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## Original Article

# CNT-based tapered optical fiber for ethanol remote sensing over 3-km optical fiber



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## ABSTRACT

Certain organic liquids like ethanol in water are considered hazardous and have an enormous environmental impact since it is toxic and classified as class I flammable liquids. Remote sensing with complex sensors is a common technique for detecting and tracking spillages of hazardous spillages. Most of the applied remote sensing methods suffer from location and control issues that force the user to be at the exact sensing spot during operation. The present work introduces a simple and highly sensitive tapered multimode optical fiber (TMOF) sensor coated with carbon nanotubes (CNT) for flammable liquids remote sensing applications. The new proposed sensor ability to transfer signals to a remote data collection center of about 3 km from the sensor location was investigated. Ethanol was utilized as the index solution to be tested in the present work. The proposed sensor was attached to 3 km multimode silica optical fiber and characterized towards different concentrations of ethanol in de-ionized water at room temperature. Various characterization techniques have investigated the detailed structural properties of the sensing layer. The experimental results demonstrated that the proposed remote sensor exhibits rapid response with recovery times of 8.7 s and 18 s, respectively, and relative absorbance of 26% upon exposure to 100% ethanol. The sensor attains an overall sensitivity of 1.3/vol% towards low ethanol concentrations in water (0.01–0.5%). Besides, the optical sensor manifests outstanding repeatability when exposed to another cycle of ethanol with concentrations of 20% and 40% in de-ionized water. The proposed optical remote sensor's superior performance via low cost and simple techniques indicates its high efficiency for ethanol detection in various industrial applications.

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## 1. Introduction

Ethanol is a clear, colorless, flammable, and one of the typical volatile organic compounds used in many applications directly attached to daily human activities, including fuel, beverage, food, and pharmaceutical industries [1,2]. Recently, ethanol production has risen substantially due to the rise in demand for hand sanitizers due to Coronavirus's ongoing pandemic (COVID-19). This growing demand for ethanol production will raise concerns regarding possible misusing practices by individuals and industrial facilities, causing massive spillage and pollution challenges in the near future. Standard ethanol sensors are electrically based and widely reported in the chemical sensing literature [3–5]. For example, an electrical-based ethanol sensor was reported using two Au electrodes coated with SnO<sub>2</sub> as a sensing layer prepared by a facile hydrothermal method [6]. The sensor showed a response of 24.9 with rapid response and recovery times of 3 s and 24 s, respectively, toward 100 ppm of ethanol at 230 °C operating working temperature. Kuchi and co-workers introduced an electrochemical ethanol sensor based on a different ratio of PbS: SnS<sub>2</sub> nanocomposite was investigated at room temperature [4]. They showed that the reported sensor's response was 45.64–100.3% upon exposure to 60–1600 ppm of ethanol. On the other hand, Dimitrov and his team introduced an electrochemical sensor using two plane-parallel electrodes coated with ZnO and ZnO doped with copper (Cu) thin films [7]. Even though these sensors are considered as an economically feasible solution to the ethanol detection problem, but they still suffer from several drawbacks that restrict their use in practical applications, for instance, no remote sensing facility, high operating temperature, low selectivity, and prone to electromagnetic interference that can be addressed by an optical sensor.

Many of the mentioned drawbacks associated with the electrical-based sensors are addressed using chemical sensing aided by optical fibers. Optical fibers chemical sensors attracted much attention in the past few years due to their low manufacturing and operation costs, compact size, ability to work in a harsh environment, and remote and distributed sensing [8–10]. The main concept of optical fiber remote chemical sensing is to continuously detect, observe and verify

the behavior of target chemical from a central station located away from the sensor's critical site without the need for electrical power feeds in the remote locations. Thus, it allows instant hazard detection compared with the currently used electrical remote sensor, which suffers from complexity and slow response times [11].

Manipulating the optical fiber structure can further improve the sensing performance by increasing the light travels in the fiber core and/or having a long interaction length [12]. Therefore, many configurations have been reported, for example, uncladded [13], tapered [14], and side-polished optical fibers [15]. Among these configurations, tapered optical fiber is gaining more attention in the sensor designs due to its simplicity of fabrication and ability to operate without a complex setup [8,16,17]. Moreover, the tapered fiber small waist diameter can improve the sensor performance by promoting the interaction of evanescent waves with the external medium. Besides, the loss of light propagation is minimal due to its uniform cylindrical structure [18]. Thus, tapered optical fiber sensors represent a suitable candidate for a seamless sensing system solution with exceptional performance.

