

A SIMPLE SYNTHESIS OF NICKEL OXIDE NANOTUBE USING HIGH VOLTAGE ELECTROLYSIS

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ABSTRACT

Recently, the development of nanoparticle nickel oxide has increased due to their potential application such as biosensors, catalysts, solar cells, supercapacitors, and batteries. In this work, the addition of CTAB for nickel oxide nanoparticle synthesis using electrolysis was investigated. The nickel plates were used as anode and cathode in the electrolysis process. The process was operated at a constant voltage of 60 V for 30 minutes. The XRD result showed conformity with the Nickel oxide diffraction pattern. Meanwhile, the impurity from nickel hydroxide peaks still appeared. From FTIR characterization also indicates the band of Ni-O stretching vibration. The morphology characterization of nickel oxide using Scanning Electron Microscopy (SEM) showed the nanotube structure, while Transmission Electron Microscopy showed the nanoparticle size from 250.44 to 325.60 nm in length. On the other hand, the transformation of Ni(OH)₂ to NiO has been shown using TGA characterization.

Keywords: nanoparticles; nickel oxide; electrolysis

Introduction

Recently, the high demand for small electronic devices has made research on nanotechnology growing rapidly. Moreover, nanotechnology became more interesting due to its large surface area and different properties than the bulk materials.¹ Nickel oxide one of the metal nanoparticles has been attracted many industries due to its potential applications in modern industries such as batteries,^{2,3} solar cells,^{4,5} catalysts,^{6,7} and sensors.^{8,9} On the other hand, Indonesia has a huge nickel mine in the world, so the use of nickel-based materials is necessary.

Research on the nickel oxide nanoparticles has been conducted since 1994,¹⁰ and continues until now due to its properties such as inexpensive material as an ion storage

material, good durability and electrochemical stability, and various manufacturing possibility.¹¹ The development of research has been conducted to obtain nickel oxide nanoparticles using various methods. The use of precursors such as NiCl₂·6H₂O,¹² nickel metal¹³, and Ni(NO₃)₂·6H₂O,¹⁴ has been conducted. On the other hand, a combination method with thermal decomposition at low-temperature to synthesize nickel oxide nanoparticles also have been applied.¹⁵ The size of the nanoparticle's product is influenced by some parameters such as pH, surfactant, and co-surfactant during the process. Surfactants are commonly used as additives in the synthesis of nanoparticles because of their advantages in reducing surface tension and stabilizing the formation

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of nanoparticles. Cetyl Trimethyl Ammonium Bromide (CTAB) is a popular cationic surfactant due to its ability to produce materials with unique characteristics than different kinds of salts, such as NaCl and Na₂SO₄. Moreover, the advantages of the CTAB surfactant can stabilize the process of nucleation, coagulation, and aggregation.¹⁶

Synthesis of Nickel Oxide nanoparticles was still being carried out at this time, but the method still takes a long time to make Nickel oxide. For this reason, we investigated the synthesis of nickel oxide nanoparticles using the CTAB surfactant and electrolysis method.

Methods

Synthesize Materials

The high purity of the nickel plate (99%) was cut into a smaller size with a dimension of 10 mm x 75 mm as the cathode and anode. The 400 ml of DI water and 10 ml sodium citrate (0.3M) were mixed as electrolyte solution in 500 ml of beaker glass. Then, 1 mm of CTAB was diluted into the electrolyte solution. The process was conducted by arranged the nickel plate and electrolyte solution as shown in figure 1.

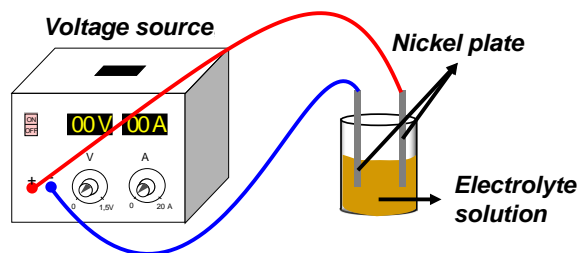


Figure 1. Schematic diagram of the electrolysis nickel plate.

The electrolysis process was carried out at a constant voltage of 60 V for 30 minutes. The solution from the electrolysis process was centrifuged and then was dried 80°C for 10 hours in the oven. After that, the sample was calcinated using a muffle furnace at 600°C to obtain the nickel oxide nanoparticles.

