MJFAS
Malaysian Journal of Fundamental and Applied Sciences

Ahmad Zaini *et al.* | Malaysian Journal of Fundamental and Applied Sciences, Vol. 21 (2025) 1709-1718

RESEARCH ARTICLE

Precision in Uric Acid Detection: Unlocking Higher Sensitivity with Zinc Oxide-Coated Tapered No-Core Fibre-Based Sensor

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Abstract In this paper, ZnO coated non tapered and tapered no-core fibre (NCF) were developed and experimentally demonstrated for the detection of changes in the uric acid (UA) concentrations. The fabricated main optical fibre-based sensor used non tapered and tapered NCF sandwiched in between SMF region. The tapered NCF by CO_2 laser technique to achieve a waist diameter of 19.17 µm. The non tapered NCF was tested under the UA solutions for comparison purposes. The non tapered and tapered NCF sensing regions were then coated with ZnO solution using the dip-coating method. The ZnO was chosen as the coating due to its remarkable properties such as high transmittance, and good electrical conductivity. It is also known as effective in immobilizing with low isoelectric point (IEP) acidic proteins. Non-tapered UV-Vis spectroscopy was used to characterize the element of the thin composite film. The sensing performance test was performed by using different concentrations of UA solutions ranging from 100 µM to 800 µM for both non tapered and tapered NCF sensors. From the results, the tapered NCF biosensor with a coating of ZnO shows an excellent sensitivity than the non tapered NCF biosensor with the highest sensitivity of 0.00775 ± 0.00072 nm/µM. The reversibility, reproducibility, and reusability test of the designated optical fibre biosensor were also analysed and discussed in this paper.

Keywords: Uric acid sensor, Tapered NCF, Zinc Oxide.

Introduction

Uric acid (UA) is known as a key product of purine metabolism and one of the prime biomolecules found in human urine up to 60 % as it is easily found in various foods and beverages including beer, peas, dried beans and peas [1,2]. A normal range of UA concentrations in human bodies in the range of 150 µM – 420 µM [3]. However, the lack of exercise and an unhealthy diet increase the UA level in the human body. The abnormal or elevated level of UA will lead to several diseases such as gout, Lesch-Nyhan syndrome, renal failure and physiological disorders [4,5]. Thus, an efficient sensor is required to help in early diagnosis and treatment. To date, there are several sensors have been developed based on colorimetric [6], fluorescence [7], spectrophotometry [8] and chemiluminescence [9]. However, all these methods are not quite reliable due to the low sensitivity, complicated sample pre-treatment and huge consumption of reagents. In the past decade, fibre-based sensors have gained significant interest and have shown to be advantageous in terms of high sensitivity, remote distribution real-time monitoring, low cost, compactness and flexible design [10]. Fibre-based sensors such as single-mode fibre (SMF) and multi-mode fibre (MMF) also become a huge well-received research focus as they have the potential to be used in universal measurement purposes due to their easy and safe installation and operation [11]. No-core fibre (NCF) is the most sensitive optical fibre sensor among all because it provides the ability of a high degree of nonlinearity, making it highly responsive and sensitive to nonlinear optical effects compared to standard fibres. NCF, being in direct contact with the external environment, can serve as a monitor of the surrounding conditions [12].

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Received: 19 Nov. 2024
Accepted: 15 Jan. 2025

Tapering the fibre structure is very advantageous as it will increase their potential to be adapted to the sensor technology [13,14]. The small diameter of the tapered fibre will allow increased interaction between the evanescent waves with the external environment as it will make the sensor very sensitive. Till now, various techniques have been developed for the tapering process such as arc discharge [15], chemical etching [16] and flame torch [17]. However, even arc discharge provides a controllable taper process, but it suffers from low symmetrical taper or distortion structure results [15]. Also, chemical etching had the disadvantage of a long duration of 1 hour to produce one tapered optical fibre [16]. Flame torch also dealt with the difficulty of producing consistent waist diameter results due to the flame instability [17]. Thus, tapering NCF with CO_2 laser was chosen in this paper as it offers a clean and consistent symmetric taper structure and short time consumption as it controllable and repeatable process [18,19].

