ISSN: 2549-6573 (Online)
ISSN: 2549-6565 (Print)
DOI: 10.17977/um0260v5i22021p008
JC-T (Journal Cis-Trans): Jurnal Kimia dan Terapannya

Electrochemical Synthesis of Nickel Hydroxide $Ni(OH)_2$ Nanoparticles Solution for Detecting Mercury (Hg)

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Article history

Received: 8th July 2021
Received in revised form:
23th December 2021
Accepted: 27th December 2021

DOI:
10.17977/um0260v5i22021p008

Kata-kata kunci:
Nanoparticle $Ni(OH)_2$,
Electrolysis
Mercury

Abstrak

Nikel hidroksida $Ni(OH)_2$ nanopartikel digunakan sebagai sensor merkuri (Hg) dalam air menggunakan instrumen spektrofotometer UV-Vis. Sintesis nanopartikel $Ni(OH)_2$ dilakukan dengan metode elektrokimia menggunakan pelat nikel sebagai anoda dan katoda dan prosesnya dioperasikan pada tegangan konstan 25 V selama 30 menit. Pada penelitian ini digunakan 2 variasi konsentrasi nanopartikel $Ni(OH)_2$ yaitu (5 mL dan 4 mL) untuk mendeteksi merkuri pada konsentrasi 0 ppm, 10 ppm, 20 ppm, 30 ppm, 40 ppm, dan 50 ppm. ppm. Perubahan nilai absorbansi nanopartikel $Ni(OH)_2$ akan diamati. Dari hasil pengamatan, terjadi penurunan nilai absorbansi nanopartikel $Ni(OH)_2$ seiring dengan peningkatan konsentrasi merkuri. Penurunan nilai absorbansi nanopartikel $Ni(OH)_2$ berbanding lurus dengan besarnya konsentrasi merkuri. Nilai limit deteksi diperoleh pada volume 5 mL dan 4 mL yaitu 45 ppm, dan 35 ppm.

Abstract

Nickel hydroxide $Ni(OH)_2$ nanoparticles were used as sensors for mercury (Hg) in water using a UV-Vis spectrophotometer instrument. The synthesis of $Ni(OH)_2$ nanoparticles was carried out by electrochemical method using nickel plates as anode and cathode and the process was operated at constant voltage of 25 V for 30 minutes. In this study, 2 variations of the concentration of $Ni(OH)_2$ nanoparticles were used, namely (5 mL and 4 mL) to detect mercury at concentrations of 10 ppm, 40 ppm, and 50 ppm. Changes in the absorbance value of $Ni(OH)_2$ nanoparticles will be observed. From the observations, there was a decrease in the absorbance value of $Ni(OH)_2$ nanoparticles along with the increase in mercury concentration. The decrease in the absorbance value of $Ni(OH)_2$ nanoparticles is directly proportional to the amount of mercury concentration. The detection limit value was obtained at a volume of 5 mL and 4 mL are 45 ppm, and 35 ppm.

INTRODUCTION

In the last 10 years nanotechnology has become interesting to study, many patents and journals have been published using several methods and material. Nanoparticles have very different properties than bulk materials, because nanoparticles have a large surface area, nanoparticles from metal like Au, Fe, Mn, Ni, are also actively studied, now we want to study detection mercury using nanoparticle nickel hydroxide. Mercury is a type of metal that is found in nature and is spread in rocks, ores, soil, water and air as inorganic and organic compounds.

Mercury is also often referred to as mercury (Hg). Mercury is corrosive to skin. This means that applying mercury to the skin will make the skin layer thinner. High exposure to mercury can include damage to the digestive tract, nervous system, and urological system. In addition, mercury also has the risk of disrupting various organs of the body, such as the brain, heart, kidneys, lungs, and immune system. Methyl mercury that stays in the human body will cause brain malfunction, anxiety / nervousness, kidneys, and liver damage during birth (birth defects). Therefore, it is necessary to develop a quick and simple analysis method to detect the presence of mercury (Hg) pollution in the environment, especially in water. So far, the methods commonly used to detect mercury contamination in water are atomic absorption spectroscopy (AAS) and gas chromatography / and ICP methods. On this occasion we studied detecting the presence of mercury metal using $Ni(OH)_2$ nickel hydroxide nanoparticles.

