Surface Activation of Coreless Termination Fibre by Wet Cleaning

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Abstract. Studying the surface treatment of sensing region optical fibre prior to designing and fabricating the complete structure of optical-based transducers is not well understood and still limited in the literature search. At the current stage of this work, preparing to clean the surface of the substrate is essential. Therefore, the surface activation of coreless termination fibre (CTF) by wet cleaning under three different conditions is studied; one of the strategies to improve the surface condition of the sensing region, as described in this paper. Throughout the experiment, three different wet cleaning procedures such as organic, alkaline, and acidic solutions for the surface of the CTF in terms of surface activation prior to preparing high-quality functional coatings thin-film onto the sensing region of CTF are evaluated. Thus, the untreated- and treated-surface of the CTFs are compared. The surface of the CTF was mainly characterised by wettability testing and further analysed by Fourier transform infrared spectroscopy, atomic force microscopy, and UV-Vis-NIR spectroscopy, further detailed. The surface treatment by wet cleaning can improve wettability, surface roughness, and optical responses corresponding to surface activation of the sensing region of CTF. In short, this is just the first step to truly understanding how the optical-type transduction principle using optical fibre works. The groundbreaking discovery could shed light on the contribution of the surface activation of CTF by wet cleaning for the reliability improvement of light-matter interactions lab-scaled dissolved oxygen (DO) optical-based sensing and monitoring devices to advanced optics research on a large scale.

Keywords: Coreless termination fibre, surface activation, wet cleaning, wettability, surface roughness, sensing and monitoring

INTRODUCTION

Detection of the amount of free gaseous oxygen, also known as the dissolved oxygen (DO) concentration present in a liquid medium, is of great importance in (i) blood oxygen level monitoring which collects oxygen from the lungs and delivers it to all parts of the body of severe and critical Coronavirus disease 2019 (COVID-19) patients, due to a novel enveloped ribonucleic acid (RNA) coronavirus, so-called SARS-CoV-2 [1-4], (ii) biological wastewater treatment to turn organic wastes into inorganic byproducts and to enhance the oxidation process by providing oxygen to the aeration basin and aerobic-anaerobic microorganisms [5-8], (iii) aquaculture water as a prerequisite for the healthy growth of aquatic microorganisms [9-12], and many more. In the literature search, DO sensing and monitoring are mainly classified into several types according to the different transduction principles, including electrochemical-type [13-16], followed by the optical-type [17-20], each of which has its outstanding characteristics. However, more transduction principles associated with various energy forms have been found for DO sensing and monitoring, including conventional electromechanical-type, thermoelectric-type, and many more. Unfortunately, these conventional detection methods have many limitations and challenges, making them unsuitable for in-situ measurement.

The optical-type transduction principle using optical fibre has emerged as a DO sensing and monitoring method of choice and gaining attention among scientists due to its good response time, stability, reversibility, repeatability, and sensitivity during the measurement. The optical-type transduction principle is divided into two sensing and monitoring mechanisms, such as (i) the fluorescence quenching effect or followed by (ii) the surface plasmon resonance (SPR) phenomenon. The first mechanism of fluorescence quenching effect of free oxygen molecules with fluorescent dyes (such as the complex of transition metals) embedded into a support matrix (such as polymer or another sol-gel matrix) to prepare oxygen-sensitive membrane is the basis for the optical-fibre based DO transducers. To explain this phenomenon, different DO concentration levels may affect the intensity of the fluorescence spectrum of fluorescent dye during the measurement. Therefore, instead of using the fluorescence quenching effect, the second mechanism shifts in SPR wavelength caused by the change in the refractive index of free oxygen molecules in a liquid medium to calculate DO concentration. As a consequence, the optical-type transduction principle using optical fibre is chosen in this study, due to the simplicity of the design and fabrication of optical-based transducers for sensing and monitoring.

However, the surface treatment of sensing region optical fibre prior functional coatings thin-film process for design and fabrication of optical-based transducers to record signals optically from the amount of gaseous oxygen, also known as the DO concentration, in water so-called light-matter interactions is poorly understood remains limited in the literature search. At the current stage, preparing to clean the surface of the substrate is essential for achieving high-quality functional coatings thin-film. Therefore, the main focus of this work, the surface activation of coreless termination fibre (CTF) by wet cleaning under different conditions such as organic, alkaline, and acidic solutions, is studied; one of the strategies to improve the surface condition of the substrate as described in this paper. Thus, the untreated and treated-surface characteristics of the CTFs are compared using the ATR-FTIR spectrometer, UV-Vis-NIR spectrometer, atomic force microscope, and contact angle with wettability measurement. This could shed light on the contribution of the surface activation of CTF by wet cleaning to the reliability improvement of optical-based sensing and monitoring devices. For the next stage of this work, we aim to develop a new simple, inexpensive, and quick method to analyse water

behaviour, which can help investigate DO change in environments such as oceans and freshwater rivers and lakes at a large scale.

