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# Fiber bundled probe for highly sensitive sensor for zinc ion detection using a concave mirror

R. MUHAMMAD[1], M. KHASANAH[2], M. ZULKARNAEN[1], M. YASIN[1,*]
[1]*Dept. of Physics, Faculty of Science and Technology, Airlangga University, Surabaya (60115), Indonesia.*
[2]*Dept. of Chemistry, Faculty of Science and Technology, Airlangga University, Surabaya (60115), Indonesia*

We experimentally demonstrate a bundled optical fiber based sensor for measuring zinc ion concentration. When zinc ions in liquid samples are shined with red laser light operating at about 630 nm wavelength it is observed that the intensity of the reflected radiation from the concave mirror, which was collected by the receiving fiber increases linearly with the increment of zinc ions concentration in the liquid sample. Our sensor has a simple architecture, it exhibits good sensitivity, linearity, and stability as it was tested with zinc ions concentration within 0% to 2%. It shows the highest sensitivity of 470 mV/% with the linearity of 92% and resolution of 0.0015%. The proposed sensor is based on optical non-destructive and on-site measurement, which may has applications in medical field.

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*Keywords:* Fiber sensor, Fiber bundled probe, Zinc ion sensor

## 1. Introduction

Zinc ion is one of important trace elements that people need to stay healthy. Of the trace elements, this element is second only to iron in its concentration in the body. $Zn^{2+}$ ions play a significant role in fundamental physiological processes as cofactors for metalloproteins and are regulators of enzyme function in neural signal transmission, and gene expression [1, 2, 3]. It is needed for the body's defensive (immune) system to properly work. It plays a role in cell division, cell growth, wound healing, and the breakdown of carbohydrates. Zinc is also needed for the senses of smell and taste. During pregnancy, infancy, and childhood the body needs zinc to grow and develop properly. Zinc also enhances the action of insulin.

Zinc ion ($Zn^{2+}$) has a low concentration in the human body yet plays a crucial role in physiology. It was estimated that the functions of more than three hundred enzymes are activated by zinc ions [4]. In particular, zinc ions are known to be vital in the male reproduction system and the nervous system. $Zn^{2+}$ is highly enriched at many glutamatergic nerve terminals. It was reported that a relatively higher concentration of $Zn^{2+}$ will be released and zinc-induced neuro- toxicity is observed following acute brain injuries, such as trauma, seizures, and ischemia. Thus, amount of free $Zn^{2+}$ can be used as a biochemical indicator for assessing the degree of brain injury.

The information on the zinc ion concentration near the injured brain region is helpful for medical intervention by neuro- surgeons. To collect such information, the zinc ion sensor has to be inside the brain. It can work together with intracranial pressure detection or stand-alone. In any case, the zinc ion sensor has to satisfy at least three criteria: i. The size has to be small for the minimally invasive surgical application. ii. The sensor has to be biocompatible and release no chemicals to the brain. iii. The sensor must respond to the zinc ion in the physiological or pathophysiological range of concentration and remains working for at least a few hours. Previously, zinc ions sensor has been reported using surface plasma resonance of surface-modified gold, which requires a complex spectroscopy system [5]. A high sensitivity zinc ions detection was also reported based on electrical approach with extended gate-AlGaN/GaN [6]. However, the electrical approach has a complicated fabrication process. The combination of the optical fiber and the hydrogel doped with probe molecules appears to be a promising approach for such three criteria [7-8]. One of the suitable probe molecules is *meso* 2,6-Dichlorophenyltripyrrinone (TPN-$Cl_2$).