Nanomaterials have proven their efficacy in improving the performance of optical sensors for chemical and biological molecules [19–22]. Several reports have found that coating TMOF with nanomaterials may dramatically enhance the sensor's efficiency [23–26]. The optical sensors show significant sensing performance regarding response time and sensitivity due to the alteration in the chemical, physical, and optical features resulting from the interaction between the active layer coated on the tapered optical fiber and the analyte molecules. Table 1 summarized some of the recently published results of an in-site optical fiber sensor using different optical fiber structures coated with various nanomaterials as a sensing layer to detect ethanol in water. The comparison between the sensor's performance shows that the sensors exhibited good optical performance using simple in-site preparation toward high ethanol concentrations only (5–100%). Despite that, all the listed results are in-situ optical fiber sensors. The limit of detection (LOD) and shelf life for most of the devices reported in the table were not mentioned.

Despite all the distinguished advances and continuous developments reported in remote sensing using optical fibers

**Table 1 – Sensing performance comparison of in-situ optical fiber-based aqueous ethanol sensors with different structures and coatings.**

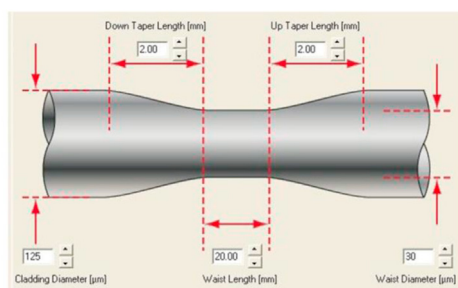
Fiber type and structure	Coating	Sensitivity (/vol%)	Ethanol concentration range (%)	Response time (s)	Recovery time (s)	LOD (%)	Reference
Tapered MMF	GO	11.47	5–40	22	20	–	[28]
Uncladded plastic fiber	CNT	0.68	20–100	6.8	13	0.2	[29]
Tapered plastic fiber	Monolayer Graphene	9.38	10–100	<30	<30	–	[30]
Tapered MMF tip	CNT	0.144	5–80	53	50	0.8	[24]
Tapered MMF	Au–Pd	0.74	20–100	13	6	–	[31]
Tapered MMF tip	GO	0.0275	5–80	19	25	–	[25]
Tapered MMF	Ag/rGO	11.432	1–100	11	6	–	[14]
Tapered MMF	GO	1.33	5–40	15	<20	–	[32]
Tapered MMF tip	rGO	0.126	20–100	40	70	–	[33]

[27], kilometers-based sensors are still not well investigated. An ethanol TMOF sensor over 3 km optical fiber will be introduced and experimentally investigated in the present work. The 3 km optical fiber will be integrated into an TMOF coated with CNT to detect low ethanol concentrations in water via a simple experimental setup.

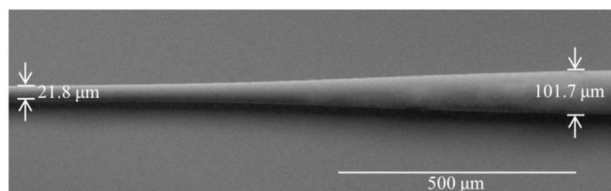
## 2. Materials and methods

### 2.1. TMOF preparation

In this work, a standard multimode optical fiber (62.5  $\mu\text{m}$  core and 125  $\mu\text{m}$  cladding) was tapered via the heat and pull principle using Vytran (Glass Processing System, Model: GPX-3000, USA) optical glass processing workstation. This machine is supported with a real-time system that provides users complete control over the uniformity of the tapers and dimension to obtain a waist diameter and length of 30  $\mu\text{m}$  and 20 mm, respectively, with fixed up and down taper of 2 mm, as illustrated in Fig. 1(a). Concisely, the bare fiber's protective coating was first removed using a fiber stripper for several centimeters and then cleaned with alcohol to eliminate any residuals. The fiber was then placed on the Vytran machine where the area to be tapered just above the filament, and the two ends of the optical fiber fixed to the fiber holding stage. After softening the fiber by the filament heat, the holding stage begins to pull the fiber ends. The power of the filament heater and pulling speed were kept constant at 38 W and 1 mm/s, respectively, to ensure the reproducibility of the tapers. Fig. 1(b) shows the scanning electron microscope (SEM) image of the proposed TMOF. The obtained tapered fiber was kept in a dry cabinet secured onto a sample holder with the tapered area hovering in the middle of the sample holder.