In addition, several coating materials have been used as coatings for the optical fibre-based UA sensor such as gold nanoparticles (AuNPs) [20], graphene oxide [21], graphene [22], and carbon nanotubes (CNT) [23]. These materials face some limitations such as low sensitivity and lack of functional groups that limit and restrict their use in sensing applications. Thus, to achieve a better sensor, ZnO was chosen as a coating material on the sensing region of non tapered and tapered NCF. ZnO offers interesting properties that attract researchers such as chemical stability, non-toxicity and electric conductivity [24]. ZnO is also known as a good material for immobilizing low isoelectric point (IEP) acidic proteins and DNA due to the large number of IEPs of 9.5 [25]. These characteristics of ZnO material make it interesting to use it as a coating on the fibre sensing region.

Therefore, in this paper, optical fibre-based sensors of non tapered and tapered NCF for UA detection have been developed and demonstrated. The main difference between these two sensors was that tapered NCF has a smaller diameter as it is expected to allow more evanescent waves to interact with the sample. Section 2 discusses a brief description of the used materials and methods, and fabrication of the optical fibre. Section 3 presents the characterization of the materials and the sensitivity of both of the optical fibre-based sensors. Section 4 presents the conclusion of this work.

Materials and Methods

Materials

For the fabrication of optical fibre sensors, NCF and SMF were used. Also, zinc oxide powder, deionized water and PVA powder were used for the production of ZnO solution for the coating immobilized on the sensing probe. Uric acid (UA) powder (crystalline ≥ 99 %, Sigma-Aldrich), and 1X phosphate-buffered saline (PBS, Sigma-Aldrich) were used for the production of UA solutions. The UA solutions' concentration will range between 100 µM to 800 µM with increments of 100 µM per sample. For the production of UA solution, 4.2027 mg of uric acid powder were dissolved in 5 ml of PBS solution to create a 5 ml stock solution of uric acid with a 5 mM molarity. Then, to get the required final volume, dilute the stock solution with additional 1X PBS solution while stirring with magnetic stirrer. Thus, UA solutions with concentrations ranging from 100 µM to 800 µM were produced for the performance analysis of the created sensor probes.

Fabrication of Tapered Fibre

For the fabrication of the fibre configuration structure of SMF-NCF-SMF, 3 cm of NCF and 4 cm of the total of 10 cm SMF were stripped by using a mechanical stripper and cleaved by using a fibre cleaver [26,27,28]. Isopropyl alcohol (IPA) solution is used to clean the fibre to remove any dirt on the fibre before the splicing process. The fibre splicing of NCF was done by utilising a fusion splicing machine as it will connect the fibre with minimal loss. There were two different configurations of fibre structures, which were the first probe of SMF-NCF-SMF and the second probe of SMF-tapered NCF-SMF. Both of the sensor probes are composed of NCF sandwiched in between SMF sections.

The tapered fibre was fabricated by using a CO_2 laser, where simultaneously heating and stretching a section of NCF as shown in Figure 1. The tapered fibre is expected to provide better sensing performance as the reduced diameter of optical fibre will allow more proportion of evanescent wave to interact with the surrounding external medium [18]. Adopting CO_2 laser as a tapering process, it was expected to create a region of tapered NCF with a reduced and clean symmetrical diameter since the CO_2 laser tapering technique provides precise, repeatable and controllable tapered results.

