Graphene Coated Optical Fiber Tip Sensor for Nitrate Sensing Application

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***Abstract*—An optical fiber tip sensing probe coated with graphene is proposed for nitrate-sensing application. The sensor was coated with graphene using dip-casting method to detect nitrate at different concentrations ranging from 0 to 50 ppm. The graphene-coated fiber tip sensor was highly sensitive in detecting nitrate ion. When the concentration of the nitrate solution was dropped onto the sensing region, the reflected optical power decreased as the nitrate concentration increased. Without the graphene layer, the uncoated fiber tip sensor only had a sensitivity of 0.0076 dBm/ppm. However, the coated sensor's sensitivity was significantly enhanced to 0.0624 dBm/ppm, eight times higher than the uncoated fiber tip sensor, mainly due to the adsorption of nitrate on the graphene surface. This graphene-coated optical fiber tip sensor has a high potential to be used in nitrate-sensing applications.**

***Index Terms*—Nitrate; Graphene; Optical fiber tip sensor; Sensitivity**

I. INTRODUCTION

Nitrate (NO_3^-), a negatively charged ion or anion, is a naturally occurring ion that is part of the nitrogen cycle. Naturally, the level of nitrate is very low in water. However, the overabundance of nitrate becomes threatening upon findings of its detrimental impact to human health. The overuse of inorganic fertilizers in modern agriculture [1] as well as disposal of untreated municipal and industrial wastes [2] have increased the level of nitrate. Ingestion of high level of nitrate reduces the blood's efficiency to carry oxygen, causing methemoglobinemia or also known as 'blue baby syndrome'.

Several methods including chromatography [3], electrochemical analysis [4] and capillary electrophoresis are used for nitrate detection in a variety of samples but the need of sample pre-treatment, derivatization reaction, and preanalysis separation reduces the practical viability of these techniques.

This study presents an alternative method to determine nitrate concentration at part per million (ppm) level in water by applying optical fiber tip sensor without the complexity of chemical process to detect the nitrate ions.

In this study, we introduce an optical sensing technology using graphene-coated optical fiber tip sensor to determine the concentration of nitrate at 0-50 ppm level in water. Our main interest is the application of graphene as the sensing layer and the optical fiber tip itself as a chemical sensor where the optical response changes by changing the chemical composition around the sensing region.

II. METHODS

The tip of a standard single mode optical fiber was cleaved to produce a flat-end surface. This surface acted as the sensing head. The tip was coated with graphene using the dipcasting method and left for drying at room temperature for 1 h. Potassium nitrate powder [KNO3, Analytical Reagent Grade, R&M Chemicals] was used to prepare the aqueous solutions containing nitrate concentrations at 0-50 ppm.

The experimental setup for the proposed nitrate sensor is shown in Fig. 1. Detailed embodiment of the proposed nitrate sensor is illustrated in the inset of Fig. 1. In the experiment, a continuous spectrum of amplified spontaneous emission (ASE) source operating at 1559 nm region was used as a light source in conjunction with an optical spectrum analyzer [Yokogawa (AQ6370D), 0.02 nm resolution] to monitor the optical sensing response.

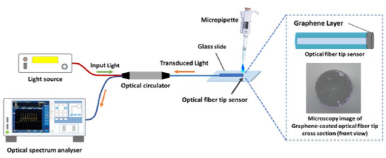

Fig. 1. Experimental setup for nitrate sensing.

The sensor was fixed on a glass slide to reduce any physical disturbance. Throughout the experiments, the nitrate solution was dropped onto the sensing region using a 100 μL micropipette. The sensitivity of the sensor for nitrate detection was measured from the reflected signal response.

III. Result & Discussion

A. Uncoated Optical Fiber Tip Sensor Response

In the first stage of the experiment, a version of optical fiber tip sensor without coating was used to perform the measurement to compare the effect of graphene as the sensing layer. The nitrate concentrations were increased from 0 to 50 ppm during the experiments.