MATERIALS AND METHOD

The prototype of the sensing devices was designed and fabricated. The chemicals used in this experimental study are analytical grade and used as is without purification procedure. The undoped, pure silica glass with approximately 99.99% purity of coreless termination fibre (CTF) diameter of CTF used in this work was 125 ± 1 μm. The core and cladding diameter of single-mode fibre (SMF) was 8.5 μm and 125 μm, respectively. The lead-in SMF was spliced with CTF using a fusion splicer (Model: Fujikura, Arc Fusion Splicer, FSM-175), and then spliced with the lead-out SMF structure. The length of each side of the SMF and the sensing region of CTF were 20 cm and 2 cm, respectively. The SMF/CTF/SMF /schematic diagram is described in Fig. 1.

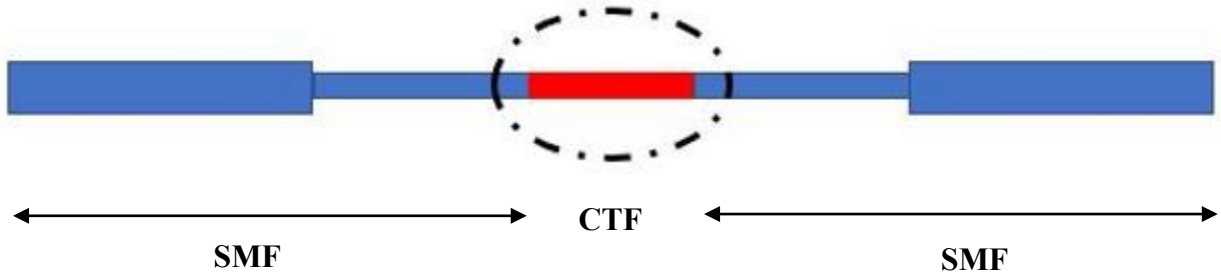

FIGURE 1. The schematic diagram (not to scale) of the SMF/CTF/SMF structure. The red colour shows the sensing region of the CTF, cascaded with SMF.

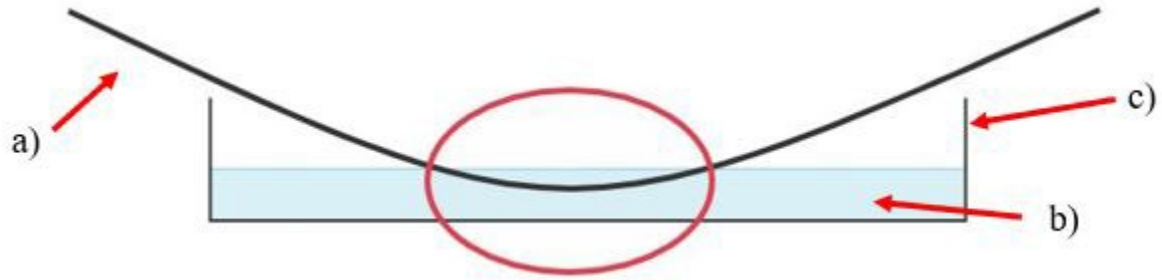

FIGURE 2. The schematic diagram (not to scale) of the side view of the surface activation of CTF by wet cleaning. The red circle in the diagram showing only the sensing region of the CTF structure will be fully immersed in the prepared solution. a) is showing the SMF/CTF/SMF structure, b) is showing the different prepared solutions of STEP1: ethanol/isopropyl alcohol, STEP2: 1M sodium hydroxide, STEP3: 1M sulphuric acid, and c) is showing the petri dish.

