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Effect of Coated Tungsten Disulfide (WS_2) on Tapered Optical Fibre for Urea Detection

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ABSTRACT

It is crucial to detect urea in clinical samples because urea levels in biological fluids are clinically linked to a number of disorders, including dehydration, heart failure, and renal dysfunction. Here, a tungsten disulfide (WS_2)-coated tapered optical fibre sensor with increased sensitivity is suggested and demonstrated. The findings have shown that the sensing sensitivity is greatly improved with WS_2 coating on the tapered optical fibre. This improvement is made possible by the unique optoelectronic capabilities of the WS_2 layer and the tapered diameter size. The coating of the 20 µm tapered diameter with 20 µL WS_2 results in the maximum sensitivity (up to 0.069 dBm/(mg/dL)) in the experiment. This result demonstrates a sensitivity improvement of 60% compared to the scenario in which a WS_2 coating was not present. Analysis and discussions are conducted on the sensing performance improvements brought about by the tapered diameter size and the WS_2 coating. In addition, the WS_2 tapered optical fibre sensor possesses a linear correlation coefficient of 99.8 % in a concentration range of 10–50 mg/dL.

1. Introduction

In recent years, researchers have shown increased interest in urea detection due to its high application in clinical diagnosis and the food industry, environmental analysis, and healthcare [1-5]. Urea is present mainly in the urine of mammals. It is a water-soluble, nitrogen-containing waste product generally filtered by healthy kidneys from the blood into the urine [4,6,7]. Urea is naturally produced during the breakdown process of proteins by the liver [8,9]. Due to this process, amino

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groups are removed from the amino acid present in the proteins. These amino groups are converted to ammonia, which is highly toxic, and the ammonia is ultimately converted to urea. In clinical diagnosis, urine and blood serum test are usually used to check the level of urea concentration. The result shows an indicator to detect early renal dysfunction, heart failure, dehydration, hypovolemic shock, gastrointestinal bleeding, and a catabolic condition [10]. Moreover, there was a report showing findings of the presence of urea in samples of milk in India that affected consumers' health. In order to increase the protein composition in their milk, some profit-seeking producers choose to do immoral activities by adding urea to the milk [11]. Therefore, attempts have been undertaken to create exceptionally accurate and sensitive urea-level detection techniques. Several analytical methods, including electrochemical, optical, and piezoelectric, have been developed for detection [12-14].

The remarkable properties of optical fibres have been inherited by fibre-optic sensors such as surface plasmon resonance, micro bending, and FTIR, which have been extensively explored and implemented as a sensor, including a urea sensor [15-18]. Tapered optical fibre has recently gained significant interest for biosensors due to their better structural rigidity and easy fabrication [19]. The current trend studies show that the research in urea is growing particularly valuable for sensing applications. Since the power of its evanescent wave (EW) in its cladding is greater, a tapered fibre optic is more sensitive to its surroundings. This is due to the shape and dimensions of tapered fibres, which permit a more significant proportion of the evanescent field to interact with the external environment, thereby making them more sensitive to variations in external forces. Figure 1 shows the structure of the tapered fibre. Impregnating the tapered fibre with the appropriate material of the correct thickness will intensify its sensitivity to the physical quantity to be measured.