The general challenge for the probes in the hydrogel is the resolution and reproducibility with a low concentration of zinc ions [9-11]. So far there are few reports on the solid-state zinc ion sensor working at the physiological or pathophysiological conditions. The basal zinc ion concentration in the brain extracellular compartment is extremely low (~$10^{-8}$ M), but it can increase dramatically under pathological conditions [12]. On stimulation by lipopolysaccharides (LPS) or oxygen/glucose deprivation (OGD), mimicking brain inflammation or ischemia, the concentration can reach the level of $10^{-6}$ M. For medical applications, the working concentration of the sensor must therefore cover the range of $10^{-6}$ to $10^{-5}$ M. In previous reports for the hydrogel-based zinc ion sensor one-time detection is observed for $10^{-7}$ and $10^{-6}$ M [11], but quantitatively such detection is not reproducible for several independent periods of the same concentrate Reliable detection was obtained only for concentration over $10^{-5}$ M. Such poor detection and resolution limit are related to the photo-degradation of the probe molecules under the intense excitation light. The sensitivity needs to be improved for at least one order of magnitude in the intracranial detections for brain insult patients.

During recent years, fiber optic sensors have gained escalating interest in the field of biosensor applications due to their potential to monitor analytes in real-time and in situ with minimum interruption to sample with higher sensitivity and desire selectivity. For instance, fiber optic sensors based on surface plasmonic resonance (SPR) and micro-bottle resonator have been proposed and demonstrated for formaldehyde liquid sensing [14-15]. In this paper, we demonstrate a bundled optical fiber-based sensor for measuring zinc ion concentration in liquid. The proposed sensor is based on optical non-destructive and on-site measurement, which is obtained using a very simple detection scheme based on collecting the intensity of laser light reflected from the concave mirror, which is placed inside the liquid sample. Compared to the free space reflective configuration, the use of bundled fiber increases the sensitivity of the sensor due to the better control of transmitting and receiving light. 630 nm laser is used in this work due to the stability and low cost of the laser.

## 2. Sample preparation and experimental setup

Samples of $Zn^{2+}$ solution with different concentrations ranging from 0% (0.1530 M) to 6% (0.9177 M) were prepared with a volume of 100 ml from zinc sulfate heptahydrate ($ZnSO_4.7H_2O$) as shown in Fig. 1. Table 1 summarizes the composition of the prepared $Zn^{2+}$ solution. This solution was used as a sample for testing the fiber optic sensor based on bundle fiber probe using a concave stainless mirror reflector. The 2.5 ml of the sample volume is placed in a concave mirror while a zero displacement is set at position where the bundle fiber is non-touching the surface of the solution.

*Fig. 1. Zn sample for various concentration (0-6%) (color online)*

*Table 1. Composition of $Zn^{2+}$ solution.*

| Zn Concentration (%) | Mass (gram) | Total solution volume (ml) |
|---|---|---|
| | $ZnSO_4.7H_2O$ | |
| 0 | 0 | 100 |
| 1 | 4.5 | 100 |
| 2 | 9.1 | 100 |
| 3 | 13.6 | 100 |
| 4 | 18.1 | 100 |
| 5 | 22.6 | 100 |
| 6 | 27.2 | 100 |
| 7 | 31.7 | 100 |

We used a Helium-Neon laser emitting in the visible wavelength of 633 nm as a light source to illuminate a zinc sample through a 2 ports fiber bundle probe; the intensity of the light reflected from the zinc was collected through the second port of the fiber bundle probe and measured by a silicon optical detectors (Newport SL- 818), which was connected to a standard digital voltmeter. The laser provides an average output power 3.0 mW, beam diameter 0.75 mm and beam divergence 0.92 mRads. The multimode plastic bundle fiber consists of two cores with a length of 2 m and core radius of 0.25 mm and it was fixed using a holder, which was attached to a translation stage. The stage supports axial displacement with a micrometric movement, thus the distance between the probe and the sample can be finely controlled. The zinc liquid samples are transferred on a concave mirror, which was attached to stainless-steel plate and fixed in a translation stage using a pipette. A schematic of the experimental setup is shown in Fig. 2. The laser was warmed up for about one hour before use to minimize the influence of optical source power fluctuation while the measurement was carried out in a dark room to avoid background radiation. The working principle of the sensor is based on the following equation [11].

$$E(D,u) \approx 1 - \exp\left(-\frac{D^2}{2(u+z_a)^2\theta^2}\right) \tag{1}$$

where $D$, $\theta$, $z_a$ *and* $u$ are a diameter of concave mirror, an acceptance angle, a distance from original source to the fiber surface, and is distance from fiber surface to concave mirror, respectively.

