Sodium nitrate (NaNO 3) sensor based on graphene coated microfiber

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Sodium nitrate ($NaNO_3$) sensor based on graphene coated microfiber

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ABSTRACT

We demonstrate the coating of graphene onto the silica optical microfiber sensor for improving sodium nitrate detection at room temperature. The graphene obtained from graphene- polylactic acid filament was coated onto the microfiber based on drop casting technique. In the proposed sensor, the graphene acts as cladding to interact with analyte as well as functions to trap either sodium cation or nitrate anion and increases the effective refractive index of the cladding. The proposed sensor shows a good sensitivity of 1.29 dBm/% and resolution of 0.049%. The sensitivity, repeatabilty and reversible response of the sensor were improved by the coating of graphene layer. The presence of graphene on the surface microfiber works as passive cladding and influence the light propagation through the microfiber.

1. Introduction

Sodium nitrate have been used widely as food additives to cured the meat color, flavour, and inhibiting rancidity by restrain lipid peroxidation. Carcinogenic substances such as N-nitroso compounds (NOC) in sodium nitrate can cause cancer and methemoglobinemia if consumed in high concentrations [1,2]. In order to avoid cancer risk in the human body, the monitoring of sodium nitrate level is essential [3], also dietary nitrate can significantly reduce systolic blood pressure [4]. According to European Union, 150 ppm concentrations of sodium nitrate are permitted for addition to meat products, whereas in the United States in most product nitrite is permitted as a curing agent around 100 ppm [5]. It has been reported by NDEA, levels permitted sodium nitrate in bacon is 0.067, 0.149 ham, and 0.040 μg/100 g in sausage [6]. Therefore, the need for sodium nitrate biosensors is pressing.

Graphene has several excellent optical characteristics including thermo-optic, optical adsorption, molecular adsorption, surface plasmon, photolumination, strong nonlinearity, and photovoltaic effect [7–9]. For instance, it sensitive to changes in ambient temperature due to the high thermal conductivity. The refractive index of graphene will also change clearly when the temperature changes [10]. The graphene has also the absorption rate of 2.3% per layer, which is useful for many applications. Theoretical research shows that monolayers and multiple layers of graphene absorb less TM mode in total internal reflection than TE light modes. Resolution and sensitivity can be significantly enhanced by evaluating the difference in reflected light intensity from the TE light modes and TM mode [10–12]. Graphene as smart material shows the potential for biochemical sensing such as for detection of magnesium [13], glucose [14], and cholesterol [15].

On the other hand, optical microfibers provide controllable waveguide dispersion [16], tight optical confinement [17], and strong evanescent fields [18]. This unique property makes them more sensitive to the environment and thus useful for various sensing application including refractive index sensor. The great evanescent fields in the microfiber have been demonstrated as physical, chemical and biological optical sensors [19–25]. The small diameter of the microfiber waist within a few of micrometers enable a higher amount power of evanescent field to interact with the surrounding making them more sensitive to the variation of outer stimulus. The evanescent field of the microfiber can be further enhanced if it is coated with the sensitive material [26–31]. For instance Sun et al. demonstrated a sensitive temperature sensor by depositing graphene onto the microfiber [32]. In another work, Zhou et al, has developed an improved γ-aminobutyric acid

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(GABA) sensing approach based on graphene oxide interface on an optical microfiber [33]. Different kinds of gas sensors were also proposed and demonstrated by depositing graphene onto microfibers so that it can interact with the evanescent field [34].

In this paper, we report on the development of new evanescent wave sensor for sodium nitrate detection using an optical microfiber, which was coated with thin layer of graphene. The graphene was obtained using a new low cost approach based on Graphene-Polylactic acid (PLA) filament which was normally used for 3D printing. Compared to the previous works [32–34], the proposed techniques are simpler. In this work, graphene nanocomposite is used as the passive cladding material to interact with the specimen. The sensitivity response, resolution and the stability of the sensor is studied accordingly.