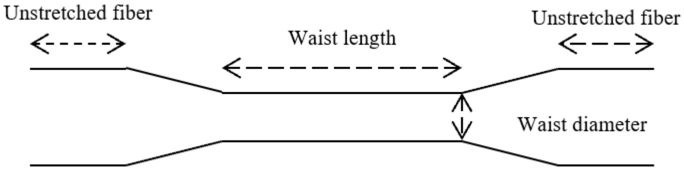
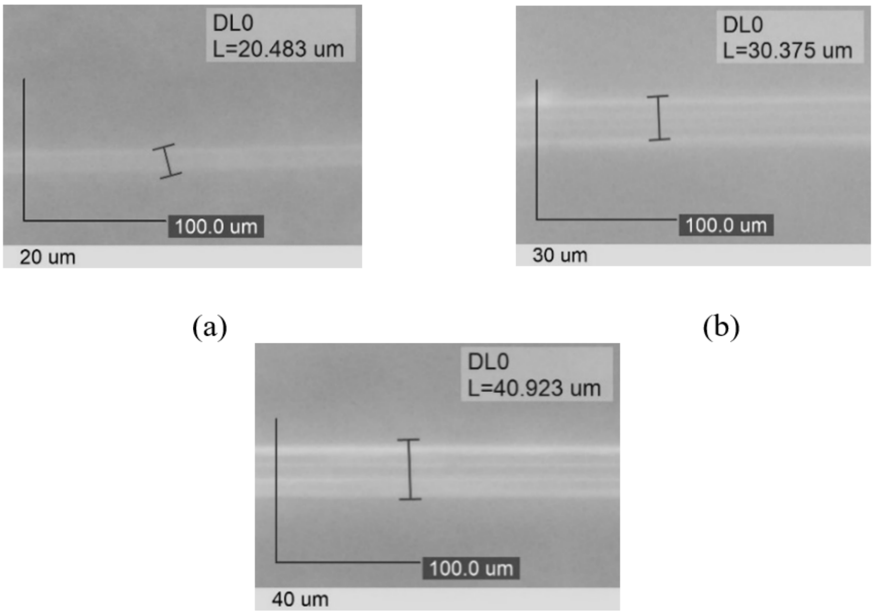


Fig. 1. The structure of a tapered fibre

Numerous materials have been used to enhance the sensitivity of the sensor, namely cobalt oxyhydroxide (CoOOH), graphene oxide (GO), gold nanoparticles (AuNPs), manganese dioxide (MnO_2), molybdenum diselenide ($MoSe_2$), tungsten disulfide (WS_2) and few other materials found by other researchers [20-24]. Current trend studies show that the research in tungsten sulfide is growing. Since their initial discovery in 1992 during the heating process of tungsten in a hydrogen sulfide environment, a range of concentric polyhedral and cylindrical formations were developed and displayed by the transmission electron microscope [25]. This material has emerged as a new class of fascinating 2-dimensional transition metal dichalcogenides due to its unique properties, which include direct band gaps, superior flexibility, moderate carrier mobility, biocompatibility, and layer-dependent electrical and optical properties. It is a promising candidate for future applications in electronic devices, particularly in sensing technology [26-28]. This study hypothesizes that the amount of tungsten disulfide (WS_2) coating an optical sensor has may affect the sensitivity to different urea concentrations. The experiment investigates and analyses the sensor's performance by comparing the tapered size and volume of WS_2 coating.

2. Experimental Setup

In this work, tapered multimode optical fibre was fabricated with core and cladding sizes 62.5 µm and 125 µm, respectively. The tapering optical fibre process employed a heat-pulled technique. Due to its uniformity and precise manipulation, the Vytran GPX3400 optical glass-processing machine performed the tapering. The performance of tapered optical fibre (TOF) before and after coatings is analysed for different tapered diameters. The tapered optical fibres were coated with Tungsten Disulfide (WS_2) by varying the coating volume. The WS_2 coating material was prepared on the solution and purchased from the Graphene Supermarket. The tapered optical fibres were coated with 20 µL and 40 µL at the tapered region using the drop-casting technique and left for two hours for drying and uniform coating under room temperature. The experiment is performed under ambient temperature. Three different tapered size diameters were used in this experiment. They are 20, 30, and 40 µm, as depicted in Figure 2.



(a) (b)

(c)

Fig. 2. Microscope images of the TOF which have cladding diameters (a) 20, (b) 30 and (c) 40 µm

For the experimental setup, the Ocean Optics (DT-MINI-2-GS UV-VIS-NIR) light source with a range of 200–2000 nm was used together with an optical power meter (OPM) (Thorlabs S-145) as an output. The TOF was placed between the light source and OPM as a sensor probe. A computer was used to collect data from the OPM, and the data will be analysed accordingly, as depicted in Figure 3. For preparing the 40 mL urea solution, a urea (NH4CONH2) pallet from Sigma Aldrich (M.W=60.06) was diluted with de-ionized water to form 10, 20, 30, 40 and 50 mg/dL concentrations. Each coated and uncoated TOF of different sizes was placed in a petri dish and immersed in 40 mL urea solution. For data collection, the immersion time is set for 30 minutes and repeated three times to measure the sensor's stability at an ambient temperature of 25 °C. Figure 3 shows the experimental setup to investigate the performance of uncoated and coated TOF under different urea concentrations for different TOF size diameters.