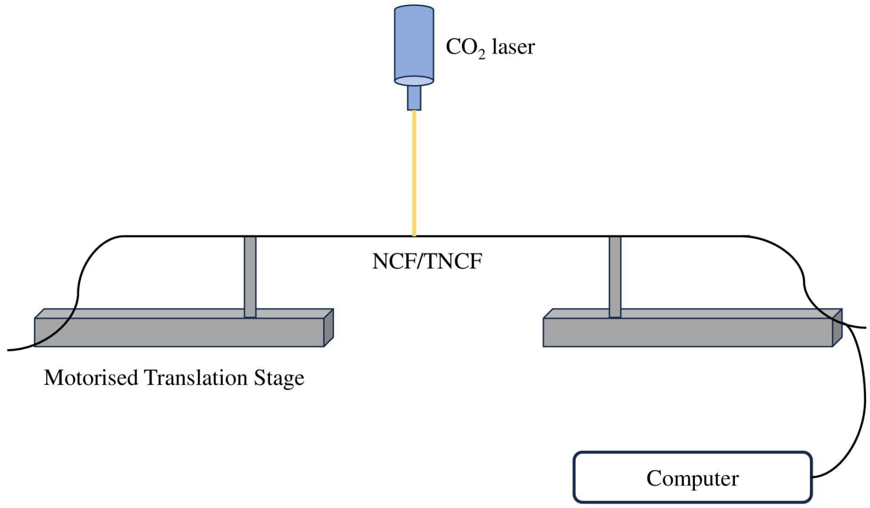

Figure 1. Schematic diagram of the tapering system by CO_2 laser

In this process, the input power of CO_2 was set to 30 W out of 40 W, the maximum power of the CO_2 laser machine. Continuous operation close to the 40 W maximum power may put stress on the laser, accelerating the deterioration of optical components and shortening the laser system's lifespan. Thus, for this experiment, 30 W was chosen where the resulting diameter was optimum for the biosensor. A high-precision motorised translation stage and a microcontroller were also involved in the process of tapering fibre as this equipment controlled the speed and moved the stage in a certain direction at a speed of 500 rpm and a pulling distance of 5 mm. The reason behind optimum diameter is that extremely thin fibres might have larger bend losses, limit practical use in the industry and become too fragile. These modifications were made to produce a quality diameter value of tapered NCF. Figure 2 shows the microscopic image of non tapered and tapered NCF with different diameter values.

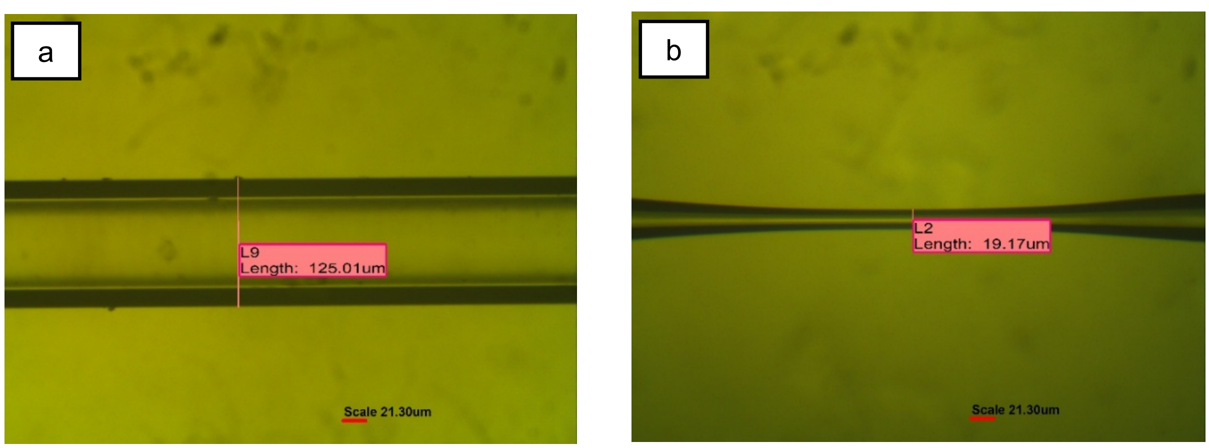

Figure 2. Microscopic images of (a) non tapered NCF; (b) tapered NCF

Preparation of Zinc Oxide/PVA Composite Thin Film onto the Fibre

Polyvinyl alcohol (PVA) solution was produced by using 0.2 g of PVA powder and 10 ml of deionized water. The solution was then mixed for 1 hour at 80 °C by using a magnetic stirrer until the transparent solution was obtained. Zinc oxide in powder form weighing 20 mg was then combined with the solution. Then, the optical fibre sensor probes were then cleaned by using the isopropyl alcohol (IPA) solution to remove any dirt or impurities on the surface of the substrate. By the dip-coating method, the fibres were immersed into the 2 mg/ml of ZnO solution for 30 minutes by dip-coating method, before being left to dry for 24 hours at room temperature. This will help to make sure that the stable composite film with the thickness of 70 nm is produced onto the optical fibre structure as shown in Figure 3.