Fig. 2 shows the reflected optical response of the uncoated sensor. In Fig. 2(a), the reflected sensing spectrum of the uncoated sensor has slightly distinguished the different nitrate concentrations. The responses were expected to be unique due to the changes in the nitrate refractive index. Since there were no significant wavelength changes observed throughout the nitrate concentration variation, we only analyzed the reflected light at 1559 nm peak wavelength, according to the available ASE light source. Fig. 2(b) shows the relationship between reflected optical power and nitrate concentration. As the nitrate concentration range was changed from 0 to 50 ppm, the reflected light power decreased from -47.97 to -48.33 dBm, resulting to 0.36 dBm of total reflected power difference. The measured sensitivity achieved by this sensor is 0.0076 dBm/ppm and the calculated linear regression (R2) value is 0.9361.

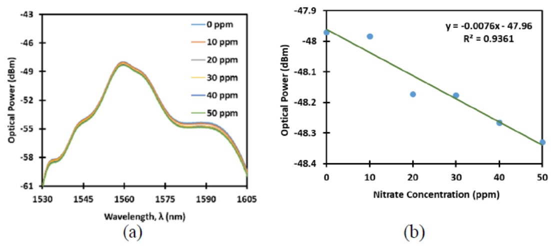

Fig. 2. The analysis of uncoated optical fiber tip sensor for nitrate detection (a) The spectral image of the reflected light for nitrate concentration in the range of 0 – 50 ppm; (b) The relationship between reflected optical power and nitrate concentration.

B. Nitrate-Sensing Response with Graphene Coating

In the second stage of the experiment, graphene-coated optical fiber tip sensor was used to perform the measurements. Fig. 3 shows the relationship between the reflected light intensity and nitrate concentration for graphene-coated fiber tip sensor. Fig. 3(a) shows the sensing spectrum of the coated sensor. There was a significant spectral change shown by the coated sensor. Fig. 3(b) shows the relationship between reflected light power and nitrate concentration. The reflected light decreased gradually with the increase of nitrate concentration. As the nitrate concentration range was increased from 0 to 50 ppm, the reflected light power decreased from -49.4233 to -52.0467 dBm, giving a total value of 2.6234 dBm of power loss. The calculated sensitivity achieved by this sensor is 0.0624 dBm/ppm. All the experimental data fit well with the line of best fit, thus giving an R2 value of 0.9666.

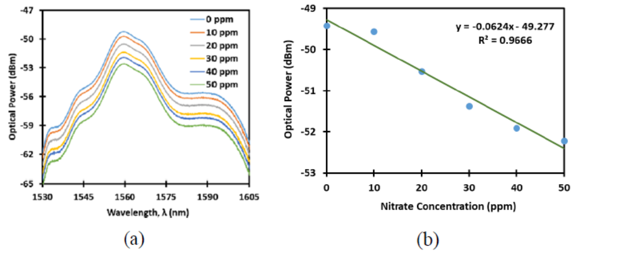

Fig. 3. The analysis of graphene-coated optical fiber tip sensor for nitrate detection (a) sensing spectra of nitrate concentration ranging from 0-50 ppm; (b) the relationship between reflected optical power and nitrate concentration.

Overall, the reflected optical power of the optical fiber decreased when the concentration of nitrate increased. However, the experimental results indicate that the sensitivity of the graphene-coated fiber tip was enhanced, and the total change of the reflected light power was higher compared to the uncoated sensor. This is because graphene has been proved as an excellent material for nitrate adsorption [5]. Moreover, graphene-coated fiber tip also provided a better linearity with higher linear regression. Hence, the presence of graphene layer significantly enhanced the sensitivity of the nitrate sensor eight times higher than the uncoated fiber tip sensor.

IV. Conclusions

The nitrate sensor was experimentally demonstrated using graphene-coated optical fiber tip. Nitrate adsorption onto the graphene surface modulated light reflectivity with a maximum nitrate detection sensitivity of 0.0624 dBm/ppm. The presence of graphene layer greatly enhanced the sensor's sensitivity to eight times higher than the uncoated fiber tip sensor. This shows that the graphene-coated optical fiber sensor has a high potential for use in nitrate-sensing. The sensor has a promising potential to be viable in advanced sensor applications. In future, the goal is to exploit this probe sensor for wastewater-monitoring applications.

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