Detection of Mercury Ions in Water using a Plastic Optical Fiber Sensor

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ABSTRACT

Research has been carried out on the detection of mercury ions in water using plastic optical fibers. Detection of mercury ions is done by immersing the optical fiber sensor in the HgCl₂ solution, where both ends of the sensor are connected to LED and phototransistor. LED as a light source will emit light along with the optical fiber which will be received by the phototransistor. The optical light received by the phototransistor is converted into an electric voltage and given again in the differential amplifier. The output voltage in the form of an analog signal is converted into a digital signal on the Arduino UNO to be read on a computer. The optical fiber as a sensor is made in two configurations, namely U configuration and spiral spring configuration. The jacket and the fiber optic cladding are peeled off and then covered with chitosan. Each configuration will be given a variation of the curve to analyze the characteristics of the sensor. The curvature can cause a large power loss resulting in attenuation of the light intensity of the LED received by the phototransistor. Apart from the effect of indentation on optical fibers, the output voltage measurement results are also influenced by the level of HgCl₂ concentration. The best measurement results for mercury ion sensors in water using plastic optical fibers are obtained in a spiral spring configuration with a chitosan cladding with a variation of 6 coils a sensitivity of 104.065 mV/ppm.

Introduction

Mercury is a toxic heavy metal that is found in several resources on earth and released through natural processes such as volcanism, weathering, and fire. Several studies have shown that mercury levels in the environment have tripled since industrialization. About half of the anthropogenic mercury released comes from burning coal for energy. In the air, elemental mercury has a long residence time and can disperse greatly from its source making this problem a global problem. The effect for the health of consuming water that containing mercury cause several disorders such as damaging the fetus in women, disrupting the baby's brain development and kidney function [1,2]. The detection of metallic mercury in waters is very important, because of its dangers.

Many sensors used to measure mercury levels in the environment are sensitive enough but expensive and complicated to use. The shortcomings of conventional sensors can be
overcome with optical sensors. This optical sensor has advantages including not using electrical signals, low noise, high sensitivity, and can be connected to a data communication system via an interface device [3]. Another advantage is that it has a high light transmission speed, resistant to corrosion, and relatively affordable [4]. Because of these advantages, optical fiber has been developed as a sensor system, such as temperature sensors [5,6], UV radiation sensors [7], pressure sensors [8,9], water turbidity sensors [10], etc.

Several studies about mercury ion sensors in water using optical fibers have been carried out by Raj et al (2016) about utilizing hybrid PVA gold nanoparticles using the Surface Plasmon Resonance (SPR) method [11]. The research was also carried out by Jia (2017) by utilizing gold-nanoparticles core-satellite nanostructure using the Plasmon Coupling Effect method [12]. Likewise, Jia (2018) by utilizing gold nanoparticles-DNA conjugates using the Localized Surface Plasmon Resonance (LSPR) method [13]. However, these studies are difficult to do, because of the use of expensive supporting tools and a complicated fabrication process.

In this research, a Plastic Optical Fiber (POF) sensor system will be developed to detect mercury ions in water by utilizing chitosan using the evanescent wave absorption method. The evanescent wave absorption method is a method for testing optical fiber sensors that take advantage of the effect of the cladding’s refractive index on the intensity of light transmitted in the optical fiber [13]. The sensor has been made with a variety of configurations. This variation aims to see the best configuration for detecting mercury ions with high sensitivity. The advantage of using plastic optical fiber as a sensor is an easy fabrication, low cost, high sensitivity, and simple measurement system.

**Experimental Method**

This research used a plastic optical fiber made of polymethyl methacrylate (PMMA) with a jacket coating diameter of 2.2 mm, a cladding of 1 mm, and a core of 0.98 mm. This research also uses several supporting devices, namely the power supply circuit, infrared Light Emitting Diode (LED) type IF-E91A, phototransistor type IF-D92, differential amplifier, Arduino UNO microcontroller, and computer. The sample used was a HgCl$_2$ solution. Mercury solution was prepared by weighing 1.5 mg of HgCl$_2$ powder. The weighed HgCl$_2$ powder was dissolved with 500 ml of distilled water, this solution has a concentration of 3 ppm as the stock solution. The concentration of the 3 ppm solution was diluted using the dilution equation to obtain the measured concentration of mercury solution. In this study, six variations of the measured concentration of mercury solution were used, namely 0.1 ppm; 0.3 ppm; 0.5 ppm; 0.7 ppm; and 0.9 ppm. Mercury solution was prepared with the same solvent volume, namely 150 ml for each concentration.

