2(s)} + H_{2(g)} \quad (3)$$

The product, green solutions were separated by centrifuge to separate water and colloid $Ni(OH)_2$ nanoparticles, then dried in a vacuum oven at 80°C for 12 hours, the final product was green powder, and continue calcination 600ºC to get NiO nanoparticles. Figure 1 shows TGA curves of the decomposition NiO nanoparticles.

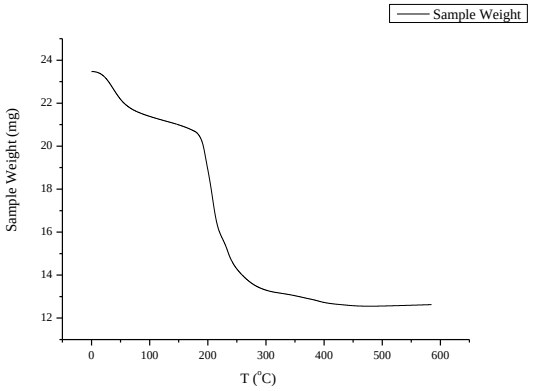

Figure 1: TGA curves obtained for thermal behavior of NiO nanoparticles

In the thermal gravimetric analysis (TGA) curves looks a decrease in mass (m_1 = 1.3455 mg) starting temperature of less than 100ºC, the mass loss is expected due to the desorption of physically bound water on the surface of the particle, around 325ºC been reduced mass (m_2 = 4.6024 mg) of the initial weight, the result has been the formation of Ni $(OH)_2$, the reaction is:

$$Ni(OH)_2.\ n\ H_2O \longrightarrow Ni(OH)_2 + H_2O \quad (4)$$

The third sharp decrease in mass at a temperature of 425ºC (m_3 = 0,9233 mg) occurred due to the glass transition of component (not have amorphous shapes like crystals) phase into a monoclinic structure, then at a temperature of 425ºC to 600ºC temperature has begun to form NiO. The reaction is:

$$Ni\ (OH)_2 \longrightarrow NiO + H_2O \quad (5)$$

There is no mass loss in the temperature range 600-900ºC.

SEM images of the $Ni(OH)_2$ nanoparticles obtained that was prepared at voltage 55 volt are shown in Figure 2. Figure 3 is TEM images of NiO nanoparticles, the morphology of NiO nanoparticles is spherical.

Diffractogram of $Ni(OH)_2$ nanoparticles obtained at voltage 55 volt is shown in Figure 4. The XRD diffractogram obtained represent $Ni(OH)_2$ nanoparticles has crystal formed. All characteristic diffraction peaks well consist with the hexagonal $Ni(OH)_2$ (International Centre for Diffraction Data file no.117). All of the peaks could be indexed as tetragonal. The difference in diffraction relative intensity between the samples indicates their difference in microstructure and morphology [12]. Additional diffractogram of NiO which obtained after calcination of $Ni(OH)_2$ at 600ºC All characteristic diffraction peaks well consist with the hexagonal NiO (match file no.96-101-0382), are also shown in Figure 5.

