Characterization of sensitivity and response time

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Abstract. The paper presents an investigation of sensitivity and response time of a chitosan coated-plastic optical fibre (POF) sensor to cadmium ion concentration. The sensor working principles is based on the change of light intensity transmitted by the chitosan coated POF due to the change of cadmium ion concentration. Three sensor probes were fabricated with various coating thickness which are 100.24μm (sensor A), 131.97 μm(sensor B) and 376.24μm (sensor C). The characterization was done by exposing the sensor to cadmium ion solution for various concentrations. The results showed that sample C provides the lowest sensitivity while sample B showed the highest sensitivity which are 15.04mA/ppm and 65.64mA/ppm, respectively. In terms of response time, it was showed that sample A has the highest average response time which is 20.5seconds.

1. Introduction

Development of sensor for heavy metal ions detection has been investigated extensively due to the harmful effect of heavy metal ions to human life. The most widely used method for heavy metal ions detection is usually based on spectroscopy method such as inductively coupled plasma - optical emission spectroscopy (ICP-OES), inductively coupled plasma - mass spectrometry (ICP-MS), atomic absorption spectrometer (AAS), graphite furnace atomic absorption spectrometer (GFAAS) and X-ray fluorescence spectrometry [1]. However, the methods are complicated, laborious and need expensive and bulky instrumentation, which not support on-line, real-time and remote monitoring. On the other hand, to save time and cost, real-time, on-line and remote measurement is important in water quality monitoring. Therefore, various efforts have been done to overcome the limitation. The most promising technology that have been intensively investigated is optical fibre sensor technology due to its advantages such as electromagnetic interference independence, compact size, electrically passive operation, low cost and suitable to be used for remote sensor, real time measurements and can be arranged in distributed and multiplexed system.

Optical sensors to detect heavy metal ions have been developed by various methods such as reflectance [2, 3], fluorescence [4] and absorption of evanescent wave [5-7]. The disadvantages of reflectance method are that the design is complex and not suitable to be used in multiplexed systems. Fluorescent method is done by coating the optical fibre with a material that has properties of fluorescent. This method is quite interesting because of the high sensitivity. However, fluorescent methods have drawbacks of complicated fabrication process and high costs. Compared to other methods mentioned above, the evanescent wave method has advantage of simple fabrication process.

Evanescent wave based-sensor for heavy metal ions detection has been fabricated by using *U*-bend silica optical fibre[8]. However, due to its small core diameter (~ 50μm), the silica fibre sensor is very

fragile. To overcome the drawback of silica fibre sensor, the silica fibre can be replaced by plastic optical fibre (POF) which has higher core diameter (~1mm). POF based-sensor has been proposed for various applications such as liquid level sensor [9], ammonia sensor [10] , biosensor [11], chemical sensor [12] and refractive index sensor [13]. For heavy metal ions detection, POF sensor was proposed to detect mercury by using dithizone [14]. The measurement was done by observing the absorption spectrum using spectrometer. In this work, more simple measurement method is proposed by adopting evanescent wave-based POF sensor. Characterization was done for sensitivity and response time for cadmium ion detection as cadmium is one of the main harmful heavy metal ions.

2. Methods

To use POF as cadmium sensor, POF should be modified so that it gives response to the change of cadmium concentration. Realization of POF for cadmium sensor using evanescent wave method is done by removing the cladding and then replacing it by other material which sensitive to cadmium ion. In this work, the cladding of POF with total diameter of 1 mm was replaced by chitosan since chitosan is a low cost material which can absorb heavy metal ion [15]. Prior to coating,part of the fibrejacket was stripped for 2 cm. Then, the cladding was removed by etching it using acetone for 90 seconds. After cleaned the unclad fibre with alcohol, the fibre was then coated with chitosan using *dip-coating* method. The chitosan solution was prepared by diluting 5g chitosan powder with 150ml of acetate acid at temperature of 90°C on a magnetic stirrer for 4 hours. The fibre was dipped into the chitosan solution for 10 seconds and then full out to let it dry in room temperature for 2 hours. Then, the coating process was repeated to get thicker coating layer. To get various coating thickness, coating process was repeated 5 times for sensor A, 10 times for sensor B and 15 times for sensor C which result in coating thickness of 100.24 µm, 131.97 µm and 376.24 µm, respectively. Scanning electron microscope (SEM) image shows that the chitosan was well-homogenously coated on the fibre as depicted in Figure 1.