(a)



(b)

Fig. 1 – (a) TMOF tapering parameters, and (b) SEM image of the proposed TMOF.

### 2.2. Preparation and deposition of CNT sensing layer

To formulate carbon nanotubes (CNTs), Pristine Multi-walled carbon nanotubes (MWCNTs) (purchased from Hangzhou Company, China) were chemically treated by sulfuric acid ( $\text{H}_2\text{SO}_4$ ). The formulated CNTs contain different carboxylic acid groups (COOH) contents attached to the surface and the ends of the CNTs, making them ideal for molecular bonding with other materials. Chemically treated CNTs were dispersed in ethanol using ultrasonication for 1 h at room temperature. Before starting with the coating process, the fiber was heated in a 70  $^\circ\text{C}$  oven for 15 min, and then the tapered area was mounted on a hot plate with a temperature of 50  $^\circ\text{C}$  to ensure homogeneous distribution and formation of the CNT sensing layer. CNT solution of 2 ml with a concentration of 0.25 mg/ml was sprayed onto the tapered area via a simple airbrush kit. The sensor was then returned to the oven for 1 h at 70  $^\circ\text{C}$  and kept in a closed environment at room temperature for 24 h to dry before usage.

### 2.3. Measurement setup

Fig. 2 illustrates the ethanol optical sensing setup for the developed TMOF coated with CNT nanomaterial over 3-km optical fiber. A multimode fiber (MMF) pigtail was spliced to one end of the proposed optical sensor and connected to an Ocean Optics™ halogen light source, HL-2000, with the wavelength in the range of 360–2400 nm. In contrast, a 3 km distance MMF was spliced to the other end of the sensor and connected to an Ocean Optics™ spectrophotometer (USB-4000), with spectral range between 200 nm and 1100 nm, through optical fiber cables.

### 2.4. CNT micro-nano characterization

Field emission scanning electron microscope (FESEM), energy-dispersive X-ray spectroscopy (EDX), atomic force microscope (AFM), Raman spectroscopy analysis (WITec, Alpha 300 R) using a laser excitation source with  $\lambda = 532$  nm), transmission electron microscopy (TEM), and High-Resolution Transmission Electron Microscopy (HRTEM) were all employed to characterize the CNT coating. The CNT sensing layer's roughness was also investigated via AFM by covering a portion of the tapered area with aluminum tape during the spray coating process to distinguish between the coated and uncoated area TMOF.

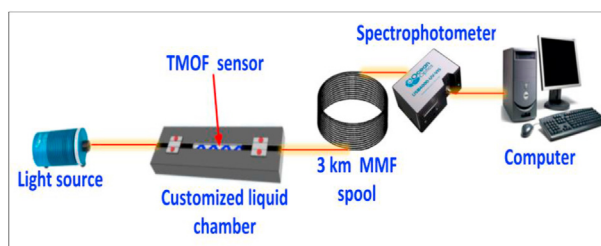


Fig. 2 – Experimental setup.

### 3. Result and discussion

#### 3.1. CNT characterization results

Fig. 3 shows the FESEM characterization of the proposed optical sensor. Fig.3(a) indicates that the CNT successfully adhered to the surface of the TMOF with no defects, thereby confirming the effective deposition of the sensing layer onto the proposed optical fiber. From Fig. 3(b), it can be observed

that the CNTs appear as long, densely, and tangled nanotubes formed into bundles with high porosity. Therefore, the active layer has a high surface area and provides more sites for the ethanol molecules.

To better understand the deposited material's elemental compositions of the proposed CNT sensing layer deposited onto TMOF, EDX analysis was performed. The inset figure (Fig. 3(b)) reveals the existence of Carbon (C) and Oxygen (O) peaks, which affirms that there is no damage or contamination to the sample.

