

Article

Optical Response Analysis at Various Pb^{2+} Concentration by Using Fiber Optic Displacement Sensor

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Abstract—A simple and low-cost Fiber Optic Displacement Sensor (FODS) using reflective intensity modulation technique was developed to analyze various concentrations of Pb^{2+} , a compound classified under heavy metal ions. Lead is harmful to the environment including to human but is used in the cosmetic field for beauty without realizing and considering the hazardousness of lead as it would cause a long-term effect. Therefore, a feasible way has been identified in this study to demonstrate the level of Pb^{2+} concentration in cosmetics field by employing the theory of modulation of intensity as a function of displacement sensor. The permissible limit according to Malaysian Cosmetics Guidelines and ASEAN Cosmetic Directive was 20 ppm. The concentration sensor's system exhibits 0.0018 V/ppm sensitivity with a linearity of 96% and 94% respectively, for both peaks. Meanwhile, the sensitivity was 0.034 V/ppm for the first peak and 27.72 V/ppm for the second peak, with slope linearity of more than 96% for surface tension parameter. The credibility of these optical response curves data might be useful, especially in the cosmetic's industrial application.

Keywords— optical sensor; fiber optic displacement sensor; lead.

I. INTRODUCTION

Optical fiber is created in a cylindrical form and with a signal arrays at 6 Mb/s near at 10 km far away. It can also be called as a dielectric waveguide and in the form of light, it produces electromagnetic (EM) energy. George et. al stated that fiber optic sensor is widely used in chemical sensing due to its simplicity, inexpensive, and easy to use [1]. Detection of the concentration and refractive index of a liquid solution has been produced widely using various types of optical fiber sensor.

According to Krishnan et. al, FODS has better characteristics, which possess a high sensitivity, high response, low cost, able to work in a hostile environment, immune to electromagnetic interference, and non-contact interaction [2]. FODS can be categorized into two types, namely interferometry-based and intensity-based sensor. Two optical waves are merged to produce interference fringes for the interferometry-based FODS. On the other hand, the intensity-based FODS is easier to construct since it uses less costly component and achieves very high bandwidth. By using bundle fiber, the amount of light collected is directly correlated with the displacement between the fiber and the reflective surface. Generally, for the sensing principle, a pair of fiber is bundled together to construct FODS with one of the optical fibers will be the receiving fiber while another fiber will be the transmitting fiber.

Lead nitrate is a very stable inorganic compound that is mainly used in the industrial sectors in the production of pigments for lead paints due to its less toxicity of titanium dioxide. It contains higher lead, high solubility, and inexpensive compared to lead acetate and lead chlorate that

possesses low solubility in water. Known as a toxic chemical, heavy metals cause large impact to a genetic system, cell energy balance, and protein metabolism. There are some issues that arise in middle East Pakistan and parts of Africa on the use of Kohl (Surma) as a traditional eye cosmetic that contains a high level of leads [3]. It blows the nervous system by incriminating towards etiology or pathology by folding the disorders protein. According to Malaysian Cosmetics Guideline and ASEAN Cosmetic Directive, the permissible limit of lead in the cosmetics is around 20 ppm. In Malaysia, all products are controlled under the Control of Drugs and Cosmetics Regulation 1984 [4].

There are several classical techniques that have been extensively applied to detect Pb^{2+} such as atomic emission spectrometry (AES), atomic absorption spectrometry (AAS), inductively coupled plasma mass spectrometry (ICP-MS), X-ray fluorescence spectrometry (R-FS), and ion chromatography [5]. All these methods are either not sensitive enough for biochemical analysis or require special laboratory setup due to the use of expensive gases, sophisticated and high-end equipment [6].

Therefore, this study proposes a light reflectance-based displacement approach with various Pb^{2+} concentration level to isolate cosmetic product with an issue by implementing the Malaysian Cosmetic permissible guideline. The response curve from the changes of intensity modulation will be exploited. Hence, this work might be a significant milestone in cosmetics applications to determine the hazard level of Pb^{2+} .