The prototypes of SMF-CTF-SMF structure were cleaned with different cleaning and surface activation methods are shown in Fig. 2. First, for initial cleaning (STEP1), the surface of the sensing region was immersed in ethanol for 5 minutes. Then, it was followed, the surface of the sensing region was immersed in isopropyl alcohol for 5 minutes and rinsed with ultrapure deionised water (Milli-Q, Gradient A10 18.2 M-Ohm, Millipore Inc). Next (STEP2), the surface of the sensing region was immersed in 1M sodium hydroxide for 5 minutes and rinsed with ultrapure deionised water. After that (STEP3), the surface of the sensing region was immersed in 1M sulphuric acid for 5 minutes and rinsed with ultrapure deionised water. The post-rinsing process for these three samples was carried out in the ultrasonic bath for 5 minutes. Finally, they

were wiped with industrial-grade tissue wipers and dried under ambient pressure and temperature in the air.

The efficiency of the cleaning and surface activation methods was verified with Attenuated Total Reflection Fourier-Transform Infrared (ATR-FTIR) Spectrometer (Spectrum 400, Perkin Elmer), UV-Vis-NIR spectrophotometer (UV-3600 Plus, Shimadzu), Atomic Force Microscope (AFM-SPI13800N, Seiko) with cantilever type of OMCL-AC200TS-C3, and static contact angle and wettability measurement (VCA Optima XE, AST Products). All the measurements were conducted under ambient pressure and temperature in the air. The spectra were plotted using the Origin 2021 - Data Analysis & Graphing software.

RESULTS AND DISCUSSION

Before the cleaning and surface activation procedure is carried out, the surface optical fibre (specifically SMF-CTF-SMF structure) may contain different contaminations such as metal impurities or organic and various unknown particles, including dust which is lied to the surface by van der Waals force or weak electrostatic forces [21]. After proposing different cleaning and surface activation procedures, the contaminations are hypothesised completely to be removed on the sensing region of CTF, resulting in a change in functional groups characterised by ATR-FTIR, surface roughness characterised by AFM, wettability evaluated by contact angle and wettability testing, and optical behaviour measured by UV-Vis-NIR spectroscopy. However, prior to achieving this goal and producing good functional coatings, the sensing region of the CTF must be free of adherent particles and be chemically receptive to the desired condensing and nucleating atoms. Furthermore, the surface of the substrate to be coated must be appropriately cleaned and well-prepared, basically to ensure strong adhesion bonds between the coating materials and substrate. We have four types of samples, including the untreated-surface CTF (known as the bare-CTF) and three treated-surface CTFs after exposure to different cleaning and surface activation procedures (cleaned-CTF STEP1, STEP2, and STEP3), that would be analysed in this work. Thus, the untreated- and treated-surface of the CTFs are compared.

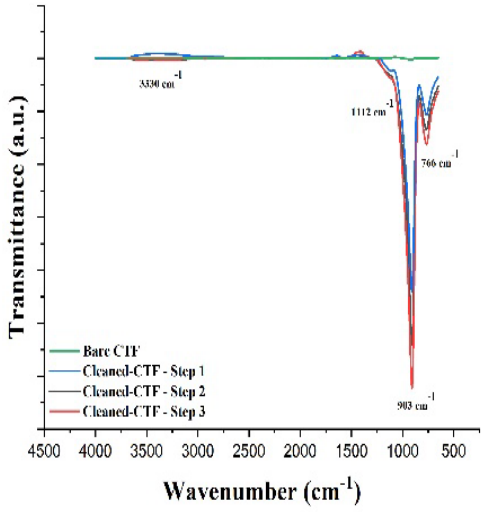

FIGURE 3. FTIR spectroscopy analysis of the untreated- and treated-surface of the CTFs.

By investigating using ATR-FTIR spectroscopy at ambient conditions, we confirmed the activation effect on the surface of the sensing region of CTF; after exposure to organic, alkaline, and acidic solutions. The graph of transmittance spectra of ATR-FTIR spectroscopy analysis was plotted from 650 to 4000 cm^{-1}, with a data interval of 1 cm^{-1} and a resolution of 1 cm^{-1}. This analysis aims to analyse the surface activation by observing the functional groups of all the absorption peaks, as shown in Fig. 3. The occurrence of the band position around 903, 766, and 3330 cm^{-1} were attributed to the assignment of C-H bending, C-O bending, and N-H stretching vibrational modes, respectively. In addition, routine interstitial oxygen measurements on silica-based optical fibre have revealed a peak at 1112 cm^{-1}. The Si-O stretching vibration mode in the silicon lattice is responsible for this absorption band. This mainly originates from oxygen precipitates inside the CTF during the fabrication process. The untreated surface of the CTF was analysed. Broad peaks with low intensity at 903 and 1112 cm^{-1} of the CTF untreated surface were observed in the spectrum. This measurement shows that the condition of an untreated surface of the CTF was found to be in an '*inert*' state due to almost being free from functional groups attached to it. Therefore, it is possible to relate a change in peak intensity to the activation effect on the surface of the sensing region of CTF. Next, the treated surface of the CTFs were then analysed after exposure to ethanol/isopropyl alcohol, sodium hydroxide, and sulphuric acid. The increase in this intensity of the band position around 903 of the C-H bending vibrational mode, 766 cm^{-1} C-O bending vibrational mode, and 1112 cm^{-1} Si-O stretching vibrational mode support C-H, C-O, Si-O bonds are formed at the surface during the treatment with the activation solutions. The 1M sulphuric acid was the most potent in Si-O, C-H and C-O group formation when the different wet cleaning solutions were compared. Unfortunately, there is not much difference in the intensity of the band position around 3330 cm^{-1} of the N-H stretching vibrational mode when the different wet cleaning solutions were compared.

