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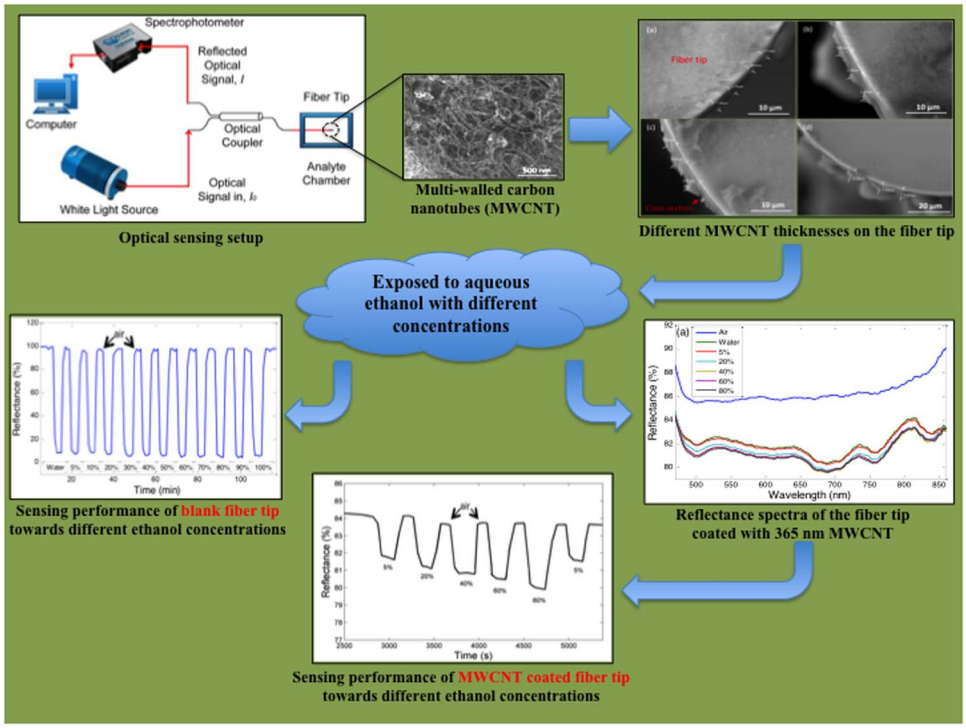

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Reflectance Response of Optical Fiber Coated With Carbon Nanotubes for Aqueous Ethanol Sensing

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Abstract: Ethanol is a highly flammable chemical and is widely used for medical and industrial applications. In this paper, optical sensing performance of aqueous ethanol with different concentrations is investigated using multimode fiber coated with carbon nanotubes (CNT). The multimode optical fiber tip is coated with CNT via a drop-casting technique and is annealed at 70 °C to improve the binding of the nanomaterial to the silica fiber. The optical fiber tip and the CNT sensing layer are microcharacterized using field emission scanning electron microscopy, Raman spectroscopy, and X-ray diffraction techniques. The reflectance response of the developed fiber sensor is measured using a spectrophotometer in the optical wavelength range of 500–800 nm. Upon exposure to ethanol with concentration ranges of 5%–80%, the sensor reflectance reduced proportionally. The dynamic response decreased by 4% when the sensor is exposed to ethanol with concentration of 80% in distilled water. It is found that the sensor shows fast response and recovery as low as 38 and 49 s, respectively.

Index Terms: Optical fiber sensor, multimode fiber, carbon nanotubes, ethanol sensing, reflectance.

