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# Label-Free Detection of Dissolved Carbon Dioxide Utilizing Multimode Tapered Optical Fiber Coated Zinc Oxide Nanorice

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**ABSTRACT** A label-free detection for dissolved carbon dioxide ($dCO_2$) is developed using a tapered optical fiber sensor. The tapered region of the optical fiber is coated with the zinc oxide (ZnO) nanorice and used as a probe for $dCO_2$ sensing. The sensor probe was exposed to different concentrations of $dCO_2$ solution ranging from 10 to 100 ppm. ZnO nanorice can adsorb $dCO_2$ via strong hydrogen bonding due to the presence of plenty of oxygen atoms on its surface layer. The interaction between ZnO nanorice and $dCO_2$ changes the optical properties of the ZnO nanorice layer, resulting in the change in reflectance. From the experiment, the result shows that there is an improvement in the sensitivity of the sensor when higher concentration was used. A broad linear trend ranging from 0 to 60 ppm ($R^2 = 0.972$) is observed for the sensor probe that is coated with 1.0 M of ZnO nanorice compared with the 0.1 M and 0.5 M ZnO nanorice concentrations. The sensor sensitivity obtained is 0.008 mW/ppm. The sensor demonstrates a response and recovery time of 0.47 and 1.70 min, respectively. Good repeatability is obtained with the standard deviation in the range of 0.008–0.027. The average resolution calculated for this sensor is 4.595 ppm.

**INDEX TERMS** Optical fiber sensor, tapered optical fiber, zinc oxide, dissolved carbon dioxide.

## I. INTRODUCTION

Carbon dioxide ($CO_2$) sensing, both in gaseous and dissolved phases are crucial for environmental monitoring and protection. Excessive amount of $CO_2$ in the atmosphere triggers global warming, climate change and elevation in ocean acidity [1], hence affecting negatively on human, marine life and environment. Gaseous $CO_2$ can be measured directly using near-infrared (NIR) spectroscopy at 4200 and 4400 $cm^{-1}$ wavelengths. Nevertheless, this technique suffers from limitation of measuring the dissolved $CO_2$($dCO_2$) concentrations [2], due to the strong interference from the water vapour [3].

Optical sensor technique is one of the reliable options for the detection of $dCO_2$ due to its highly sensitive characteristics and low-cost fabrication. However, to date, most of the reported works on dissolved $CO_2$ sensing rely on the usage of the fluorescent indicator such as hydroxypyrenetrisulfonic acid (HPTS) for labeling the target chemical analyte. Burke *et al.* (2006) [4], used poly(dimethylsiloxane) doped with HPTS for detection of $dCO_2$ and reported a response and recovery times of 3 and 10 minutes, respectively. Oter *et al.* (2006) [5], developed the detection of $dCO_2$ using HPTS dissolved in green chemistry reagent and obtained a good sensor stability for $dCO_2$. Later in 2008, a simpler optical technique using evanescent wave for detection of $CO_2$ in gas phase was reported by Chu and Lo (2008) [6]. This technique involved the utilization of the optical fiber integrated with the fluorinated xerogels doped with the HPTS as the sensing material. A good response and recovery times of 1.7 s and 38.5 s were reported, respectively. A good detection limit of 0.03 % was also reported. Although the existing techniques exhibit excellent sensor performance, the preparation of the labeling materials are complex and time-consuming [4]–[6]. Most of the labeling techniques are not suitable for in-situ application. Furthermore, the use of the standard optical fiber leads to low sensitivity of the sensor due to the thick cladding layer, thus limiting the interaction between the contact surface and substrate. To improve this limitation, modification of the standard optical fiber is essential in which the evanescent field is exposed beyond the surface of the sensing region [7]. One of such modification is by tapering the fiber. Orgichi *et al.* [8] fabricated a sensor for $CO_2$ in gas and liquid phases using tapered optical fiber without material coating for sequestration application. The sensing mechanism is based on the temperature variation of the water in the tank. The solubility of the $dCO_2$ is dependent on the variation of the temperature, hence producing different output signal. A good agreement is obtained between the experimental and theoretical results. However, there are various chemical analytes other than $dCO_2$ present in the water. Detection of $dCO_2$ based on the temperature changes alone does not ensure the selectivity of the sensor due to the possible interference from other chemical analytes. One way to avoid this issue is by coating the tapered optical fiber with the material that is selective to $dCO_2$ [7], [9].

