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STUDY OF EDC/NHS IMMOBILIZATION FOR PLUMBOUS DETECTION USING SURFACE PLASMON RESONANCE

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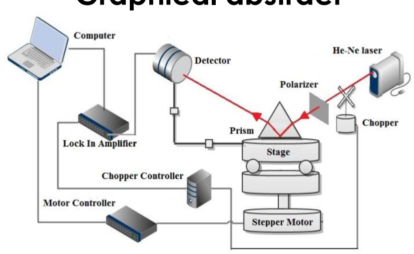
Graphical abstract



Abstract

The presence of plumbous (Pb^{2+}) in Irrigation water is harmful for the environment as well as human health. Herein, a simple yet effective sensor for Pb^{2+} detection is presented utilizing a surface plasmon resonance technique. The proposed sensor consists of a combination of 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrochloride (EDC) and N-hydroxysuccinimide (NHS) were attached to a gold layer offers a new option for heavy metal detection. The EDC/NHS works as a sensing layer that able to detect Pb^{2+} down to 15 ppm that matches with the United States Environment Protection Agency.

Keywords: Surface plasmon, heavy metal, plumbous detection

Abstrak

Pencemaran air disebabkan unsur plumbous (PB2) berbahaya terhadap alam sekitar dan kesihatan manusia. Justeru, pembinaan sensor yang mudah dan berkesan untuk mengesan PB2 dibentangkan dengan penggunaan teknik permukaan plasmon resonans. Sensor yang dicadangkan terdiri daripada gabungan 1-etil-3 (3-dinethylaminopropyl) karbodilimide hidroklorida (EDC) dan N-hidrooksisuccinimide (NHS) yang dilampirkan dengan lapisan emas. Pembinaan sistem ini menawarkan satu pilihan baru untuk mengesan logam berat. EDC atau NHS berfungsi sebagai lapisan yang dapat mengesan PB2 sampai tahap 15 ppm, sepadan dengan Agensi Perlindungan Alam Sekitar Amerika Syarikat.

Kata kunci: Permukaan plasmon, logam berat, plumbous mengesan

1.0 INTRODUCTION

The worldwide emission of heavy metals can be divided into industrial source, such as irrigation water, industrial activities and wastewater [1], and also natural source for instance volcano particles, windblown dust, forest wild fires and vegetation [2]. These toxic materials can cause serious damage to immunity system of human body as well as fatal cancer types [3]. Therefore, detection of heavy metals in water has become a vital issue globally [4], [5].

Different types of sensor have been used to detect the heavy metals in water and soil [6]. One of popular techniques is surface plasmon resonance (SPR) which has a minimum detection limit up to part per billion (ppb) [7]. SPR is the most economical test comparable with other methods that require special laboratory facility and qualified staff [8].

In angular modulation SPR, a monochromatic light single wavelength excites a surface plasmon wave. Strength of coupling in SPR sensor is intensity modulation. In intensity modulation, both angle of incidence and the wavelength of incidence are stable and the intensity of light wave works as a sensor output and that relies on the sensing materials and tested solution[9], [10].

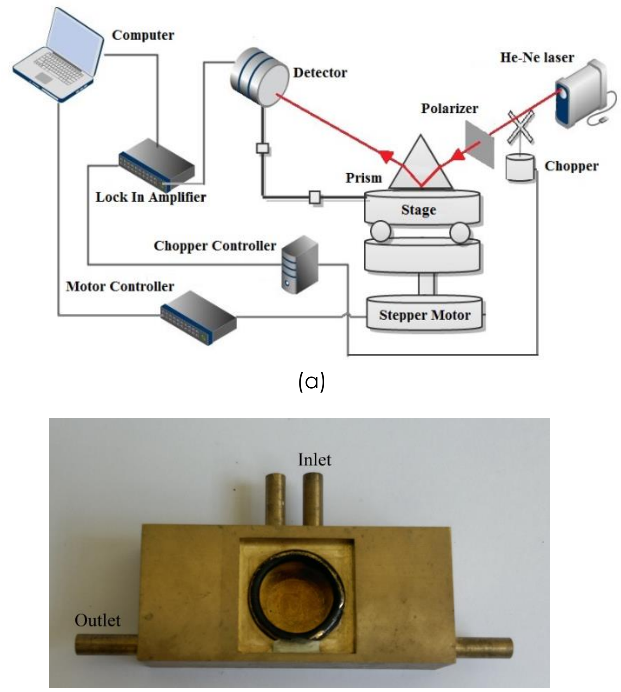
The coupling strength between incident light and surface plasmon wave is important for maximum depth of resonance dip. This condition depends on the sensing materials and wavelength, thus the optimum metal thickness can be reached [11]. The stability and sensitivity of SPR technique is also has been enhanced by the advancement of new sensing properties using nanocomposite materials [14], [15], [16].