1. Introduction

Carbon nanotubes (CNT) are widely used for the improvement of electrochemical sensors [1]. The structures of CNT have been organized in two categories: multi-walled carbon nanotubes (MWCNTs) and single-walled carbon nanotubes (SWCNTs) [2]. They are attractive materials due to the capability of detecting various types of chemicals. Excellent chemical/mechanical stability and optical-electrical features are some of the other attractive and unique properties of the CNT [2]–[7]. Optical fiber sensors have exceptional advantages such as immunity toward

TABLE 1

Preparation parameters of CNT and substrates

Preparation parameters	
Temperature (°C)	70
Annealing time (min)	15
Substrate structure	MMF & SiO_2
MMF tip diameter (μm)	125
Surface area of the fiber tip (m^2)	1.97×10^{-6}

electromagnetic interference, temperature, and large bandwidth, as compared to other sensing platforms [8]. It is known that sensitivity increases by combining nanomaterial with optical fiber sensors [9]. However, the integration of CNT with optical fiber as a chemical sensor has yet to be fully explored.

Ethanol (C_2H_5OH) sensors are widely implemented in the industrial areas. They are employed to detect ethanol leakages in the areas of biomedical, chemical, and food industries [3], [10]–[12]. Apart from that, ethanol sensors are also deployed as analyzer for health tests and in scientific laboratories. Nevertheless, most of the ethanol sensors are based on the electrical domain. Only a few studies are reported on the ethanol sensors based on the optical domain [13].

Most of the existing works focus on the electrical based sensor coated with CNT. Penza *et al.* [14] have designed and characterized surface acoustic waves (SAW) based sensors coated with either SWCNT or MWCNTs toward chemicals at room temperature. The CNT films were deposited by a spray-painting method. Yeh [10] has developed an electrical sensor prototype using CNT synthesized by thermal and chemical vapor deposition. The sensor prototype was investigated for its sensitivity toward different chamber pressure of ethanol vapor at room temperature. In this research, a highly sensitive ethanol sensor based on optical multi-mode fiber tip coated with CNT via drop-casting technique is developed. The effects of the structure and morphology of the CNT films on the fiber tip are also discussed. The optical sensing performance under investigation includes the dynamic reflectance response of the fiber coated with CNT thin films toward different concentrations of ethanol (5%–80%) in distilled water.

2. Experimental Details

2.1. Deposition of CNT Nanostructured Thin Films

The CNT powder (from Hangzhou Company, China) is soaked in nitric acid (HNO_3) as a treatment process in order to obtain functionalized CNT which can be easily dispersed in acetone [15]. Subsequently, the acid soaked CNT powder is dispersed in acetone by ultrasonication process. The concentrations of CNT used in this experiment are 0.002 g/ml, 0.008 g/ml, 0.014 g/ml, 0.02 g/ml, and 0.026 g/ml deposited onto silica (SiO_2) substrates via drop-casting technique for characterization purposes and on the multimode fiber (MMF) tip for ethanol sensing investigation. Prior to the CNT deposition, the SiO_2 wafer substrate and the fiber tip are cleaned with alcohol and heated in an oven at 70 °C for 6 min. to enhance the binding of the nanomaterial. After drop casting the CNT, the samples are annealed up again in the oven for 15 min at 70 °C and left to dry in the air to improve the film adhesion. Table 1 summarizes the details of the CNT deposition and substrates parameters. The different concentrations of CNT are drop-casted on optical fiber tips, while the other parameters (temperature, volume, and annealing time) are kept constant in order to analyze the thickness of the CNT layer.

The micro-characterization results of the CNT thin films in this experiment are investigated employing techniques such as field emission scanning electron microscopy (FESEM), Raman and X-ray diffraction (XRD) analysis. The morphology of the CNT thin films is studied using a FEI Nova NanoSEM 230. The FESEM images are also characterized using ImageJ (image processing software) to extract the average diameter of the nanotubes. Raman analysis is performed using a laser of 514 nm wavelength (Horiba Jobin Yvon, LabRam HR800). Optical properties of the sensor are measured with Ocean Optics spectrophotometer.