Sample Characterizations

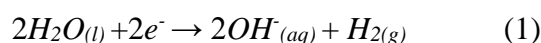
The crystal structure of the sample was analyzed by Philips Xpertpor X-ray

diffraction (XRD). A Scanning Electron Microscopy (FEI Nova SEM 230) and Transmission Electron Microscopy (JEOL-JM 140) was used to know the morphology of the sample. The functional groups of the sample were analyzed by Fourier Transform Infrared Spectroscopy (Thermo Scientific Nicolet iS 10 spectrometer).

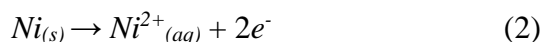
Result and Discussion

The reactions in the electrolysis process at the constant voltage of 60V for 30 minutes were shown by equation 1 and 2.^{17,18}

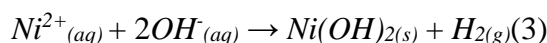
Cathode



Anode



In the solution, the Ni(OH)₂ was formed as shown by Equation 3.



XRD Analysis

The XRD pattern of the Nickel oxide was shown in figure 2.

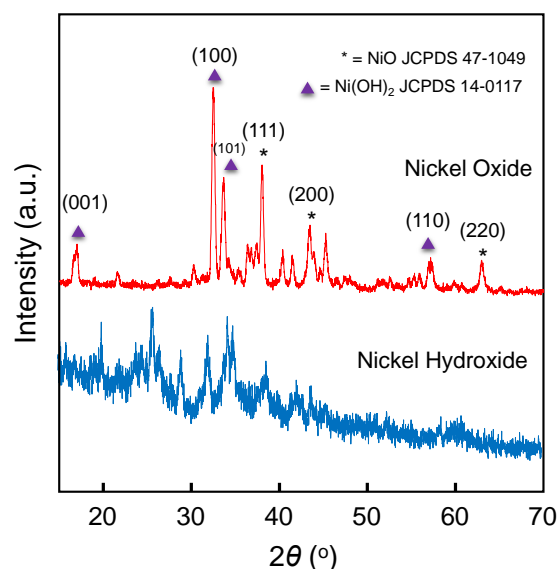


Figure 2. The X-Ray Diffraction pattern of Nickel Oxide (NiO)

There corresponding some peaks to the nickel oxide nanoparticle diffraction pattern (JCPDS 47-1049) indicate that the nickel oxide has been formed.¹¹ The appearing peaks

at $2\theta=37.94^\circ$, 43.51° , and $62,87^\circ$ can be indexed as (111), (200), and (220). Meanwhile, the impurity from nickel hydroxide peaks still appeared in the nickel oxide XRD pattern (JCPDS 14-0117), indicating the nickel oxide has not been perfectly formed.¹⁹

FTIR Analysis

Figure 3 shows the functional group of NiO and Ni(OH)₂. The NiO material had a transmittance peak at wavenumber 3397 cm^{-1} and 1640 cm^{-1} which represent the functional group of O-H vibrations and H-O-H bending vibration mode.²⁰ The appearance of O-H vibration and H-O-H bending vibration mode indicated that the sample absorbed moisture. On the other hand, the peaks at 1422 cm^{-1} and 999 cm^{-1} likely showed some precursor remnant materials in the synthesized NiO process.²¹ Meanwhile, the existence peak of Ni-O and Ni-O-H wavenumber 534 cm^{-1} and 693 cm^{-1} respectively indicate that the nickel oxide was present in the sample.^{22,23}

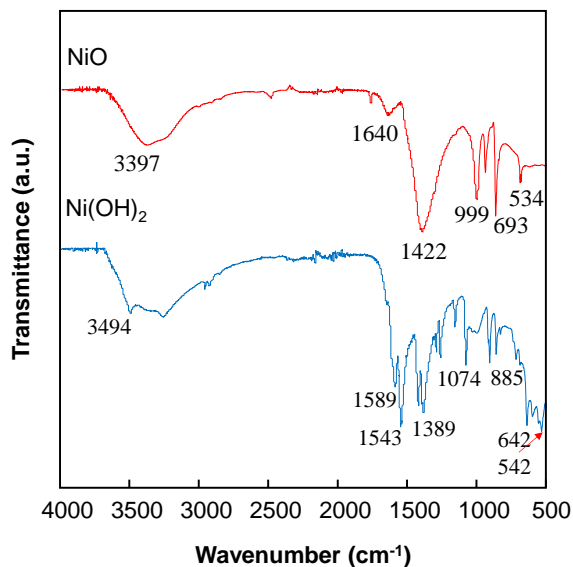


Figure 3. FTIR spectra of NiO and Ni(OH)₂.

SEM and TEM Analysis

The morphology of the NiO sample is shown in Figure 4.

