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Advanced Uric Acid Detection by No-Core Fiber (NCF) and Single-Mode Fibre (SMF) Based Sensor Coated with Graphene Oxide

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ABSTRACT

In this paper, optical fibre-based sensors incorporating with no-core fibre (NCF) and single-mode fibre (SMF) were developed for the detection of uric acid (UA). The primary sensor probe was fabricated by sandwiching NCF between two SMF segments, resulting in an SMF-NCF-SMF based structure. As for comparative analysis, a second sensor consisting solely of an SMF structure was developed. To enhance sensitivity, the sensing regions of both NCF and SMF were immobilized with a layer of graphene oxide (GO) coating. The functional groups of GO i.e., hydroxyl, carboxyl, and epoxy groups facilitated dispersion in polyvinyl alcohol (PVA) and increase the surface area available for biomolecule attachment, making it high effective for use in optical fibre sensor. The sensor performance was evaluated using UA concentrations ranging from 100 µM to 800 µM, respectively. The results demonstrated that the NCF-based biosensor with GO coating exhibited superior sensitivity compared to the SMF-based sensor, achieving a maximum sensitivity of 0.00420 ± 0.000118 nm/µM. The reproducibility and reusability of both the SMF and NCF biosensors were thoroughly assessed and discussed in this paper.

1. Introduction

Urid acid (UA) plays a vital role in human health, serving as an important indicator for evaluating metabolism and diagnosing various disorder. UA is a byproduct of the metabolism of purine nucleotides and can be found in human urine or serum [1]. Purines usually can be found in beer, mackerel fish, dry beans, and peas. These foods and beverages cause the body to generate urate crystals in human [2]. According to reports [3], the normal concentration of UA in human bodies is range in between 150 µM to 420 µM.

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Unwelcomely high amount of UA in the body, due to lack of exercise activity and an improper diet, will cause a number of illnesses. There are several diseases that are made up of this condition, such as gout, Lesch-Nyhan syndrome, renal failure and physiological disorders [4,5]. Therefore, there is a need to develop a simple yet effective technique for accurately diagnosing UA levels in the human body. Till now, several techniques have been developed for UA level measurement including spectrophotometry [6], chemiluminescence [7], capillary-based electrophoresis [8], and colorimetric [9]. However, the disadvantage in these sensors is their reliance chemical oxidation, which lead to reduced selectivity and relatively slow response time.

To date, the use of optical fibre-based sensor has been the subject of much interest because of the enhancement sensing application characteristics. It has the ability to harness the photonics properties of light, offering advantages over conventional electrical sensing methods [10]. High sensitivity, stability, remote sensing, electromagnetic immunity, and small size of fibre-based sensor also proves to be better [11]. A vast well appropriated research areas of optical fibre-based sensor are single mode fibre (SMF), multimode fibre (MMF), and no-core fibre (NCF) since they have the possibility to be used for general measuring purposes as they are easy to be kindly install and use [12].

With regards to all the listed optical fibres, SMF and NCF are considered the best fibres. SMF can deliver information in large distances with a low level of signal degradation with the core diameter that confines light within a single path of transmission. It features a lower modal dispersion characteristic that leads to cleaner data transmission along long distance compared to that of MMF [13]. As for NCF, it does not imply a chemical corrosion process that is necessary for sensor sensors based on multimode interference (MMI), which simplifies processing and safety factors [14]. NCF also offers a high level of non-linearity, which make it a very effective medium for non-linear optical phenomena a great deal more efficiently than any other type of fibre. For fibre-based sensing, high non-linearity can improve the attenuation of light to the sensing element.

In recent years, various materials had been employed as coating for UA detection including zinc oxide (ZnO) [15], gold nanoparticles (AuNPs) [16], carbon nanotubes (CNT) [17], graphene [18], copper oxide (CuO-NPs) and silver nanoparticles (AgNPs) [19]. Even so, these coatings have some drawbacks such as the biomolecules are not well absorbed over the coatings, also limiting and reducing the sensitivity of the sensor. Thus, sensitivity of the sensor can be improved by incorporating the graphene oxide (GO) as the layer of coating over the fibre. GO with functional groups includes hydroxyl, carboxyl, and epoxide functional group. These functional groups enable the GO to disperse in the water, have good biocompatibility of the bio-reaction, and also, offers extra surface area on the optical fibre to attach biomolecules onto the surface of fibre [20,21].