The main process of this research is the manufacture of a mercury ion sensor in water using a plastic optical fiber without jacket and cladding, then coated with chitosan gel. The chitosan as a coating of the sensor was carried out by a dip-coating technique for 30 minutes and dried at room temperature. Production of chitosan gel was done by mixing 2 grams of chitosan powder with 100 ml of 2% acetic acid, and then stirring using a magnetic stirrer until the solution became homogeneous and orange-yellow color. A schematic of a mercury ion sensor in water using optical fiber is shown in Figure 1.
Figure 1. The schematic of the mercury ions sensor in water using a plastic optical fiber

The power supply as a voltage source is connected to the LED. LED as a light source connected to one end of the optical fiber. The LED emits light along with the fiber optic sensor. Optical fiber is immersed in a HgCl2 solution with various concentrations. While at the other end of the optical fiber is connected to a phototransistor. The phototransistor is a detector that receives the light emitted by the LED on the optical fiber. The light intensity will change due to the difference in the refractive index. The intensity of light traveling along the optical fiber will be detected and received by the phototransistor and then converted into a voltage that the differential amplifier will amplify. The voltage in the form of an analog signal will be converted into a digital signal by the microcontroller then displayed on the computer.

In this study, POF without cladding was used with two different configurations, namely U and spiral spring as shown in Figure 2. Several testing was carried out on the sensor without cladding and testing of plastic fiber optic sensors coated with chitosan. Its aim to determine the optimal sensor to detected mercury in water using a POF sensor.

Figure 2. Mercury sensor design based on POF using a variety of (a) Spiral Spring Configuration; (b) U Configuration.

Result and Discussion

The experimental setup has been done and obtains the mercury sensor with a variety of configurations and coated with chitosan gel. The sensor for mercury ions in water uses optical fibers with two configurations, namely the U configuration and the spiral spring configuration. In the U configuration, the POF used is 15 cm then peeled off 3 cm in the middle of the jacket and then formed into a U configuration. In the spiral spring configuration, the optical fiber used is 18 cm then peeled 8 cm in the middle of the jacket and then formed into a spiral spring configuration. The U configuration has been made with variations in the indentation diameter, while the spiral spring configuration has been made
by varying the number of coils. Each configuration was treated without cladding and coating with chitosan as a cladding.

The U configuration has been made with variations in diameter, namely 0.5 cm, 1 cm, and 1.5 cm. Measurements were carried out for 30 seconds at each concentration of HgCl₂ solution. Each measurement results in a different output voltage response. Figure 2 shows the output voltage of each concentration in the U configuration.

**Figure 2.** Output voltage in the U configuration (a) without cladding (b) with chitosan cladding
Figure 2 shows a graph of the change in output voltage from the measurement results using a fiber optic sensor with a U configuration. The graph shows that the higher the concentration of mercury solution, the smaller the sensor output voltage. Changes in the concentration of HgCl₂ result in power losses that affect the measured light intensity transmitted from the optical fiber. The intensity of light is affected by changes in the refractive index of the cladding. The change in refractive index was caused by the presence of chitosan coating as a cladding on the optical fiber and changes in the concentration of the HgCl₂ solution. Besides, the presence of chitosan as a cladding causes the optical fiber to be able to absorb mercury metal ions in solution, impact the sensor more sensitive to mercury ions. So that the substances measured on the optical fiber are mercury ions present in the solution and other substances are not measured on the sensor. While the change in diameter in the U configuration will affect the angle of incidence and angle of reflection along with the optical fiber.

The results of the measurement data can be analyzed by characterizing each sensor variation consist the range, sensitivity, and resolution values. The range value for the sensor is determined using the following equation [14]:

\[ \Delta = V_{\text{max}} - V_{\text{min}} \]  

(1)

where \( V_{\text{max}} \) is the maximum output voltage and \( V_{\text{min}} \) is the minimum output voltage.

The sensitivity value for the sensor is determined using the following equation [10]:

\[ S = \frac{V_{\text{max}} - V_{\text{min}}}{K_{\text{max}} - K_{\text{min}}} \]  

(2)

where \( K_{\text{max}} \) is the maximum concentration and \( K_{\text{min}} \) is the minimum concentration.

The resolution is the smallest change in value measured on the sensor. The resolution value for the sensor is determined using the following equation [10]:

\[ R = \frac{N}{S} \]  

(3)

\( N \) is the smallest scale of the measuring instrument, which is 0.001 V and \( S \) is the sensitivity of the sensor’s sensitivity.

The results of the calculation of the range, sensitivity, and resolution of the U configuration fiber optic sensor with variations in indentation diameter are shown in Table 1.

Table 1. Characterization of mercury ions sensor in water based on U configuration plastic optical fiber with the variations of indentation diameter

| Indentation Diameter (cm) | U configuration without cladding | U configuration with chitosan cladding |
|--------------------------|----------------------------------|---------------------------------------|
|                          | \( \Delta \) (mV) | \( S \) (mV/ppm) | \( R \) (ppb) | \( \Delta \) (mV) | \( S \) (mV/ppm) | \( R \) (ppb) |
| 0.5                      | 40.241 | 50.301 | 19.880 | 59.791 | 74.738 | 13.379 |
| 1.0                      | 39.752 | 49.690 | 20.124 | 58.651 | 73.313 | 13.640 |
| 1.5                      | 39.263 | 49.078 | 20.375 | 57.836 | 72.295 | 13.832 |
Table 1 shows the effect of bending on the sensor output, where a change in this diameter will result in a change in the bending angle. If the indentation given is bigger, it will cause the sensor to be more sensitive. The greater the sensitivity of the sensor, the value of the range will increase and the resolution of the sensor will decrease. In Figure 2, the change in the output voltage obtained is very small. So that the sensitivity value and range value generated for each variation of the indentation diameter are also small.