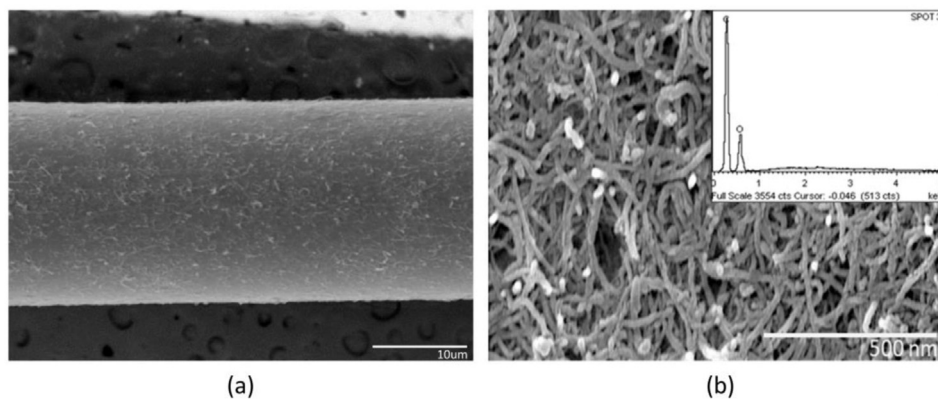


Fig. 3 – (a) FESEM images of the (a) proposed TMOF coated with CNT, and (b) CNT coating. The inset image shows the EDX spectrum of the CNT coating.

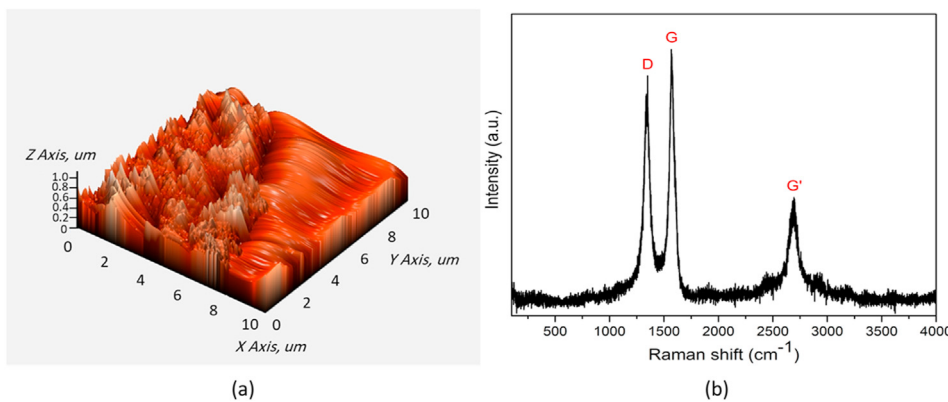


Fig. 4 – (a) AFM 3D image, and (b) Raman spectrum of the CNT sensing layer.

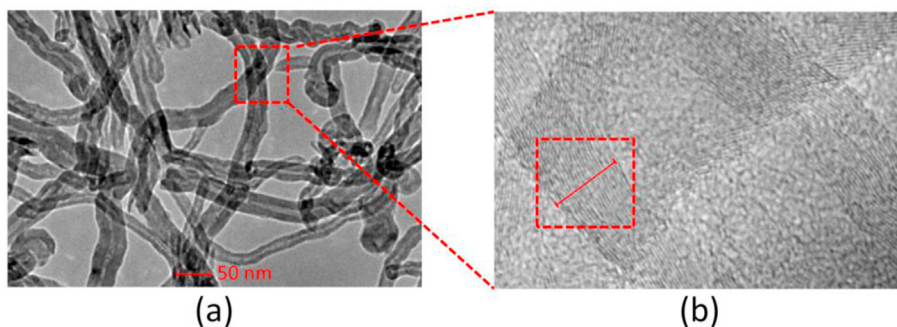
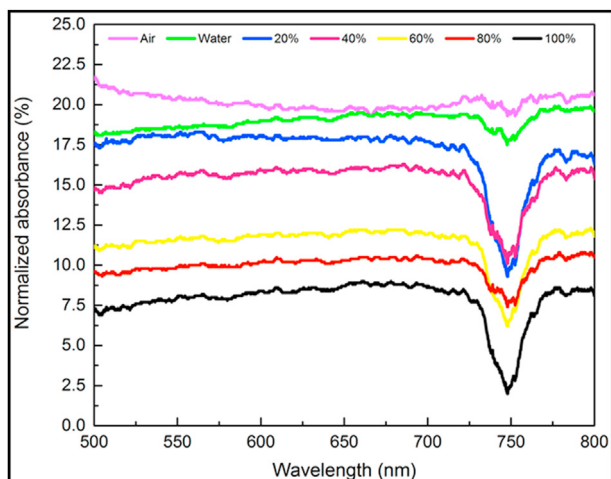