The incorporation of GO into polymers like polyvinyl alcohol (PVA) enhances the properties of the composite film, resulting an improved performance and long-term stability [22]. The presence of oxygen functional groups within the structure of the GO allows the PVA to enter and interact between the layers of the GO to produce a thin film. These characters of GO material make it interesting to use it as a coating on the fibre sensing region.

This study intends to address the lack of research on the synergistic potential of integrating graphene oxide with NCF and SMF structures for UA detection, despite previous studies showing the effectiveness of graphene oxide coatings in improving sensor sensitivity. Hence, in this paper, two distinct unique optical fibre-based sensors for UA detection have been developed and presented. The first sensor probe experiment was solely SMF sensor with GO coating and the second probe the NCF sensor was also coated with GO thin film. Section 2 explains the types of material used and the fabrication method of the optical fibre and the experimental arrangement used. Section 3 describes characterization of the materials and analyses the performance of the fibre-based sensors. The whole experiment will be concluded with details in Section 4 of the paper.

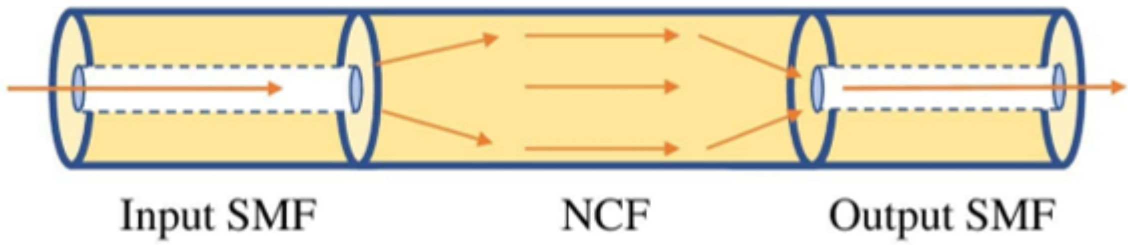
2. Methodology

2.1 Materials

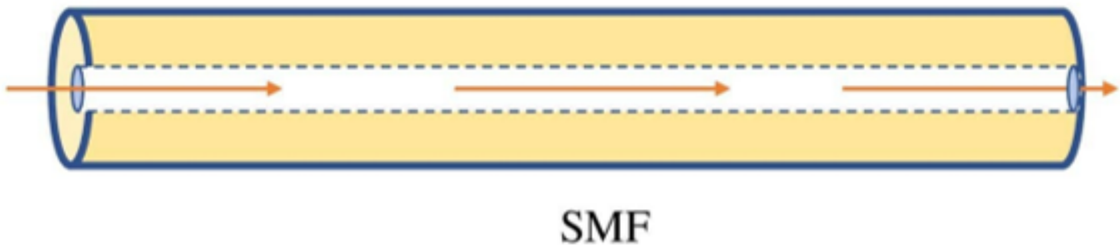
NCF and SMF was employed in the process of developing the fibre sensor probes. GO in the form of ultra highly concentrated single-layer GO, deionized water, and PVA powder was used in preparing the GO/PVA solution for the coating process. UA powder (crystalline ≥ 99 %), phosphate-buffered saline (PBS), were used for the preparation of UA solutions at various concentrations ranging from 100 µM to 800 µM. All UA solutions were diluted by using 1X PBS solutions of specific pH of 7.4.

2.2 Optical Fibre Fabrication

As for the fabrication of the sensors, SMF was chosen because the low in mode interfering noise value [23]. The fabrication of SMF-NCF-SMF sensor involves stripping 3 cm of NCF and 10 cm of SMF. The splicing process is carried out on fibre that has first been applied in isopropyl alcohol (IPA) solution, which is then washed to ensure that it is clean from any dust. As for splicing process, fibre structure would be spliced using fusion splicing machine as it would join the fibre in the most optimal way to avoid excessive loss. Figure 1 (a) shows the primary fibre sensor consists of NCF sandwiched in between SMF region and (b) is the second sensor solely SMF region.



(a)



(b)

Fig. 1. Schematic diagram of the optical fibre structure (a) SMF-NCF-SMF; (b) SMF

The working principle of the entire optical fibre sensor is associated with total internal reflection (TIR) and the interaction of evanescent waves with the external environment. The light in an optical fibre travel in the cladding, hence a fraction of the electromagnetic field extends into the surrounding medium called evanescent field. Noted that the intensity the evanescent wave in the fibre, E decays exponentially with $1/e$ as expressed in Eq. (1):

$$E = E_0 e^{-ab} \tag{1}$$

where E_0 is known as initial evanescent wave, *a* is the distance from the fibre surface, and *b* is the attenuation coefficient.