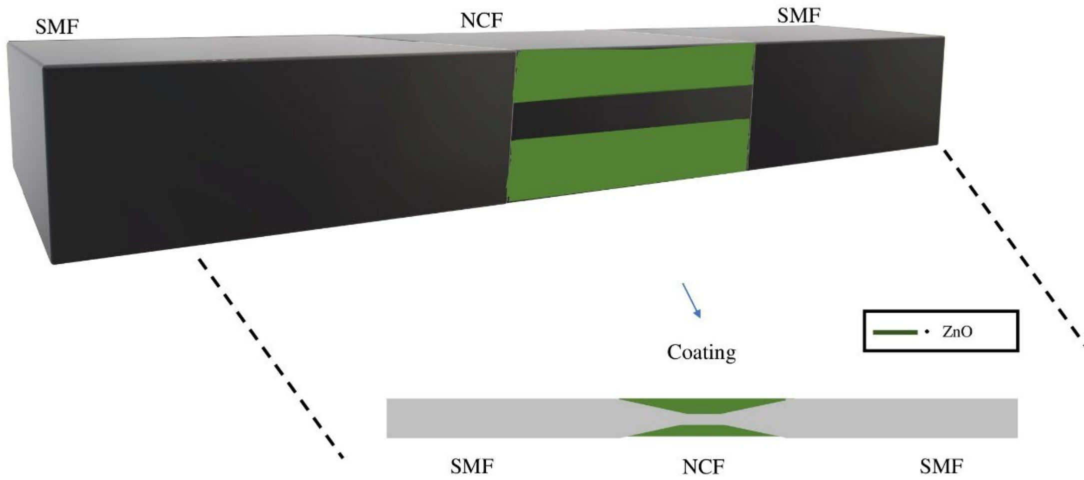

Figure 3. ZnO coating on the fibre sensing region

Characterization

The ZnO solution was fully characterised using an ultraviolet-visible (UV-Vis) spectrophotometer. This stage is essential for assessing its absorption spectrum. By using the water in the cuvette as a reference, the characterisation was completed in order to do the absorption measurement. The presence and calibre of the coating can be verified by UV-Vis by locating particular absorbance peaks at particular wavelengths. In order to maximise the fibre's sensor effectiveness, it is crucial to keep an eye on these absorption wavelengths to make sure the coating is uniform and has the capacity to interact with light at the appropriate wavelengths.

Experimental Setup

Figure 4 shows the experimental configuration setup for the suggested UA detection sensor. The light source used in this experiment is an Ocean Optics HL-2000 Halogen-Tungsten white light source with a wavelength range of 360 nm to 2400 nm. ZnO-coated non tapered and tapered NCF fibre-based sensors were installed inside the container. The output spectrum was detected using the Thorlabs spectrometer CCS 200, which operates between 200 and 1000 nm. The concentrations of UA solutions used in this experiment differ from 100 μM to 800 μM. UA solutions are placed inside the container, where the optical fibre-based sensor will interact with changes in UA concentrations of each sample. Thus, no further movement was made throughout the data intake if not necessary to retain the setup adjustment.