2. Experiment

The graphene Polylactic acid (PLA) filament has a volume resistivity of 0.6 Ohm-cm and diameter of 1.75 mm. At first, a graphene-PLA filament was extruded using 3D printer nozzle (0.4 mm diameter) at 210 °C to reduce the diameter from 1.75 mm to 200 μm. Then, the 25 mg of extruded Graphene-PLA was mixed with 2 ml of Tetrahydrofuran (THF), and in order to dissolve the PLA and produce the graphene-THF suspension, the mix solution is ultrasonic for 2 min. The graphene THF solution was drop onto a glass slide for Raman spectroscopy and FESEM measurements. Fig. 1(a) shows the Raman spectrum obtained after THF evaporated at ambient temperature. The distinct peaks of Raman shift for D band, G band and 2D band are observed at 1348 cm^{-1}, 1582 cm^{-1} and 2699 cm^{-1}, respectively. The Raman spectroscopy also revealed the Intensity of G peak and 2D peak which is at 389 and 287, respectively with the ratio of I(G)/I(2D) was about 1.35 indicating that the sample is a multi-layer Graphene with the number of Graphene layer (nGL) of around 25 layer [18] A peak is also observed at 1453 cm^{-1} due to the glass slide. Fig. 1(b) shows the FESEM image showing the morphology of the graphene slurry solution. The prepared graphene solution was deposited into the optical silica microfiber by using drop coating method for sensing application. The droplets were left for a few seconds to let the THF to evaporate, leaving only the graphene coated onto the microfiber outer surface. The prepared graphene coated microfiber was kept at room temperature for at least 48 h before it can be used for the experiment to ensure that the graphene thin layer is not washed out when immersed in $NaNO_3$ solution.

Fig. 2 shows the schematic experimental setup for the proposed sodium nitrate sensing. A silica microfiber with diameter 6 μm and length of 2 cm was fabricated by flame brushing technique [26] using single mode fiber (SMF corning 28). One end of the microfiber was connected to the amplified spontaneous emission (ASE) as light source while another end was connected to the optical spectrum analyzer (OSA) for the measurement. Various concentrations

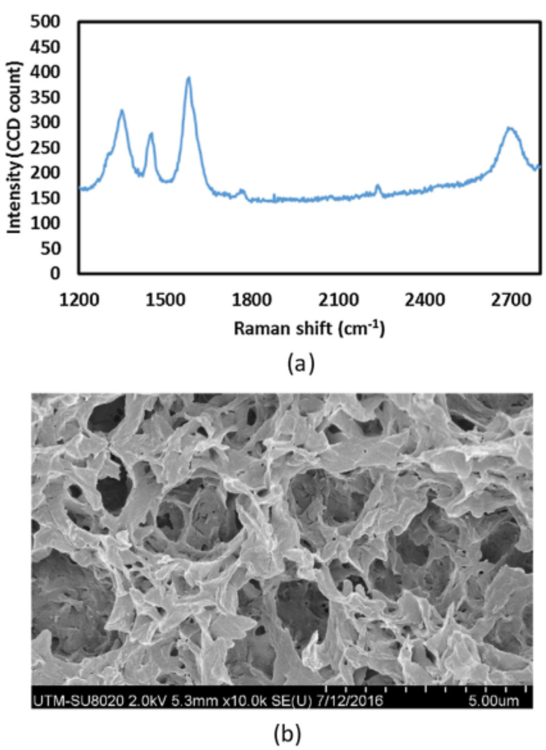


Fig. 1. Characteristic of the prepared graphene solution (a) Raman spectrum (b) FESEM image.

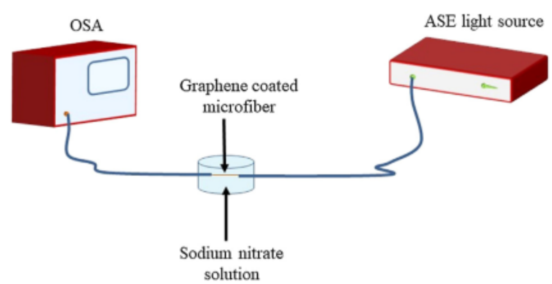


Fig. 2. Schematic experimental setup used for sodium nitrate sensing.