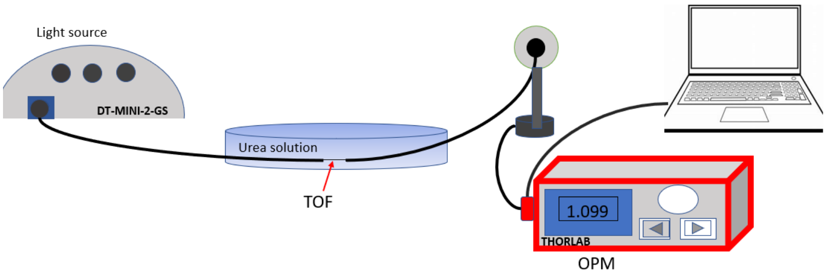
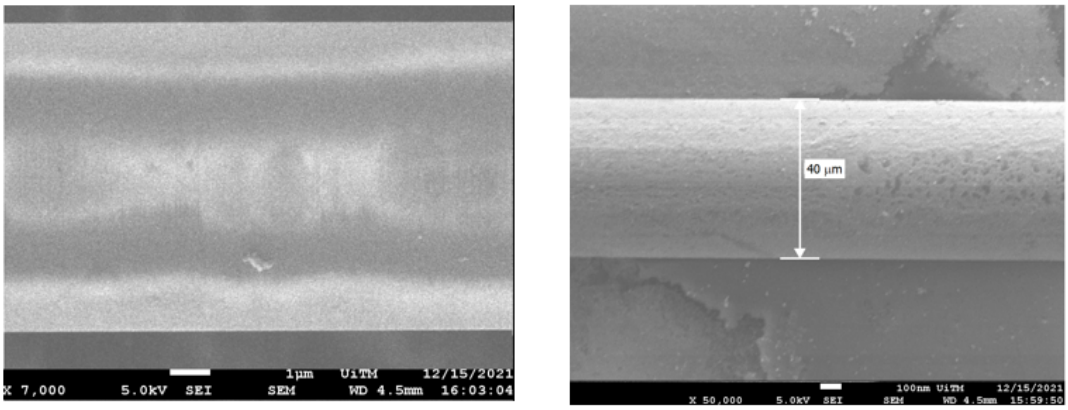


Fig. 3. Schematic diagram of the experimental setup for uncoated and coated TOF under different urea concentrations

3. Results and Discussion

The performance of TOF before and after coatings were compared with different tapered size diameters. The Ocean Optics (UV-VIS-NIR) light source was used to characterize the WS_2-coated TOF with a wavelength range of 250 nm to 2000 nm. The optical power meter (OPM) collected the output power using a photodetector, which converts light into an electrical current. The comparison between uncoated and coated TOF was examined using field emission scanning electron microscope solutions FESEM, as shown in Figure 4, for the 40 µm TOF size diameter.



(a) (b)

Fig. 4. Comparison of surface morphology for (a) uncoated and (b) coated 40 µl WS_2 on TOF surface

The first part of the experiment will compare the sensor performance for different diameter sizes of uncoated TOF for different urea concentrations. Based on the result obtained, decreasing the diameter size of TOF will improve the performance of the sensor at different urea concentrations, as depicted in Figure 5. It is found that the sensitivity of 20 µm TOF is 0.02173 dBm/(mg/dL) with a slope linearity of 99.91%. On the other hand, the sensitivity of 30 µm and 40 µm TOF is 0.0158 dBm/(mg/dL) and 0.01233 dBm/(mg/dL) with linearity more than 99% each. Based on this observation, the smallest diameter is more sensitive due to it allowing lighter to be refracted over TOF boundaries in the sensing mechanism [29]. So, decreasing the TOF size diameter will improve the sensitivity of the sensing mechanism for TOF. Furthermore, TOF will provide easy access to the evanescent wave mode propagation through the tapered regions and facilitate interaction with the surrounding medium for different analytes [30].