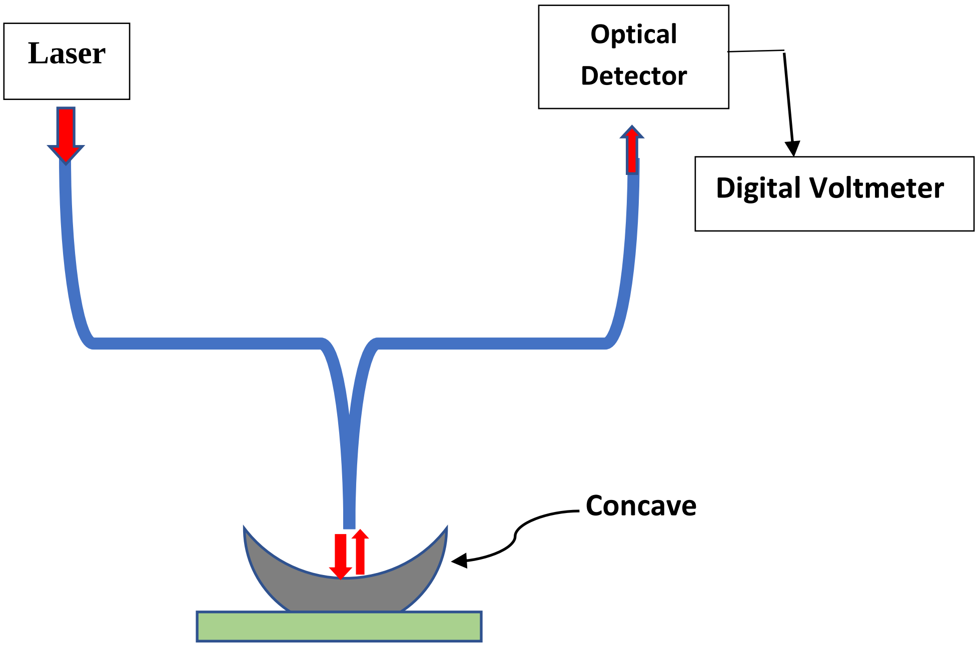


*Fig. 2. Experimental setup of fiber sensor (color online)*

## 3. Results and discussions

### 3.1. Displacement characteristic

Fig. 3 shows the reflected light intensity versus distance of the reflecting concave mirror from the fiber optic probe without zinc concentrations. The displacement curves exhibit the peak voltage value decreases and moves towards the right as the displacement increases. However, the back slope appears to be the latter steeper than the first. In the near field sensing, most of the transmitted light from the bundled fiber is reflected as long as the incident light cone is within the reflecting surface area of the mirror. However, as the displacement increases the incident light cone drops larger. The maximum reflected light power from the mirror decreases due to the limited reflecting surface area of the mirror and subsequently influences the displacement response of the sensor.

The displacement sensor characteristics were carried out without a sample (0% of zinc concentration) which was only a stainless steel concave mirror (as a sample reflector) with a concave mirror shift of 0 - 22 mm for 3 measurements as in Fig. 3. From the characterization results, a second peak appears at a distance of about 9 mm, this result can be used to estimate the focal length of a concave mirror (2f = 9 mm) so that the concave mirror used has a focal length of about 4.5 mm.

