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## SYNTHESIS NICKEL HIDROXIDE BY ELECTROLYSIS AT HIGH VOLTAGE

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### ABSTRACT

Nickel hydroxide nanoparticles have been synthesized electrochemically. The synthesis based on electrolysis system which bare nickels were used for both cathode and anode. The potential applied during electrolysis was from 10 - 55V. The variation of sodium citrate concentration, i.e. 0.1 M; 0.2M; 0.3 M; 0.4 M; 0.5M, was used to study optimal condition of nickel hydroxide nanoparticles formation. UV-Vis spectroscopy, X-ray Diffraction (XRD), Transmission Electron Microscopy (TEM) and Fourier Transform Infrared Spectrometer (FTIR) were used to characterize the microstructure and morphology of the products. Spherical nanoparticles were obtained by this method. The generated particles are nearly spherical with a mean size 60 nm depending on synthesis condition. A stable product with no agglomeration in the long term was obtained using condition 0.3 M sodium citrate at 55 Volt for 30 minutes.

**Keywords:** nickel hydroxide nanoparticles, electrolysis, high voltage.

### INTRODUCTION

Nickel hydroxide nanoparticles have been widely studied in some varieties of application: material science, chemistry, physics, biology, medicine and environmental science. Nickel hydroxide nanoparticle was also applied in modern industries such as sensors, electronics, super capacitors, catalysts, and batteries [1]. Several techniques have been used to obtain nickel hydroxide nanoparticle. Synthesis of nickel hydroxide was done by urea hydrolysis using homogeneous alkalization of nickel (II) nitrate solutions [2]. Motlagh, et al. (2012) reported that synthesis nickel hydroxide used microwave and sodium hydroxide as the precipitating agent [3]. Khan et al. (2011) used hydrothermal method to synthesize nickel hydroxide from NiSO<sub>4</sub> and NH<sub>3</sub>. H<sub>2</sub>O<sub>2</sub> was used as precipitating agent [4]. However, some techniques require high pressure, high temperature, and soluble polymer as protective agent which increase production cost. In this paper a combination of high voltage electrochemical methods (electrolysis) and sodium citrate as electrolyte were applied for nickel hydroxide synthesis. This method has some advantages in order to easily scaling up and economical process because only use bare nickel and power supply unit. Each of the nickel hydroxide samples obtained was characterized by UV-Vis spectroscopy and X-ray Diffraction (XRD).

### EXPERIMENTAL

Pure nickel metal sheet with 1 mm thickness was bought from PT. INCO, Indonesia. The nickel metal sheet was cut into dimension of 1 cm x 7.5 cm. sodium citrate purchased from MERCK was used directly without any purification. The deionized water was used during all the experiments

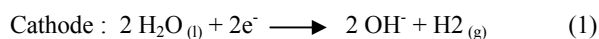
400 ml of H<sub>2</sub>O and 10 ml of Na-citrate at various concentrations, i.e. 0.1 M, 0.2M, 0.3 M, 0.4 M and 0.5M, were placed in 500 ml beaker glass. The electrolysis system used nickel metal sheet as the cathode and the anode which dimension was described at section 2.1.

Initially, the sodium citrate solution was heated at 300 °C, and then the power supply unit was operated at constant potential (i.e. 10V; 15V; 20V; 25V; 30V; 40V; 45V; 50V; and 55V). Continues stirring was applied during the electrolysis process. The reaction was conducted for 30 minutes. The solution obtained was cooled in room temperature and characterized

The plasmon band peak of nickel nanoparticles obtained from electrolysis process was analyzed by Genesys 10S UV-Vis spectrophotometer. Philips X' Pert MPD (Multi Purpose Diffractometer) XRD using Cu K<sub>α</sub> radiation (λ = 0.15418 nm), provided 40 kV and 40 mA with scan step of 0, 02 ° (2θ) was used to characterize the nanoparticles structure. Absorption spectrum was recorded from 400 to 4000 cm<sup>-1</sup> on a Shimadzu Fourier Transform Infrared (FTIR) spectrometer. The shape of the nickel nanoparticles obtained was observed using JEOL JM140m HR-TEM test (High Resolution Transmission Electron Microscope).