The penetration depth, d_p of the light in the SMF or MMF, which can be conveyed at a particular mode incident angle, θ_i is given as:

$$d_p = \frac{\lambda}{2\pi\sqrt{[(n_{co} sin\theta_i)^2 - (n_{cl})^2]}} \tag{2}$$

where n_{co} and n_{cl} is known as the core and cladding refractive index values, respectively.

The determination of the wavelength shift can be calculated by using Eq. (3), where λ is called the incident operating wavelength and the subsequent two parameters as n_{ext} and n_{eff} are categorized externally reflecting index and effective refracting index of fibre.

$$\Delta\lambda = \lambda \frac{\Delta n_{ext}}{n_{eff}} \tag{3}$$

Accordingly, the variation of wavelength shift when the fibre sensor is measuring the UA concentrations can be determined in Eq. (3). It can be used to calculate the wavelength shift value, $\Delta\lambda$ for both SMF and NCF sensor as the UA concentration varied from 100 µM up to 800 µM.

2.3 GO/PVA Coating Preparation

GO with ultra highly concentrated single-layer solution with initial 6 mg/ml concentration was diluted using deionized water to the concentration of 2 mg/ml. 0.2 g of PVA powder was then added into the solution. The solution was stirred at 30 °C for 6 hours by magnetic stirrer until which a uniform mixture of GO/PVA solution was obtained.

Subsequently, the SMF and NCF was cleaned with the IPA solution to remove any deposited dust of dirt on the surface of the fibre substrate. This step will assure that the coating glued well with the fibre structure. SMF and NCF were then immersed into 5 ml of GO/PVA solution for 30 minutes by dip-coating technique and being left to dry at the room temperature for 24 hours. This will assist in achieving an assurance that the stable composite film formed on the fibre structure.

2.4 Experimental Setup

The light source in this study covered a wavelength range from 360 nm to 2400 nm. The fibre sensing region covered with the GO/PVA film was placed fix on the glass slide in order to conserve the characteristics of the fibre. Using a Thorlabs CCS 200 spectrometer with an operating wavelength range of 200 to 1000 nm, the output spectrum was detected and subsequently processed. The UA solution was dropped at the middle part in the fibre structure, precisely at the SMF/NCF region as shown in Figure 2. The UA concentrations, in this work, were gradually increased from 100 µM up to 800 µM since it would affect the shifting of wavelength changes in the output spectrum. Without necessity, no further movement was made throughout the data intake in order to maintain the adjustment of the set-up.