Metal oxide nanomaterials are the most promising candidate for this purpose and commonly used in the construction of the sensor devices due to their high thermal stability, non-toxic nature, photo catalytic properties [10] and good optical behavior [11]. In the previous study, it was reported that metal oxides able to adsorb $CO_2$ species on its surface [12], [13]. There are many kinds of metal oxide nanomaterials and zinc oxide (ZnO) is one of such materials. ZnO has a high surface area and a wide bandgap II-IV semiconductor ($\sim$3.37 eV) which makes it suitable for the optoelectronic application [14]. Other than that, ZnO is also known as a better ultraviolet (UV) emitting phosphor than gallium nitride (GaN) because of its high exciton binding energy ($\sim$60 meV) at room temperature [11]. This leads to a reduced UV lasing threshold and yields a higher UV emitting efficiency at room temperature [15]. ZnO nanostructure can be manipulated into various structures such as nanorods, nanotubes, nanocombs, nanosaws and so forth, where it is an important feature as a sensing material for optical sensor [16]. In optical sensing, ZnO nanorod is a natural candidate for an optical waveguide because it has near-cylindrical geometry and large refractive index ($\sim$2.0) [17].

This paper reports on the development of a label-free technique for $dCO_2$ sensing based on evanescent wave, by using a tapered multimode optical fiber coated with a thin layer of ZnO nanorice. The diameter size of the tapered multimode optical fiber is fixed to one size which is 50 $\mu$m as suggested by Biazoli *et al.* [18] to ensure the presence of a large fraction of the evanescent field on the surface of the tapered optical fiber. ZnO nanorice is used as the sensing material for detection of $dCO_2$ at different concentrations. To assess the sensor performance, the coating concentration of the ZnO nanorice is varied. As a result, a good sensitivity of the sensor is achieved. The dynamic response and the repeatability of the sensor is studied accordingly.

## II. MATERIALS AND METHODS

### A. PREPARATION OF ZINC OXIDE (ZnO) NANORICE SOLUTION

ZnO nanorice solutions was synthesized using a sol-gel technique following method developed by Abdullah *et al.* (2009) [16] with some modifications. Zinc nitrate hexahydrate, $Zn(NO_3)_2 \cdot 6H_2O$ and urea, $CH_4N_2O$ were dissolved in 60 mL of ethanol, $C_2H_5OH$. The solution was stirred on a hotplate for 3 hours at room temperature. To adjust the pH of the solution to 4, nitric acid was added slowly during the stirring process. A clear solution was formed after the reaction was completed. The ZnO solution was prepared in three different concentrations, which were 0.1 M, 0.5 M and 1.0 M.