II. SENSOR MECHANISM

Fig. 1(a) shows the detected solution is a curvature with pore meniscus at center point due to the minimization of surface energy subsequently leading to a curved shape in the square cuvette. In the vicinity of the cuvette corners, the curvature is relatively high as to interfere with the wetting action leading to the changes in the contact angle, causing the liquid to attach to the cuvette wall. Meanwhile, Fig. 1(b) shows the geometrical structure of a sensor using a bundled fiber as a sensor probe and liquid surface as a virtual reflector. The longitudinal axis of the sensor probe coincides with the normal axis of the pore meniscus.

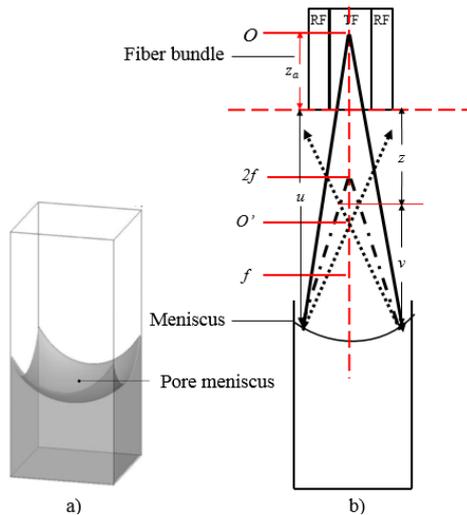


Fig. 1: (a) pore meniscus in a square cuvette (b) geometry of intensity modulation mechanism using a bundled fiber probe on liquid surface

The assumption is made for the curvature to resemble like a concave mirror having a virtual focal point and a center, located at two times of virtual focal point. When the light from the original point O and at a distance of z_a of TR is shone directly on the meniscus, the light concentrated at point O' after being reflected by the meniscus and becomes another emitting point source virtually, and is collected by the RF [7–8]. The amount of collected light varies as the sensor probe tip is brought away from the meniscus surface.

The sensor probe tip should be positioned as close as possible to the meniscus surface to optimize the light-coupling quality. It should be noted that the curve reflection phenomenon is used to clarify the working principle of the displacement sensor. For the analysis, the concentration-surface tension variation depends on the intensity of the output voltages demonstrated by the shape of the response curve. It is based on the probe shifting from the surface passing through its virtual focal point. The shift of the sensor probe away from the fixed reference point will produce two peaks of voltages. The two peaks of voltages are the maximum intensity occur due to the reflected light from the meniscus corresponding to the minimum surface energy on the surface.

The intensity collected is a result of increasing the size of the acceptance angle as the probe moves away from the meniscus. The larger the acceptance angle, the bigger the cone of the light hence more lights can be captured by the RF.

III. METHODOLOGY

In this study, FODS system is employed to study the effect of different concentration level and surface tension in Pb^{2+} . Light intensity modulation technique can be effectively employed as a diagnostic tool by providing a calibration curve of the concentration-surface tension parameter. The light-path of the optical fiber-based displacement sensor runs directly with almost no air obstruction through the surface of the sample liquid. The optical characteristics earn by the sensor system are, therefore, contingent upon the various types of the sample surface (Pb^{2+}), which may be flat or curved, depending on the meniscus present at the air-liquid interface.

During Pb^{2+} sample preparation, approximately 0.8 g of lead nitrate is placed into a 1000 ml volumetric flask. The compound is diluted by adding distilled water. Then, a stock solution of the lead was prepared according to the formula of $m g = (\text{mass molecule} \times \text{ppm} \times \text{volume}) / \text{mass atom}$ to get the lead solution. In order to prepare a working solution of lead, the dilution process plays a role. The concentrations of the lead were varied using the dilution formula below:

$$M_1 V_1 = M_2 V_2$$

where M_1 is a concentration in molarity of the concentrated solution, M_2 is a concentration in molarity of the diluted solution ($Pb(NO_3)_2$), V_1 is a volume of the concentrated stock solution, and V_2 is a volume of the diluted working solution. Every lead concentration have a similar volume to maintain the respective concentration during the measurement. The flow preparations of the diluted working standard solution are listed below:

a) For 100 ppm:

Pipette accurately 20 ml of 500 ppm stock standard solution into a 100 ml volumetric flask and make up the volume with distilled water until it achieves the calibration mark.

b) For 200 ppm:

Pipette accurately 40 ml of 500 ppm stock standard solution into a 100 ml volumetric flask and make up the volume with distilled water until it achieves the calibration mark.

c) For 300 ppm:

Pipette accurately 60 ml of 500 ppm stock standard solution into a 100 ml volumetric flask and make up the volume with distilled water until it achieves the calibration mark.

d) For 400 ppm:

Pipette accurately 80 ml of 500 ppm stock standard solution into a 100 ml volumetric flask and make up the volume with distilled water until it achieves the calibration mark.

e) For 500 ppm:

Pipette accurately 100 ml of 500 ppm stock standard solution into a 100 ml volumetric flask.

The flow of Pb^{2+} sample preparation is shown in Figure 2 below.

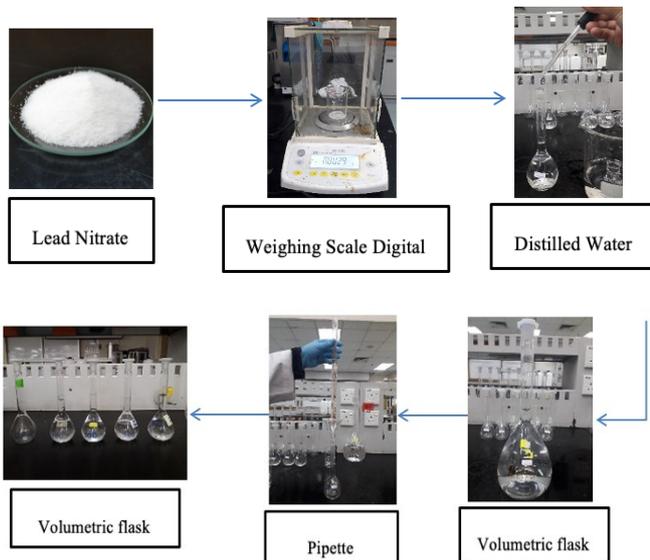


Fig. 2: Sample preparation flow

The experiment set up is shown in Figure 3. It consists of an optical fiber bundle probe, light source, photodiode, an automotive displacement controller, and an oscilloscope.

A 650 nm red laser diode and a concentric type fiber bundled probe with a transmitting fiber (TF) surrounded by 16 receiving fiber (RF) are employed. The operation starts when the light from diode laser is attached into a TF and is emitted at the end of fiber bundle to illuminate the liquid meniscus. The Pb^{2+} liquid meniscus acts as a virtual concave mirror with an interesting behavior of light reflection properties. Various solution in the range of 0-500 ppm has been prepared by a dissolution method. The reflected light from the liquid meniscus is then collected by 16 RF that maximize a light collection due to its concentric arrangement surrounding the TF.

The signal collected from RF will be captured and displayed in a waveform by the oscilloscope. The changes of intensity modulation-based displacement are controlled using an automation controller of Cytron Technologies software.

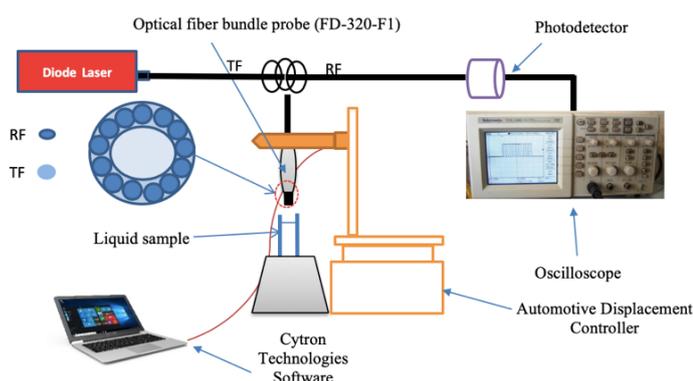


Fig. 3: Experimental setup of reflectance-based displacement sensor [9]

IV. RESULTS AND DISCUSSION

The discussion will be focused on the overall performance displayed by the optical response towards various concentration of Pb^{2+} .