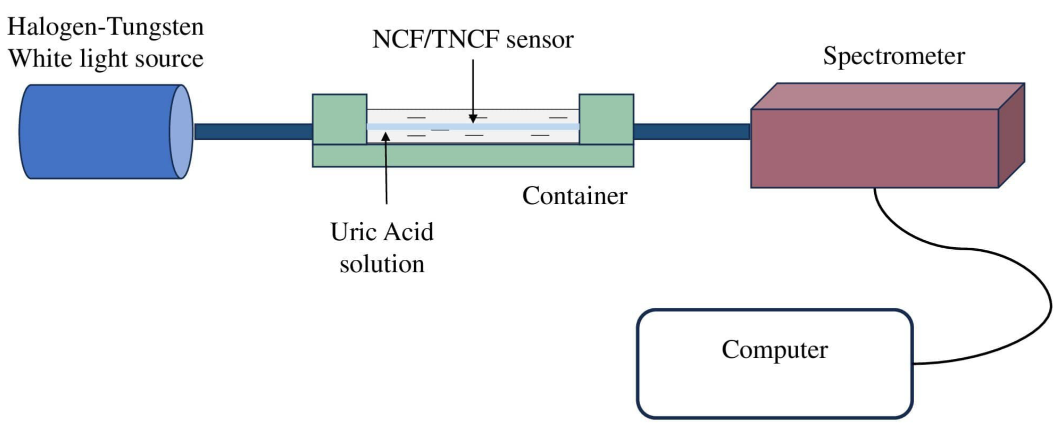

Figure 4. Schematic diagram of the experimental setup

Results and Discussion

Characterization of the ZnO Thin Film by UV-Vis

In this section, the characterizations of the immobilized optical fibre probe and the functional testing of the ZnO solution were discussed. ZnO solution was characterized by using a UV-Vis spectroscopy. The absorbance measurement was recorded from 300 nm to 800 nm wavelength, where the peak wavelength was obtained at 340 nm as shown in Figure 5. Theoretically, the bulk absorption of ZnO should appear at the wavelength of 380 nm [29]. Regarding the change in the theoretical and actual value, it can be observed that a blue shift occurs from 380 nm to 340 nm as ZnO tend to exhibit a strong absorption band.

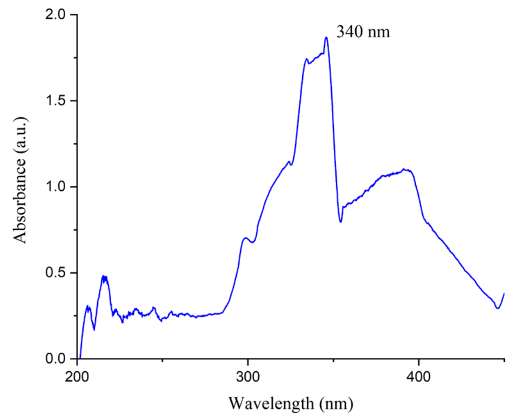

Figure 5. Absorption spectrum of zinc oxide

Sensors System's Performance

An optical fibre-based sensor of non tapered and tapered NCF coated with ZnO thin film was employed as a biosensor for UA detection. The ZnO thin film was well dispersed over the fibre-sensing region which enhanced the performance of the sensor. ZnO is known as a wide bandgap semiconductor material with a value of 3.37 eV, where if ZnO particles react with UA solution, the optical properties such as refractive index (RI) value will change. This change will be easily detectable by the sensor. In this paper, the concentrations of UA solutions varied from 100 µM to 800 µM, since it influences the output spectrum wavelength shift. For the analysis of the developed optical fibre-based sensor for UA detection, 100 µM was applied first onto the fibre sensing region and the respective intensity wavelength output spectrum was recorded. Then, 1X PBS solution was used to clean the fibre sensor and remove any suspension of the previous solution. Based on the output spectrum results, it was observed that there was a wavelength shift, which is known as a "red shift" as the concentration of the UA solutions changes as shown in Figure 6 for both non tapered and tapered NCF sensor probes. The magnitude of the wavelength shift is known to be proportional to the increases in the UA solutions as the RI of the solution changes.

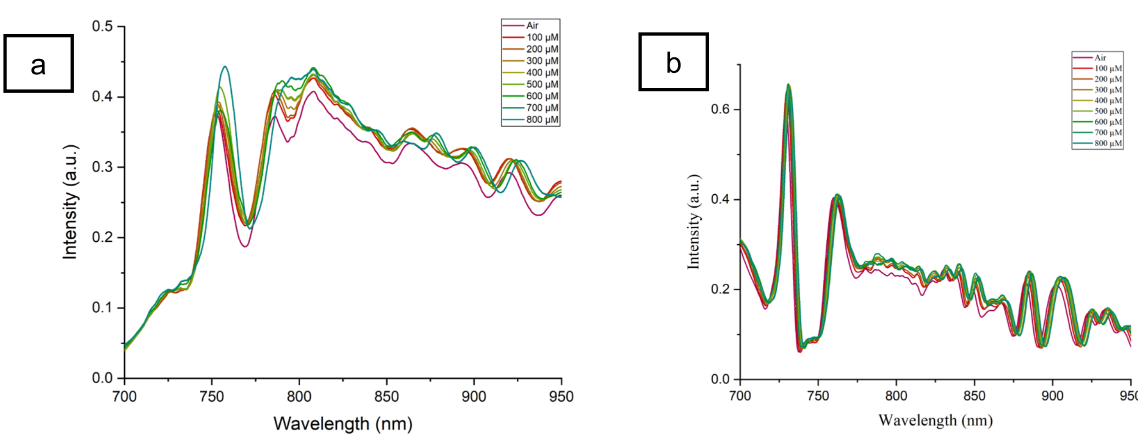

Figure 6. Output spectrum of (a) non tapered NCF-ZnO sensor probe; (b) tapered NCF-ZnO sensor probe