Fig. 5 – (a) TEM image of CNTs, and (b) high-resolution transmission electron microscopy (HRTEM) image of a CNT showing the morphology and structure of the MWCNTs as well as detail showing the number of walls measured for sensing layer.

Fig. 4(a) shows that the coating's average roughness was found to be 60.85 nm, and the thickness of the proposed sensing layer was approximately  $1.8 \pm 0.32 \mu\text{m}$ . On the other



**Fig. 6 – Absorbance vs wavelength of the CNT based TMOF remote sensor exposed to different aqueous ethanol concentrations ranging from 0 to 100% at room temperature.**

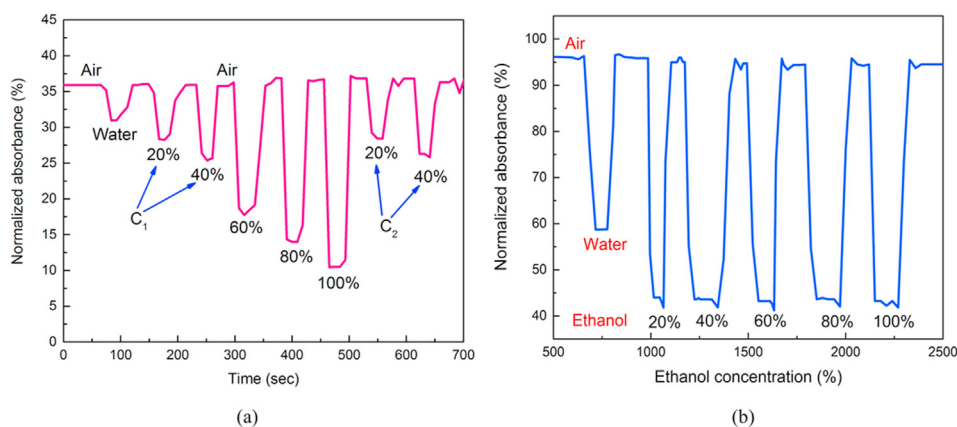
hand, Fig. 4(b) depicts the proposed sensing layer's Raman spectrum, which shows the strong characterization peaks (D, G, and G') of CNT. The D-band located at  $1349 \text{ cm}^{-1}$  proves defects on the CNT sidewalls; thus, the nanotubes exhibited a certain degree of disorder [34]. The G-band, which is located at  $1588 \text{ cm}^{-1}$ , corresponds to the stretching mode of the  $-\text{CC}-$  bond in the graphitic nature of the synthesized sample and indicates that the C atoms are well-ordered. Furthermore, this band is typically the most intense peak in the CNT Raman spectrum [35]. Another peak was observed around  $2700 \text{ cm}^{-1}$ .

This peak is the G' band, which is the overtone of the defect-induced D band resulting from double resonant Raman scattering with two-photon emission [36]. The well-defined intensity of G and D bands indicates that the CNT exhibited a well-ordered stacking of layers [37].

Fig. 5 shows that the CNT's inner and outer diameters were in the range of 9.5 and 26 nm, respectively, and the number of walls was 24, as shown in Fig. 5(b).