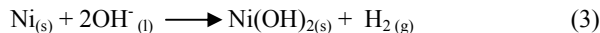
### RESULT AND DISCUSSIONS

Electrolysis usually were performed at low potential (below 10V) to induce chemical reaction because of the reactant potential reduction standard was also low. In this experiment the electrolysis was performed at high potential (more than 10 V) because at low potential the redox reaction will not take place to produce nickel hydroxide nanoparticles. The formation of Nickel hydroxide nanoparticles during electrolysis can be observed from the color change that occurred in the solution. The color of the solution changes from colorless to green gradually. The reactions that occur in the both electrodes as Eqn. (1) and (2):

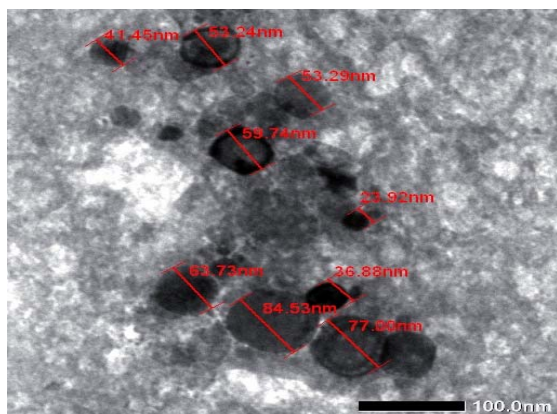




The products from cathode and anode diffused in the solution and then mixed together to form nickel hydroxide nanoparticles as Eqn. (3)

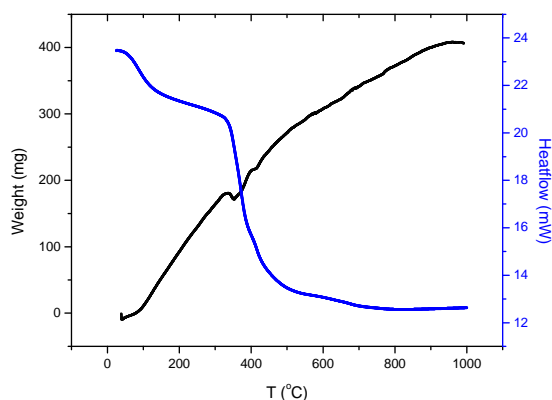


In the electrolysis process, the oxidation reaction occurs at the anode. The oxidation number of nickel increase from 0 to 2+, while at the cathode the water reduced to OH<sup>-</sup> and H<sub>2</sub> (it can be observed by the production of bubbles at the cathode during electrolysis process). The addition of sodium citrate induced the formation of nickel nanoparticles, because there will not be any product without the sodium citrate addition. The average size of nickel hydroxide nanoparticles is 60 nm which can be seen in Figure-1.



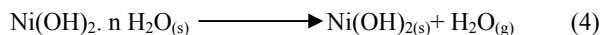
**Figure-1.** TEM image of nickel hydroxide nanoparticles obtained from the electrolysis process in 0.3M Sodium citrate at 50V for 30 minutes.

The DTA/TGA analysis was studied to have a better understanding on thermal stability of the product obtained. Thermogram of the decomposition of nickel hydroxide nanoparticles can be seen in Figure-2.

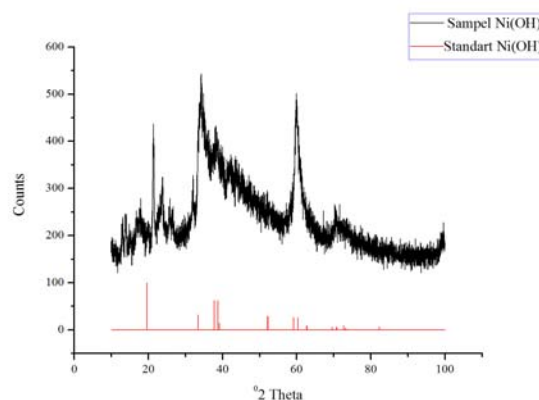
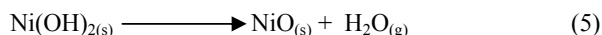