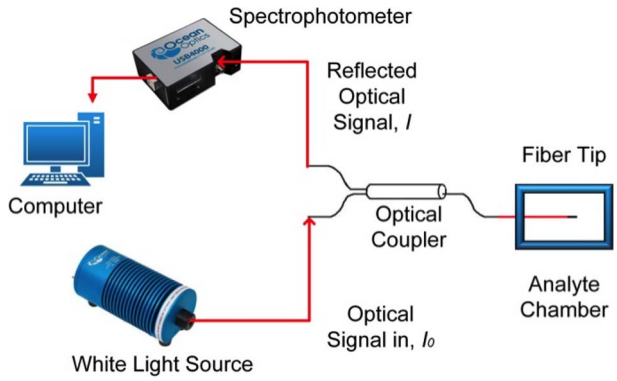

Fig. 1. Optical sensing setup.

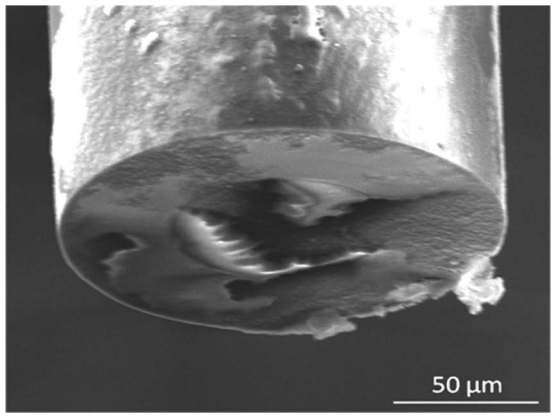

Fig. 2. FESEM image of the MMF tip coated with the CNT layer via the drop-casting technique.

2.2. Experimental Setup

In this experiment, a standard MMF fiber tip with core and cladding diameter of 60.2 and 125 μm, respectively, is deployed as the transducing platform. Different concentrations of ethanol are prepared by diluting ethanol with appropriate amount of distilled water and followed by rigorous stirring. The concentrations of ethanol in water are varied from 5%–80%. The coated fiber tip is immersed in the ethanol with different concentrations. Fig. 1 shows the experimental setup for the reflectance measurement of the fiber tip. It consists of tungsten halogen white light source (HL-2000, Ocean Optics, USA) with wavelength emission range of 360–2500 nm, a spectrophotometer (USB4000-VIS-NIR, Ocean Optics, USA) with spectral response from 200–1100 nm, 2 $\times$ 1 coupler (50 : 50 ratio), and a personal computer. The spectrophotometer is linked to the computer system via a USB communication port. The reflected light from the optical sensor passed through the coupler and collected by the spectrophotometer. Subsequently, the signal from the coated fiber tip is processed by the computer via SpectraSuite software.

3. Results and Discussion

3.1. CNT Film Characterizations

The morphologies of the CNT thin films are observed via FESEM. The FESEM is also used to characterize the MMF fiber coated with MWCNTs, as shown in Fig. 2. This image shows the layer of the MWCNTs coated on the fiber tip. Fig. 3 shows the image of the CNT deposited on the SiO_2 substrate. A densely entangled bundled structure of MWCNTs can be seen clearly in

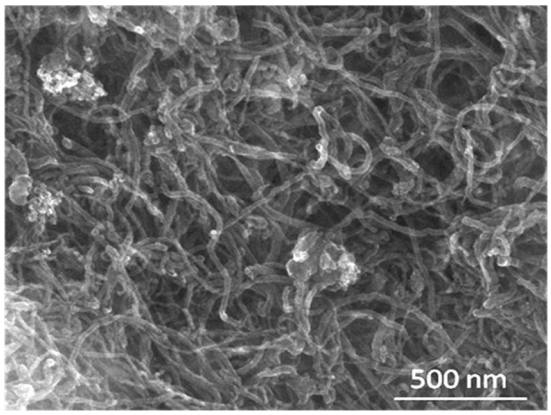

Fig. 3. FESEM image of CNTs deposited on the SiO_2 substrate.