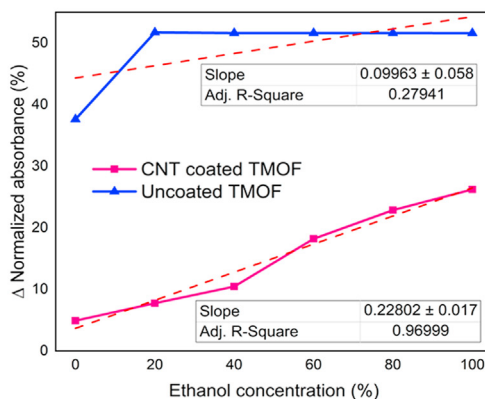
The outcomes confirm that the CNT was pure with no impurities and that it was long, cylindrical in shape, entangled with different bends and curvatures due to its long structural length and hollow in the middle of the tube.

Conclusively, the characterization results show that the formulated CNT completely coated the optical fiber, remained undamaged, and retained its properties after the coating and annealing processes as well as during the tests.



(a)

(b)



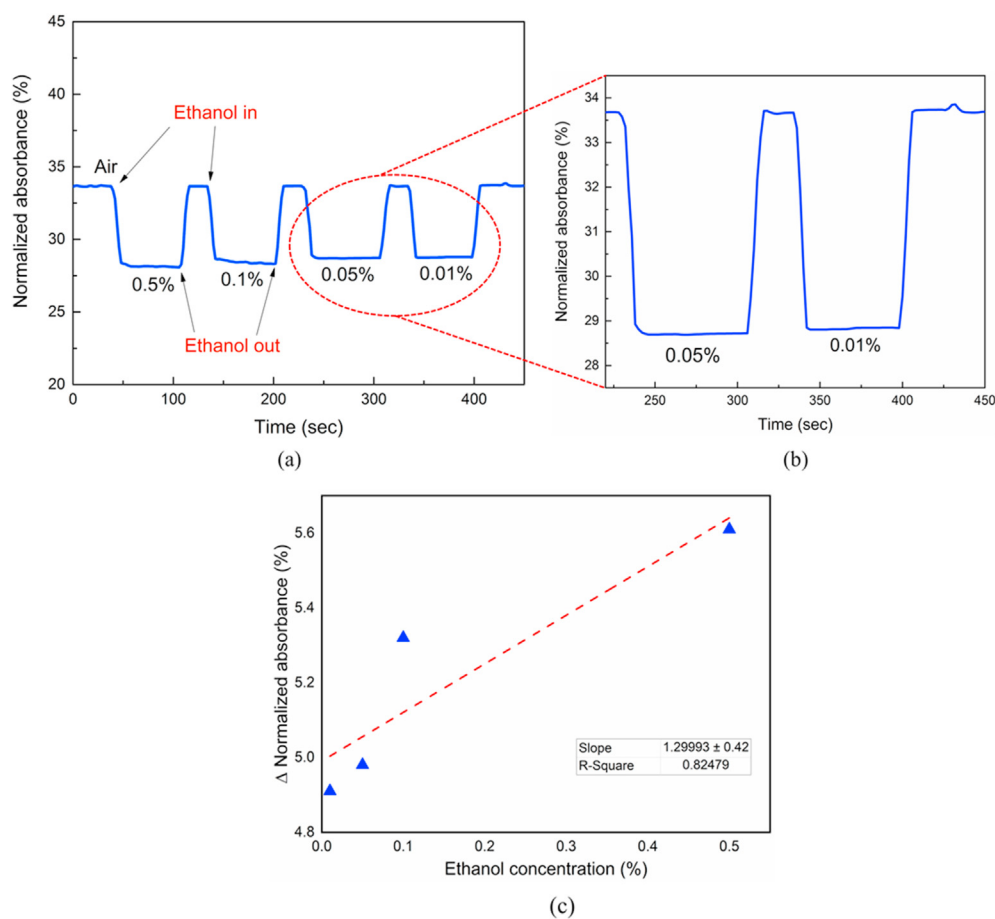
(c)

**Fig. 7 – Dynamic responses of the (a) CNT coated on TMOF and (b) uncoated TMOF exposed to different ethanol concentrations over 3-km optical fiber. (c) Absorbance response change as function of ethanol concentration of the TMOF remote sensor with and without CNT coating.**