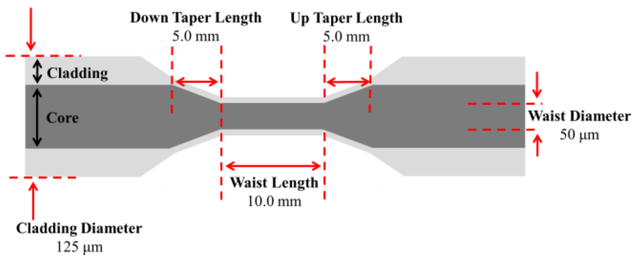


**FIGURE 1. Sketched image of tapered optical fiber parameter.**

### B. PREPARATION OF THE TAPERED MULTIMODE OPTICAL FIBER

A multimode type of optical fiber is used throughout the study. A tapered optical fiber has a reduced diameter of waist region than that of the standard optical fiber (Figure 1) due to the thinning of the cladding at the tapered region. This is essential to ensure the evanescent field is exposed beyond the surface of the sensing region. The tapered optical fiber was prepared using a Vytran GPX-3400 optical glass fiber processor. The Vytran machine uses a filament heater made of graphite to heat up a standard graded-index optical fiber while both sides of the fiber were pulled simultaneously. The tapering process was fully automated, and the desired taper profiles were specified on a graphical user interface as shown in Figure 1. In this work, an optical fiber with a cladding/core size of 125 $\mu$m/62.5 $\mu$m was tapered to produce biconical tapers with waist diameter of 50 $\mu$m. The transition region and waist lengths were fixed at 5 mm and 10 mm, respectively. The tapered optical fiber was characterized using a Hitachi S-3400N scanning electron microscopy (SEM) as shown in Figure 2.

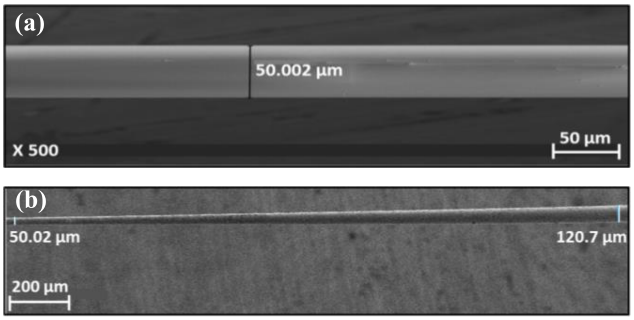


**FIGURE 2.** SEM images of (a) transition region and (b) waist region of the tapered optical fiber.

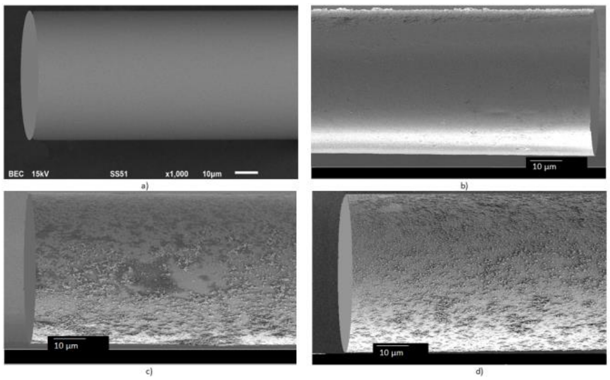


**FIGURE 3.** FESEM images of the tapered optical fiber (a) without coating, coated with (b) 0.1 M (c) 0.5 M and (d) 1.0 M of ZnO nanorice.

### C. FABRICATION OF THE SENSOR PROBE

To enhance the sensing area of the optical fiber, the tapered region of the optical fiber was dip-coated with the ZnO nanorice solution using a millimeter grade programmable dip coater (PTL-MMB01, MTI Corporation, USA) which was attached to the drying oven (WHL-30B, MTI Corporation, USA). Prior to the dip-coating process, the tapered optical fiber was cleaned using isopropanol to ensure the surface was free from dirt and dust. In addition, no pre-treatment process was applied to the surface of the tapered optical fiber before it was coated with the ZnO nanorice. Different concentrations of ZnO nanorice solutions; 0.1 M, 0.5 M and 1.0 M were used for coating. The speed of the dip coater was set to constant for all samples and the coated optical fibers were left for 48 hours in the oven at room temperature. Once the dipping process was completed, the coated optical fiber was dried in the oven at 150 °C. The morphology of the ZnO nanoparticles is dependent on the solvent used for the dispersion of the ZnO nanoparticles due to the interaction of solvent with the zinc nitrate salt [19]. In this work, ethanol was used as a dispersion medium for the ZnO nanoparticles. The chemical interaction between ethanol and zinc nitrate salt produces nanorice-like structure of ZnO nanoparticles on the surface of the tapered optical fiber as shown by field emission scanning electron microscopy (FESEM) (Zeiss SUPRA 55VP, Germany) images in Figure 3. In Figure 3(d), a uniform distribution of ZnO nanorice on the surface of the tapered optical fiber is observed for 1.0 M of ZnO nanorice in comparison to 0.1 M (Figure 3(b)) and 0.5 M (Figure 3(c)) of ZnO nanorice.

