

Optical Fibre Tip Sensor Coated With Chitosan For Lead Ion Detection

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Abstract. In this work, we demonstrate an optical fibre tip sensor coated with chitosan for lead ion (Pb^{2+}) detection. The sensor was fabricated using a tip of standard single mode optical fibre. The fibre tip region was cleaved and dipped into chitosan solution to form a sensitive layer. The coated sensor was exposed to different Pb^{2+} concentration ranging from 0 to 70 ppm. In order to compare the sensor's performance, the uncoated optical fibre tip sensor was firstly used to perform the measurement. The chitosan-coated tip sensor has greatly enhanced the sensor's sensitivity 7 times higher compared to uncoated the sensor with high linearity up to 97%. As the proposed sensor shows a promising potential in detecting Pb^{2+} , we believe the application of the sensor can be extended for environmental and wastewater monitoring.

INTRODUCTION

Lead is a type of heavy metals that can be considered as the most toxic element in the environment. Due to its important physio chemical properties, lead has been used widespread and it seems difficult to give up its usage. Lead has been used in industries such as battery manufacturing [1], lead based painting and boat building [2]. However, due to its non-biodegradable nature and continuous usage, its concentration accumulates in the environment which increases hazards. Human exposure to lead and its compounds can bring harm to human health. This highly poisonous metal can damage the functioning of the brain, lungs, kidney, liver, blood composition and other important organs [3].

To date, methods to trace lead ion (Pb^{2+}) have been established such as atomic absorption spectroscopy (AAS) [4], inductively coupled plasma-mass spectroscopy (ICP-MS) [5] and inductively coupled plasma-optical emission spectrometry (ICP-OES) [6]. Nevertheless, these techniques are very expensive, require trained personnel to work and require multi-sample preparation [7]. Therefore, the development of low cost, simple and reliable sensor is necessary. This study proposes an optical fibre sensor to determine the concentrations of Pb^{2+} at ppm level without the complexity of chemical and technical process.

Optical fibre sensor has attracted considerable attention in the new sensor systems as it offers numerous advantages due to its thin and small size, biological inertness in nature and the safeness due to absence of electric current at the sensing point. Nowadays, this sensor is being widely used as chemical sensor for contaminant ion detection such as nitrate (NO_3^-) [8], cadmium (Cd^{2+}) [9], copper (Cu^{2+}) [10], fluoride (F^-) [11], mercury (Hg^{2+}) and chromium (Cr^{3+}) [12]. Based on these references, we determined it is feasible to detect the Pb^{2+} by using the optical fiber tip sensor.

In this work, we employ the optical sensing technology using an optical fibre tip sensor coated with a natural biopolymer; chitosan to determine the concentrations of Pb^{2+} at 0 to 70 ppm level in water. The main interest is the application of fibre tip sensor as a chemical sensor where the optical power changes by changing the chemical composition of measured solution around the sensing region.

METHODOLOGY

Figure 1 shows the experimental setup and the illustration of the proposed sensing probe. As shown in Fig. 1(a), the experimental setup consists of amplified spontaneous emission (ASE) [Fiberlabs, FL7004] as a light source, an optical circulator to navigate the reflected light from the sensing region to the optical spectrum analyzer (OSA) [Yokogawa, AQ6370D, 0.02 nm resolution] to monitor the sensing signal. Details structure of the sensing probe is illustrated in Fig. 1(b). The sensor was fixed on the glass slide to avoid any physical disturbances during measurement. The experiment was conducted at constant room temperature.

The Pb^{2+} stock solution (1000 ppm) was prepared by dissolving 1.598 g of $Pb(NO_3)_2$ in 1L of de-ionized water. Further, the dilution was diluted with to obtain Pb^{2+} concentration from 0 to 70 ppm for use in our experiment.