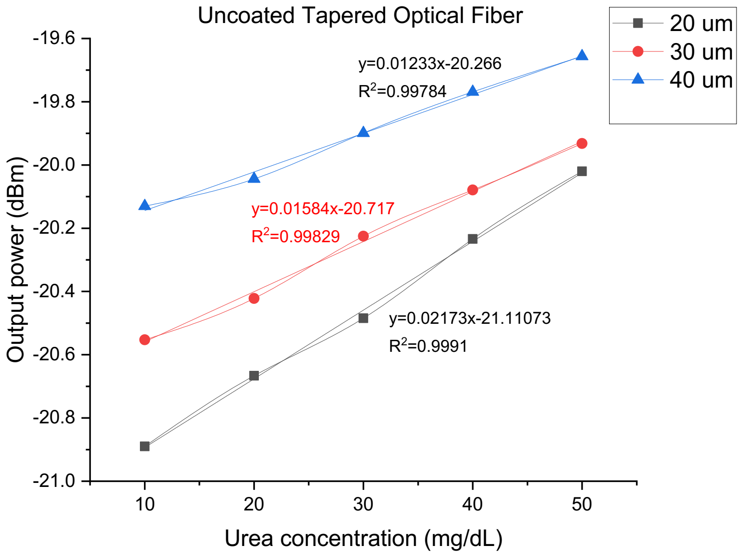
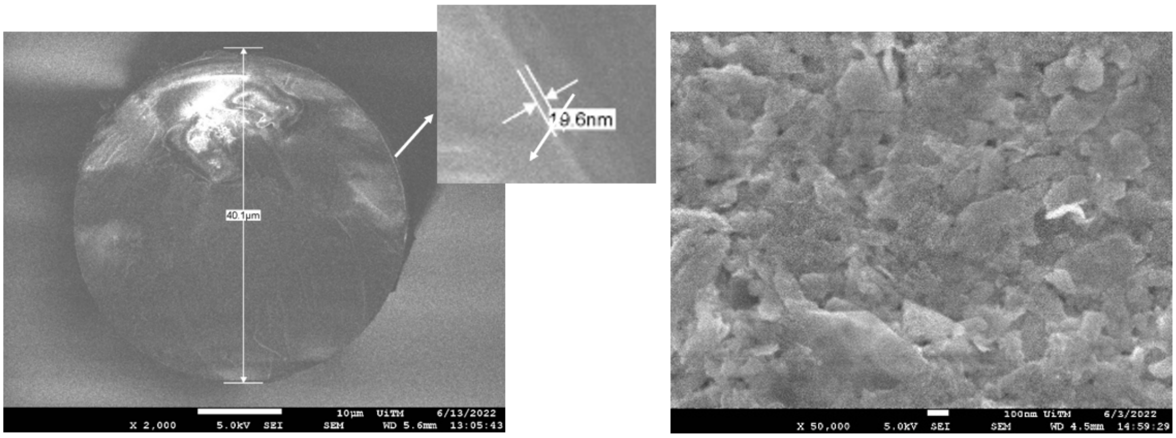


Fig. 5. Output voltage against urea concentration for uncoated TOF

Next, the performance of TOF coated with WS_2 with different volumes will investigate for different TOF size diameters. Figure 6 (a) shows the cross-section of TOF with a 40 µm diameter and 19.6 nm thickness of WS_2 coated on the surface of TOF. Furthermore, the morphology of WS_2 also has been examined under FESEM, as depicted in Figure 6 (b).