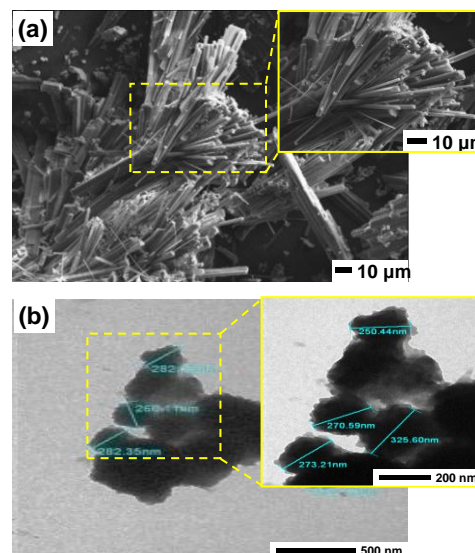


Figure 4. Characterization result of (a) SEM and (b) TEM from NiO nanotubes.

The synthesis of the NiO sample shows a tube shape in the SEM image (figure 4(a)), indicating the formation of NiO nanotubes.^{24,25} In the higher magnification, the sample has confirmed the agglomeration of one-dimensional nanotubes, and the rough surface may be caused by the set of NiO nanoparticles.²⁶ However, the sample has inhomogeneous shapes nanotubes. The inhomogeneous shapes were suspected by the existence of Nickel hydroxide and other remnant materials in the sample corresponding to the XRD and FTIR characterization. On the other hand, to know the NiO stabilized with CTAB size, the TEM characterization was conducted (figure 4(b)). TEM image showed the spherical structure of NiO with a particle size of 250.44 to 325.60 nm, indicating the sample was the nanoparticle's materials.

Thermogravimetric Analysis

The effect of heating treatment to mass changing of Ni(OH)₂ to NiO was investigated using thermogravimetric analysis. Figure 5 shows the Thermogravimetric curve of NiO at a temperature range of 100-900 °C. The first and declining points can be seen at a temperature of 170 °C with a reduced mass of about 4.0403 mg. This step is indicating the elimination of water content from the sample.²⁷ In the temperature of 320°C, the

1.180 mg of mass reduced due to the decomposition of sodium citrate.^{17,18}

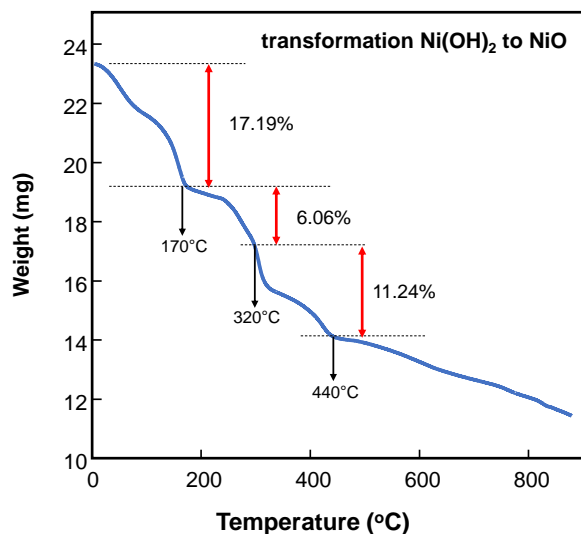
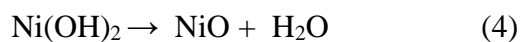


Figure 5. The thermogravimetric curve of NiO at the temperature range of 100-900 °C

Meanwhile, the last declining point can be seen at the temperature of 440°C with the 2.0536 mg mass loss. The reduced mass was possible due to the crystallization of the sample.²⁸ The conversion of Ni(OH)₂ to NiO was approximately formed at the temperature of 200-400°C (Eq. 4).²⁹



After the temperature reaches 600°C, the thermogravimetric curve became flattered. It indicates that there was not a mass loss in this temperature range of 600-900°C.

Conclusion

A simple synthesis of spherical nickel oxide (NiO) nanotubes has been successfully produced by electrolysis at high voltage techniques. The XRD result showed conformity with the Nickel oxide diffraction pattern. From FTIR characterization indicates the band of Ni-O stretching vibration. The morphology characterization of nickel oxide using Scanning Electron Microscopy (SEM) showed the nanotube structure, while Transmission Electron Microscopy showed the nanoparticle size from 250.44 to 325.60 nm in length. On the other hand, Thermogravimetric analysis (TGA) showed the transformation of Ni(OH)₂ to NiO.

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