

An optical fiber based surface plasmon resonance technique for sensing of lead ions: A toxic water pollutant

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ABSTRACT

Lead is a very toxic element whose presence in aqueous solutions in high concentration levels leads to several undesirable consequences. In this work, we report a detection mechanism of lead ion through an optical method. This SPR based technique measures output response of the sensor probe in terms of voltage of different analytes such as arsenic, copper, cadmium, nickel, mercury, iron and lead. The proposed sensor probe is coated with chitosan and glutathione. It is observed that this sensor probe has good voltage response for lead ions solutions in particular. This technique has limit of detection 1.3 ppb with a sensitivity of 0.28 mV/ppb. Use of optical fiber, LED and photodiode has made this technique low cost, easily hand able and environment friendly.

1. Introduction

Lead is a toxic non-biodegradable water pollutant, intake of which can cause cancer, kidney and brain disease. Application of lead compounds in pipe lines, battery etc. is the major sources of lead pollution in developing countries [1]. The permissible values of lead ions in water are 15 ppb and 10 ppb fixed by Environment Protection Agency (EPA) and World Health Organization (WHO) [2]. For detection of lead ions in water, several techniques such as atomic absorption spectrometry, inductively coupled mass plasma spectrometry, electrochemical methods are available. The problems with these methods are that they are costly, time consuming and require well established laboratory [3]. Optical fiber surface plasmon resonance (SPR) based sensors are low cost, label free, low weight and easily hand able that can be applied for real time analysis [4]. During the past decades, optical fiber SPR sensors are widely used in various fields of sensing like as bio-molecules sensing [5], gas sensing [6], refractive index sensing [7], temperature sensing [8] etc. There are various types of optical fiber SPR sensors depending upon the shape of the sensor probe. The most commonly used optical fiber sensor probes are tapered, D-shaped and U-shaped. Although these sensor probes have good sensitivity, from the experimental results it is known that U-shaped sensor probe has higher sensitivity than the others [9].

In our work, we have described experimentally an optical fiber based SPR sensor for detection of lead ions in aqueous medium. During the experiment, we have utilized U-shaped probe as sensor unit. From the literature review, it is found that, based on U-shaped sensor probe

till now, no work has been reported for the detection of lead ions. An environment friendly and biodegradable material namely chitosan is chosen as the sensitive material being accompanied by glutathione. This assembly has been coated on U-shaped sensor probe. Experimentally, we have shown that glutathione increases lead ion binding capability with chitosan, resulting lower limit of detection.

2. Experimental section

2.1. Materials and chemicals

A plastic optical fiber of core diameter 600 μm and numerical aperture (NA) ~ 0.37 is bought from Thorlabs (U.S.A). Chitosan of medium molecular weight and glutathione are purchased from Merck (U.S.A). Lead chloride, sodium arsenite, mercury chloride, nickel chloride, copper chloride and cadmium chloride were bought from Sigma Aldrich (U.S.A) and ferric chloride from Lebecem (India). Aqua regia solution is used to clean glass wears. For sample preparation, double distilled water is utilized.

2.2. Preparation of sensor probe

To fabricate sensor probe, a 15 cm long plastic optical fiber is taken and 3 cm middle portion of it, is decladed using a sharp surgical blade [10]. Afterwards, this unclad portion is heated using a wax candle and bent to a U-shape of radius ~ 1 cm as described in Fig. 1(a).

Due to its' insolubility in water, we prepare chitosan solution in

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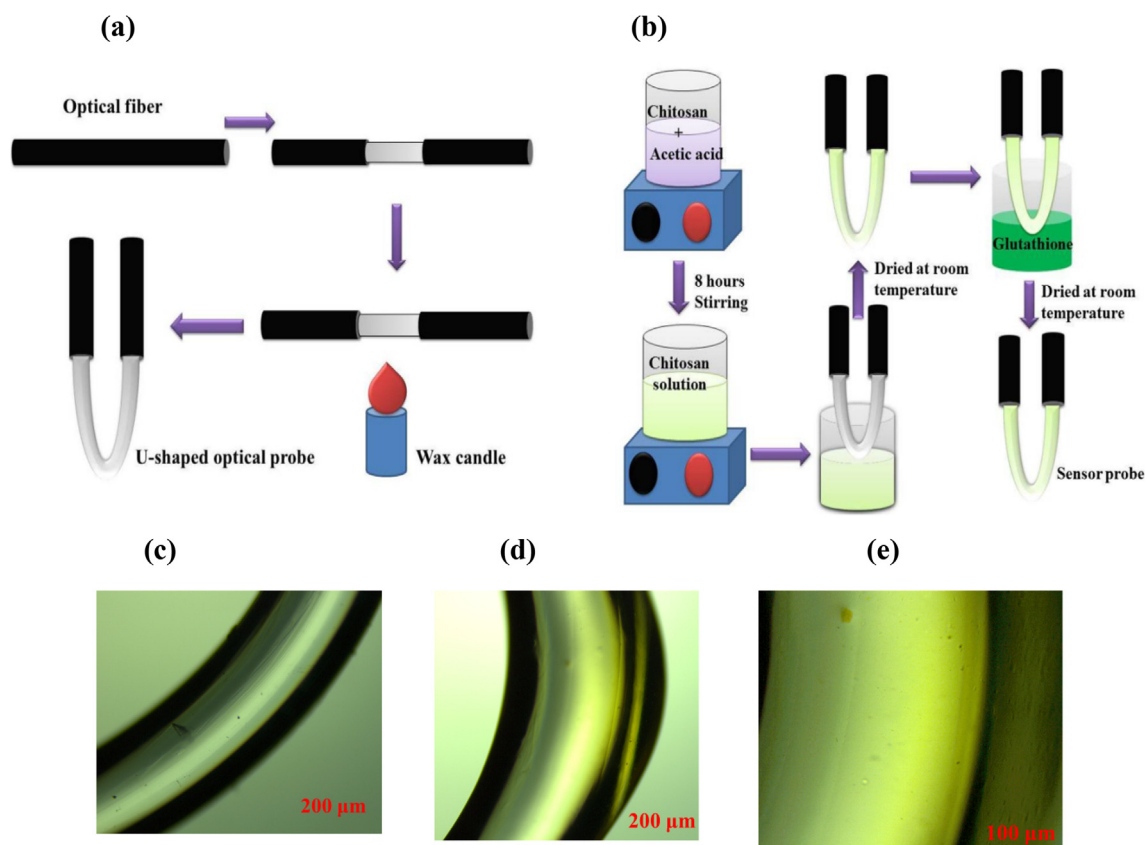