METHODS

$Ni(OH)_2$ nanoparticles were prepared using a set of electrolysis cells with 2 nickel electrode plates that act as anode and cathode with a purity of 99% as proven by XRD characterization. The synthesis of nickel nanoparticles was carried out by adding 1 mL of 0.3 M sodium citrate solution into 400 mL of water under boiling (heating using a hot plate) and stirring using a magnetic stirrer (Pramana et al, 2020). At the time of adding Na-citrate, simultaneously the on button is pressed on the power supply which has previously been adjusted to the voltage of 25V. The reaction begins when the sodium citrate solution is added to the water. The reaction of the formation of $Ni(OH)_2$ nanoparticles can be observed from the color change that occurs in the solution. The color of the solution will change from colorless to green. This color change (Fig 1) indicates that $Ni(OH)_2$ nanoparticles have formed. The darker the color of the solution, the more $Ni(OH)_2$ nanoparticles are formed in the solution. Preparation of 1000 ppm mercury main solution (Hg), Making mercury (Hg) solution was done by taking as much as 73 µL of Hg metal using a micropipette and then putting it into a beaker. Added 50 µL of concentrated HNO_3 (37%) into the beaker which had previously been given a little demineralized water to dissolve the Hg metal. Shake the beaker slowly until Hg metal dissolves completely, after that put the solution into a 1 L volumetric flask using a funnel and then add demineralized water to the volumetric flask until the limit mark for dilution.

(a) (b)

Figure 1. Color changes in the synthesis of $Ni(OH)_2$ nanoparticles at 0 minutes (a), 30 minutes (b).

RESULT AND DISCCUSION

Analysis of $Ni(OH)_2$ Nanoparticles

Diffractogram of Nickel plate shown in Fig 2a, from The XRD results showed that nickel plate has purity 99 %. The diffractogram shows 3 peaks at 2 theta 44.68; 51.73 and 76.47. This is in accordance with the ICDD nickel standard (*International Center of Diffraction Data no 00004-0850*). Diffractogram of $Ni(OH)_2$ nanoparticles obtained at 25 V are shown in Fig. 2b All characteristic diffraction peaks are well consisted with the hexagonal $Ni(OH)_2$ at 2θ = 34,22; 38,30; 60,37; 70,87, ICDD (*International Centre for Diffraction Data* file no.117). TEM images of the $Ni(OH)_2$ nanoparticles obtained as prepared at voltage 25 V are shown in Fig. 2d The morphology of $Ni(OH)_2$ nanoparticles is spherical. The results of characterization using UV-Vis spectrophotometer can be seen in Fig 2c. In this curve, information is obtained in the form of the absorbance value and the maximum wavelength of the $Ni(OH)_2$ nanoparticles. The maximum wavelength is the highest peak where $Ni(OH)_2$ nanoparticles can absorb the maximum light. This maximum wavelength will be used as the reference wavelength for further measurements. Based on the curve, the maximum wavelength of $Ni(OH)_2$ nanoparticles is 387 nm. In addition to the maximum wavelength, data is also obtained in the form of absorbance. The absorbance is a large size that can be absorbed by $Ni(OH)_2$ nanoparticles. This absorbance will be used as a comparison for the addition of additional mercury solutions that will be carried out next. From the curve, the absorbance is 0.127.