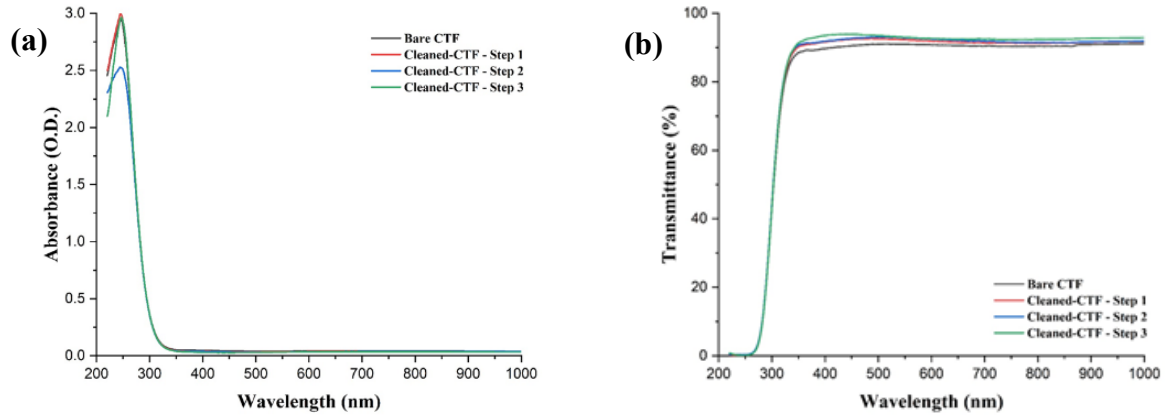

FIGURE 4. (a) Absorbance and (b) transmittance spectra of UV-Vis-NIR spectroscopy analysis of the untreated- and treated-surface of the CTFs.

The ambient conditions UV-Vis-NIR absorbance and transmittance spectra recorded in the range of 200 to 1000 nm of the untreated- and treated-surface of CTFs were presented in Fig. 4 a) and b). Similar trends were observed for both graphs. Furthermore, both spectra showed insignificant changes in the optical behaviour of the samples in the visible region of the electromagnetic spectrum lies between the UV and the IR regions. Thus, the results verify that

the untreated and treated surface of the CTFs were not damaged after wet cleaning procedures were employed, resulting without harming their optical characteristics.