Using different non tapered and tapered NCF sensing probes, the measurement was carried out three times. The graph of the average sensitivity of the sensor probes was plotted in Figure 7, based on the wavelength shift output spectrum. The sensitivity of the non tapered NCF-ZnO sensor probe was 0.00502 ± 0.00022 nm/μM, with the linearity of R^2 of 0.98863. On the other hand, the performance of the tapered NCF sensor probe yields the linearity of R^2 = 0.95140 and linear as the concentration of UA solution increases. The achieved sensitivity of the tapered NCF-ZnO was measured to be 0.00775 ± 0.00072 nm/μM.

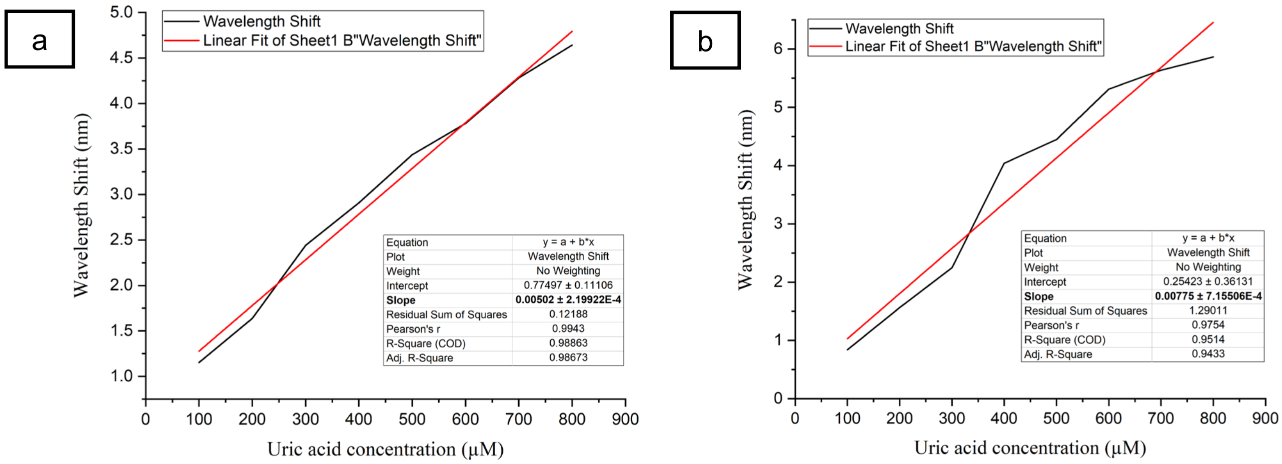

Figure 7. Sensitivity of (a) non tapered NCF-ZnO sensor probe; (b) tapered NCF-ZnO sensor probe

Results demonstrated that the sensitivity of the sensor system is influenced by the NCF's diameter and also the coating material of ZnO. A smaller tapered NCF diameter results in a larger contact area between the sample and the evanescent field, which boosts sensitivity in identifying RI variations. The findings also emphasise how crucial the functional ZnO coating and the diameter size of the tapered NCF sensing region are to improving the performance of the sensor system. The way the evanescent wave interacts with the surrounding medium depends on the ZnO thin film's thickness. An ideal thickness improves wave-medium interactions and boosts sensor performance, whereas an excessively thick thin layer can attenuate the evanescent wave and decrease sensitivity. In a comparison of both non-tapered and tapered NCF sensing probes, the performance of tapered NCF with ZnO composite thin film is better in terms of the sensitivity of the sensor as shown in Table 1.

Table 1. Comparison of sensors non tapered and tapered NCF' performance

	Non tapered NCF-ZnO	Tapered NCF-ZnO
Sensitivity (nm/μM)	0.00502	0.00775
Linearity (%)	98.8	95.1
Standard deviation (%)	0.11	0.36
Limit of detection (μM)	72.31	153.29

Reversibility, Reproducibility and Reusability Tests

Since the purpose was to improve the understanding and evaluation of the sensor's performance, several tests were conducted to see the reversibility, repeatability, and reusability of the sensor. The reversibility of manufactured optical fibre sensors of non tapered and tapered NCF was investigated. Sensor reversibility is another valuable aspect, especially when the sensor will need to be exposed to multiple cycles of measurements and further, the property being measured may change with time. The concentration of the same UA in the ascending and decreasing order negatively affected the data at almost the same level, hence the reversibility of both the manufactured optical fibre sensors, as illustrated in Figure 8.