In this study, we investigate the performance of sensing layer on the fluid circulation and the sensitivity of SPR sensor based on 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrochloride (EDC) and N-hydroxysuccinimide (NHS) sensing layer in order to identify heavy metal ions limiting factors and understand the interaction between tested ion water and sensing layer.

2.0 EXPERIMENT

In Turbadar-Kretschmann-Raether sensor application, the Au layer is directly coated on a prism. In our experiment, a Deckglaser Microscopic cover glass slide was used as the base for coating Au layer and was attached to a prism which has refractive index 1.77861. A glass slide of 22x22 mm with 99.99% transparency was utilized after cleaning with hot water and methanol for 20 minutes. It has thickness in the range between 0.13 to 0.17 mm. Matching index fluid of 100cps viscosity was injected between prism and glass slide. The Au layer was coated using a sputter coater, Turbo K575X. The deposition was performed at 20 mA for 67 second to yield the desired 50 nm of Au thickness.

The gold coated glass slide was then immersed in EDC/NHS coupling agent liquid for 15, 30, 45, 60 minutes at fixed temperature ~8°C. After immersing in coupling agent liquid, the Au-coated surface was washed at room temperature in distilled (DI) water for 1 minute. Then, it was dried for 10-15 minutes before using in SPR test. The activing agent (EDC/NHS) was prepared by mixing 50 mole of EDC and 20 mole of NHS. In order to prevent liquid-liquid separation, the EDC/NHS coupling was kept in a refrigerator.



(a)

(b)

Figure 1 (a) Schematic diagram of prism-based SPR and (b) flow cell used in the SPR setup

For the SPR setup as shown in Figure 1(a), the Kretschmann configuration was used. A Helium-Neon (He-Ne) laser light *(*633 nm) with output power of 2 *mW*, was employed as the excitation source. The rotational prism base was configured to rotate with resolution of 0.184 degree angle with a stepper motor (Sigma Koki, SG SP-60YAW-0b). The Pb ions solution was pumped into the flow cell as shown in Figure 1(b). All measurement data was registered in a computer program through a lock-in-amplifier (Stanford Research, SR530).

3.0 RESULTS AND DISCUSSION

In our initial experiment, the detection performance of 15 ppm Pb^{2+} ion was investigated with different immersion times of EDC/NHS layer as depicted in Figure 2. In this case, the thickness of EDC/NHS is directly proportional to the immersion time. The experimental findings show that the resonance angle was not changed for the first 15 minutes immersion of gold layer in EDC/NHS. The angle shift is 0.184° was observed for the 30 minutes immersion time. There was no significant change of angle shift even though the immersion time was increased from 30 to 60 minutes. Therefore, the coating time of 30 minutes was chosen as the optimized one for our next experiment.