**Figure-2.** Thermogram of nickel hydroxide nanoparticles obtained from synthesis.

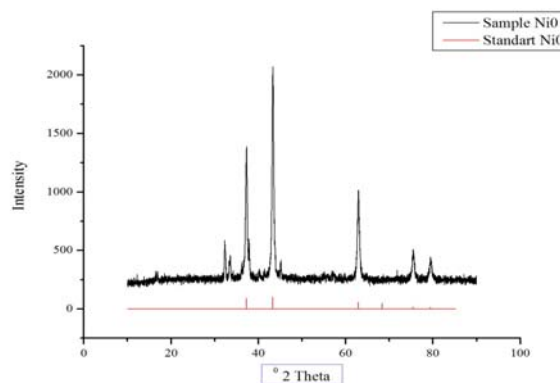
At the beginning of the heating until 120°C, the water which physically bound to nickel hydroxide nanoparticles is evaporated (6.5% of the sample). In the other side the hydrated water from nickel hydroxide nanoparticles was evaporated at higher temperature (from 120°C - 323°C). This is in accordance with the XRD analysis that showed nickel hydroxide anhydrate product (Figure-3) as described in reaction as Eqn. (4):



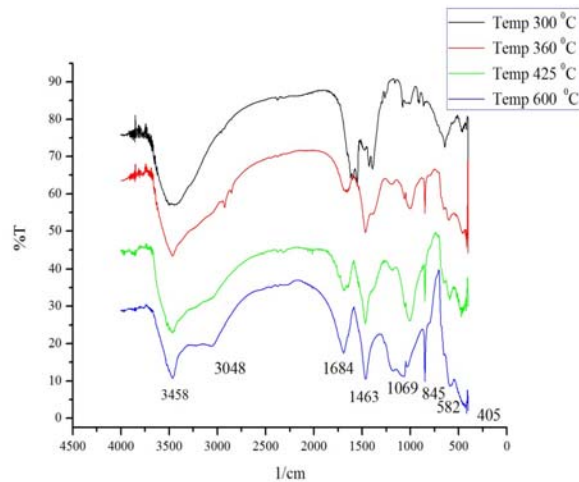
There are two valleys which appear from DTA result, i.e. at 372 °C and 414 °C with a weight loss of 23.86 % due to evaporation of crystalized water. The decomposition of nickel hydroxide nanoparticles starts from 400°C with weight loss of 19.67% due to formation of H<sub>2</sub>O and NiO which analyzed by XRD analysis (Figure-4). The decomposition reaction is described as Eqn. (5).



**Figure-3.** XRD of nickel hydroxide nanoparticles at a temperature of 300 °C.



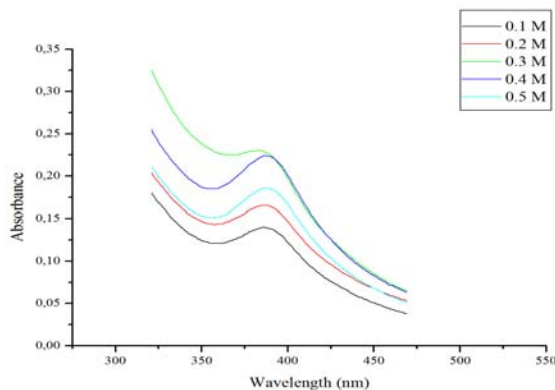
**Figure-4.** XRD of nickel oxide at a temperature of 600 °C.



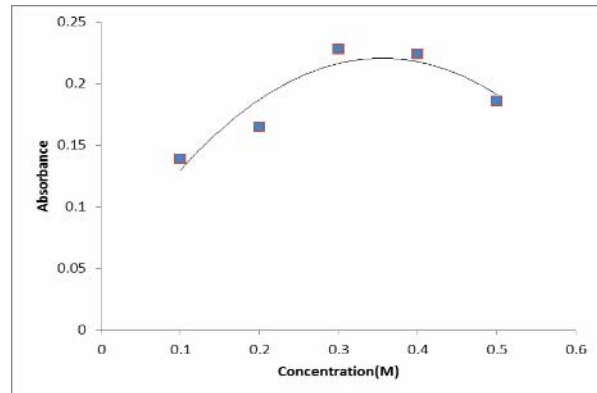
**Figure-5.** FTIR spectra of nickel hydroxide nanoparticles.

The FTIR analysis of the nickel nanoparticles obtained by electrolysis process is shown in Figure-5. The vibrational peaks at 3458 and 3048  $\text{cm}^{-1}$  due to O–H stretch [4]. Peak around 405  $\text{cm}^{-1}$  and 582  $\text{cm}^{-1}$  are described as Ni–O stretching and Ni–O–H bending vibration, bending vibration at 1684  $\text{cm}^{-1}$  due to adsorbed water molecules [5]

Nickel hydroxide nanoparticles in water have a light green color due to its plasmon band. The nanoparticles formation can be observed by its colors change, so it can be measured by UV-Vis spectroscopy, see Figure-6. The concentration of nickel hydroxide nanoparticles can be determined by its absorbance, see Figure-7.