(a) Cross section of 40 µm TOF

(b) Surface morphology of coated WS2

Fig. 6. TOF surface: (a) cross section of TOF with 19.6 nm WS_2 thin layer (b) surface morphology of WS_2

The same uncoated TOF is used in this experiment. The TOF was cleaned using acetone and rinsed with DI water to avoid contamination from the previous experiment. Next, 20 µl of WS_2 was cast dropped on uncoated TOF and dried under ambient temperature for approximately 2 hours. Then, the coated TOF will immerse in different concentration urea solution for 30 minutes based on the setup mentioned above for uncoated TOF. Figure 7 shows the sensing result of the output power from different TOF size diameters coated with 20 µl of WS_2. It is found that the sensitivity of coated 20 µm TOF is 0.0642 dBm/(mg/dL) with linearity of 99.896%. On the other hand, the sensitivities for 30 and 40 TOF are observed 0.01202 dBm/(mg/dL) and 0.0142 dBm/(mg/dL) with linearity above

98%. From the results obtained, the sensing performance of coated TOF is increased by approximately 60% compared to the uncoated 20 µm TOF size diameter. Meanwhile, the performance sensing for other TOF improved 6% compared to uncoated TOF.

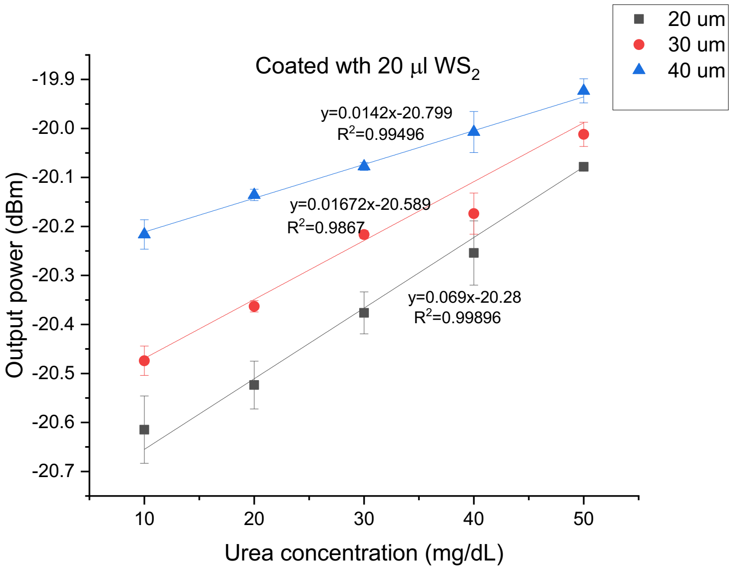


Fig. 7. Output power vs. urea concentration for the proposed TOF with 20 µl WS_2 with different TOF size diameter

Next, the same coated TOF is used and cleaned using DI water. For preparing 40 µl WS_2 TOF, another 20 µl WS_2 is dropped cast on the surface of 20 µl coated TOF. It took approximately two hours to dry WS_2 at room temperature. The performance of coated TOF is examined by immersing different sizes of TOF in various urea concentrations. By analysing the result, the sensitivity of coated 20 µm TOF is 0.0164 dBm/(mg/dL) with a linearity of 99.979%. On the other hand, the sensitivity of the sensor was reduced for 30 µm and 40 µm TOF, which is 0.0161 dBm/(mg/dL) and 0.0010 dBm/(mg/dL) with linearity of more than 99.4%. The plotted graph is shown in Figure 8 for analysis of coated 40 µL of WS_2. The sensing performance decrease while increasing the volume of WS_2. The performance decreases rapidly for TOF size diameter 20 µm, which is 75% compared to 20 µl WS_2 volume. Meanwhile, the performance for another coated TOF decreased slowly. In order to investigate the sensor performance for coated WS_2, another 20 µl is dropped on the coated 40 µl TOF. As result, the sensor becomes insensitive by increasing the volume of WS_2 with a sensitivity 0.002dBm/(mg/dL).