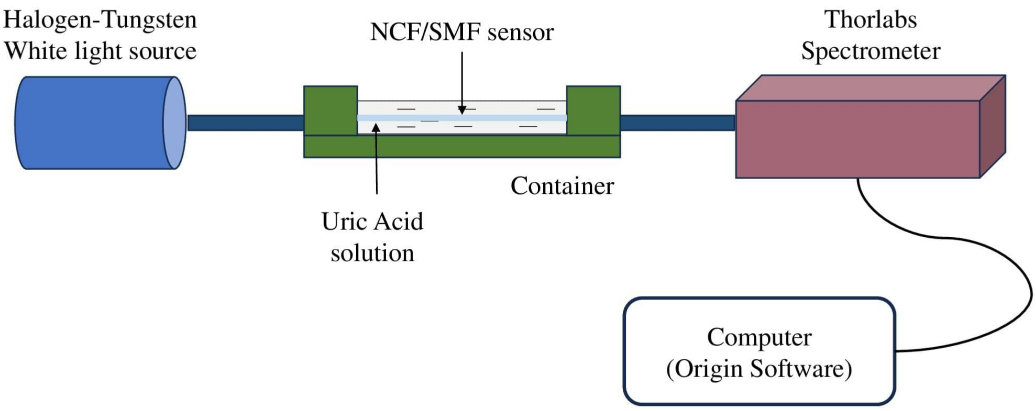


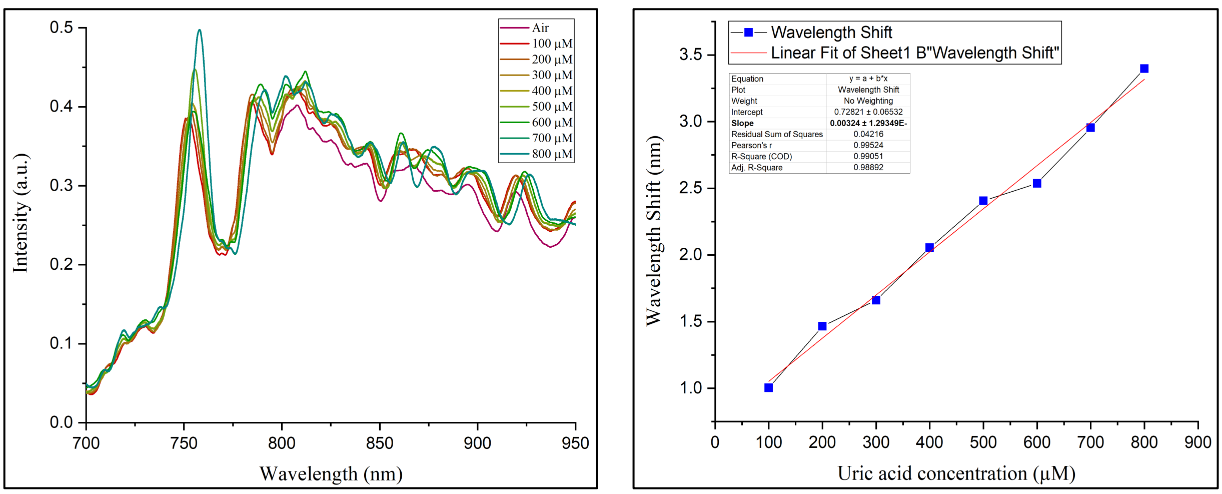
Fig. 2. Experimental setup.

3. Results and Discussion

3.1 Uric Acid Detection

The sensing mechanism of this sensor was established by incorporating the GO/PVA thin film on the NCF-SMF sensing region. The layered GO was found to be well dispersed into the PVA matrix resulting stable film of coating through hydrogen bonding interactions. The functional groups of the GO make the sensor be more sensitive due to its ability to offer extra surface area to the bio-reaction on the surface of the optical fibre. The increasing number of wavelengths shifts for different UA concentrations ranging from 100 µM to 800 µM was obtained.

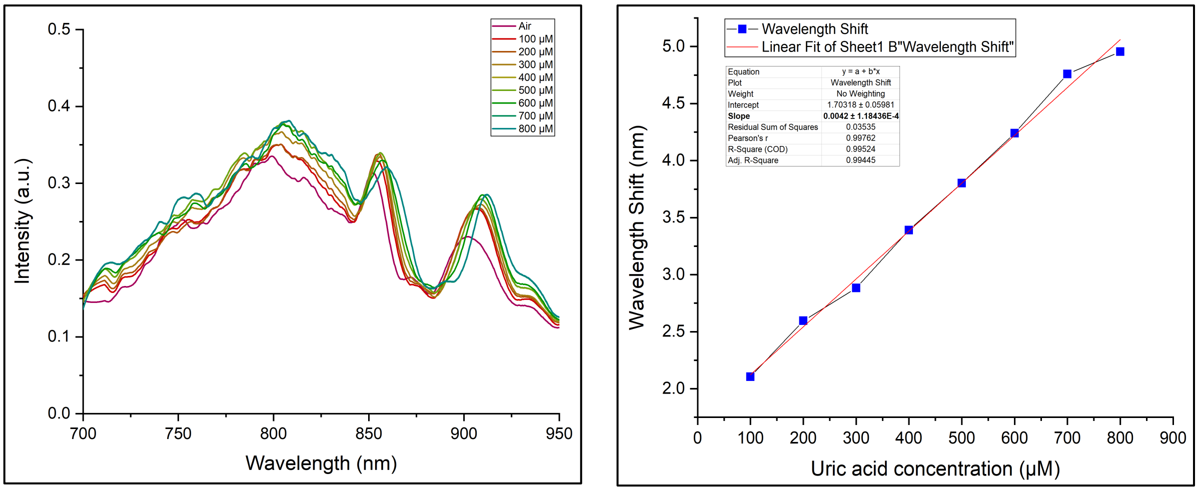
The sensing performance of the sensor probes was initially examined using an SMF with a GO/PVA sensor. Over the sensing region, 100 µM concentration of UA solution was placed on it, and the output spectrum's matching transmitted intensity was recorded. By using PBS solution, the fibre sensor was then cleaned to get rid of any remaining solution and it was allowed to dry at room temperature. Upon adding the UA solution, a 'red shift' was observed, attributed to the changes in solution concentration. The experiment was repeated three times using different SMF sensor probes and the spectra is presented in the Figure 3(a). The change in wavelength with varying concentrations of UA solutions is illustrated in Figure 3(b). The sensitivity of the SMF-GO sensor probe was recorded as 0.00324 ± 0.000129 nm/µM, with a linearity of R^2 = 0.99051.