### D. EXPERIMENTAL SETUP FOR DISSOLVED $CO_2$ SENSING

Different concentrations of $dCO_2$ in the range of 10 to 100 ppm with steps of 10 ppm were prepared by dissolving sodium hydrogen carbonate ($NaHCO_3$) in deionised water as suggested by Müller and Hauser [20]. All the aforementioned $dCO_2$ samples were freshly prepared prior to the experiment to avoid contamination. Figure 4 shows the schematic arrangement of the experimental setup for the sensor probe characterization. A tunable light source (ANDO-AQ4312A) was used as the laser source where it was connected to the port 1 of the optical circulator. The sensor probe that was connected to the port 2 was immersed in the bottle containing $dCO_2$ solution while port 3 was connected to an InGaAs photodetector (PM16-144, Thorlabs, USA) for the conversion of light to current. The wavelength range of the photodetector is from 800 to 1700 nm with the resolution of <1 nW. The photodetector was then connected to the optical power meter (PM100D, Thorlabs, USA) to detect the changes of the output power. The wavelength used was 1550 nm.

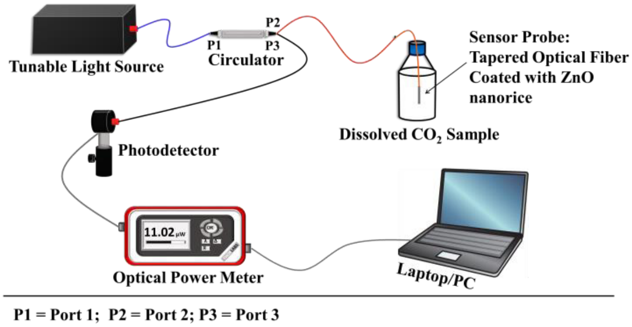


**FIGURE 4.** Experimental setup for $dCO_2$ sensing using tapered optical fiber coated with ZnO nanorice.

### E. SENSING MECHANISMS OF DISSOLVED $CO_2$

In this work, the sensing mechanism is based on the interaction between the evanescent fields with different concentrations of the chemical analytes. Light that propagates in the optical fiber undergoes total internal reflection at the interface between the two media with different refractive indices; core and cladding [8]. Near to the core-cladding interface, a stronger evanescent field exists and the penetration depth decays with 1/e into the cladding. Due to the radiation field of the mode field diameter, the main guided mode is confined in the core of the optical fiber. However, the radiation field also extends partially into the cladding, thereby causing the cladding mode to appear.

Based on this principle, the optical fiber is tapered to thin down the cladding to enhance the interaction of evanescent field with the chemical analytes. Hence, this method provides a stronger interaction between the evanescent field and the light-sensing material with the chemical analytes. Figure 5(a) shows the bare tapered fiber without the ZnO