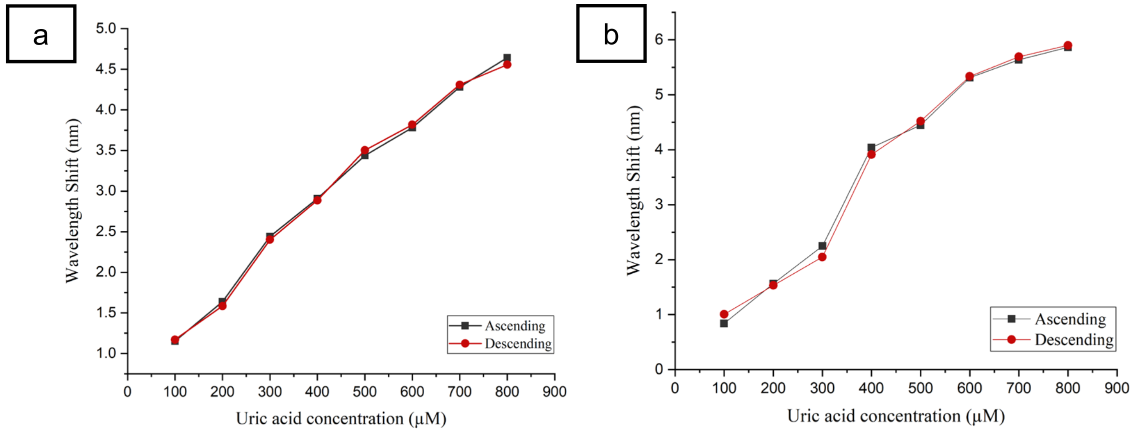

Figure 8. Reversibility test for (a) non tapered NCF sensors; (b) tapered NCF sensors

The fabricated sensors also were analysed for the reproducibility test with three different sensor probes of non tapered and tapered NCF. Three different sensor-1, sensor-2 and sensor-3 were used to measure 100 µM concentration of UA solution as shown in Figure 9. The results show that the output spectrum of non tapered and tapered NCF have a nearly similar spectrum and the results indicate that the sensor probes have a good reproducibility.

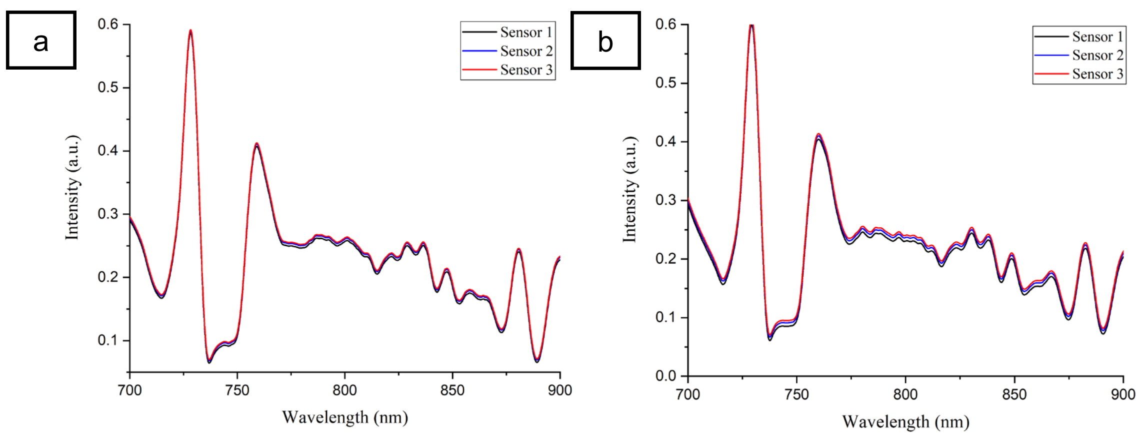

Figure 9. Reproducibility test at 100 µM for (a) non tapered NCF sensors; (b) tapered NCF sensors

The reusability of the fabricated optical fibre sensors also was tested by using two different concentrations of UA solution, which were 400 µM and 500 µM. The test was done by dropping 400 µM UA concentration over the sensing region and observing the output spectrum of it. By using 1X PBS solution, the fibre sensor will be rinsed before being tested again with the same UA concentration. The step was repeated likewise for the 500 µM. From Figure 10, the result indicates that the sensors possess a good reusability property as the results show the nearly identical spectrum for both concentrations of UA solutions.