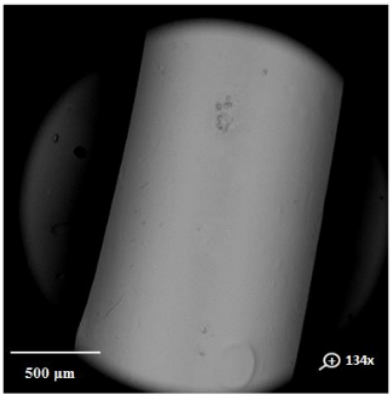
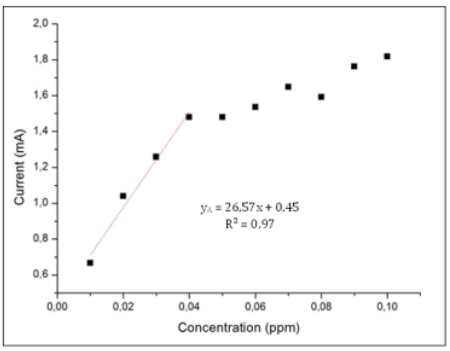


Figure 1. SEM image of chitosan coated POF

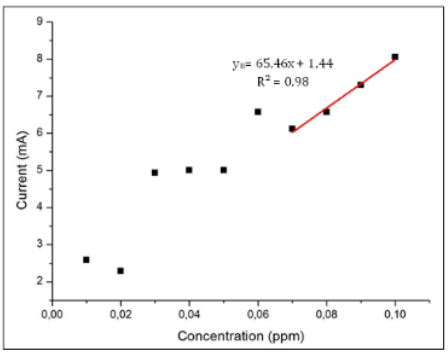
To characterize the sensor response to cadmium ion i.e. sensitivity and response time, ten cadmium ion solution with various concentration were prepared by dissolving $Cd(NO_3)_2$ with distilled water. The cadmium solution concentration was varied for 0.01 ppm to 0.1 ppm with increment of 0.01ppm. The fabricated sensors were then characterized by launching 650nm light from light emitting diode (LED) to input end of the POF while immersing them in the cadmium solution. To measure the output light intensity, the other end of the POF was connected to photodiode (PD) and ampere meter. The light intensity which is represented by the current was recorded every 5 seconds. After100 seconds, the cadmium solution was replaced by other solution with other concentration.

3. Result and Discussion

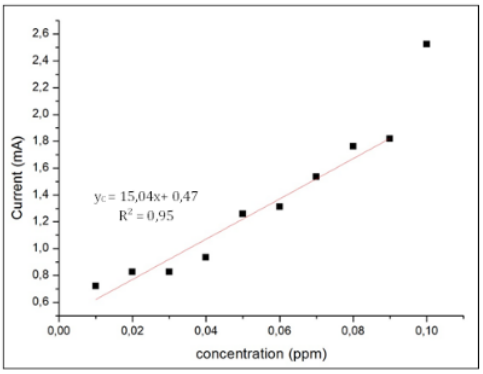
Figure 2 shows the current vs. cadmium concentration for sensor A, B and C. It is shown that the curve for sensor A has a linear region in the range of 0.01ppm-0.04 ppm with sensitivity of 26.57mA/ppm and correlation coefficient of 0.97. Meanwhile, sample B has a linear region in the range of 0.07ppm-0.1ppm with sensitivity and correlation coefficient of 65.46mA/ppm and 0.98, respectively. Much broader linear range is provided by sensor C, which is in the range of 0.01ppm-0.09ppm. However, despite its broad working range, sensor C has the lowest sensitivity and the lowest correlation coefficient which are 15.04 mA/ppm and 0.95, respectively. It is also shown that all sensors have positive sensitivity values which mean that as cadmium concentration increases, the current also increases. Since the resistance of PD decreases as more light intensity is received by the PD, then increasing current is indicating that the light intensity transmitted by the sensor increases. The increase of light intensity occurred since the refractive index of chitosan decreases as chitosan adsorb cadmium. When chitosan adsorb cadmium, cadmium ion is attached to the polymer chain forming chelate which has low density. Thus, it further results in decreased refractive index.



(a)



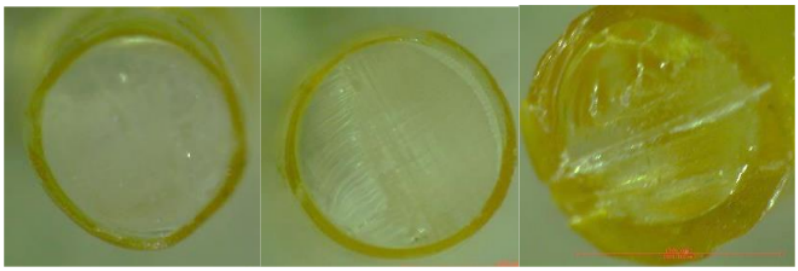
(b)



(c)

Figure 2. Current vs cadmium concentration for sensor A (a), sensor B (b) and sensor C (c)

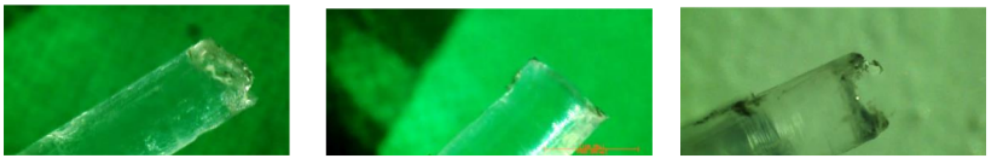
It is clearly shown from Figure 2 that sensor B provides the highest sensitivity. The main factor that affects sensitivity is chitosan layer properties such as thickness and its homogeneity. From the CCD microscope image, it is shown that chitosan layer of sensor B is the most homogenous layer compared to that of sensor A and C, as shown in Figure 3. Meanwhile, coating layer of sensor C is the most inhomogeneous, consequently its sensitivity is the lowest among others.