(a) (b)

Fig. 3. (a) Output spectrum of SMF-GO/PVA sensor probes; (b) wavelength variations against different concentrations of UA solutions

The NCF using a GO/PVA sensor probe was then analysed to access and compare the performance of both NCF and SMS sensor probes. Figure 4(a) present the spectra for UA solutions. The output spectrum graph shown the wavelength shift as UA concentration varied is illustrated in Figure 4(b). From the analysis of data collected, a linear graph with R^2 = 0.99524 is obtained and linear concentration of UA gives rise to increase in UA concentration. The sensitivity obtained for the NCF-GO/PVA was found to be 0.00420 ± 0.000118 nm/µM. In terms of sensor sensitivity, the NCF with GO/PVA outperforms the SMF sensing probes.



(a) (b)

Fig. 4. (a) Output spectrum of NCF-GO/PVA sensor probes; (b) wavelength variations against different concentrations of UA solutions

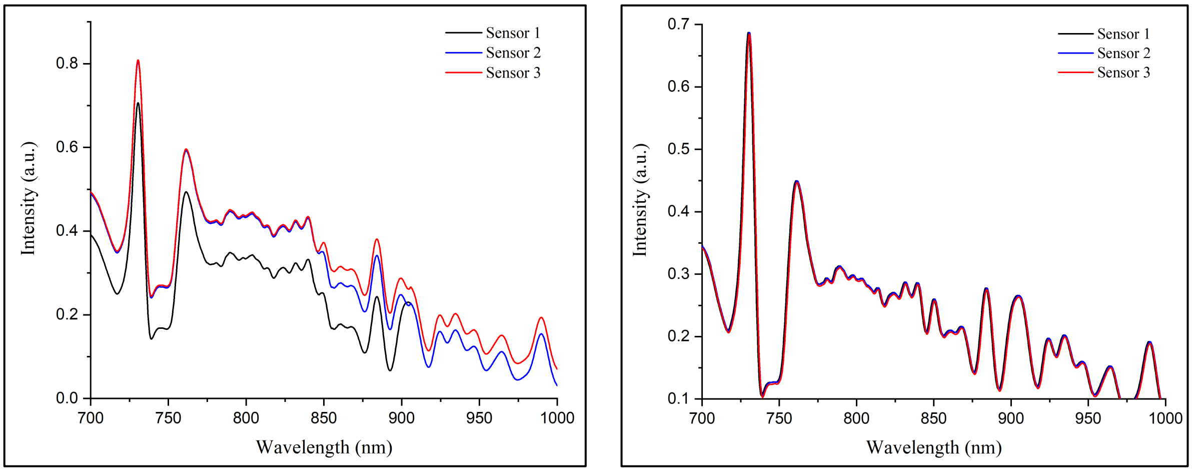
Table 1 demonstrates that the sensitivity of the NCF sensor is higher than that of the SMF sensor structure. In NCF, light is transmitted through an optical fibre without a core, allowing for greater light coverage by the surrounding material (coating or solution). This results in a strong light-matter coupling, therefore improving the sensing ability of the sensor to fluctuations in the UA solution.

Table 1
Comparison of sensors' performance

	SMF-GO	NCF-GO
Sensitivity (nm/µM)	0.00324	0.00420
Linearity (%)	99.1	99.5
Standard deviation (%)	0.06	0.05
Limit of detection (µM)	66.53	46.99