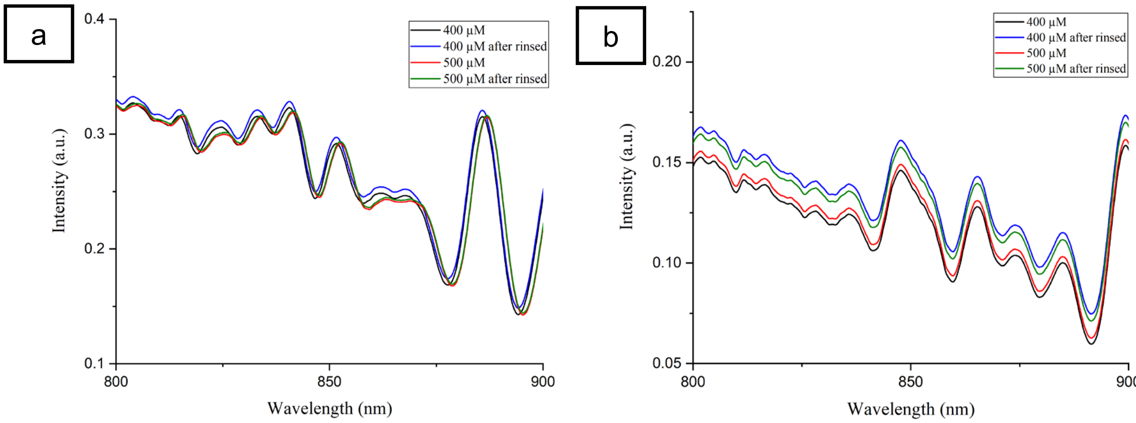

Figure 10. Reusability test for (a) non tapered NCF sensors; (b) tapered NCF sensors

Conclusions

The study concludes that advanced detection of UA using a tapered NCF coated with ZnO thin film is far more effective than a non tapered NCF sensor. The unique structure of the tapered NCF sensor enabled a higher degree of light interaction with the ZnO coating and resulted in superior sensitivity of the sensor with the value of 0.00775 ± 0.00072 nm/µM. Comparatively lower sensitivity to UA concentration changes was found on the non tapered NCF sensor with the value of 0.00502 ± 0.00022 nm/µM, as it had a more confined light guiding core. With the presence of ZnO coating, the light interaction of the tapered NCF improved responsiveness, resulting in higher wavelength shifts but lower detection accuracy. While both have good UA detection capabilities, the tapered NCF sensor outperforms the non tapered NCF sensor in terms of sensitivity, reversibility, and dynamic range, to be used 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 tapered NCF-ZnO sensor outperformed the non tapered NCF-ZnO sensor.

To facilitate early diagnosis and monitoring, ZnO with a large surface area and special electrical characteristics enables the sensor to detect even low UA concentrations. This sensor development leads to more easily accessible, portable diagnostic sensor probes for clinics and possibly at-home testing equipment. By doing so, patient outcomes will be improved since testing will be faster and more frequent as its enable continuous monitoring of patients at risk for problems from increased UA. Future studies could also modify the sensor to detect more than one biomarker at a time to enable metabolic profiling. Perhaps also lower healthcare expenditures in the country for delayed diagnosis and treatment. Here, we comprehensively study the tapered NCF as a highly efficient platform for biomedical sensors to detect biomolecules like UA. Although there is potential to increase sensitivity using the non tapered and tapered NCF arrangement in a medical industry application, there remains further investigation needed to obtain a full understanding of the effects of a diameter range of fibre and coating thicknesses.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

Acknowledgement

Universiti Teknologi Malaysia in standings of facilities and finances by UTMNexus scholarship and the UTM Encouragement Grant vot no. Q.J130000.3854.31J54 supported this research.

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