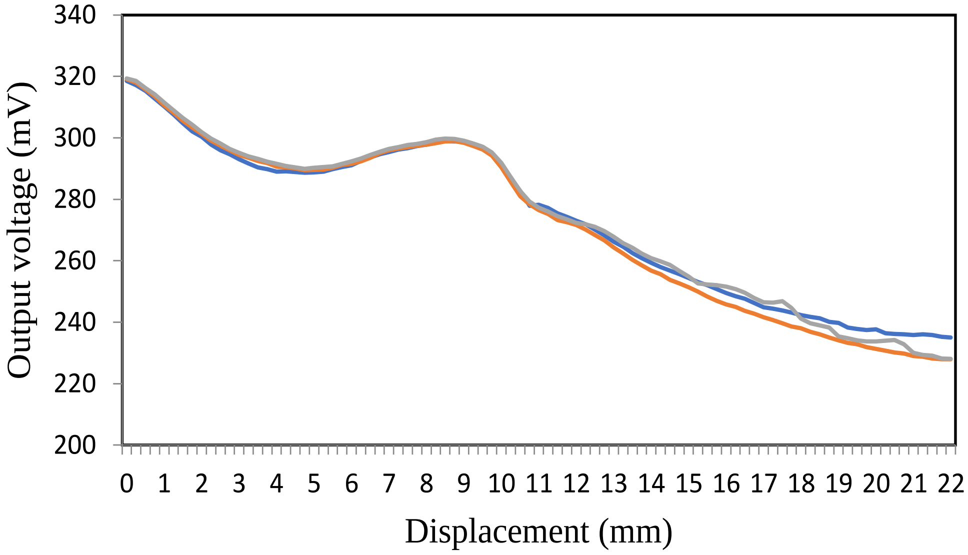


*Fig. 3. Characteristic of the sensor displacement without samples (color online)*

### 3.2. Characteristic of the sensor for zinc ion concentration measurement

The displacement sensor characterization produces a graph with one valley and one peak (at a distance of 9 mm) with a displacement overall range of 22 millimeters from the bottom of the container to the farthest point. After that, the sensor characterization was carried out using a sample of various zinc solutions (0-6%) and the shift range was 14 mm because the zero point was taken from the surface of the solution without touching the probe bundle. This is done so that the probe bundle is not immersed in the solution and affects the measurement results, as well as for efficiency if there are certain events. Fig. 4 shows the output voltage against the displacement for various zinc concentration ranging from 0 to 6%. It is shown that there is a decrease in the sensor output voltage for all concentration variations (0-6%) as the bundled fiber tip is shifted from the zero point (initial) to 14 mm. In this case, the farther the fiber bundle probe is from the sample, the lower the output voltage. It is also obtained that the higher the concentration of Zn solution, the higher the sensor output voltage value.

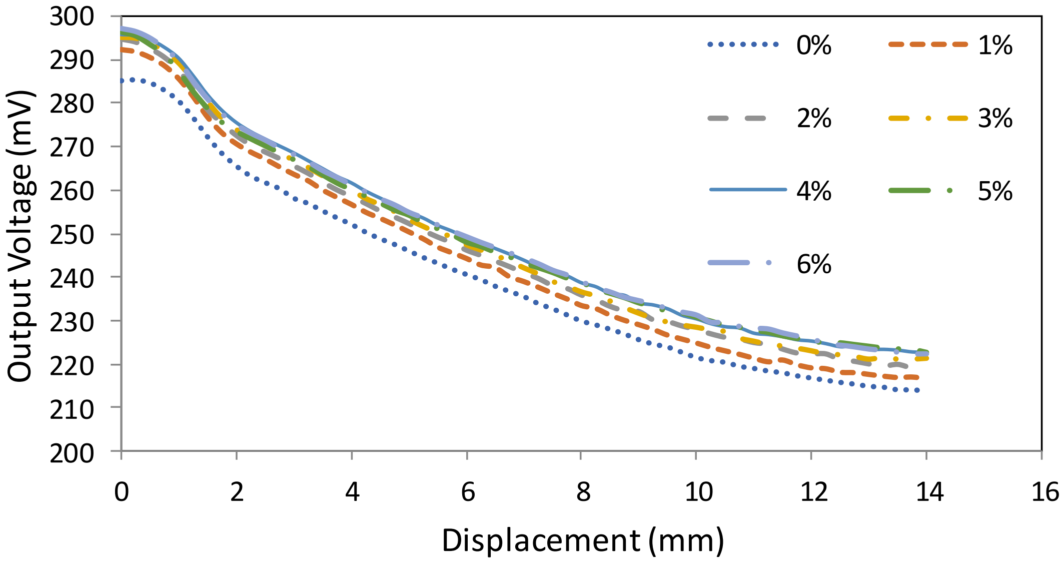


*Fig. 4. Voltage of output signal (in mV) versus displacement (mm) for various zinc ions concentrations (0-6%) (color online)*