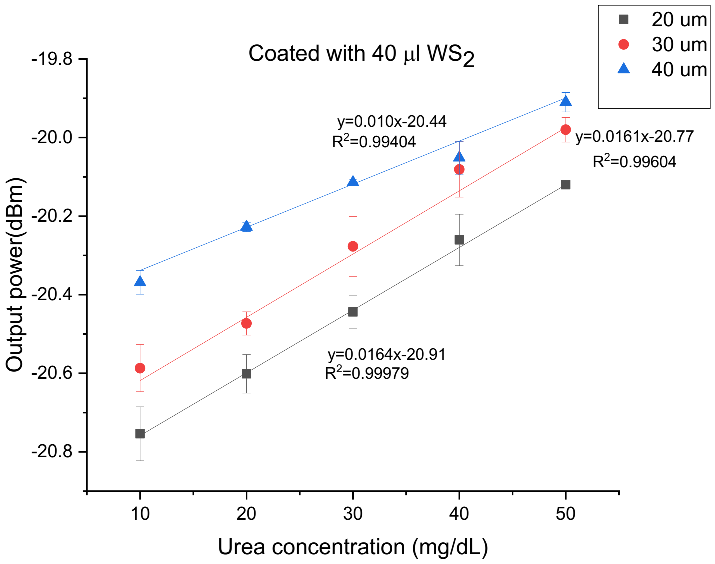


Fig. 8. Output power against urea concentration for the proposed TOF with 40 µl WS$_2$

Based on this experiment, coating WS$_2$ increased the performance of the sensor. However, too much-coated WS$_2$ on TOF will decrease the performance of the sensor. In order to prove coating limitation, an additional experiment is performed by adding another 20 µl of WS$_2$ on top of 40 µl coated TOF. After completing this experiment, the sensor performance decreased drastically with a sensitivity 0.002 dBm/(mg/dL) with linearity of 88%. The performance of the sensor in terms of sensitivity, linearity and LOD will summarize in Table 1.

Table 1
The performance of sensing different concentration of urea

	UNCOATED			COATED						
TOF SIZE (µM)	40 µm	30 µm	20 µm	40 µm			30 µm		20 µm	
VOLUME (µL)				20	40	60	20	40	20	40
SENSITIVITY (DBM/(MG/DL)	0.0123	0.0158	0.0217	0.014	0.01	0.002	0.017	0.016	0.069	0.016
LINEARITY (%MG/DL)	99.7	99.8	99.99	99.5	99.4	88.2	98.67	99.6	99.8	99.9
LOD (MG/DL)	1.884	1.472	2.085	1.638	2.3268	25.71	1.393	1.44	0.6559	2.76

Table 1 summarises the performance of tapered optical fibre based on the sensitivity, linearity and limit of detection (LOD). Coated 20 µl on the 20 µm TOF has sensitivity and LOD, which are 0.069 dBm/(mg/dL) and 0.6559 mg/dL respectively. Based on the result, several factors affect the sensor's performance. The first one is the size diameter of TOF, which can be observed that 20 µm is more sensitive compared to 30 and 40 µm. The results obtained are match well with that size diameter TOF and tapered length influence the sensor performance [28,31]. Furthermore, the sensor's performance can be improved by coating it with nanoparticles or nanomaterial also has been discussed by other researchers [32-34]. In the end of the paper clearly mention and discovers the advantages of tapered optical fibre to improve sensitivity due to simple fabrication and improving sensor performance using WS$_2$ was investigated with optimal coating material also has been clarified.

4. Conclusions

Simple and reliable sensors are validated and evaluated using coated and uncoated tapered optical fibres by varying their size diameter and WS_2 coating volume. The tapered optical fibre was fabricated using Vytran GPX3400. These TOFs were coated with different volumes of WS_2 using a drop casting technique to investigate the sensor's performance for coated and uncoated TOFs. The sensing sensitivity increases by decreasing the 10 µm TOF size diameter with an improvement of approximately 10%-20%. For coated TOF with WS_2, the highest sensitivity of up to 0.0642 dBm/(mg/dL) was obtained with the 20 µL of WS_2 coating using the 20 µm tapered diameter size. In addition, the tapered optical fibre sensor that has been proposed has a linear correlation coefficient of 99.8 percent in the concentration range of 10 to 50 mg/dl. Hence, it can be seen that an effective technique for improving the sensor's sensing sensitivity performance is by using a straightforward coating procedure. The strategy was successful in enhancing the sensor's performance. It is essential to highlight that the proposed WS_2-coated tapered optical fibre sensors could hold considerable potential in clinical detections by further exploiting the additional benefits of WS_2, such as biocompatibility and layer-dependent electrical and optical properties.

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