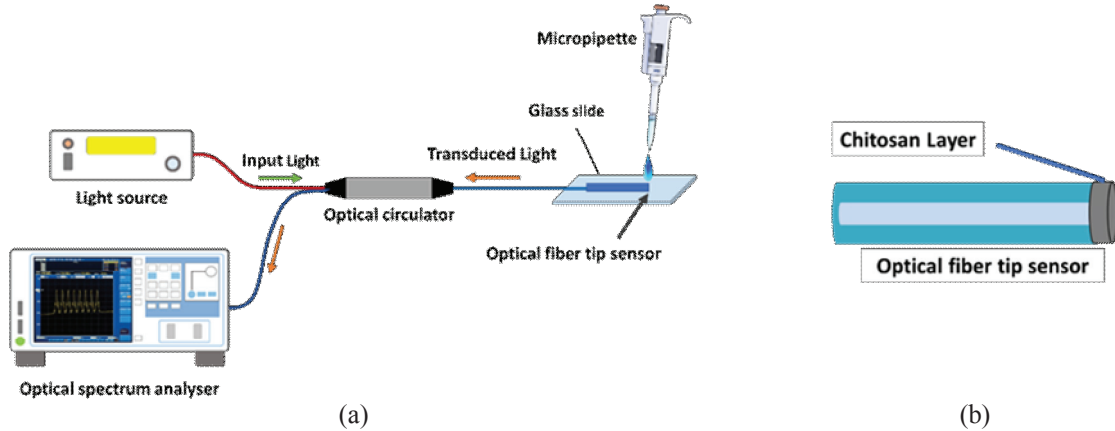


FIGURE 1. a) Experimental setup and b) an illustration of proposed sensor

In sensor fabrication process, the sensor was firstly prepared by cleaving the standard single mode fibre using high precision cleaver to produce a flat end surface. The well cleaved fibre was dipped into chitosan solution with 0.20 g/mL of concentration for 5 minutes and left for drying for 5 hours to ensure the chitosan layer is totally dried. As shown in Fig. 2, chitosan can act as excellent adsorbent for Pb^{2+} due to the presence of chelating ligand (i.e., amine group (NH_2) and hydroxyl group (OH)) in chitosan chemical structure [13,14]. This natural biopolymer exhibits strong binding affinity toward Pb^{2+} through the chelation effect. As a result, the refractive index (RI) of the chitosan will be modulated by the amount of Pb^{2+} present in the analyte. This RI will subsequently modulate the Fresnel reflectivity at the fibre tip region. When the fibre tip is coated with chitosan, Fresnel reflectivity (R) at fibre tip can be described in Eq. (1):

$$R = \left(\frac{n_1 - n_2}{n_1 + n_2} \right)^2$$

Here, n_1 and n_2 are the refractive index of the optical fibre core and chitosan respectively.

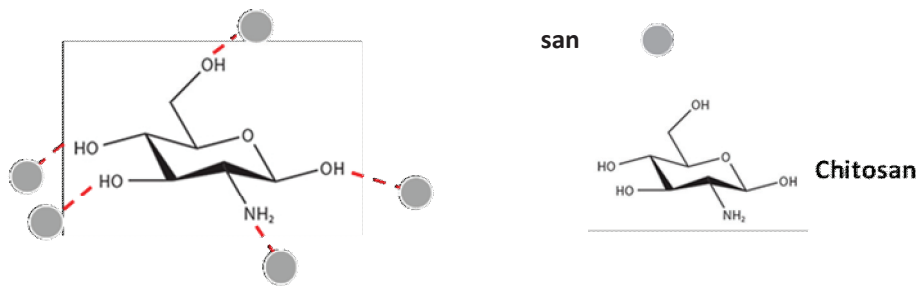


FIGURE 2. Chemical interaction between chitosan and Pb^{2+}

RESULTS AND DISCUSSION

Figure 3 shows the scanning electron (SEM) images of the proposed optical fiber tip sensor before and after chitosan coating process. Figure 3(a) depicts the SEM image of the flat uncoated sensing surface. The flat end surface is formed after cleaving the fiber by using a high precision cleaver. The chitosan layer deposited on the flat end surface can be seen from Fig. 3(b). This image confirms the successful fabrication of the chitosan-coated optical fiber tip sensor.