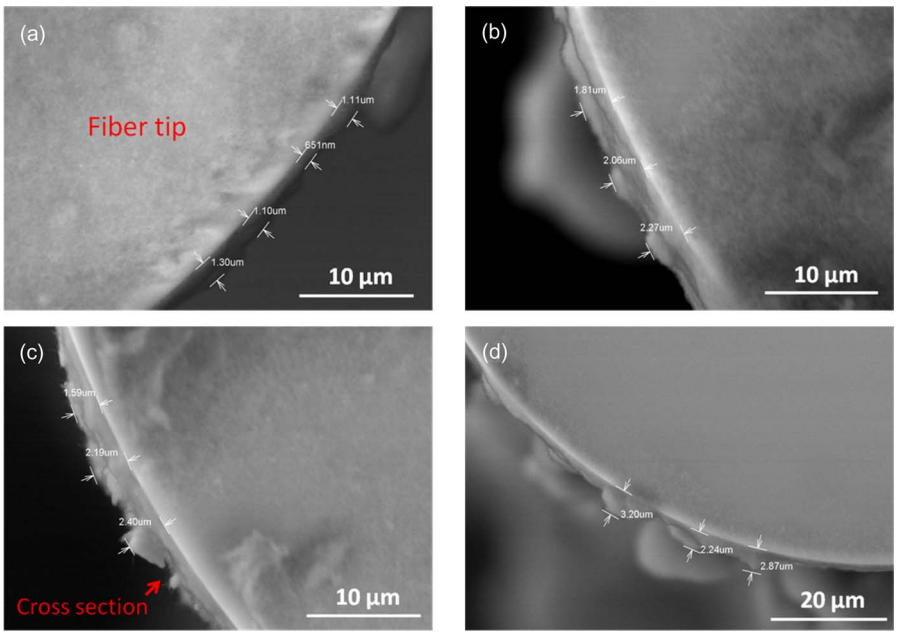

Fig. 4. Cross-sectionalFESEM images of CNT layers deposited on optical fiber tips for different CNT concentrations. (a) 0.008g/ml, (b) 0.014 g/ml, (c) 0.02 g/ml, and (d) 0.026 g/ml.

the FESEM image. The CNTs are organized in bundles with individual nanotubes arranged in a dense net with a good adhesion to the substrate as similarly reported elsewhere [16]. The FESEM image is also processed using ImageJ to extract the average diameter of the tubes that is approximately ~24 nm. The thickness of the CNT layer on the fiber tip as in Fig. 2 is estimated to be 365 nm for the CNT solution of 0.002 g/ml. Different CNT layer thicknesses are achieved for the CNT deposition with different concentrations. Fig. 4 shows the cross-sectional FESEM images of the optical fiber tip coated with CNT layer deposited with concentrations of (a) 0.008 g/ml, (b) 0.014 g/ml, (c) 0.02 g/ml, and (d) 0.026 g/ml. The average thickness of the sensing layer measured is 1.03 μm, 2.05 μm, 2.06 μm, and 2.77 μm for the CNT concentrations of 0.008 g/ml, 0.014 g/ml, 0.02 g/ml, and 0.026 g/ml, respectively. In general, the thickness of the sensing layer increases with the increase of the CNT concentrations deposited onto the fiber tip.

Raman spectroscopy with laser wavelength of 514 nm (Horiba Jobin Yvon, LabRam HR800) is used for the characterization of the MWCNTs. Fig. 5 shows the Raman spectra of CNT on

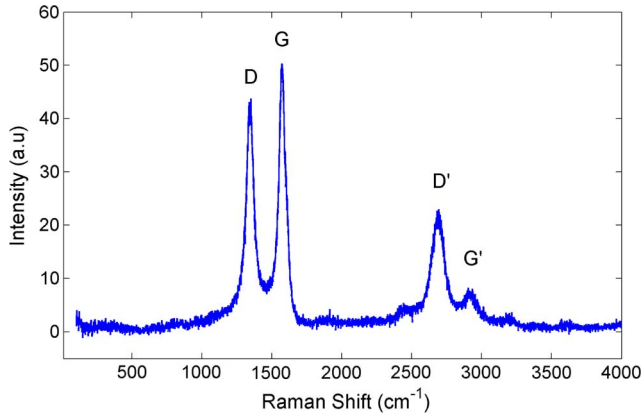

Fig. 5. Raman shift of the CNT nanostructured thin films.