Fig. 1. Schematic of (a) sensor probe preparation, (b) synthesis of chitosan and coating technique of sensitive material to sensor probe (c) microscopic image of sensor probe without coating (d) image of sensor probe coated with chitosan-glutathione and (e) Zoomed image of coated sensor probe.

acetic acid solution. Accordingly, 10 ml of 2% acetic acid solution is prepared. To this solution, chitosan is added and stirred for 8 hour which yields light yellowish color solution. This solution is kept at room temperature. To impregnate the sensor probe with functional materials, at first U-shaped probe is initially dipped into chitosan solution for 5 minutes and it is dried at room temperature for 30 minutes, as illustrated in Fig. 1(b). Then, the sensor probe is dipped into 1 mM glutathione solution for 10 min, which is prepared in double distilled water. Again, the sensor probe is dried at room temperature for 30 minutes and kept at desiccator, for sensing application [9]. Fig. 1 (c), has shown the microscopic image of sensor probe without coating materials. From the Fig. 1(d) and 1 (e), it is clearly noticeable that the sensor probe is coated with chitosan, incorporated with glutathione.

3. Principle of sensing

Optical fiber propagates light by total internal reflection which is basically dependent on core and cladding refractive index. When light propagates through an optical fiber, a small portion of light is on the core – cladding interface and generates an electromagnetic wave, known as evanescent wave. This evanescent wave decays exponentially as distance from core to cladding increases [11]. Surface plasmon also known as surface plasma wave, is the collective oscillation of conduction electrons. This surface plasmon wave can be excited using light source such as light emitting diodes. When the wave vector of propagating light wave matches with the wave vector of conduction electrons oscillations, resonance occurs which leads to surface plasmon resonance (SPR) [12].

Surface plasmon resonance depends on refractive index of surrounding medium. If the refractive index of surrounding medium changes, SPR response also changes. This eventually yields modulated light. In Fig. 2(a), an optical set up is schematically depicted for lead

ions detection. This set up consists of white LED light as light source and a photo detector to measure optical output in terms of voltage. The white LED is bought from Thorlabs. The wavelength range of this LED is 430–660 nm, which is nearly visible wavelength region of the electromagnetic spectrum. There is a sample holder where we have put the metal ions solutions. When the sensor probe is dipped into the lead ions solutions, then lead ion interacts with glutathione mediated chitosan layer as described in Fig. 2(b). During this interaction, some lead ions are bonded by glutathione since glutathione highly interacts with lead ions. This interaction changes local refractive index of the sensor probe and thus emerges variations in output voltage [13,14]. This change in output voltage gives the concentration of lead ions in the sample solution.

4. Results and discussion

For the measurement of lead ions in aqueous medium, we have prepared 1, 3, 5, 7, 9 and 11 ppb lead ions solutions from lead chloride. In the sample holder, lead ions solutions are taken and then sensor probe is placed into sample holder. The output response in terms of voltage of sensor probe is shown in Fig. 3(a). From output curves, it is evident that output voltage increases with time, but after 5 minutes, it attains saturation. The saturated values of voltage for each ppb are shown in Fig. 3(b). From Fig. 3(a) and (b), it is also observed that, as the concentration of lead ions increases, output voltage response is also increased but after 7 ppb it has become saturated. This phenomenon can be explained in terms interaction mechanism of lead ions with the sensor probe. In our experiment, we have prepared our sensor probe coated with chitosan mediated with glutathione.

Chitosan is a biocompatible, non-toxic biopolymer that has several functional groups. These functional groups are amino ($-NH_2$) and hydroxyl ($-OH$) groups. Due to these groups, chitosan acts like a heavy

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