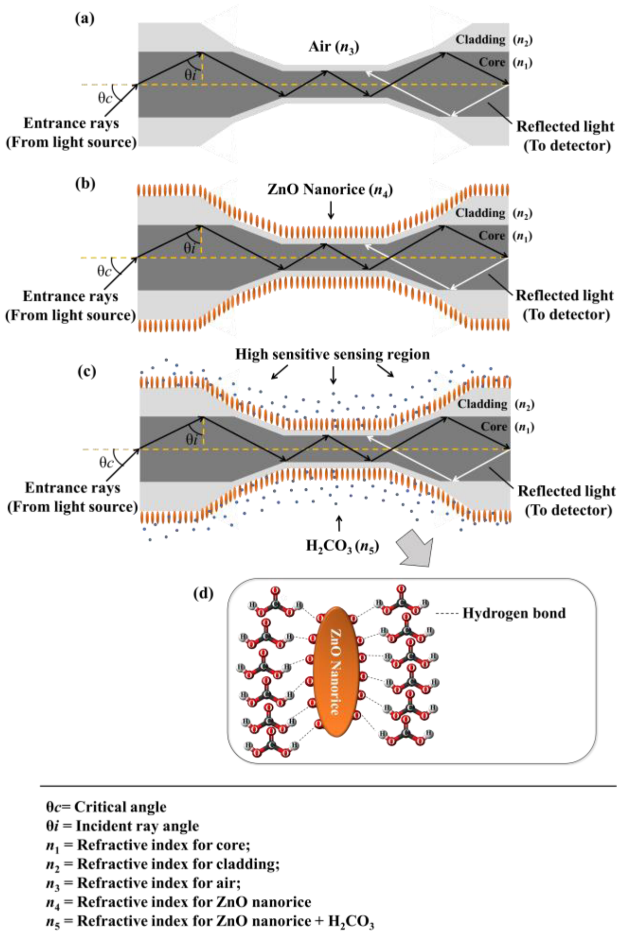


**FIGURE 5. Schematic diagram of $dCO_2$ sensing mechanism for the proposed tapered optical fiber sensor in the (a) bare tapered optical fiber, (b) tapered optical fiber coated with ZnO nanorice, (c) presence of $dCO_2$ on the surface of the tapered optical fiber coated with the ZnO nanorice and (d) molecular interaction of $dCO_2$ on the surface of the ZnO nanorice during the adsorption process.**

nanorice coating on the surface. In this case, the light is guided in the core of the optical fiber. The chemical analyte may react with the evanescent field on the cladding surface of the optical fiber. Upon encountering the end of the fiber, the light is reflected, following the Fresnel reflection law. This signal propagates to the detector, producing a measurable optical signal output. In figure 5(b), the tapered region of the optical fiber is coated with the ZnO nanorice. The similar principle light propagation applies but, in this case, there is interaction between the evanescent field and ZnO nanorice with chemical analyte. Variation of chemical analyte binding causes variation of reflectance of optical signal.

Before discussing on the chemical interaction mechanism between ZnO nanorice and $dCO_2$ that occurs on the surface of the tapered optical fiber, it is worth-highlighting the chemical nature of the $dCO_2$ in water prior to the reaction with the ZnO nanorice. In water, $CO_2$ gas dissociates into carbonic acid ($H_2CO_3$) and hydrated $CO_2$ gas ($CO_2$ (aq)) [5]. However, in this work, the term $*H_2CO_3$ is used to represent the total amount of $dCO_2$ in the solution ($H_2CO_3$(aq) *plus* hydrated $CO_2$ (aq)) as described in equation 1.

$$CO_2\,(g) + H_2O\,(l) \rightleftharpoons *\,H_2CO_3\,(aq) \tag{1}$$

Detection of $dCO_2$ is measured based on the adsorption of $*H_2CO_3$ on the surface of the ZnO nanorice. The amount of $*H_2CO_3$ that is adsorbed on the surface of the tapered optical fiber coated with ZnO nanorice is proportional to the concentration of $dCO_2$. When the surface is exposed to the different concentrations of $dCO_2$(Figure 5(c)), electrostatic interaction occurs between oxygen atom of ZnO nanorice and hydrogen atom of $*H_2CO_3$. These two atoms are linked via strong hydrogen bond as shown in Figure 5(d). This phenomenon changes the refractive index of the sensor, hence producing variations in optical signal output [21]. The sensitivity, resolution, dynamic response and repeatability of the proposed sensor is discussed accordingly.