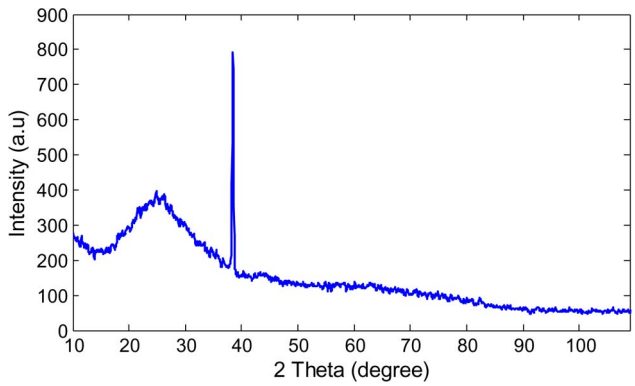

Fig. 6. XRD patterns of CNT nanostructured thin films.

SiO_2 substrates. It shows the sharp peaks that are the distinguishing features of the MWCNTs. The D-band is located at 1350 cm^{-1}, and it is as a result of disorder occurred from the tubes. The G-band is located at 1600 cm^{-1} which is corresponds to graphitic and well-ordered carbon atoms [17] and [18]. The tangential stretch of G-band is one of the most significant features in Raman spectra for CNT. Another sharp peak is also visible at 2700 cm^{-1}, which is the D'-band and the second order disorder as a result of the overtone of the D-band. A small peak is also visible at 2950 cm^{-1} which is called the second order G'-band. The appearance of the G'-band is also an indication of high purity carbon nanotubes [17]. The I_d to I_g ratio is 0.9, which indicates highly graphitized and less defective carbon nanotubes [18].

The MWCNTs deposited on SiO_2 substrates is confirmed by XRD, as presented in Fig. 6. The patterns of the CNT, typical of well-crystallized material, revealed the presence of two peaks at 26.4° and 39.3°. These peaks correspond to the inter-graphitic interlayer spacing of the nanotubes d (002) and to the d-spacing of the family planes (100), respectively [19]–[21].

3.2. Optical Reflectance Spectra Performance

Fig. 7 shows the reflectance spectra of the MMF tips coated with different CNT thicknesses when exposed to air, distilled water and different concentrations of ethanol (5%–80%) at room temperature. Overall, the magnitude of the reflectance in the fiber sensors is reduced with the increase in the CNT thickness. This indicates that the CNT layer absorbs more light as its thickness is increased. The reflectance spectra of the CNT coated fiber tips also decrease when exposed to increasing concentrations of ethanol. The interaction between the CNT layer on the