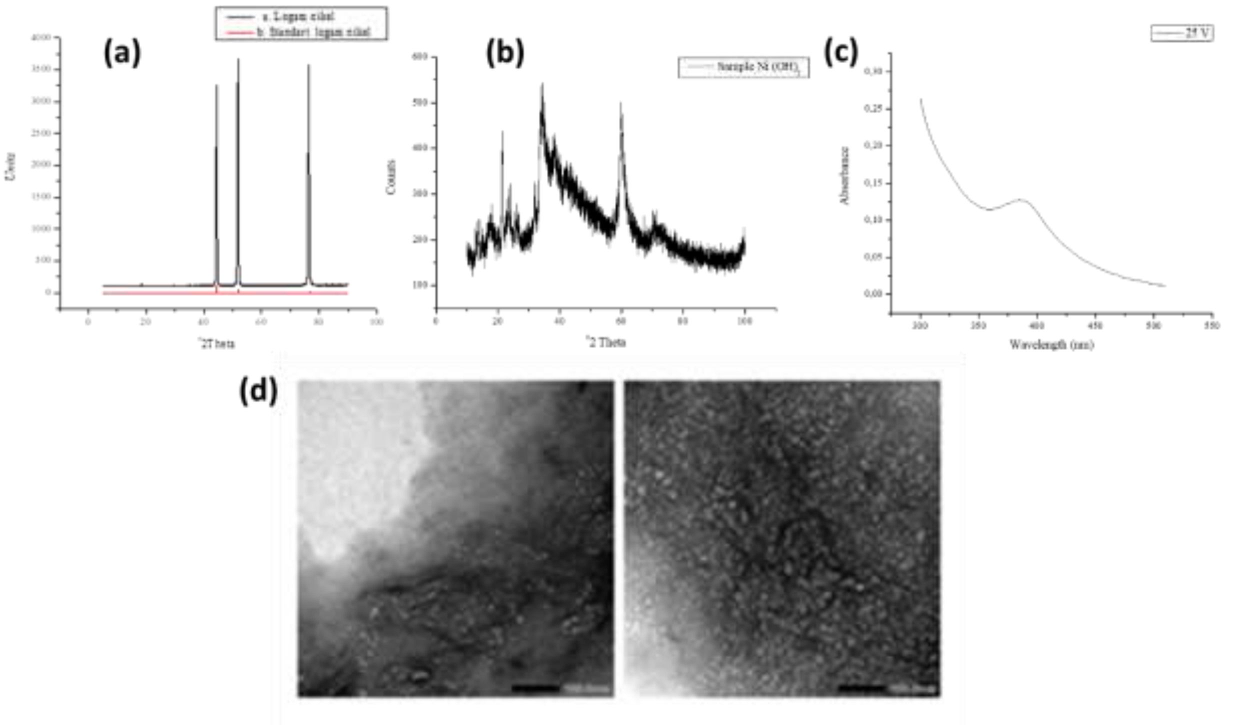

Figure 2. (a) XRD of Nickel Plate, (b) XRD of Nickel hydroxide nanoparticles, (c) TEM images of nickel hydroxide nanoparticles obtained from electrolysis using 0.3 M sodium citrate with a potential of 25 V for 30 minutes, (d) UV Vis spectrum $Ni(OH)_2$ nanoparticles obtain by electrolysis at 25 V

Analysis of Mercury using $Ni(OH)_2$ Nanoparticles Solution

In this sensor solution test, 2 variations of the volume of $Ni(OH)_2$ nanoparticles and 5 variations of the concentration of mercury (Hg) were used. The volume variations of $Ni(OH)_2$ nanoparticles used were 5mL; and 4 mL. While the concentration variation for the mercury (Hg) solution used is 0 ppm, 10 ppm, 20 ppm, 30 ppm, 40 ppm, and 50 ppm. A total of 5 mL of $Ni(OH)_2$ nanoparticles were piped using a micropipette and put into each of the 6 small bottles. Then the mercury solution was added with a volume according to the variation used and demineralized water was added until the total volume reached 5 mL. The absorbance of the mixture in the 6 small bottles was measured using a UV-Vis spectrophotometer. The absorbance value obtained will be observed how the effect of adding mercury solution on the absorbance value of $Ni(OH)_2$ nanoparticles. After all the solution has been mixed, shake the bottle slowly then let stand for 5 minutes. Furthermore, the absorbance of each solution was measured using a UV-Vis spectrophotometer. The treatment was repeated for $Ni(OH)_2$ nanoparticles with other volume variations.

The absorbance value data of the measurement results for a solution with a concentration of 0 ppm Hg can be seen in table 1 where the detection limit calculation uses three standard deviations (3σ). The linearity curve of the absorbance response to the concentration of mercury in mercury analysis using a solution of $Ni(OH)_2$ nanoparticles with a volume of 5 mL can be seen in Figure 3 and equation of the line $y = -0.00003x + 0.1615$.

Absorbance at detection Limit = a + 3 σ

$= 0.159 + 3. (0.00541603)$

$= 0.175248$

Where (a) is the intercept and (σ) is the standard deviation. The absorbance value of the detection limit is substituted as the y value in the equation of the line:

$$y = -0.00003x + 0.1615$$
$$0.175248 = -0.00003x + 0.1615$$
$$x = (-0.175248+0.1605):(-0.0003)$$
$$x = 45 \text{ ppm}$$

Table 1. Measurement of the Absorbance Value of Mercury Analysis using $Ni(OH)_2$ nanoparticles Solution at a volume of 5 mL

Concentration Hg (ppm)	Absorbance (A)		
	1	2	3
10	0.159	0.159	0.159
40	0.151	0.151	0.151
50	0.147	0.147	0.147