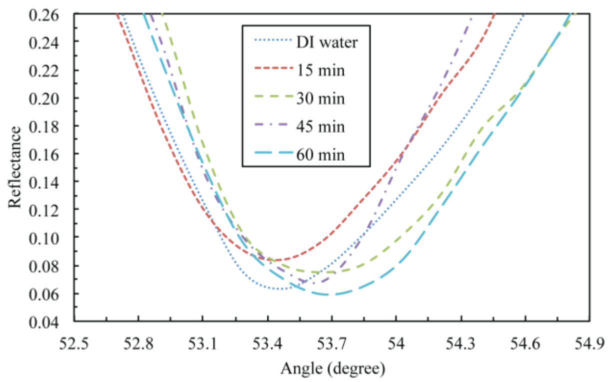


Figure 2 SPR curve at different immersion Au layer

In order to prove the sensitivity of sensor towards Pb^{2+} ion, a comparative analysis was made between this proposed sensor and the conventional SPR sensor (Au layer alone, 50 nm thickness). The same Pb^{2+} concentration of 15 ppm was used and the experimental results are shown in Figure 3. It is obvious that the resonance angle of SPR signal using Au layer alone was not changed in the presence of Pb^{2+} ion [Figure 3(a)]. On the other hand, referring to Figure 3(b), the resonance angle shift of 0.184° was obtained for the proposed sensor with Au-EDC/NHS layer.

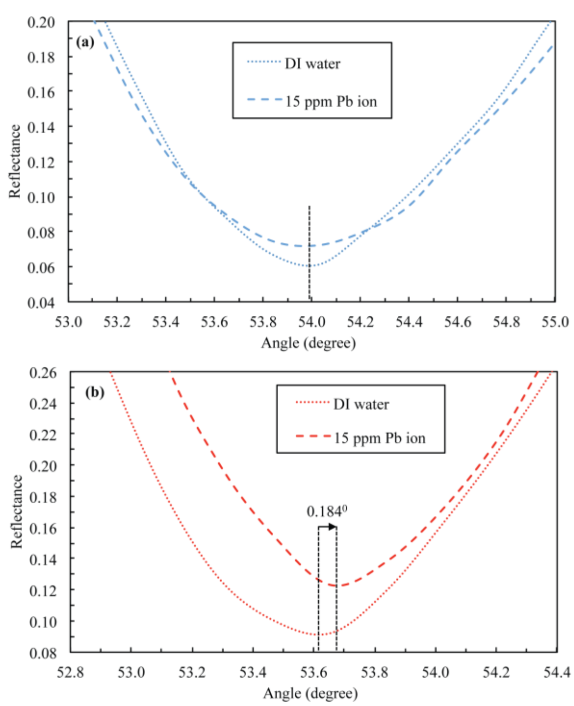


Figure 3 SPR response of DI water and 15 ppm Pb^{2+} ion with (a) Au sensing layer and (b) Au-EDC/NHS sensing layer

Finally, the proposed sensor was tested with different standard solutions that represent various refractive indexes. The findings from this experimental work are illustrated in Figure 4. By increasing the refractive index from 1.30 to 1.39 the SPR curves shifted to the larger resonance angle from 52.182° to 56.817°. The refractive indices can lead to change in strength of coupling (degree of overlapping between two fields). By increasing the strength of coupling between surface plasmon wave and incident light, the resonance depth becomes deeper [11], [12].

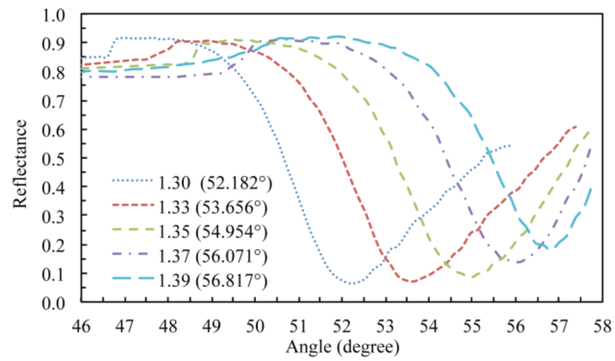


Figure 4 SPR curves at different refractive indices with Au-EDC/NHS sensing layer

4.0 CONCLUSION

A simple approach of detecting Pb^{2+} ion in water using prism-based SPR technique was demonstrated. The traditional SPR sensor based on Au layer was unable to detect the presence of Pb^{2+} ion in water. The sensitivity of SPR sensor was then improved by coating EDC/NHS layer on top on the Au layer. The thickness of EDC/NHS layer was directly proportional to the immersion time and 30 minutes was found to be the optimum immersion time. The minimum detection of Pb^{2+} was 15 ppm which is complies with the United States Environment Protection Agency. This technique has great potential of detecting Pb^{2+} in water irrigation systems.

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