(a) (b) (d)

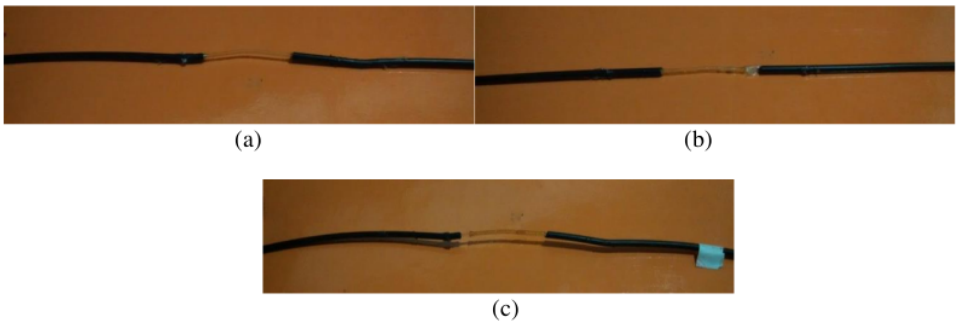
Figure 3. Cross-sectional image of chitosan coated POF of sensor A (a), sensor B (b) and sensor C (c) with magnification of 100 times

In terms of intensity, it is also shown that sensor B transmitted high light intensity as indicated by high current value. From figure 2 (b), it is shown that the current of sensor B is in the range of 2mA-8.5mA. These values are much higher than that of sensor A and C which is in the range of 0.6mA-2mA. Light intensity transmitted through the sensor highly depends on the incident angle of the light with normal of the surface of the input end. If the incident angle is higher than fibre numerical aperture (NA), then the light will not be transmitted through the fibre. As shown in Figure 4, sensor B has the smoothest surface, therefore, more light is transmitted inside the fibre. Moreover, light intensity also depends on loss due to bending. It is shown in Figure 5 that sensor A and C are suffered from bend loss since there are bending with high curvature radius around the chitosan coated part.



(a) (b) (c)

Figure 4. CCD microscope image of input end surface of sensor A (a), sensor B(b) and sensor C (c)



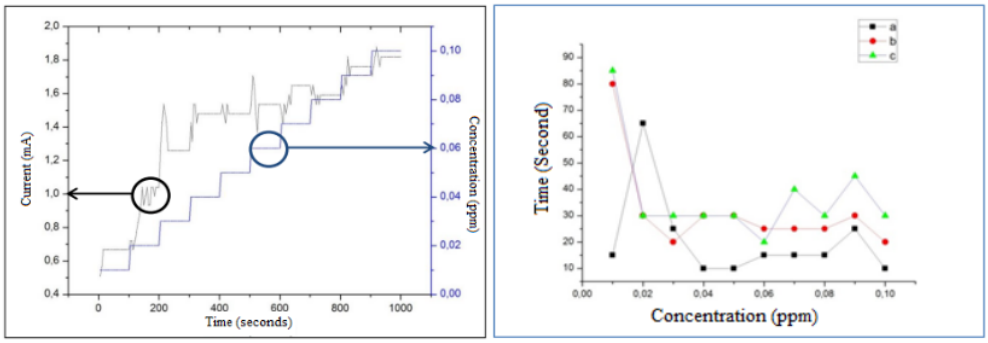
(a) (b)

(c)

Figure 5. Physical appearance of sensor A (a), sensor B (b) and sensor C (c)

Response times of the sensors are depicted in Figure 6. Response time is time taken for the sensor to reach 90% of its final value. The results show that the current fluctuated before it reached stable value (Figure 6(a)). The fluctuation occurred due to several factors such as fluctuation of the

power that generate LED and the unstable refractive index of the coating layer due to cadmium adsorption process. It is also shown that the thicker chitosan layer, the higher the response time as depicted in Figure 6(b). The results are reasonable since as chitosan layer is thicker, the more polymer chain is involved in capturing cadmium ion, therefore more times is required. The average response time for sensor A, sensor B and sensor C are 20.5 seconds, 32.5 seconds and 37.5 seconds, respectively. This value is much higher than the silica-based sensor which is above 5 minutes [8].



(a) (b)

Figure 6. The current vs. time for various concentration for sample A (a) and response time of sensor A, B and C (b)

4. Conclusion

Characterization of POF sensors for cadmium ion detection has been done. The results show that the sensor provides high sensitivity and fast response time. The highest sensitivity is 65.46mA/ppm for sensor B with coating thickness of 131.97 µm. Meanwhile, the fastest response time is 20.25 seconds which is obtained from sensor A with coating thickness of 100.24 µm. However, temperature cross sensitivity and selectivity to other heavy metal ions need further investigation.

Acknowledgment

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