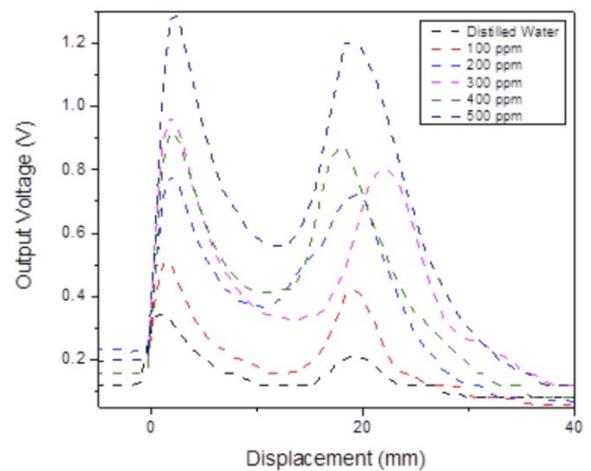


Fig. 4: Sensor pattern as a displacement feature

Figure 4 shows a graph of output voltage versus displacement at different concentration of Pb^{2+} from 0 ppm to 500 ppm. Based on the graph, as the concentration increases, the light cone angle of the transmitting fiber and the distance of the refracted light from the normal line increases [10]. The increment in the displacement affects the intensity modulation in the receiving fiber hence causes larger size and volume of intersection. At zero displacement, the light cone is unable to transmit into receiving fiber hence the output voltage is zero.

The response curve represents the variation of voltages with probe distances and its dip like-shaped is the indicator of the meniscus characteristics. A similar pattern can be observed at which all concentrations represent two maximum output voltages located at two different positions. It is clearly shown that liquid meniscus varies according to the displacement response curve. At small displacement, only a minimal voltage reading was observed due to only a small emitted light cone entering the RF due to the probe position and meniscus is nearly closed to each other. At a larger displacement, as the probe starts to move away from the meniscus, the rapid increases in the intensity of the voltage reading cause it to reach the first maximum point as the overlapping region among the emitted light cone and the core of RF become larger. After reaching the first maximum peak, the intensity reduces following the inverse square law relationship. At one point, called local minima (the virtual point source position moves far away from the probe), the output voltage increases making the second concentrated point that is the second peak voltage.

The peak voltage at the optimized displacement sensor is a marker of the maximum reflectivity. Therefore, the peak voltage will be the indicator for this calibration curve as depicted in Figure 4.