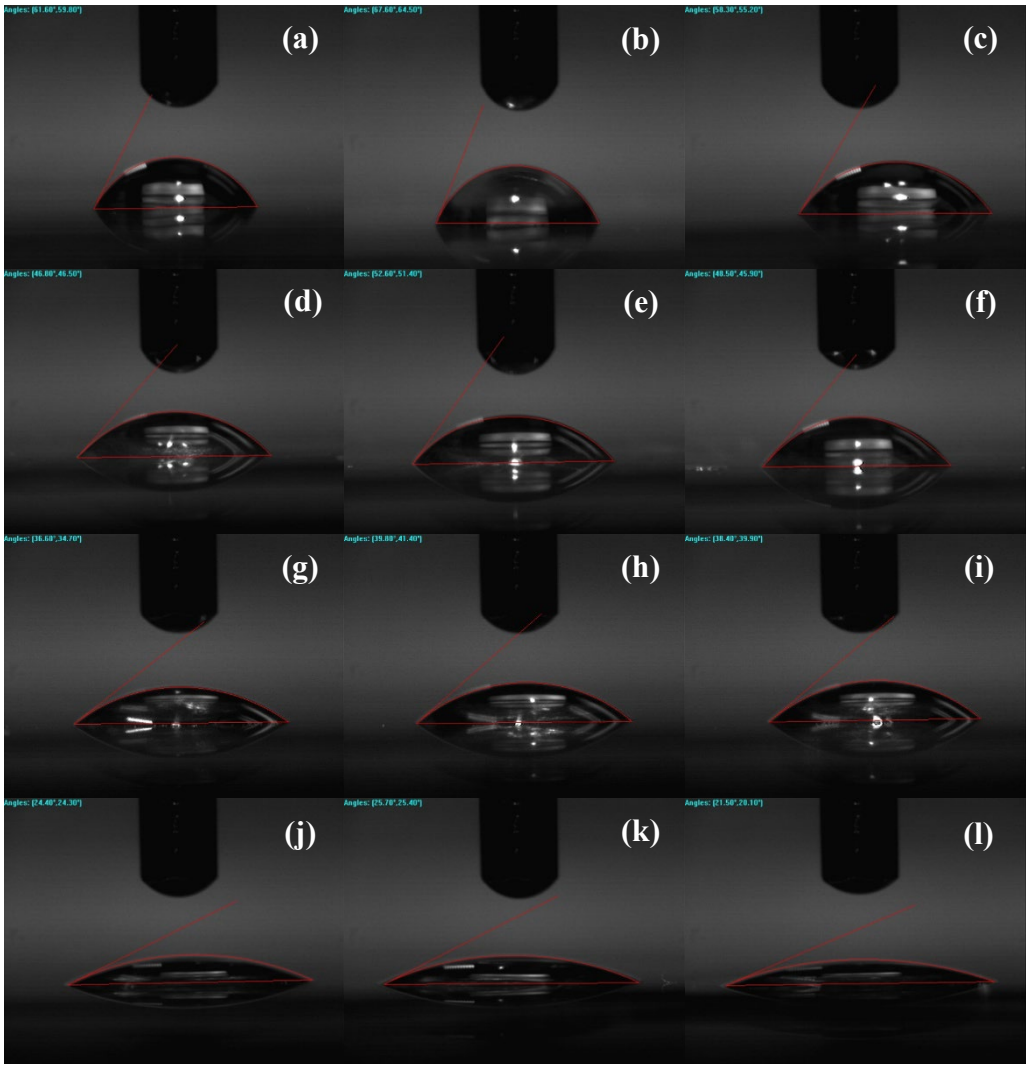

FIGURE 5. Deionised water drops and contact angles measurement on the untreated-CTF surfaces for three trials (a to c), the treated-CTF surfaces with ethanol/isopropyl alcohol for three trials (d to f), the treated-CTF surfaces with sodium hydroxide for three trials (g to i), and the treated-CTF surfaces with sulphuric acid for three trials (j to l), for different wet cleaning procedures.

The static contact angles were measured based on the 1 µL deionised water sessile drop method to understand the activation effect on the surface of the sensing region of CTF correspond to wettability characteristic; after exposure to organic, alkaline, and acidic solutions. The images and contact angles were taken within 30 s after the sessile drop formation and at three different locations for each sample, which shows consistency of the measurements. The usefulness of the surface treatment in the processing of clean surfaces with enhanced wettability is discussed. The images are presented in Fig. 5, and the corresponding values of the data are given in Table 1. The initial average contact angle reading for the untreated sample was 62.50^{o}. And then, several activation solutions were applied during a wet cleaning procedure. After exposure to ethanol/isopropyl alcohol, sodium hydroxide, and sulphuric acid, samples resulted in average contact angles further decreasing to 49.30, 38.40, and 28.87^{o}, respectively, which effectively cleans the surface and leads to a highly hydrophilic interface. After treatment, the

wetting envelopes of the deionised water widened, indicating increased wettability. The contact angles noticeably decrease after the samples have been treated. In other words, to support these findings compared with the previous ATR-FTIR measurement, the wet cleaning procedures alter the free energy of the surface by converting the surface groups. As a result, the surface of the sample is more likely to react with deionised water, resulting in a hydrophilic surface.

TABLE 1. Contact angle measurements for different wet cleaning procedures. The measurements were conducted at three different locations of the sensing region for each sample.

