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Development of Optical Fiber Sensor for Water Salinity Detection

Muhammad Safwan Mohammed Nor[1], Abdul Ali Khan[1], Suraya Mohamad[1], Punithavathi Thirunavakkarasu[1*]

[1]Communication Technology Section, British Malaysia Institute, Universiti Kuala Lumpur, 53100 Gombak, Malaysia

Abstract. This project addresses the lack of a real-time, low-cost sensor to detect salt levels in water. The authors aim to develop an optical fiber sensor for water salinity detection. The sensor employs the principles of absorption spectroscopy using a broadband light source and spectrometer to detect changes in the optical spectrum of the sensor in the presence of varying concentrations of sodium chloride ions. A D-shaped sensor is fabricated by modifying the circular structure of a plastic optical fiber. Functionalized carbon nanotubes are drop casted over the D-shaped sensing region. Both uncoated and CNT-coated POF sensors are exposed to different concentrations of sodium chloride in water, and the spectral response is recorded. The results show that the sensors exhibited a strong correlation in their intensity response towards varying concentrations of sodium chloride salt ranging from 0 to 25%. The uncoated sensor had a sensitivity of 31A.U./% salt, and the CNT-coated Sensor had a sensitivity of 114 A.U/% salt. The functionalized CNT layer increased the sensitivity of the POF sensor by approximately 4 times. The outcome of this research provides a cost-effective and reliable method for water salinity detection in industrial and environmental applications.

Keywords: Carbon Nanotubes (CNT); D-shape optical fiber; Optical fiber sensor; Sodium chloride ions, Sensitivity; Water salinity detection

1. Introduction

Water salinity plays a crucial role in numerous fields, including environmental monitoring, industrial processes, and agriculture. Precise and continuous measurement of water salinity is essential for effective water resource management and ensuring optimal conditions for various applications. However, conventional sensors are affected by electromagnetic interference, radio frequency interference, and real-time monitoring may also poses potential risks, such as short circuits when used in an aqueous environment (Arrieta, Barrera and Mendoza, 2023; Abuzairi *et al.* 2022; Hardi and Rahman 2020).

To address these constraints, the development of an optical fiber sensor (OFS) for water salinity sensing has emerged as a promising solution. This sensor employs absorption spectroscopy principles and integrates a D-shaped optical fiber sensing zone to detect changes in optical parameters when exposed to dissolved salts. Moreover, the coating of the OFS with carbon nanotubes (CNT) has shown remarkable potential in enhancing sensitivity and improving detection capabilities.

*Corresponding author's email: punitha@unikl.edu.my, Tel.: +603 – 6184 1000; Fax: +603 – 6186 4040
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Optical fiber sensors utilize the interaction between light and the analyte in the surrounding environment to sense alterations in the surrounding refractive index (SRI). The versatility of optical fiber sensors enables various sensing mechanisms such as (absorption-based, reflection-based, evanescent wave, and surface plasmon resonance (SPR)) sensors (Liu and Peng, 2021). Absorption-based sensors rely on the principle that the absorption spectrum of water changes with salinity. The salinity level can be determined by measuring the intensity of light transmitted through an optical fiber immersed in the water sample.

Refractive index [RI] sensors, on the other hand, exploit the refractive index variations caused by changes in water salinity (Tan *et al.,* 2014). Thus making it viable by measuring the intensity or phase of light reflected or transmitted through the fiber. Evanescent wave sensors make use of the electromagnetic field extending beyond the fiber core. When the fiber is exposed to a water sample, the RI of the evanescent field changes due to salinity variations, leading to alterations in the transmitted or reflected light. SPR sensors utilize the excitation of surface plasmons on a thin metal film deposited on the fiber surface.

The interaction between plasmons and the salinity of a water sample leads to changes in RI, which enables the measurement of water salinity levels. When designing optical fiber sensors for this purpose, researchers must consider several key factors. Among these, the choice of fiber is of utmost importance. In this study, plastic optical fibers were selected due to their robustness, large core size, and flexibility. Additionally, the selection of materials for the sensor probe, including coatings or sensing layers, plays a vital role in enhancing sensitivity and stability.

Sensor geometry and configuration have a significant impact on the sensor's performance. Various modifications, such as tapered fibers, long-period gratings, or microstructured fibers, can be used to increase the interaction between the fiber and the surrounding medium, leading to increased sensitivity (Khan *et al.*, 2022). Additionally, techniques such as wavelength modulation, intensity modulation, or interferometry can be incorporated into the sensor design to increase the measurement accuracy and dynamic range.