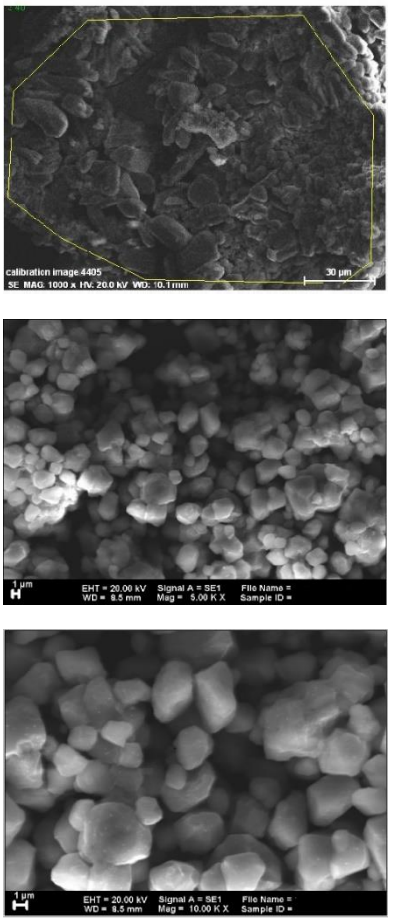

Figure 2: SEM micrograph of the $Ni(OH)_2$ nanoparticles

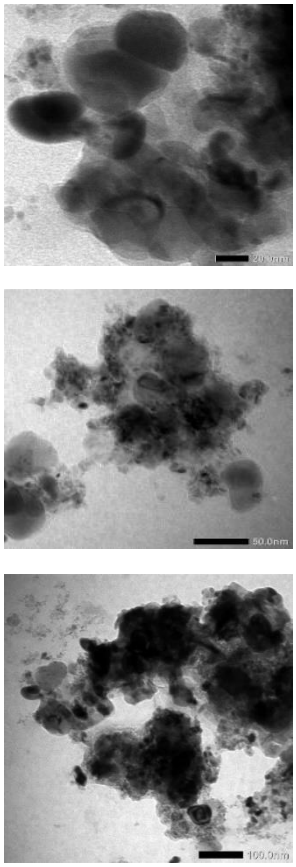

Figure 3: TEM images of the NiO nanoparticles

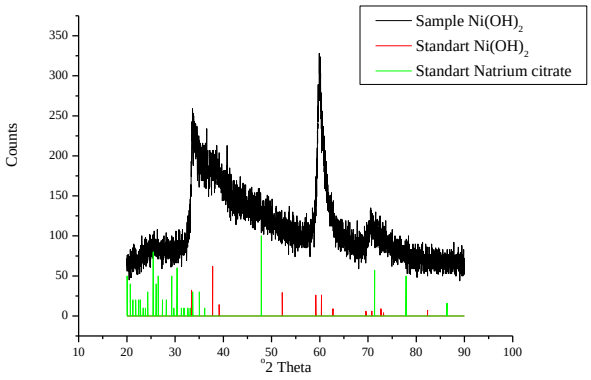

Figure 4: Diffractograms of $Ni(OH)_2$ nanoparticles

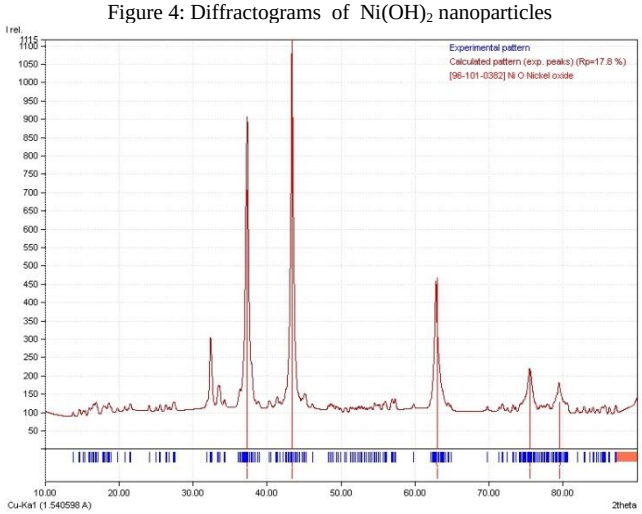

Figure 5: Diffractograms of NiO nanoparticles which were obtained after calcination of $Ni(OH)_2$

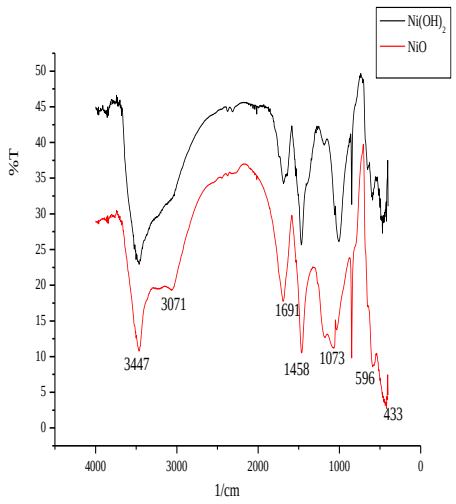

Figure 6: FTIR spectrum $Ni(OH)_2$ and NiO nanoparticles.

Figure 6 is FTIR images of $Ni(OH)_2$ and NiO nanoparticles The narrow band at 3447 cm^{-1} indicates O–H group. The band at 1691 cm^{-1} is due to bending vibration of the adsorbed water molecules. The weak absorption band at 433 cm^{-1} and around 596 cm^{-1} is assigned to Ni–O stretching and Ni–O–H bending vibration [2].

BET (Brunauer, Emmet and Teller) surface area measurements were also performed on samples from the optimal reaction range, and pore distribution calculation using BJH (Barrett, Joyner and Halenda) method. BET also gives results that can be used as a complement to the SEM, TEM and XRD results, where the distribution of particle size is very narrow, the specific surface areas are around 13.249 m^2/g.

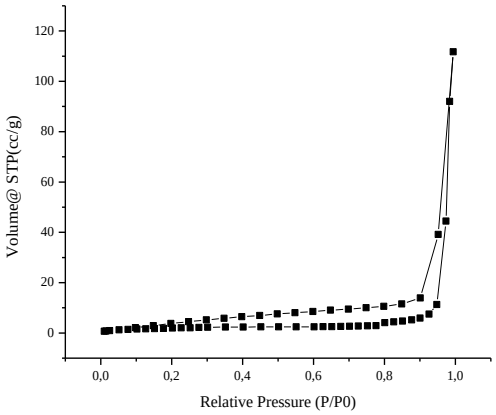

Figure 7: BET curve of NiO nanoparticles

Figure 7 is BET curve of NiO nanoparticles, BET curve adsorption and desorption curves are quite parallel without any flat branch for the high-pressure area. BET analysis showed these nanoparticles have a surface area of 7.238 m^2/g, and the average pore diameters are above 16.984˚ A. Then, the curve in Figure 7 included type IV isotherm in the Brunauer, Emmet and Teller classification.

IV. CONCLUSION

Nickel oxide nanoparticles can be prepared using electrolysis method. Voltage 55 volt and concentration 0.3M natrium citrate give the best result because it is stable for long periods. TEM analysis indicates that particles size 30-50 nm and the morphology of nickel oxide nanoparticles are spherical.

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