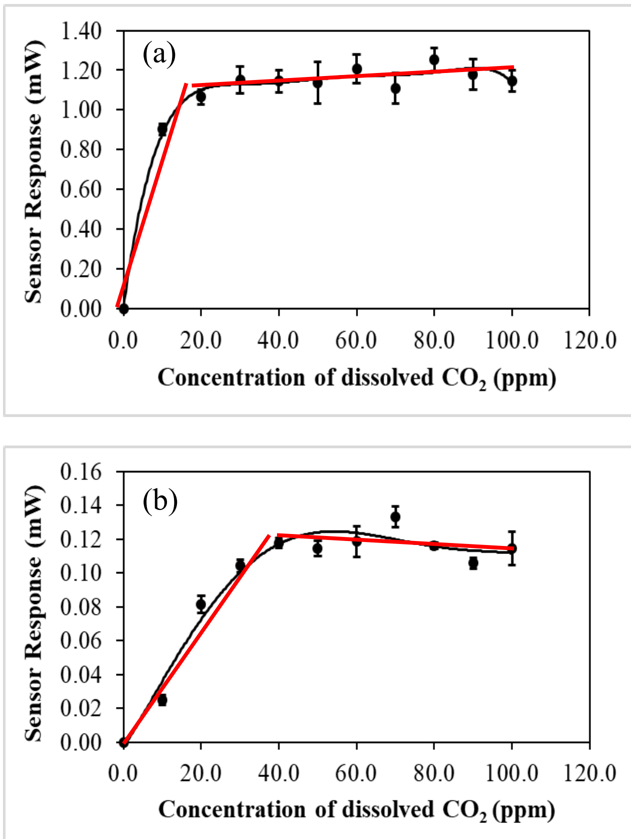


**FIGURE 6. Sensor response of $dCO_2$ detection using (a) bare tapered optical fiber and (b) tapered optical fiber coated with 0.1 M ZnO.**

## III. RESULTS AND DISCUSSIONS

### A. SENSOR RESPONSE

Figure 6 shows the sensor response (SR) of (a) bare tapered optical fiber and (b) tapered optical fiber coated with ZnO probes when exposed to the different concentrations of $dCO_2$ in the range of 0 to 100 ppm. The calculation of sensor response is adopted from Chu and Lo (2008) [6]. The sensor response for bare tapered optical fiber in Figure 6(a) is linearly dependent on [$dCO_2$] in two phases of concentration (i.e. 0 ppm to 20 ppm and 20 ppm to 100 ppm). The calibration curve for both phases are given by the equations (2) and (3), with $R^2 = 0.862$ and $R^2 = 0.253$ for 0 ppm to 20 ppm and 20 ppm to 100 ppm, respectively.

$$SR = 0.053[dCO_2] + 0.123 \quad 0\text{ ppm} \leq [dCO_2] \leq 20\text{ ppm} \tag{2}$$

$$SR = 0.001[dCO_2] + 1.095 \quad 20\text{ ppm} \leq [dCO_2] \leq 100\text{ ppm} \tag{3}$$

Albeit the bare tapered optical fiber exhibits high response towards $dCO_2$ at low concentration levels (i.e. 0 – 20 ppm), the reflectance signals started to reach saturation at 20 ppm concentration due to the fully occupied adsorption site on the surface of bare tapered optical fiber, hence producing a stagnant curve in reflectance signal thereafter. On the contrary, by coating the surface of bare tapered optical fiber with 0.1 M of ZnO nanorice as shown in Figure 6(b), a better linearity range for $dCO_2$ sensing is obtained (i.e. 0 ppm to 40 ppm and 40 ppm to 100 ppm) due to the more adsorption sites available contributed by the ZnO nanorice on the surface of the optical fiber. The saturation of the adsorption site only occurs after 40 ppm concentration. The calibration curve for both phases are given by the equations (4) and (5), with $R^2 = 0.969$ and $R^2 = 0.011$ for 0 ppm to 40 ppm and 40 ppm to 100 ppm, respectively.

$$SR = 0.004[dCO_2] - 0.003 \quad 0\text{ ppm} \leq [dCO_2] \leq 40\text{ ppm} \tag{4}$$

$$SR = 4 \times 10^{-5}[dCO_2] + 0.113 \quad 40\text{ ppm} \leq [dCO_2] \leq 100\text{ ppm} \tag{5}$$

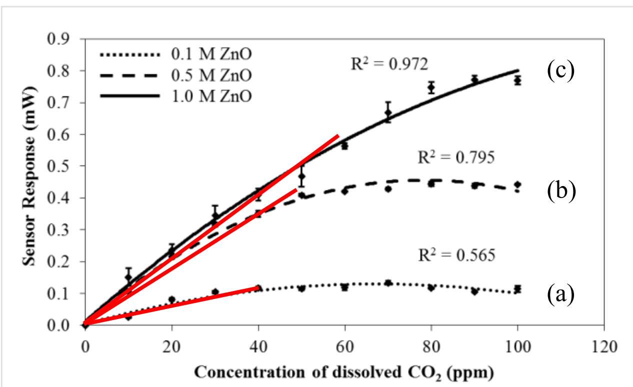


**FIGURE 7.** Effect of (a) 0.1 M, (b) 0.5 M and (c) 1.0 M ZnO coating concentrations on the tapered optical fiber for $dCO_2$ sensor.