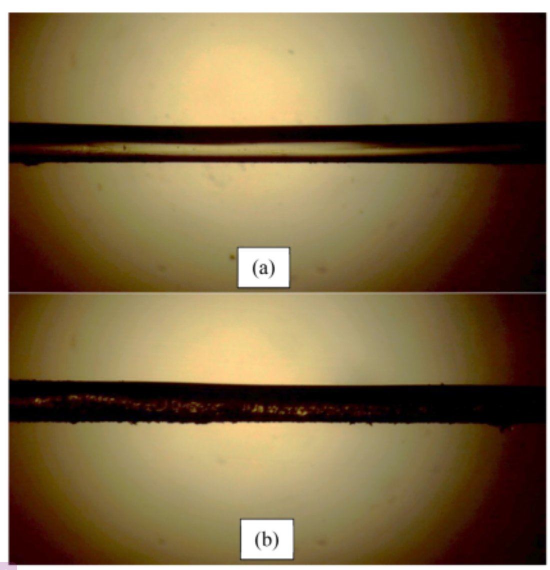
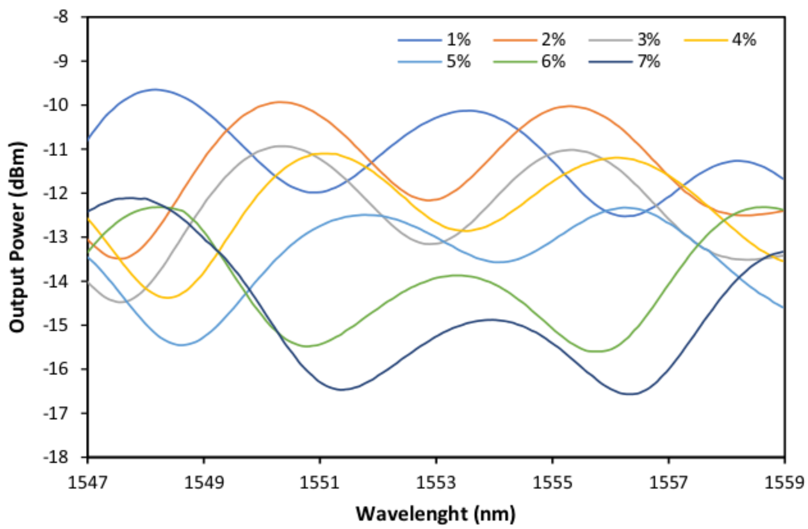
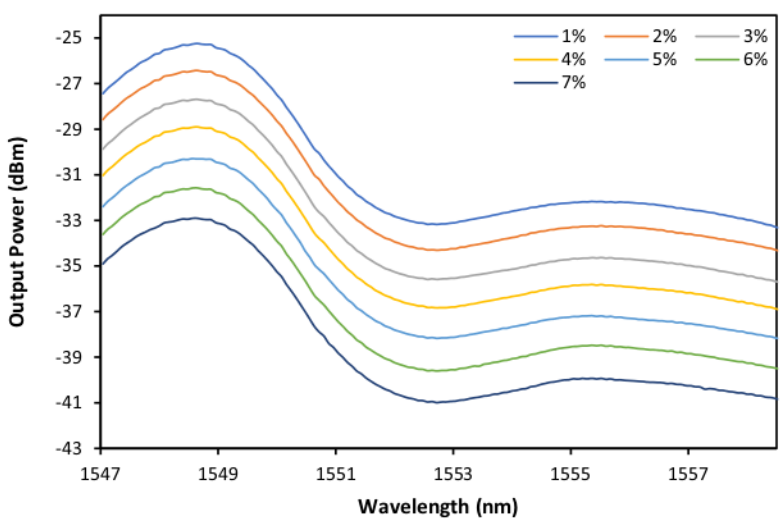


Fig. 3. Microscopic image of microfiber with (a) waist diameter of 6 μm, (b) presence of a graphene layer at the microfiber.



(a)



(b)

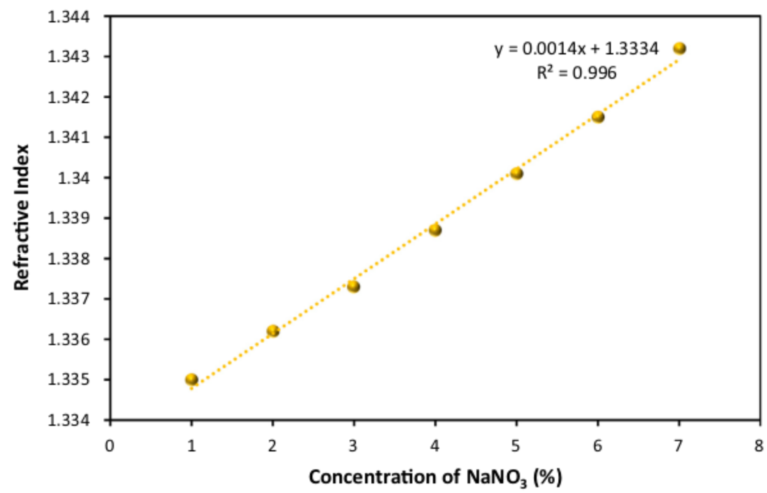
Fig. 4. Transmission spectra from (a) uncoated (b) graphene layer coated microfiber based sensors.