### 3.2. TMOF sensor performance

Fig. 6 shows the absorbance versus wavelength of the CNT-based TMOF remote sensor upon exposure to different concentrations of ethanol diluted in DI water ranging from 0 to 100% at room temperature (25–26 °C). The results show that the absorbance decreases significantly as the ethanol concentration increases over the wavelength range between 500 and 800 nm. A noticeable dip occurred in the absorbance spectrum of the CNT coating in the range of 721 nm–774 nm. This can be attributed to the changes in the refractive index of the proposed coating [38]. The TMOF remote sensor's dynamic response was achieved by integrating the absorbance change over a wavelength between 500 and 800 nm. From Fig. 7(a), the absorbance change increased correspondingly with ethanol concentration, which is believed to be due to the combination tone of O–H stretched absorption and the transition of the overtone. In chemical sensing applications, it is essential to calculate the response and recovery times where response time is defined as the duration for the sensor response to rise 90% of the maximum absorbance when target chemical is pumped into the customized testing chamber, whereas recovery time is described as the duration the sensor takes to recover from the maximum absorbance value to 10% above its baseline. For 100% ethanol in water,

the proposed sensor has response and recovery times of 8.7 s and 18 s, respectively, while its relative absorbance was 26%. The CNT based remote sensor exhibits good repeatability when exposed to ethanol concentrations of 20% and 40% as an additional cycle ( $C_2$ ). Fig. 7(b) demonstrates the dynamic response of the uncoated (blank) TMOF integrated with 3-km optical fiber when exposed to different ethanol concentrations over a wavelength ranging from 500 to 800 nm at room temperature. However, the remote sensor shows high sensitivity towards ethanol, the response between different concentrations cannot be differentiated. It is expected that the uncoated TMOF's response towards ethanol was only due to the change of the ethanol refractive index. The sensitivity of the coated and uncoated TMOF remote sensors was approximately 0.23/vol% and 0.1/vol%, with slope linearity of 98% and 27%, respectively, as demonstrated in Fig. 7(c). The CNT coated TMOF remote sensor achieved higher sensitivity than that of the uncoated one and the in-situ tapered fiber tip coated with CNT reported in [24]. It is believed that the superior sensor performance is due to the CNT high surface area and porous structure, which gives a better opportunity for the analyte molecules to diffuse into or out of the sensing layer easily. Besides, TMOF provides a long interaction region between the altitude evanescent field waves, CNT, and ethanol molecules.



**Fig. 8 – (a) Normalized dynamic response of the TMOF based CNT exposed to low ethanol concentrations at room temperature. (b) Enlarged image of the normalized absorbance response towards 0.05 and 0.01% ethanol concentration. (c) Absorbance response change as function of ethanol with low concentration ranging from 0.01 to 0.5%.**

To further investigate the proposed remote sensor's performance, Fig. 8(a) demonstrates the overall normalized optical response of the TMOF sensor exposed to low ethanol concentrations of 0.5%, 0.1%, 0.05%, and 0.01% at room temperature. LOD of the developed sensor was found to be 0.01% ethanol concentration. The absorbance response change was approximately 4.91% for low ethanol concentration of 0.01%, with regard to the baseline of the developed TMOF sensor over 3-km optical fiber. Fig. 8(b) shows enlarged view of the absorbance response towards ethanol concentrations of 0.01 and 0.05% in water. It can be noticed that the proposed sensor demonstrates a small change in its absorbance that result in a detectable increment in the output signal. The optical remote sensor's overall sensitivity was approximately 1.3/vol% towards low ethanol concentrations ranging from 0.01 to 0.5% (Fig. 8(c)).

To investigate the fabricated sensor's shelf life, the CNT-based TMOF sensor's sensing response was evaluated every week for three months. The sensor was stored in a sealed container at 23 °C in a dry cabinet to prevent contamination of the sensor surface. The sensing findings are well demonstrated in Fig. 9. The CNT based TMOF sensor showed

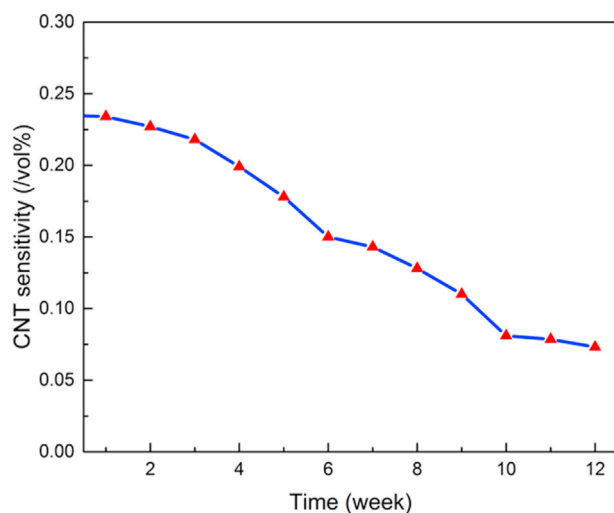


Fig. 9 – Sensitivity of the proposed TMOF sensor-based CNT for 12 weeks.

degradation in sensitivity of approximately 1.56% after three months.