### B. OPTIMIZATION OF ZnO NANORICE COATING CONCENTRATIONS

To ensure a good performance of the sensor, various concentrations of ZnO nanorice coated on the surface of the tapered optical fiber is studied. Various concentrations of the ZnO nanorice solutions used affect the distribution of the ZnO nanorice on the surface of the tapered optical fiber [22]. Figure 7 shows the sensor response of different ZnO nanorice coating concentrations at (a) 0.1 M, (b) 0.5 M and (c) 1.0 M for detection of $dCO_2$. In the figure, the line (a) shows the calibration curve for 0.1 M of ZnO nanorice coating concentration on the surface of the tapered optical fiber. The linearity range observed for 0.1 M of ZnO nanorice is 0 – 40 ppm ($R^2$=0.565). Meanwhile, as the concentration of the ZnO nanorice increases to 0.5 M as seen in line (b), the linearity range increases from 0 – 50 ppm ($R^2$=0.795). The highest range of linearity is given by the 1.0 M of the ZnO nanorice coating concentration as seen in line (c) which is from 0 ppm to 60 ppm ($R^2$=0.972).

This phenomenon is due to the distribution variations of ZnO nanorice coatings on the surface of the tapered optical fibers. At higher concentration, ZnO nanorice is distributed evenly on the surface of the tapered optical fiber as shown by FESEM analysis in Figure 3(d). This leads to a better and effective collision between ZnO nanorice and $dCO_2$, hence producing a better sensor response. In contrast, a lower sensor response is observed for 0.5 M and 0.1 M of ZnO nanorice concentrations which is due to the uneven ZnO nanorice distribution on the surface of the tapered optical fiber as shown by FESEM analysis in Figure 3(b) and 3(c). The sensitivity of the sensor is the ratio of the change in the SR to the change in the concentration of $dCO_2$ ($\Delta SR:\Delta dCO_2$) and can be obtained from the gradient of the graph. The sensitivity for $dCO_2$ sensor utilizing 0.1 M, 0.5 M and 1.0 M of ZnO nanorice are 0.001 mW/ppm, 0.004 mW/ppm and 0.008 mW/ppm, respectively.

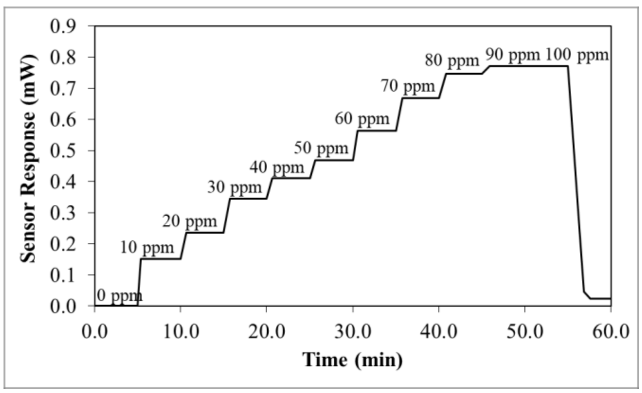


**FIGURE 8.** Response and recovery times of the $dCO_2$ sensor probe by alternating between $dCO_2$ solution and deionized water.