Great progress has been made in the field of optical fiber sensors for water salinity measurement (Flores, Janeiro, and Viegas, 2019). Researchers have concentrated on the development of novel sensing configurations, enhancing sensitivity and selectivity, and incorporating optical fiber sensors with other technologies for multi-parameter measurements, such as using nanomaterial coatings on the fiber surface to increase sensitivity and enable selective detection of specific ions in saline water. However, fiber sensors have several limitations, such as low mechanical strength, especially in glass fibers, complex modification processes, and low selectivity without a sensitive film.

Researchers have also explored the combination of optical fiber sensors with microfluidics, allowing real-time monitoring of water salinity. This integration offers benefits such as smaller sample volume requirements, rapid response times, and the capability to analyze multiple samples simultaneously. CNTs are now popular as an efficient material for chemical sensing detection due to their distinctive structural and optical properties, such as strong tensile characteristics, extremely lightweight, and chemical and thermal stability (Khalaf *et al.,*2017; Pokhrel *et al.,* 2017). These special characteristics have gained substantial interest in using CNTs in research areas involving emerging nanomaterials and their applications. Optical spectroscopy techniques measure the changes in the RI of the CNTs caused by salt adsorption or desorption processes. Furthermore, functionalization is a chemical process that introduces specific functional groups onto the sidewalls of CNTs. These functional groups can be created using specific molecules or coatings, and they can enhance the selectivity of CNT-based sensors towards particular salts, thereby improving the sensor's performance (Norizan *et al.,* 2020).

Overall, carbon nanotubes provide a promising platform for salt detection, offering high sensitivity, rapid response times, and the potential for integration into miniaturized and portable sensing devices. Further research is being conducted to optimize the sensing performance, selectivity, and stability of CNT-based salt sensors for a broad range of applications, including environmental monitoring, agriculture, and healthcare. In this research, we propose a novel salinity sensor using a D-shaped polymer optical fiber with a carbon nanotube sensing layer. By optimizing the sensor's sensitivity, accuracy, and real-time monitoring capabilities, it holds the potential to offer a more efficient and cost-effective alternative to traditional methods such as microstrip moisture sensor (Jain, 2022) for the detection of salt, where a microstrip antenna is dipped in a solution containing salt, the amount of concentration is detected by the change in reflection coefficient. Additionally, the Terahertz metamaterial sensor (Deng *et al.,* 2022) relies on salt solution coverage and detects concentration changes through shifts in resonant frequency peaks. However, the performance of such sensors can be compromised by electromagnetic and RF interference.

2. Materials and Methods

In this research, MWCNTs 95%, sodium chloride 99%, nitric acid 65%, and sulfuric acid 95% were purchased from Sigma-Aldrich, and a 1000-micron diameter plastic optical fiber (POF) core and cladding made of (poly methyl methacrylate and fluorine resin) purchased from Mouser Electronics (Malaysia) was used to design a D shaped sensor. The POF was then placed in a v groove to secure it, and a fine file was used to polish the fiber down to the required size, as shown in Figure 1 (a). The POF sensing zone was fabricated to be 1 cm in length. After polishing, the cladding of the POF over the sensing region was totally removed. The side view and cross-section diagrams of the D-shaped POF are depicted in Figure 1(b and c) separately, and the resultant D-shaped sensor is depicted in Figure 1(d).

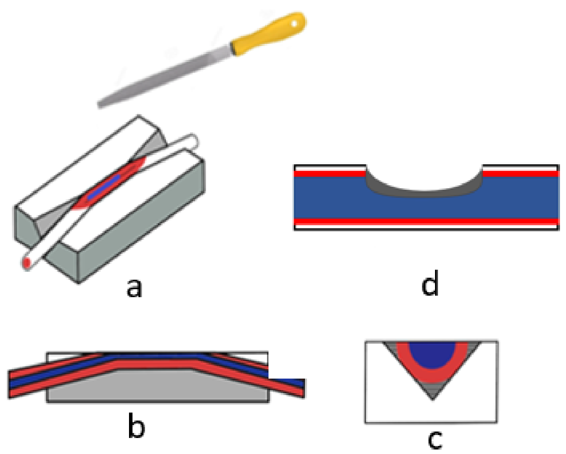

Figure 1 POF in V groove for side polishing process a) Aerial view; b) Side view; c) Cross section view d) D-shaped sensor

To functionalize CNTs, acid treatment was employed using commonly used acids such as nitric acid (HNO_3), sulfuric acid (H_2SO_4), or a mixture of both (HNO_3 + H_2SO_4) (Norizan *et al.,* 2020). The nanotubes were immersed in the acid mixture and subjected to reflux or sonication to introduce functional groups onto their surface. This functionalization process enhanced the compatibility of the CNTs with the fiber material and enabled chemical reactions.