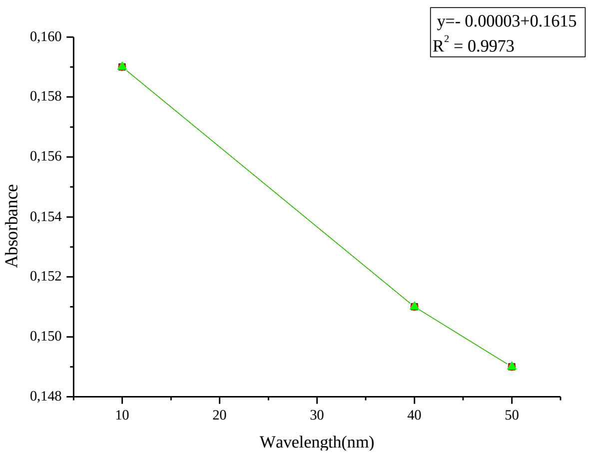

Figure 3. Linear curve analysis of mercury using a solution of $Ni(OH)_2$ nanoparticles at a volume of 5 mL

Limit of detection (LOD) For mercury concentration in mercury analysis using a solution of $Ni(OH)_2$ nanoparticles with a volume of 5 mL was 45 ppm. The absorbance value data of the measurement results for a solution with a concentration of 0 ppm Hg can be seen in table 2 where the detection limit calculation uses three standard deviations (3σ). The linearity curve of the absorbance response to the concentration of mercury in mercury analysis using a solution of $Ni(OH)_2$ nanoparticles with a volume of 4 mL can be seen in Figure 4 and equation of the line y = -0.0002x + 0.1465, Absorbance at detection Limit = a + 3 σ, from calculation Absorbance = 0.002869379, Where (a) is the intercept and (σ) is the standard deviation. The absorbance value of the detection limit is substituted as the y value in the equation of the line: y = -0.0002x + 0.1465, x= 35 ppm

Table 2. Measurement of the Absorbance Value of Mercury Analysis using $Ni(OH)_2$ nanoparticles Solution at a volume of 4 mL

Concentration Hg (ppm)	Absorbance (A)		
	1	2	3
10	0.145	0.145	0.145
40	0.14	0.14	0.14
50	0.139	0.139	0.139

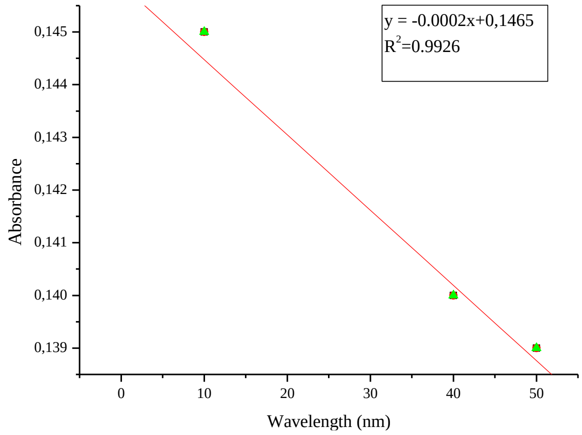

Figure 4. The absorbance curve of the mercury analysis used a solution of $Ni(OH)_2$ nanoparticles at a volume of 4 mL

From experiment obtained on the curve of the analysis of mercury using a solution of $Ni(OH)_2$ nanoparticles with 2 volume variations, there is no visible shift in wavelength as in the LSPR theory. it can be assumed that in this case there is a reaction between $Ni(OH)_2$ nanoparticles and the interference in the form of mercury that is given. The reactions that occur are:

$Ni(OH)_2(l) + 2e- \rightarrow Ni(s) + 2OH-$
$E^0 = + 0,72$

Nickel which was originally +2 will become uncharged nickel and produce OH- ions. The OH- ion generated indicates an alkaline atmosphere in the solution. This is in accordance with the measurement results, where the solution of $Ni(OH)_2$ particles has a pH value = 10. So that the reaction of mercury (Hg) in an alkaline atmosphere can be described as follows:

$Hg(l) + 2\ OH^- \rightarrow HgO(s) + H_2O + 2e^-$
$E^0 = -0,0977$

Because the size of the $Ni(OH)_2$ nanoparticles is very small, that is, on the nanoscale, mercuric oxide deposits cannot be observed. The reaction between $Ni(OH)_2$ nanoparticles and mercury solution is indicated by the color fading of the solution and a decrease in the absorbance value of $Ni(OH)_2$ nanoparticles.

CONCLUSION

Nickel hydroxide nanoparticles can be utilized to detect mercury simply, quickly and practical. *Limit of detection* (LOD) For mercury concentration in mercury analysis using a solution of nickel hydroxide nanoparticles with a volume of 5 mL was 42 ppm and for 4 ml the LOD value is 75.8 ppm.

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