### C. DYNAMIC RESPONSE

Figure 8 shows the dynamic response of 1.0 M ZnO nanorice concentration towards different range of $dCO_2$ concentrations. Response time is defined as the time taken for the sensor to reach stability immediately after the sensor probe is exposed to $dCO_2$ solution, while recovery time is defined as the time taken for the sensor to reach the baseline again when the sensor probe is exposed to deionized $H_2O$ immediately after being exposed to $dCO_2$ solution. The estimation of response and recovery times of the sensor was carried out by exposing the sensor probe in $dCO_2$ solution for 5 minutes, followed by deionized water for 10 minutes. The exposure was carried out for a few cycles for different $dCO_2$ concentrations in the range of 10 to 100 ppm. As seen in Figure 8, the sensor response increases as the concentrations of $dCO_2$ increases from 0 to 90 ppm. Upon further increasing the $dCO_2$ concentration, the sensor response becomes constant, suggesting that the sensor probe was no longer sensitive at this phase due to the fully occupied reaction sites on the surface of the ZnO nanorice. The response and the recovery times with $t_{90}$ values are 0.47 minutes and 1.70 minutes, respectively. This result is better than the previous report which is around 3 and 10 minutes [4]. Burke *et al.* [4], reported that the response and recovery times are greatly affected by the thickness of the material used during the experiment, where the interaction response between the sensing materials with the chemical analytes is dependent on the thickness of the material.

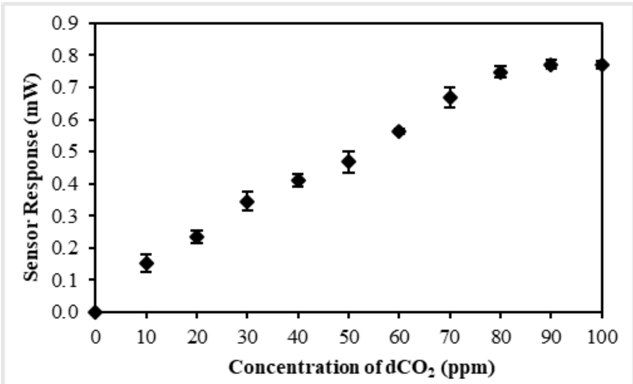


**FIGURE 9. Repeatability of the $dCO_2$ sensor probe based on different range of $dCO_2$ solution concentrations.**

### D. REPEATABILITY OF THE SENSOR

Sensor repeatability is defined as the consecutive runs made using a single sensor to ensure the ability of the sensor to produce the same response for continuous measurements. The repeatability of the sensor was carried out by using the sensor probe coated with 1.0 M ZnO for detection of dissolved $CO_2$. Five (5) measurements were taken for each $dCO_2$ concentration in the range of 10 to 100 ppm as shown in Figure 9. A good repeatability was observed for every $dCO_2$ concentrations used with small magnitude of error which has the standard deviation in the range of 0.008 to 0.027. The average calculated resolution for this sensor is 4.595 ppm.

## IV. CONCLUSIONS

In this work, we demonstrate a tapered optical fiber coated with ZnO nanorice for $dCO_2$ sensing. Ethanol was used as the solvent to solvate the ZnO nanoparticles. The chemical interaction between ZnO nanoparticles and ethanol produces a nanorice-like structure of ZnO. A good linearity range was obtained for the fiber coated with ZnO nanorice (0 – 40 ppm) in comparison to the bare tapered optical fiber (0 – 20 ppm) due to the more adsorption sites available contributed by the ZnO nanorice on the surface of the optical fiber, hence lead to efficiency of $dCO_2$ adsorption. Different concentrations of ZnO solution affect the distribution of the ZnO nanorice on the surface of the optical fiber, as observed in FESEM images. An increase in ZnO solution concentration produces a well-distributed ZnO nanorice on the surface of the tapered optical fiber, as shown by the 1.0 M of ZnO solution concentration. This leads to a higher interaction between the ZnO nanorice with the $dCO_2$, hence shows a good linearity range of the sensor in the range of 0 – 60 ppm ($R^2$=0.972). The sensor shows a response and recovery times with $t_{90}$ values of 0.47 and 1.70 minutes, respectively. The developed sensor also shows a good repeatability results with the standard deviation in the range of 0.008 to 0.027. The average resolution of the sensor is 4.595 ppm.

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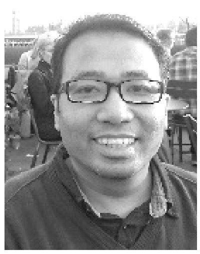

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