### 3.3. Sensor stability

The stability of the sensor is investigated after the measurements of output voltage against displacement at various zinc ions concentrations. The output voltage at a fixed position of sensor probe is monitored against time as shown in Fig. 5. The experiment was repeated three time. From this stability measurement, the maximum deviation value is 0.21 mV. This standard offset value is used to calculate the sensor resolution which is defined as follows,

$$Resolution = \frac{Std}{Sn} \tag{2}$$

where Std is Standard Deviation (mV) and Sn is sensitivity (mV/%).

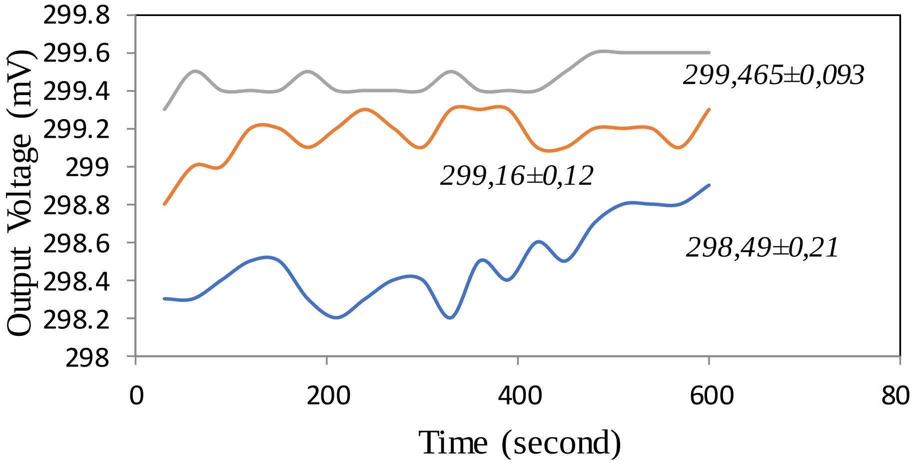


*Fig. 5. Stability of the sensor (color online)*

### 3.4. Performance of the sensor

The variation of output voltage of the sensor against the zinc ions solution concentrations is summarized in Fig. 6 for three different probe positions; 0-, 2-, and 4-mm. The result shows a trend of decreasing output voltage as the probe displacement is increased. In the experiment, the probe is located between the probe bundle and the sample surface, and the zero point is taken near the liquid surface. It was not immersed inside the liquid to avoid the probe bundle from dirt that can affect the measurement. The sensor characterization with the sample is taken for three displacement positions of 0, 2 and 4 mm. At a fixed displacement position, the relationship between the sensor output voltage and the concentration of Zn solution is also obtained.