	Trial 1 (degree)	**Trial 2 (degree)**	**Trial 3 (degree)**	**Average (degree)**
Bare CTF	61.60	67.60	58.30	62.50
Cleaned CTF – Step 1	46.80	52.60	48.50	49.30
Cleaned CTF – Step 2	36.60	39.80	38.80	38.40
Cleaned CTF – Step 3	24.40	25.70	21.50	28.87

AFM scanned all four images with an area of 4 x 4 μm^2 of CTF sensing region. Tapping mode AFM images of the surfaces treated with the different wet cleaning procedures were observed in Fig. 6. The initial surface roughness, R_a, reading for the untreated sample was 0.274 nm. After exposure to ethanol/isopropyl alcohol, sodium hydroxide, and sulphuric acid, samples resulted in R_a further increasing to 0.413, 0.625, and 1.168 nm, respectively. Thus, in this study, these three wet cleaning methods increased the surface roughness significantly. We believe that increased surface roughness with good homogeneity and improved wettability of the sample is better adhesion at optical fibre and functional coatings thin-film matrix interface.

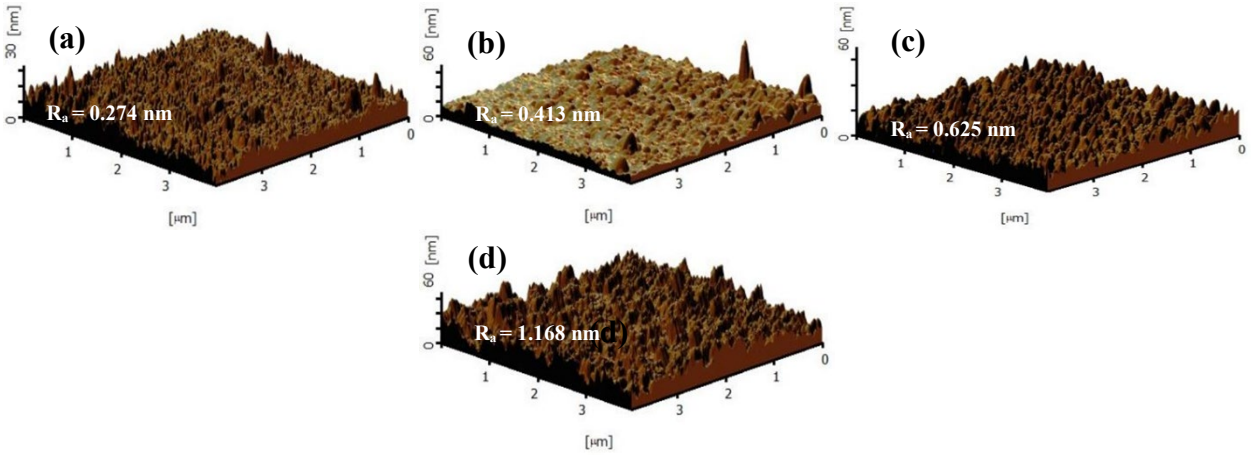

FIGURE 6. Surface roughness, R_a measurement characterised by atomic force microscopy of (a) the untreated-CTF surface, (b) the treated-CTF surfaces with ethanol/isopropyl alcohol, (c) the treated-CTF surfaces with 1M sodium hydroxide (d) and the treated-CTF surfaces with 1M sulphuric acid, for different wet cleaning procedures.

CONCLUSION

In this study, three different wet cleaning procedures by exposing the sensing region of the CTF with organic, alkaline, and acidic solutions, have been demonstrated and evaluated. We used different characterising tools such as ATR-FTIR, UV-Vis-NIR, AFM, and contact angle and wettability measurement to verify the efficiency of the cleaning and surface activation methods, prior functional coatings thin-film process for design and fabrication of optical-based transducers to record signals optically from the amount of gaseous oxygen, also known as the

DO concentration. This explains that improving the optical response, functional groups, wettability and surface roughness may achieve a better activation effect on the surface of the sensing region of CTF. From the findings, the substrate exposed with 1M sulphuric acid has shown an excellent optical response in UV-Vis-NIR analysis, the most potent in Si-O, C-H and C-O group formation in ATR-FTIR analysis, resulting in a hydrophilic surface in static contact angle and wettability measurement, and improved surface roughness in AFM measurement. Other treatments are being researched and will be discussed in future studies. Implications of the current study for the surface activation of CTF by optimised wet cleaning procedure may be essential for achieving high-quality functional coatings thin-film in future work and the reliability improvement of optical-based sensing and monitoring devices to advanced optics research. In conclusion, the research shows there is still a long way to go in DO sensing and monitoring.

DISCLOSE STATEMENT

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 manuscript.

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