Figure 3. Output voltage in the spiral spring configuration (a) without cladding (b) with chitosan cladding
The characterization of the test results for fiber optic sensors with U configuration with variations in the diameter of the indentation obtained the best sensitivity in the U configuration with chitosan with a diameter variation of 0.5 cm. Characterization results were obtained with a value range of 59.791 mV, sensitivity 74.738 mV/ppm, and resolution 13.379 ppb.

The mercury ions sensor in water based on optical fiber with a spiral spring configuration is made with several variations in the number of coils, that is 4 coils, 5 coils, and 6 coils. As in the U configuration, measurements are also carried out for 30 seconds at each concentration of the HgCl₂ solution. Each measurement results in a different output voltage response. Figure 3 shows the output voltage of each concentration in the spiral spring configuration.

Figure 3 shows a graph of the change in output voltage from the measurement results using a fiber optic sensor with a spiral spring configuration. The graph shows that the higher the concentration of mercury solution, the smaller the sensor output voltage. Changes in the concentration of HgCl₂ result in power losses that affect the measured light intensity transmitted from the optical fiber. The intensity of light is affected by changes in the refractive index of the cladding. The change in refractive index was caused by the presence of chitosan coating as a cladding on the optical fiber and changes in the concentration of the HgCl₂ solution. Besides, the presence of chitosan as a cladding causes the optical fiber to be able to absorb mercury metal ions in solution, impact the sensor more sensitive to mercury ions. So that the substances measured on the optical fiber are mercury ions present in the solution and other substances are not measured on the sensor. While the indentation in the spiral spring configuration will affect the angle of incidence and angle of reflection along with the optical fiber. A large number of indentations on the sensor will affect the amount of power loss resulting in reduced light intensity received by the phototransistor.

Changes in the output voltage with each concentration based on the spiral spring configuration variation obtain different values of range, sensitivity, and resolution. The results of testing the spiral spring configuration fiber with variations in the number of coils can be seen in Table 2.

Table 2. Characterization of mercury ions sensor in water based on spiral spring configuration plastic optical fiber with the variation in the number of coils

| Number of coils | Spiral spring configuration without cladding |  | Spiral spring configuration with chitosan cladding |  |
|-----------------|---------------------------------------------|---|---------------------------------------------|---|
|                 | Δ (mV) | S (mV/ppm) | R (ppb) | Δ (mV) | S (mV/ppm) | R (ppb) |
| 4               | 44.447 | 55.558     | 17.998  | 79.342 | 99.177     | 10.082  |
| 5               | 45.292 | 56.615     | 17.663  | 81.297 | 101.621    | 9.840   |
| 6               | 46.106 | 57.632     | 17.351  | 83.252 | 104.065    | 9.609   |

Table 2 shows the effect of bending on the sensor output, where the number of coils will result in a change in the bending angle. A large number of coils on the sensor will affect the amount of power loss resulting in reduced light intensity received by the phototransistor. Changes in power loss cause the output voltage of the sensor to be smaller. If the number of coils given is more it will cause the sensor to be more sensitive. The greater the sensitivity of the sensor, the range value will increase and the sensor resolution will decrease. In Figure 3,
the change in the output voltage obtained is very small. So that the sensitivity value and range value produced by each variation in the number of coils is also small. The characterization of the optical fiber sensor’s test results with spiral spring configuration, the variation in the number of coils, obtained the best sensitivity in the spiral spring configuration with chitosan with 6 coils. Characterization results were obtained with a range value of 83.252 mV, sensitivity 104.065 mV/ppm, and resolution 9.609 ppb.

The results of measurements in previous research conducted by Boruah and Biswas in 2018 used a U configuration with a sensitivity of 280 mV/ppm [15]. However, Boruah and Biswas made sensors to detect lead ions, while in this research, sensors were made to detect mercury ions.

**Conclusion**

Conclusively, a plastic optical fiber sensor is applied to detect mercury ions in water. It was observed that the proposed sensor probe has shown high sensitivity for detecting mercury ions. The result showed that the best results of the sensor is obtained in the spiral spring configuration with chitosan cladding at 6 coils where the range value obtained is 83.252 mV, sensitivity 104.065 mV/ppm, and resolution 9.609 ppb. The optical fiber sensor with chitosan cladding also affect to increases the sensitivities of the sensor because chitosan have an ability to absorb the mercury ion in the water. The advantages of this sensor probe are that it is easy fabrication, low cost, high sensitivity and simple measurement system.

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