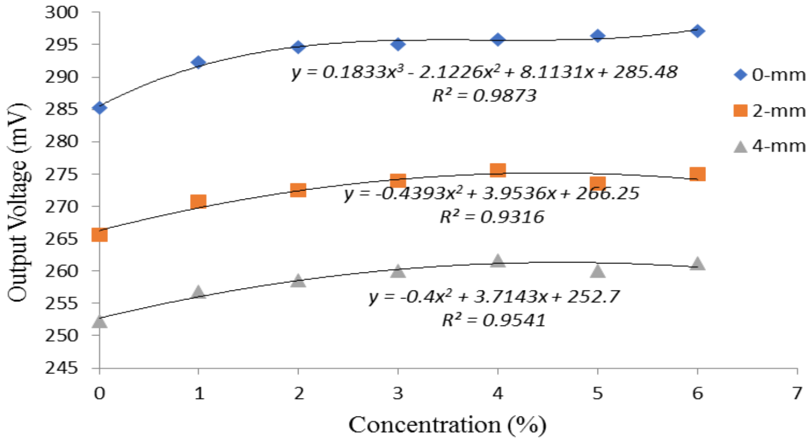
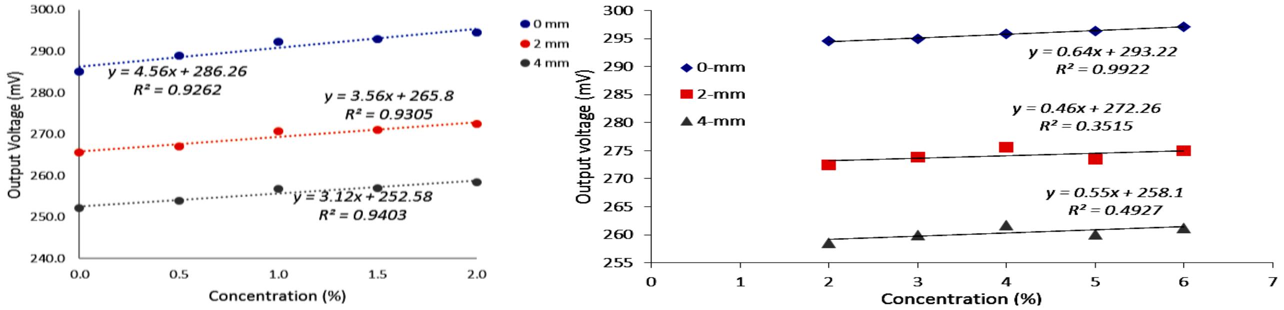


*Fig. 6. The correlation curve between output voltage of the sensor (mV) and Zn solution concentration when the displacement is fixed at 0-, 2-, and 4-mm position (color online)*

Based on Fig. 6, it is found that there are two linear ranges; 0-2% and 2-6%, the sensitivity is larger at smaller concentration range. Therefore, the output voltage against the concentration analysis is separated in Fig. 7. Fig. 7(a) and (b) shows the separate graph within a linear range of 0-2% and 2-6%, respectively at three different displacement positions of 0, 2 and 4 mm. As shown in Fig. 7(a), a linear relationship between the output voltage and the concentration of Zn solution is obtained for all three positions. The highest sensitivity of 4.56 mV/% is obtained at 0 mm position. A similar trend is also obtained as the concentration is varied within a linear range of 2-6% as shown in Fig. 7(b), but the sensor's sensitivity is significantly lower. At 0 mm position, the maximum sensitivity is obtained at 0.64 mV/%. The performance of the sensor is summarized in Table 2.



*(a)* *(b)*

*Fig. 7. A regression curve of sensor output voltage to Zn concentration within (a) 0-2 mm range (b) 2-6 mm range at different probe positions of 0, 2, and 4 mm (color online)*

*Table 2. Performance of the sensor at a fixed displacement of 0, 2 dan 4 mm.*

| Position (mm) | Linearity (%) | Sensitivity mV/%)* | Resolution (%) | Linear range |
|---|---|---|---|---|
| 0 | 92 | 4.56 | 0.046 | 0% - 2% |
| 2 | 93 | 3.56 | 0.059 | |
| 4 | 94 | 3.12 | 0.067 | |

**volume percentage*

## 4. Conclusion

We have successfully demonstrated a new fiber-optic sensor using a bundled fiber probe to measure the concentration of zinc ion solution based on measuring the reflection of laser light from a concave mirror. We observed that the intensity of reflected light increases proportionally to the increase of zinc concentration. The sensor is very stable and responds linearly to the concentration of zinc ion liquid, which was placed on the concave mirror surface. Our sensing technique is simple and low cost. It could be applied to detect zinc ion concentration in a non-invasive and non-destructive way. The resolution, linearity and sensitivity of the sensor are obtained at 0.046%, 92 % and 4.56 mV/%, respectively. The proposed sensor has potential to be further developed towards a portable device, which is suitable for applications in many fields.

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[*] Corresponding author: yasin@fst.unair.ac.id