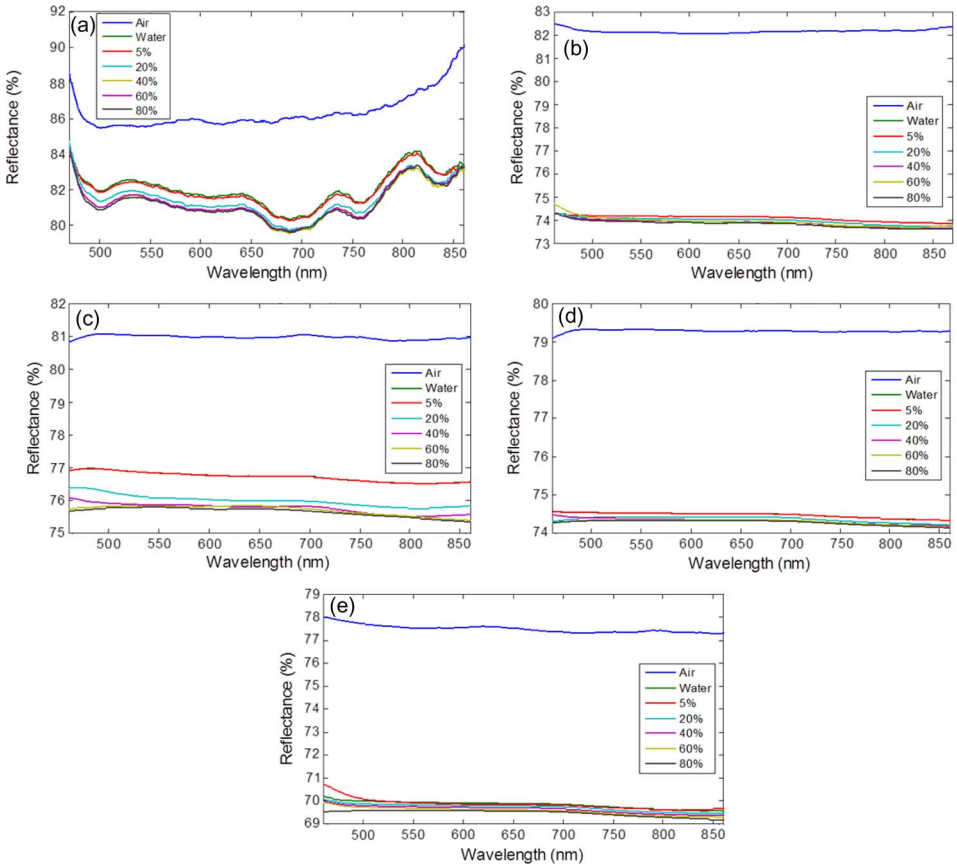

Fig. 7. Reflectance spectra of the fiber sensors coated with different CNT thicknesses of (a) 365 nm, (b) 1.03 μm, (c) 2.05 μm, (d) 2.06 μm, and (e) 2.77 μm, toward air, water, and different ethanol concentrations.

optical fiber tip and ethanol of different concentrations changes the optical characteristic of the CNT films and resulting in the proportional responses of the developed sensors. Fig. 7(a) shows a significant depth of reflectance spectrum at wavelength range 660–740 nm. It is expected that the reflection depth around this range is due to over-tone and combination tone of O-H stretched reflection centered at approximately 690 nm with the reflectance of 79.5% [22]. The peak around 825 nm is likely due to the induced reflection of water [23]–[25]. It is suggested that the hydrogen bonding of the functionalized groups on CNT with reflected molecules is responsible for this response [10].

Fig. 7(a) also indicates significant change of reflectance response for an MMF tip coated with CNT layer of 365 nm thick upon exposure to different ethanol concentrations. It is suggested that high ethanol molecules adsorption onto the sensing layer changes the CNT refractive index and induces a strong change in the evanescent field, as well as signal reflected at the tip [26] and [27]. However, the penetration depth of the evanescent field is typically small, and its change is diminishing in the fiber tips with thicker CNT layer (> 1 μm), thus, making the tipless sensitive upon ethanol exposure. As a result, the reflectance response is less significant for the tips coated with CNT thickness higher than 365 nm, as shown in Fig. 7(b)–(e).