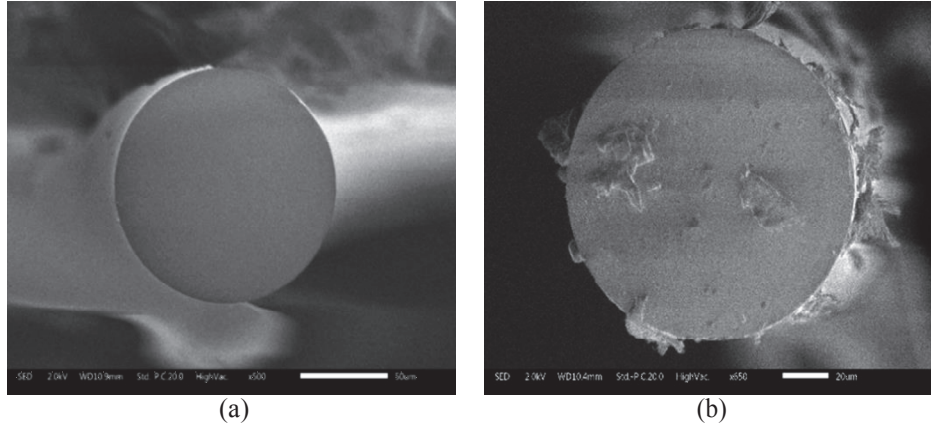


FIGURE 3. SEM images of sensor (a) before coating and (b) after coating

In order to evaluate the sensor's performance, we made a comparison between chitosan-coated sensor and uncoated sensor. Figure 4 shows the optical sensing spectrum of Pb^{2+} detection for both sensors. Figure 4(a) shows the sensing spectrum of the uncoated sensor when exposed to Pb^{2+} at different concentration ranging from 0 to 70 ppm. The response of the uncoated sensor does not distinguish the different Pb^{2+} concentration. The responses are expected to be uniquely due to the change in refractive index of Pb^{2+} concentration. As reported in Fig. 4(b), a significant reflected spectral change from the sensing region is shown by the fibre tip coated with chitosan layer. Since there are no wavelength changes, we only analysed the sensors based on the optical power changes.

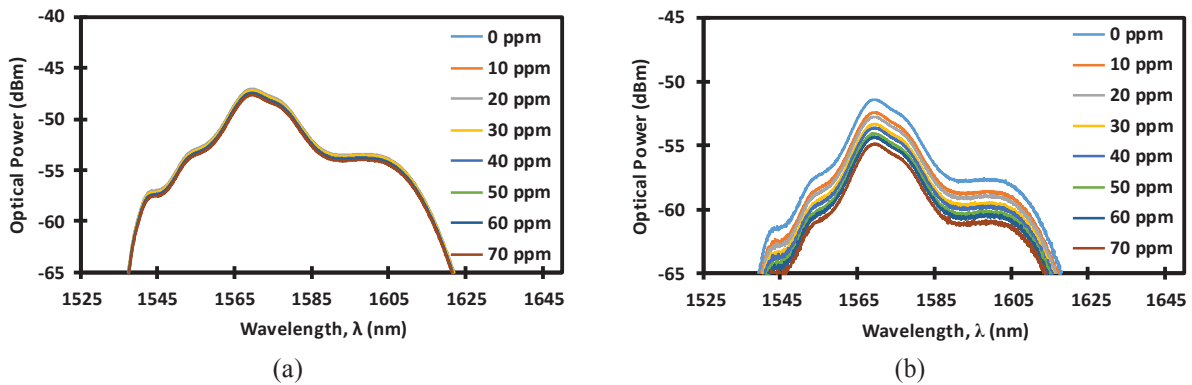


FIGURE 4. Sensing spectrum of (a) uncoated sensor and (b) chitosan-coated sensor. Both graphs are plotted in the same scale

Figure 5 depicts the optical power response of the developed optical fibre tip sensors. The total optical power loss for chitosan-coated sensor was 3.36 dBm, higher than uncoated sensor which only shows 0.30 dBm of power loss. The sensitivity of the sensor can be directly determined from the graph slope. The sensitivity achieved by the chitosan-coated optical fibre tip sensor is 0.0441 dBm/ppm, 7 times higher than uncoated sensor. The coated sensor shows a high linearity up to 97% indicates that the experimental data are fitted well with the straight line. The interaction between Pb^{2+} has greatly enhanced the sensor performance compared to uncoated sensor. The performance of the sensors is summarized in Table 1.