Thus, the developed TMOF sensor based on nanomaterials can retain a long shelf life due to the high chemical stability of the CNT that do not deteriorate over a long period.

It is believed that the physical sensing mechanism of the CNT coating is mainly based on the interaction that occurred between ethanol molecules and the proposed CNT sensing layer. TMOF provides a strong interaction between the evanescent field and the light-sensing material with the chemical analytes. Fig. 10 illustrates the direct interaction between the evanescent field and the CNT coating with ethanol molecules. Thus, different ethanol concentrations cause variation of the measured optical signal. The suggested chemical sensing mechanism of the CNT sensing layer is as follows (see Fig. 10):

- The carboxylic acid groups attached to the CNT coating surface can be produced by treating raw CNT with nitric acid used as oxidizing agents.
- The OH groups of ethanol molecules interact with the COOH bond connected to the CNT surface.
- As a result of the hydrogen bonding of the dipole–dipole interactions among the chemical analyte molecules and the polar groups on the coating surface, the proposed sensor's sensitivity towards ethanol molecules is enhanced.

#### 4. Conclusions

In summary, this work reports a simple and efficient optical fiber sensor based on CNT sensing layer coated onto TMOF operates at room temperature for the detection of ethanol remotely over 3 km optical fiber. A reproducible and straightforward tapering process was utilized to fabricate the TMOF sensing area. The optical sensor is integrated with the CNT sensing layer via the spray-coating deposition technique. The sensor's sensing performance is not only comparable to the studies listed in Table 1; it also achieved the lowest LOD at 0.01% with rapid response and recovery times. The experimental results revealed that the TMOF sensor coated with CNT exhibited high sensitivity and rapid response and recovery times of approximately 0.23/vol%, 8.7 s, and 18 s,

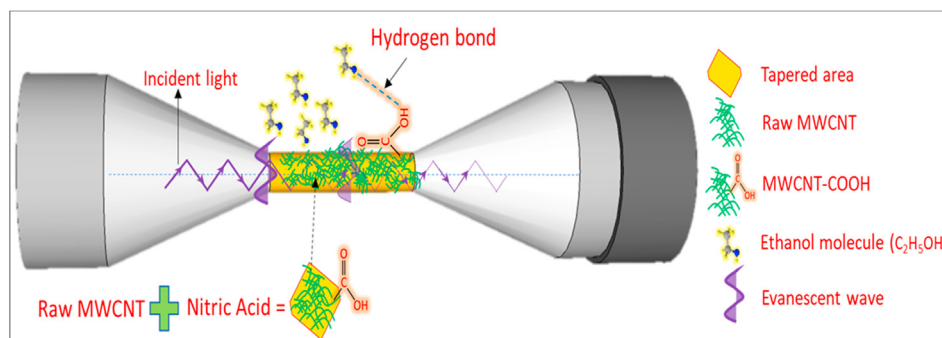


Fig. 10 – Schematic representation of the proposed interaction mechanism between ethanol molecules and CNT sensing layer.

respectively, which endows the proposed remote sensor the ability of precision and fast detection. This can be ascribed to the CNT high surface area and excellent porosity in addition to the exceptional affinity of CNT coating to ethanol molecules. The sensor resolution was calculated to be 0.01% ethanol, with an overall sensitivity of 1.3/vol% towards low ethanol concentrations (0.01–0.5%) in water. The shelf life was also investigated for 12 weeks. The significant performance obtained from the CNT based TMOF remote sensor over 3 km optical fiber distance at room temperature indicates its enormous potential to be employed in real-world applications to ensure safety standards in the industrial sectors.

### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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