3.3. Ethanol Sensing Response

The presence of ethanol is detected by both optical fiber tips without and with CNT thin films, as shown in Figs. 8 and 9, respectively. The tests are conducted at room temperature. Fig. 8

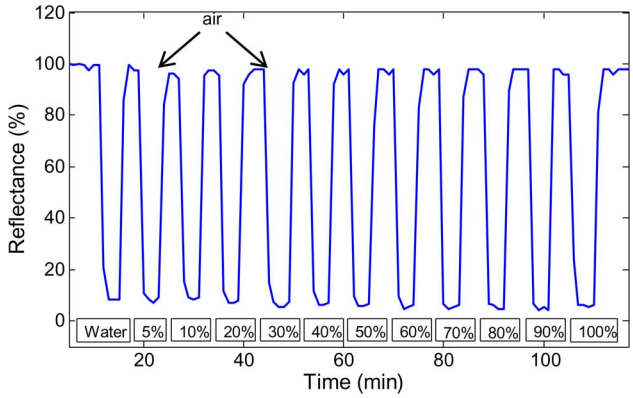

Fig. 8. Dynamic response of the uncoated fiber sensor toward distilled water and different concentrations of ethanol (5%–100%), integrated over wavelength range of 500–800 nm.

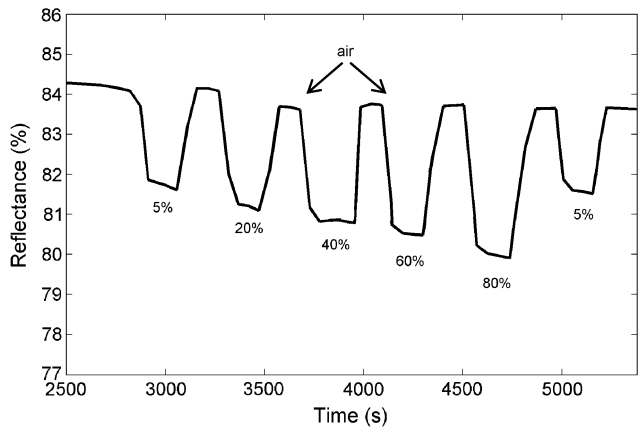

Fig. 9. Dynamic response of fiber tip sensor coated with 365 nm CNT layer toward different concentrations of ethanol, integrated over wavelength range of 500–800 nm.

shows the dynamic response of blank MMF tip exposed toward distilled water and different concentrations of ethanol in water. The measurement is taken by integrating the spectrum over a wavelength range of 500–800 nm. It indicates the optical fiber tip is sensitive toward ethanol and water with respect to air. However, the response from the blank fiber tip does not distinguish the different ethanol concentrations and water. The responses are expected to be solely due to the change in the ethanol and water refractive indices.

As reported in Fig. 7, a significant spectral change is shown by the fiber tip with CNT layer of 365 nm. Hence, the dynamic response is carried out for the tip with the specified CNT thickness. As illustrated in Fig. 9, the dynamic response of the MMF fiber tip coated with 365 nm CNT layer is proportional to the different concentrations of ethanol. The response from the CNT coated fiber tip changes according to the ethanol concentrations in the forward order. The proportional responses are expected due to both chemisorption between ethanol-CNT and the refractive index changes. A significant decrease in the reflectance is observed with respect to the response time. However, the reflectance response is decreasing with the increase in the ethanol concentrations. The dynamic reflectance response decreased by 2.3% and 4% when the sensor is exposed to 5% and 80% of ethanol, respectively. The sensor is fully recovered and returned to its initial baseline upon exposure to air. In general, the response time for the CNT coated fiber tip is less than 38 s, while the recovery time is less than 49 s. The response and recovery of the developed sensor are fast and stable.

TABLE 2

Sensitivity of the CNT coated fiber sensor tip toward different ethanol concentrations

Ethanol concentration (%)	Reflectance (%)	ΔC	ΔR	S (ΔR/ΔC)
5	81.6	-	-	-
20	81.2	15	0.4	0.026
40	80.7	20	0.5	0.025
60	80.3	20	0.4	0.02
80	79.9	20	0.4	0.02