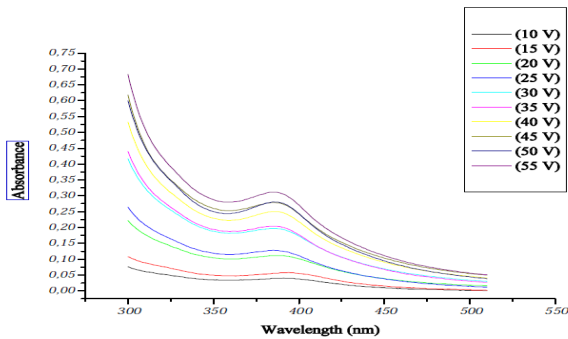
**Figure-6.** UV-Vis spectrum of nickel hydroxide nanoparticles obtained in 0.1M to 0.5M sodium citrate at 55V for 30 minutes.



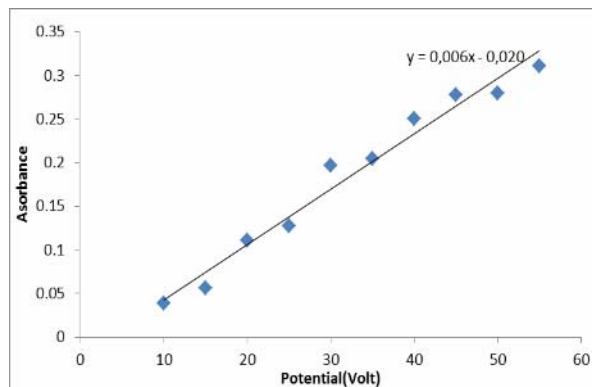
**Figure-7.** Absorbance as function of sodium citrate concentration from 0.1 M - 0.5M at 55V for 30 minutes.

The concentration of nickel hydroxide nanoparticles increase with the increase of sodium citrate concentration. It indicates that sodium citrate can induce the formation of nickel hydroxide nanoparticles. All spectrums show the similar peak pattern, it means that the size distribution of the nickel hydroxide nanoparticles, which is obtained from various concentrations of sodium citrate, is also similar. The higher sodium citrate concentration used in the solution will only influence to particles growth suppression by the formation of many nuclei. More nuclei formed at the same time and grow at the same rate will increase the concentration of the obtained particles. Too many nuclei formed at the same time will cause the particle's surface potential cannot inhibit the agglomeration [6]. Therefore, the nanoparticles produced from the solution probably are unstable and will grow by coagulation process to form bigger particles. Thus initial concentration of  $\text{Ni}^{2+}$  can determine the particle number of the resulting product in the formation of nickel hydroxide nanoparticles. The proper  $\text{Ni}^{2+}$  concentration is required to get well-dispersed nickel powder without particle agglomeration. In this research, 0.3 M is the optimum condition.

The higher electrolysis potential applied, the higher absorbance peak of nickel nanoparticles obtained, as shown in Figure-8 and Figure-9.



**Figure-8.** UV-Vis spectra of nickel hydroxide nanoparticles obtained in 0.3 M sodium citrate at 10 V to 55 V for 30 minutes.



**Figure-9.** Absorbance as the function of potential applied from 10 V to 55 V in 0.3 M sodium citrate for 30 minutes.

The constant distribution of the particle size and morphology due to the differences of sodium citrate concentration and electrolysis voltage are explained by the reaction in nucleus. The diameter of the particle size increases until definite value, and will agglomerate and deposited in the bottom of the vessel. In the other side the nickel hydroxide nanoparticles will remain in the solution. The increasing concentrations of sodium citrate and voltage from 10 volts to 55 volts will only influence to the number of generated nucleus which was explained with the increase of the absorbance peak. Based on the results, it can be concluded that nickel nanoparticles can be synthesized by electrochemical method in sodium citrate solution as the electrolyte. This method produced nickel hydroxide nanoparticles with a nearly spherical shape.

## CONCLUSIONS

Nickel hydroxide nanoparticles can be prepared by electrochemical method in sodium citrate solution. Electrolysis at 55 volt in 0.3 M sodium citrate produces stable product in the long term. TEM analysis indicates that average particles size is 60 nm.

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