The functionalized CNTs were then deposited onto the fiber's sensing zone using the drop-casting method. The coated fiber was subjected to a heat treatment at 70 degrees Celsius for 2 hours, followed by a gradual 24-hour cooling process at room temperature. This thermal treatment ensured proper adhesion and stability of the CNT coating on the fiber. To characterize the coated fiber, we conducted comprehensive analyses using scanning electron microscopy (SEM) to examine the dispersion and adherence of CNTs on the fiber. Energy-dispersive X-ray spectroscopy (EDX) analysis was performed to determine if the material coated on the fiber was CNT or had undergone any changes or contamination during the functionalization phase. X-ray diffraction (XRD) analysis was also done to study the crystalline phases of the CNTs. SEM analysis at different magnifications allowed for the examination of the CNT thickness, observation of the structure, and verification of proper coating uniformity. The EDX analysis provided valuable information regarding the elemental composition and the presence of impurities in the coated fiber. The XRD analysis yielded essential insights into the crystalline phases present in the carbon nanotubes. By combining the results from SEM, EDX, and XRD analyses, a comprehensive characterization of the coated fiber was achieved, enabling a thorough assessment of the structure, composition, and crystallinity of the carbon nanotubes on the fiber's sensing zone.

Figure 2 shows the sensing setup for this research. The sensing region of the POF sensor was positioned in a liquid chamber. The POF was connected to a broadband light source at one end and a spectrometer at the other end.

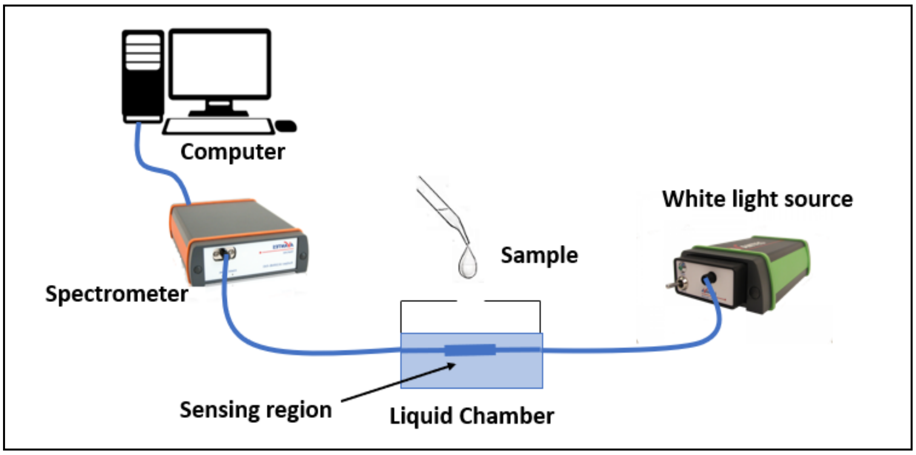

Figure 2 Optical sensing setup for salinity detection

The AvaLight-HAL-S-MINI Tungsten-Halogen Light Source has a spectral range of (400-2000 nm) and the spectrometer (AvaSpec-ULS2048CL) has a range of (400-1100) nm. The resulting data was analyzed using dedicated software on a PC. The sensitivity of the sensor was evaluated by measuring the response to salt solutions of varying concentrations ranging from (0-25)%. The measurements were taken after 20 sec for each concentration repeated for both the uncoated fiber and the CNT-coated fiber. The collected data was further analyzed, compared, and calculated to determine the sensitivity of the sensor.

The described material and method provide a systematic approach for the fabrication, functionalization, characterization, sensing, optimization, and analysis of the optical fiber sensor, with functionalized carbon nanotubes (CNTs) playing an important part in enhancing the sensor's performance for water salinity detection.

3. Results and Discussion

Figure 3 presents the magnified image of the POF that had been coated with CNT. This SEM with a magnification of 90 times shows that the CNT has fully coated the fiber in a uniform manner. There are no uncoated patches or peeling that can be observed. It can be concluded that the POF has been successfully coated with CNT.