3.2 Reproducibility and Reusability Tests

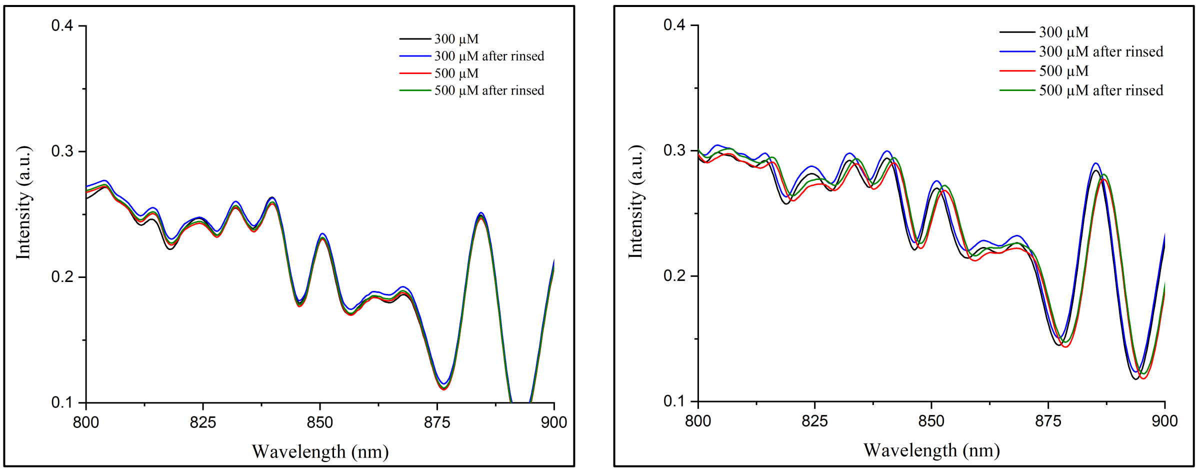
The reproducibility test of the fabricated fibre-based sensors was conducted using three different sensor probes of SMF and NCF. In this study, three distinct sensors, namely sensor-1, sensor-2, and sensor-3 were employed to measure a 100 µM concentration of UA solution, as illustrated in Figure 5. The results indicated that the output spectra of the SMF and NCF sensors were nearly identical, confirming the good reproducibility of the sensor probes.



(a) (b)

Fig. 5. Reproducibility test of (a) SMF; (b) NCF sensors

The reusability of the fabricated optical fibre sensors also tested using two different concentrations of UA solution, 300 µM and 500 µM, respectively. The test was carried out onto the sensing region, where 300 µM UA concentration was applied over it. The output spectrum from the spectrophotometer was recorded for comparison purpose. The fibre sensor was then washed with a 1X PBS solution and tested again with the same concentration of UA. The same process was repeated for 500 µM concentration as well. As shown in Figure 6, the sensors exhibit high reusability, with the spectra generated displaying similar patterns across different concentrations of UA solutions.



(a) (b)

Fig. 6. Reusability test of (a) SMF; (b) NCF sensors

4. Conclusions

The study concludes that advanced detection of UA using a NCF coated with GO thin film is far more effective than a solely SMF sensor. The unique structure of the NCF sensor enabled a higher degree of light interaction with the GO coating and resulted in superior sensitivity of the sensor with the value of 0.00420 ± 0.000118 nm/µM. Comparatively lower sensitivity to UA concentration changes was found on the SMF sensor with the value of is 0.00324 ± 0.000129 nm/µM, as it had a more confined light guiding core. With the present of GO coating, the light interaction of the NCF improved responsiveness, resulting in higher wavelength shifts but lower detection accuracy. While both have good UA detection capabilities, the NCF sensor outperforms the SMF sensor in terms of both sensitivity, linearity, and dynamic range, to be use in various applications where it is important to have accurate and very high-resolution UA detection such as in medical and health industry. The reproducibility and reusability tests indicated that the NCF-GO sensor outperformed the SMF-GO sensor. The sensor's capability to detect even low UA concentrations is improved by GO's large surface area and special electrical characteristics, which is essential for early diagnosis and monitoring. This sensor development result in more easily accessible, portable diagnostic sensor probe for clinics and possibly even at-home testing equipment. This will lead to improved patient outcomes by allowing for faster and more frequent testing.

Furthermore, patients at risk of problems from increased UA can benefit from continuous monitoring through the use of NCF-GO sensors, which can improve disease management. To enable thorough metabolic profiling, future studies could look into modifying the sensor to detect several biomarkers at once. Because of this, it would be particularly useful in diagnostic settings where monitoring of uric acid levels in conjunction with other metabolites is necessary. Also, perhaps lower healthcare expenditures related to delayed diagnosis and treatment in the country. This study provides a comprehensive analysis of NCF-based sensors as an efficient biomedical sensing platform capable of detecting biomolecules such as UA. Although there is potential for increased sensitivity with the NCF and SMF arrangement to be used in the medical industry, more investigation is needed to completely comprehend the effects of varying fibre and coating thicknesses, as these factors may have an impact on the consistency of the sensor's performance.

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