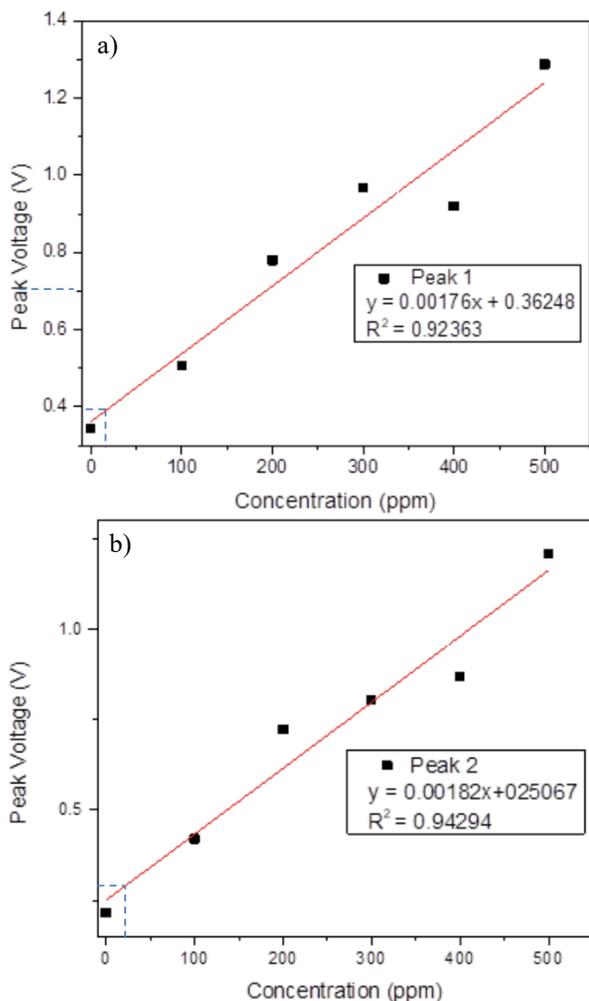


Fig. 5: Sensor calibration curve for concentration a) first peak b) second peak
 : The area of permissible limit of Pb²⁺ in cosmetics product [4]

Figure 5a shows sensor calibration line for the first peak while Figure 5b shows the second peak voltage. Both graphs demonstrate that the peak voltage increases proportionally with the concentration. The gradient of the graph is used to resolve the sensitivity of the sensor fingerprint. Based on the figure, the sensor attains approximately 100% sensitivity for both first and second peak voltage, which are 0.0018 V/ppm. This indicates the ability of the sensor to detect the identical output voltage for the peak's pattern consistently even the probe is relocated further from the surface of the liquid. In addition, the linearity of the sensor grants 96% for the first peak and 94% for the second peak with a low detection limit of 17.6 ppm and 18.2 ppm signifying that it works efficiently in detecting Pb²⁺ concentration level and is relevant to be a potential sensor for any cosmetics product application. The sensor performance of Pb²⁺ detection is tabulated in Table I.

TABLE I
 SENSOR PERFORMANCE OF Pb²⁺ DETECTION (CONCENTRATION)

Parameter	Peak Voltage (V)	
	1st Peak	2nd Peak
Sensitivity (V/ppm)	0.0018	
Measurement range (ppm)	0-500	

Linearity (%)	> 96	> 94
Resolution (mN/m)	17.6	18.2

According to Figure 5, the dotted line shows the focusing area for the permissibility limit based on the Malaysian Cosmetics Guideline and ASEAN Cosmetic Directive. This range is crucial as it might be useful for the detection of Pb²⁺ concentration level of hazardousness in cosmetics application purpose.

Further investigation then was executed to clarify the relationship between concentration-surface tension effect. Surface tension becomes an important liquid parameter in the medical area and industrial purpose. The changes in the surface tension especially in human biological fluids might be a vital sign to a pathological disorder, which correlates with the development of diseases. The exploration of meniscus formation on the surface of Pb²⁺ sample based on its relationship of the optical response curve against surface tension parameter is plotted in Figure 6. As the concentration increases, the surface tension of Pb²⁺ also increases linearly. This proved that lead contains surface tension characteristics as the surface tension value changes with the increment of its concentration.

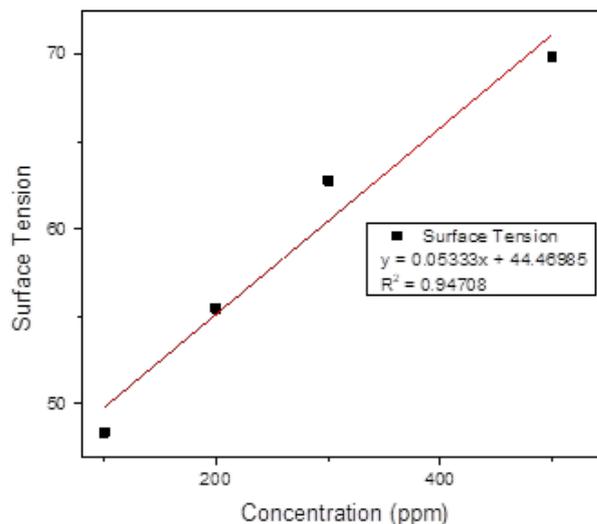


Fig. 6: Optical Response of surface tension against concentration

Figure 7a and 7b show the graphs of sensor calibration of surface tension characteristic for the first and second peak, respectively. The trend of both graphs indicates that the value of surface tension increases as the peak voltage increases. This is in a good agreement stated by Fen et. al [11] in which the group demonstrated as the refractive index increase, the concentration of the heavy metal of lead (Pb²⁺) also increases with respect to 1.3317 RIU to 1.3356 RIU. There is a great potential refractive index variant of lead affect the quantity of light being reflected on the surface tension of the lead sample.