A further magnification of 30000 times is presented in Figure 4. In this image, we can observe the CNT structure. The CNTs are tightly packed, and the diameter of the CNTs was measured to be averaging at approximately 40 nms. The twisted structure of the CNT agrees well with others reported in the literature (Norizan *et al.*, 2020).

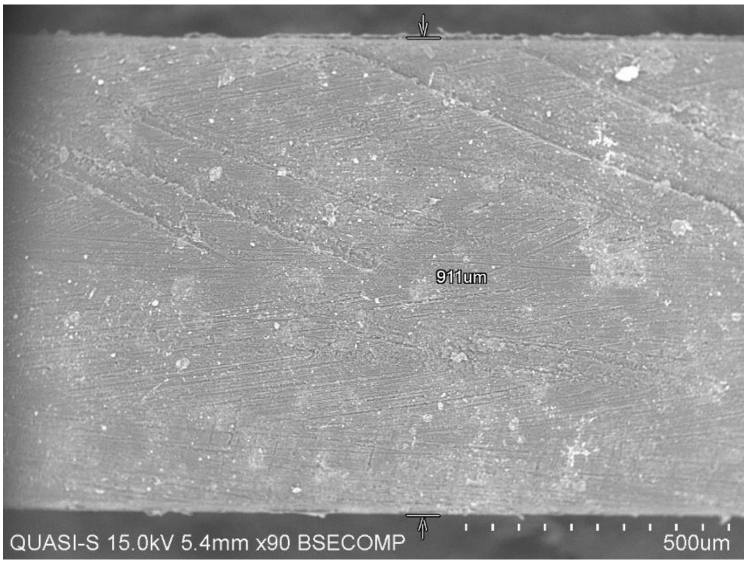

Figure 3 SEM image of CNT on POF with 90x magnification

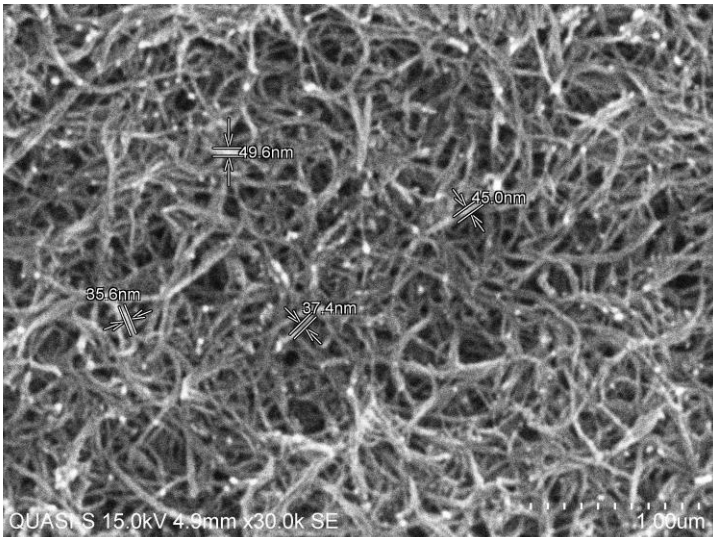

Figure 4 SEM image of CNT on POF with 30000x magnification

Table 1 shows the EDX analysis of the CNT. There are trace amounts of oxygen and sulfur detected in EDX. This could be due to contamination during the annealing and is negligible.

The material contains carbon, which is expected, and there is no other contamination present. The XRD analysis of the CNT material in Figure 5 shows the diffraction peak patterns of multi-walled CNTs. The peak patterns in MWCNTs appear at roughly 26° (002) and 40° (100) with respect to 2θ values. The presence of the 002 peaks suggests a lower degree of alignment among the nanotubes, with a crystal spacing or d-spacing of 0.34 Å, and

100 peak signifies highly aligned nanotubes with a crystal spacing, or d-spacing of 2.12 Å. These results indicate the presence of aligned and unaligned CNTs in the sample.