of sodium solution in a fix quantity was placed in the petri dish. The sensor probe was immersed into the $NaNO_3$ liquid with various concentrations ranging from 1% to 7% during the experiment while investigating the transmission spectrum and output power.

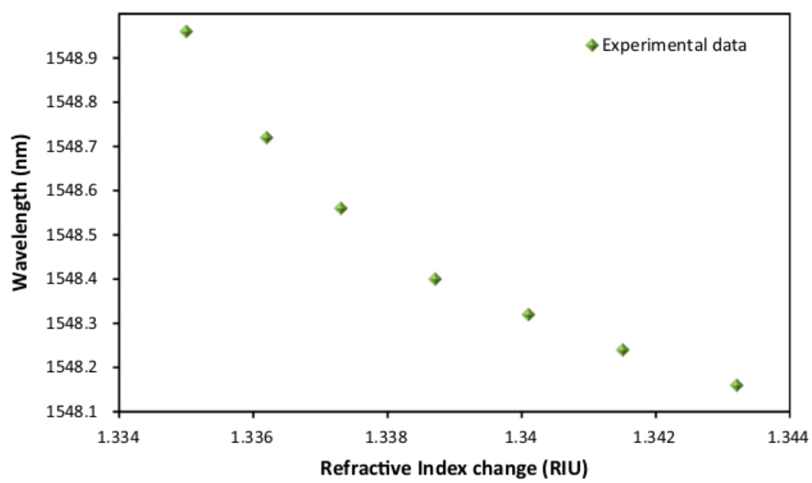
In this work, we firstly immerse the microfiber without graphene coating into the sodium nitrate solution with various concentrations ranging from 1% to 7%. Then the same experiment was repeated with the graphene coated microfiber probe. The small changes response of the sensor can be observed using small working range. We used abbe refractometer to measure the refractive index of each sodium nitrate solution. The microscopic image of the sensor probe without and with the presence of a graphene layer is shown in Fig. 3(a) and (b), respectively.

3. Result and discussion

Fig. 4(a) and (b) show the transmission spectra obtained at various concentration of sodium nitrate for the microfiber probe without and with graphene coating, respectively. It is observed that the interference comb is significantly reduced as the graphene layer is coated on the surface of the microfiber. This is happened due to the reflection at the microfiber decrease as the index contrast between two medium decrease. The refractive index of the sodium nitrate at different concentrations of solution from 1% to 7% is shown in the Fig. 5(a). Fig. 5(b) shows the resonant peak wavelength against the the refractive index of the sodium nitrate solution for graphene coated microfiber based sensor. It indicates a wavelength shift



(a)



(b)

Fig. 5. (a) refractive index against sodium nitrate concentrations (b) resonant peak against the refractive index indicating a shift to shorter wavelength when the index of the sodium nitrate is increased.

with sensitivity of the sensor 93.1 nm/RIU on the increasing refractive index. As the refractive index of the sodium solution increases from 1.335 to 1.3432, the wavelength transmission spectra shift from 1548.96 nm to 1548.16 nm.

In the proposed sensor, the evanescent field of the microfiber depends on the surrounding medium, refractive index of the fiber, and the wavelength operation. Thus the slightly increase of the refractive index reduces the transmission light output power while shifting the peak wavelength to a shorter region. The increase of $NaNO_3$ concentration reduce refractive index contrast at the sensor probe, which allows more light leakage to the cladding and air. The change of index contrast is more significant with the existance of graphene layers, which trapped more ions. It is also worthy to note that the contrast reduction of the interference spectrum may be related to the increase in higher order mode loss caused by the introduction of graphene.