The linearity of the surface tension graph against the peak voltage for first the peak is about 96% meanwhile for the second peak it is 97%. These two values of linearity show

good performance since it does not show much contrast at all. For the sensitivity analysis, the first and second peak demonstrates 0.035 V/mN/m and 27.72 V/mN/m, respectively.

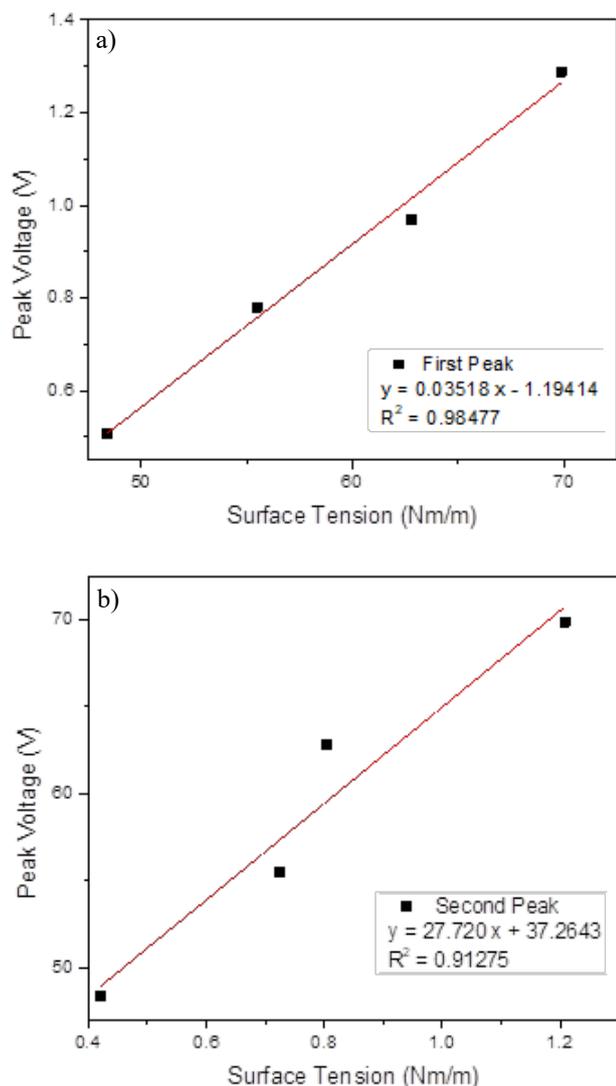


Fig. 7: Sensor calibration curve for surface tension a) first peak b) second peak

These surface tension's results confirmed that instead of concentration, surface tension also might be used widely as a potential parameter in recent sensor development as it possesses good optical response curve, sensitivity, and linearity. The summarization of sensor performance based on the surface tension characteristic is tabulated in Table 2.

TABLE II
SENSOR PERFORMANCE OF Pb²⁺ DETECTION (SURFACE TENSION)

Parameter	Peak Voltage (V)	
	1st Peak	2nd Peak
Sensitivity (V/ppm)	0.035	27.72
Measurement range (ppm)	0-500	
Linearity (%)	> 96	> 97

V. CONCLUSION

Fiber optic displacement sensor through reflective intensity modulation technique was successfully employed as a potential device to analyze the hazardousness of Pb²⁺ level. In evaluating the Pb²⁺ concentration in the range of 0 ppm to 500 ppm, two peak voltages obtained from the displacement curves illustrate outstanding features. For the concentration parameter, the sensitivity obtained for both peaks is almost similar, which is 0.0018 V/ppm with a detection limit of 17.6 ppm and 18.2 ppm and slope linearity of more than 94%. For surface tension parameter, the sensitivity gained was 0.034 V/ppm for the first peak and 27.72 V/ppm for the second peak, with slope linearity of more than 96%. This shows a promising technique with simplicity, low cost, and real time.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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