Table 1 EDX analysis of CNT

Element	Weight%	Atomic%
C K	97.04	97.9
O K	2.45	1.90
S K	0.51	0.20
Totals	100	100

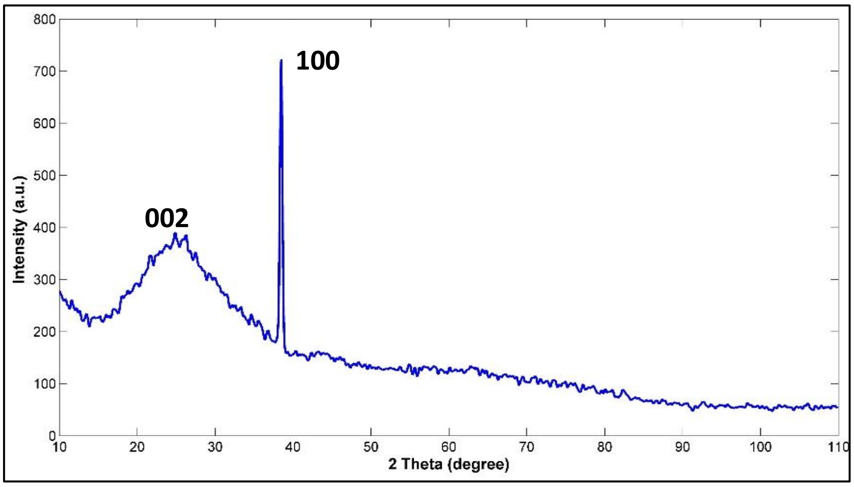

Figure 5 XRD analysis of CNT

Figures 6 and 7 present the intensity results of the uncoated and CNT-coated POF sensor towards different concentrations of salt, respectively. The observed trend reveals that as the salt concentration increases, the intensity spectrum exhibits a corresponding increase. This phenomenon can be attributed to the variations in the RI of the surrounding medium as the salt concentration is increased.

When the CNT-coated POF sensor is deployed, it can be clearly observed that the changes in intensity are more noticeable for each concentration of salt. Due to the high surface-to-volume ratio of nanotubes, it allows more salt molecules to interact. As the salt concentration increases, the salt molecules bind with the CNTs, causing greater changes to the CNT's effective RI.

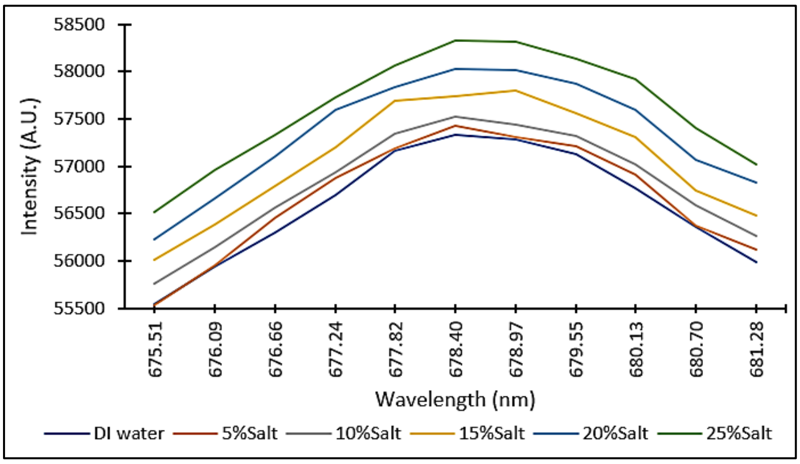

Figure 6 Spectral response of uncoated POF sensor towards salt solutions

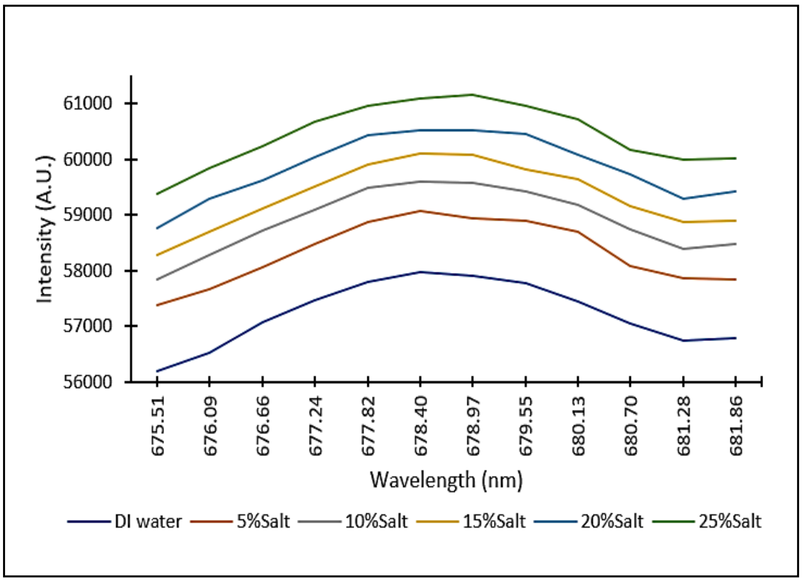

Figure 7 Spectral response of CNT-coated POF sensor towards salt solutions

Figure 8 compares the performance of the uncoated and CNT-coated POF sensor. The results clearly demonstrate the superior performance of the CNT-coated fiber in terms of intensity change when exposed to different salt concentrations.