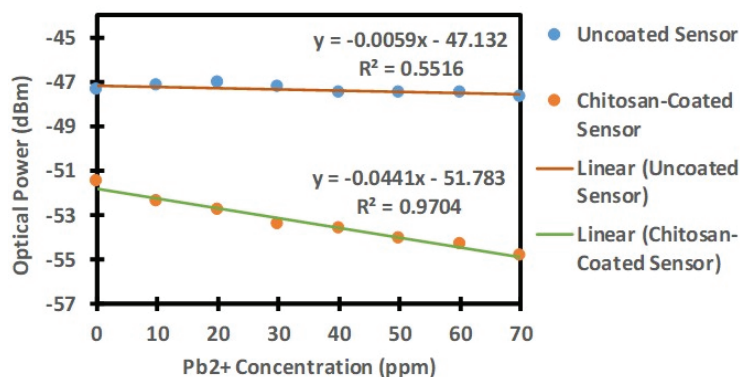


FIGURE 5. Analysis of optical power for chitosan-coated sensor and uncoated sensor

TABLE 1. Sensor performance

Characteristics	Chitosan-coated Sensor	Uncoated Sensor
Sensitivity (dBm/ppm)	0.0441	0.0059
Linearity (%)	97.4	55.16
Working range (ppm)		0 - 70

CONCLUSION

We successfully demonstrated a fabrication of optical fibre tip sensor using a standard single mode fibre coated with chitosan for Pb²⁺ detection. The presence of chitosan which has the ability to adsorb Pb²⁺ has greatly enhanced the sensor sensitivity 7 times higher than uncoated sensor. This sensor has potential to be applied in practical application for environmental monitoring and safety requirements in industries.

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REFERENCES

1. Basit, S., N. Karim, and A.B. Munshi, Occupational lead toxicity in battery workers. *Pak J Med Sci*, 2015. 31(4): p. 775-80.
2. Wani, A.L., A. Ara, and J.A. Usmani, Lead toxicity: a review. *Interdiscip Toxicol*, 2015. 8(2): p. 55-64.
3. Jaishankar, M., et al., Toxicity, mechanism and health effects of some heavy metals. *Interdiscip Toxicol*, 2014. 7(2): p. 60-72.
4. Kenawy, I.M.M., et al., Determination by AAS of Some Trace Heavy Metal Ions in Some Natural and Biological Samples after Their Preconcentration Using Newly Chemically Modified Chloromethylated Polystyrene-PAN Ion-Exchanger. *Analytical Sciences*, 2000. 16(5): p. 493-500.
5. Hinners, T.A., et al., Inductively coupled plasma mass spectrometric determination of lead isotopes. *Analytical Chemistry*, 1987. 59(22): p. 2658-2662.
6. Terrab, A., D. Hernanz, and F.J. Heredia, Inductively Coupled Plasma Optical Emission Spectrometric Determination of Minerals in Thyme Honeys and Their Contribution to Geographical Discrimination. *Journal of Agricultural and Food Chemistry*, 2004. 52(11): p. 3441-3445.
7. Kong, A.Q., et al., A novel route for the removal of Cu(II) and Ni(II) ions via homogeneous adsorption by chitosan solution. *Journal of Cleaner Production*, 2018. 192: p. 801-808.
8. Razali, N.M., et al. Etched Fiber Bragg Grating Sensor for Nitrate Sensing Application. in 2018 IEEE 7th International Conference on Photonics (ICP). 2018.

9. Yulianti, I., et al., Detection of Cadmium Ion by Evanescent Wave Based Chitosan Coated Optical Fibre Sensor. 3rd International Conference on Mathematics, Science and Education 2016, 2017. 824(1): p. 012002.
10. Tran, V.T., et al., Liquid Cladding Mediated Optical Fibre Sensors for Copper Ion Detection. [Micromachines \(Basel\)](#), 2018. 9(9): p. 471.
11. Jadhav, M.S., et al., Fluoride contamination sensor based on optical fibre grating technology. [Optical Fibre Technology](#), 2017. 38: p. 136-141.
12. Bhavsar, K., et al. Fibre optic sensor to detect heavy metal pollutants in water environments. in OCEANS 2017 - Aberdeen. 2017.
13. Bamgbose, J.T., et al., Adsorption kinetics of cadmium and lead by chitosan. *African Journal of Biotechnology*, 2010. 9(17): p. 2560-2565.
14. Findon, A., G. McKay, and H.S. Blair, Transport Studies for the Sorption of Copper Ions by Chitosan. *Journal of Environmental Science and Health Part a-Environmental Science and Engineering & Toxic and Hazardous Substance Control*, 1993. A28(1): p. 173-185.