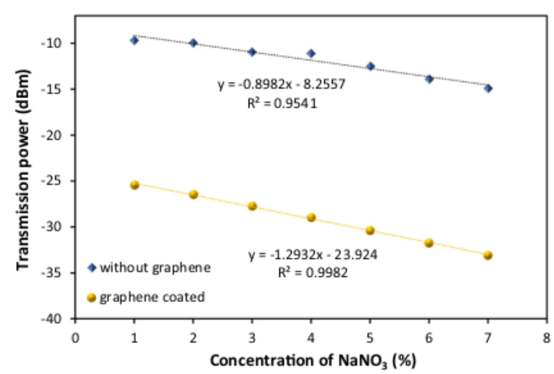


Fig. 6. Transmission power of the sodium nitrate sensor at various sodium nitrate concentrations.

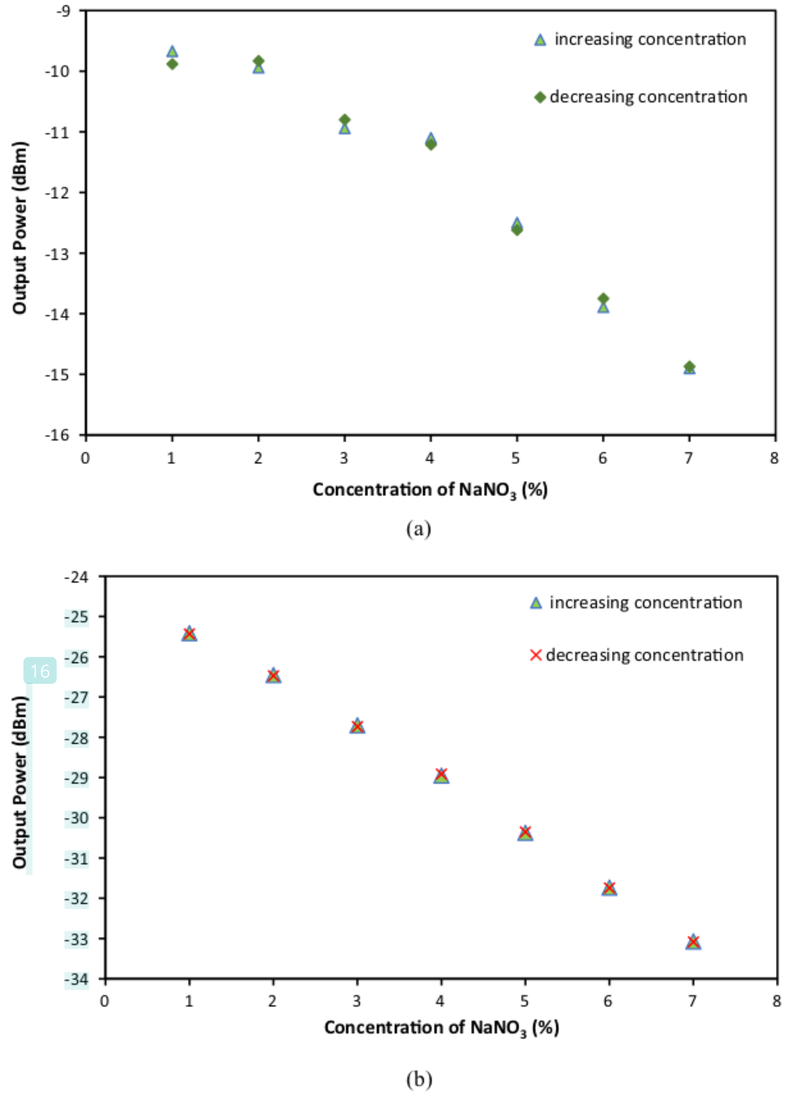


Fig. 7. Reversibility of the silica microfiber (a) without graphene coating (b) with graphene layer deposited onto the silica microfiber surface.