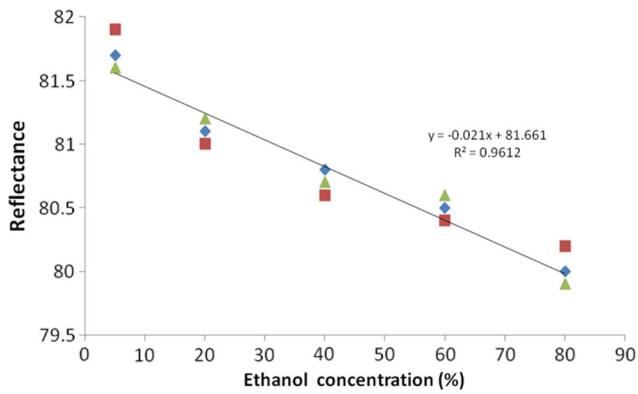

Fig. 10. Reflectance changes of the CNT coated fiber tip against ethanol concentrations.

3.4. Sensitivity, Repeatability and Linearity of the Ethanol Fiber Sensors

The developed sensor shows high baseline stability, sensitivity and repeatability. The sensitivity is mainly determined by the efficient interaction between the target analyte and the surface area of the material sensing layer. Sensitivity S is defined as the slope of the curve given by $S = R/C$, where R is the sensor response, and C is the analyte concentration in %. High surface area to volume ratio of the materials enhances the interaction between adsorbed analyte and the sensor surface, boosting the sensitivity of a sensor [28] and [29].

Table 2 summarizes the sensitivity of the fiber tip coated with 365 nm thick CNT layer toward ethanol. The fiber sensitivity is consistent (0.02) for all ethanol concentrations. The results indicate stable sensing performance of CNT coated optical fiber tip toward ethanol. Fig. 10 summarizes the reflectance response of the developed fiber sensor. The reflectance for the sensor is observed to correspond with the concentrations of ethanol and the results are consistent even after multiple ethanol cycles. The sensitivity of the CNT coated fiber sensor is measured to be 0.021/vol% and matched with the sensitivity in Table 2 with a slope linearity of more than 96%. The error bar is calculated to be 4.02%. Overall, the results show that the interaction of different ethanol concentrations with the CNT thin films make the sensor highly sensitive when compared to blank fiber. The result reveals a promising performance of CNT coated optical fiber tip as an aqueous ethanol sensor.

3.5. Ethanol Sensing Mechanism

The ethanol sensing mechanism for ethanol was proposed by Ouyang and Li [30]. It is suggested that the polar COOH groups attached onto the nanotube surface give stronger response toward ethanol. The absorption efficiency of ethanol molecules increases due to the fact that there are dipole-dipole interactions (mainly hydrogen bonding) between the COOH groups on the CNT and the polar organic molecules of ethanol. When the OH group of ethanol molecules in the liquid chamber and the -COOH group attach to the CNT interact through hydrogen bonds,

it consequently leads to the dynamic change of the ethanol sensor output. The main chemical reaction is given below [30]: $-COOH + OH^- \rightarrow -COO^- + H_2O$.

As a conclusion, the O-H stretch of the water molecules is reduced due to the presence of carbon molecules from ethanol.

4. Conclusion

An optical sensor using MMF tip coated with CNT nanostructured thin films for aqueous ethanol is successfully developed. CNT nanostructured thin films as the sensing layer is deposited by drop-casting technique onto fiber tip. Upon exposure to air and different ethanol concentrations at the room temperature, the sensor reflectance decreases as the concentrations increase. The CNT thickness around 360 nm is identified to demonstrate high sensing performance even for low ethanol concentrations as compared to thicker films $(> 1\ \mu m)$. The sensor showed high sensitivity and fast and stable responses in the visible spectrum range at room temperature. The response and recovery times of the sensor are about 38 and 49 s, respectively. The operation of the optical fiber sensor is suggested due to the chemisorption of ethanol onto the CNT films changes the film refractive index and the reflected signal on the tip. The ethanol sensing performance of the developed fiber sensors at room temperature indicate their potential for applications in the biological, chemical, health, and food industries.

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