The results indicate that the CNT-coated fiber demonstrates a significantly larger intensity change compared to the uncoated fiber. This enhanced response is attributed to the functionalization of CNTs, which leads to an improved selectivity towards specific salts. The chemical composition of the functionalized CNTs enables a binding interaction between the salt ions and the CNTs, resulting in a greater difference in the effective RI between the sensor surface and the surrounding medium.

The sensitivity of the uncoated and CNT-coated sensors is quantified using equation 4 (Arasu *et al.* 2016) and summarized in Table 2. It is noteworthy that the CNT-coated sensor exhibits approximately four times higher sensitivity compared to the uncoated sensor. This increased sensitivity is attributed to the unique properties of the functionalized CNTs, which enhance the sensing capability of the fiber. The functionalized CNTs facilitate a more precise and selective detection of salt concentrations by inducing significant variations to the effective RI of the sensor surface.

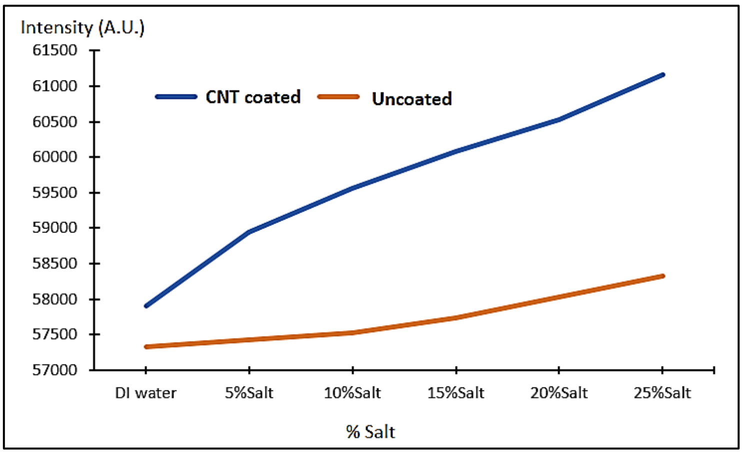

Figure 8 Comparison of the intensity shift of the uncoated and CNT coated sensor

Table 2 Sensitivity Comparison

Type of fiber	Sensitivity (AU/%salt)
Uncoated fiber	31 AU/%salt
Coated CNT fiber	114 AU/%salt

Overall, the results demonstrate the effectiveness of the CNT-coated fiber sensor in detecting and quantifying salt concentrations. The uncoated sensor had a sensitivity of 31A.U./% salt, and the CNT-coated Sensor had a sensitivity of 114 A.U/% salt. The functionalization of the CNTs enhances the sensor's (selectivity and sensitivity) making it a favorable choice for a range of potential applications requiring accurate and reliable water salinity detection.

4. Conclusions

In conclusion, the development of a D-shaped fiber sensor for water salinity detection has proven to be successful. The use of CNT coating through the drop-casting method has significantly enhanced the sensitivity of the sensor, achieving up to 4 times the sensitivity of the uncoated sensor. The CNT-coated fiber sensor's superior sensing capabilities have been established, providing a better sensing mechanism for water salinity detection. This research opens up possibilities for further exploration and improvement in the area of optical fiber sensing. Future work in this area may be focused towards the sensing layer. More research can be done towards determining the optimum thickness of the sensing layer that would produce the highest sensitivity. Varying the sensing layer with other sensitive materials such as graphene, Zinc Oxode, conductive ploymers as well as nanocomposite layers could be another direction for further work in this area. This would broaden the variety of materials for sensor coating and potentially uncovering novel sensing mechanisms. The successful development of the CNT-coated fiber sensor and the demonstrated improvements in sensitivity hold great promise for various applications requiring water salinity detection. Industries such as environmental monitoring, industrial processes, and agriculture can benefit from the cost-effective, reliable, and real-time monitoring capabilities offered by this optical fiber sensor. The simplicity and robustness of this sensor make it easy to deploy in various environmental conditions. The findings of this research contribute to the advancement of sensor technology and provide a foundation for future studies in the field of water salinity detection and related applications.

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