Fig. 6 shows the sensor response of bare microfiber and graphene coated microfiber when exposed to different concentration of sodium nitrate. As shown in the figure, the transmission power decreases from −9.67 dBm to −14.9 dBm as the sodium nitrate solution concentration increases from 1% to 7% for the uncoated sensor. With the graphene coating, the transmission power decreases from −25.41 dBm to −33.07 dBm. It can be seen that the increase in the refractive index of sodium nitrate decreases the confinement of the guided light in the microfiber, which then leads to the increase in optical loss. The graphene coating also increases the cladding refractive index, which reduces the cladding modes and degrades the resonance effect. The graphene material increases the sensitivity of the sensor by changing the refractive index at the surface of the microfiber due to the interaction between the graphene and the ions inside sodium nitrate solution. This in turn changes the effective index of the propagating mode guided along the microfiber and resulting in the shifting of the resonance peak. In the experiment, graphene coated silica microfiber has sensitivity 1.29 dBm/% with resolution of 0.049% and slope linearity of 99% which is better than the uncoated silica microfiber. The uncoated sensor has a sensitivity, resolution and slope linearity of 0.89 dBm/%, 0.143% and 95%, respectively.

The relative low transmission power level in the graphene coated microfiber when immersing in the various sodium nitrate concentrations due to the refractive index of the surrounding medium is higher than water. The graphene layer on the surface microfiber works as passive cladding since the waist of fiber reduced into 6 μm and influence the propagates light through the fiber. The higher sensitivity of the sensor caused by increasing of the evanescent field penetration of guided modes [16] and the graphene material also significantly enhancing the evanescent fields of the microfiber and is sensitive to local refractive index so that it greatly improving the sensitivity of the silica microfiber sensor with the local environment [13,32]. The core and cladding index for the

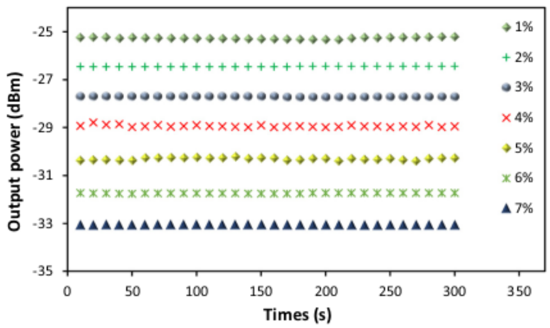


Fig. 8. Stability of the graphene coated silica microfiber for various sodium nitrate solution.

Table 1
Comparison of sensors performance.

Parameters	without graphene	with graphene
Sensitivity (dBm/%)	0.89	1.29
Standard deviation (dBm)	0.127	0.063
Resolution (%)	0.143	0.049
Linearity %	95	99
Linear ranges (%)	1–7	1–7

sensing element drops with the increment of the sodium nitrate concentrations when the refractive index of the graphene increases. The electrical properties of the graphene sensitive to the charge transfer and chemical doping of the various ions. The density of the main charge carrier in the graphene will be change due to the graphene layer interact either with electron donating molecules or electron withdrawing molecules [13,35].

Fig. 7 shows the reversibility of the sensor. It can be observed that the maximum difference between the increasing and decreasing runs probe with the graphene layer is about ±0.02 dBm, which is lower as compared to that of without graphene coating. Without the coating it has the maximum difference of about ±0.21 dBm. Both of the maximum difference still acceptable for a full scale output of 7.66 dBm and 5.23 dBm for graphene coated silica microfiber and without graphene layer, respectively. During the experiment, the temperature of the room was kept constant at 25 °C.

Fig. 8 shows the stability of the sensor as recorded for a duration of 300 s for the coated graphene layer in the sensor probe. These result show that the demonstrated sensor has high sensitivity, stability and repeatability for the refractive index sensing of sodium nitrate solution. Table 1 shows the summarized performance characteristic of the sensor. The sensor probe was observed to be sufficiently stable with standard deviation of 0.127 dBm and 0.063 dBm for uncoated and coated graphene layer, respectively. Evidently, the performance of the sensor is enhanced after being coated with graphene as shown in Table 1. It is also worthy to note that the coated sensor can be reused for another measurement due to the graphene layer is not easily fall off from the microfiber.

4. Conclusion

We have successfully demonstrated a simple optical silica microfiber sensor to detect sodium nitrate concentration. A graphene layer was integrated as passive cladding into the silica microfiber to increase the sensitivity of the sensor. As the refractive index of the specimen goes up, the transmission power of the sensor increases linearly and the transmission spectra of the sensor shifted from 1548.96 nm to 1548.16 nm. The sensitivity of the sensor after coating with graphene layer is 1.29 dBm/% with linearity of 99%. Our results show that the demonstrated sensor is useful and applicable for detection of various concentration of sodium nitrate with the ability to provide real time measurement.

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