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# Reflectance response of tapered optical fiber coated with graphene oxide nanostructured thin film for aqueous ethanol sensing

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ABSTRACT

In this work, optical sensing performance of tapered multimode fiber tip coated with graphene oxide (GO) nanostructured thin film towards aqueous ethanol with different concentrations is investigated. The tapering process of the optical fiber is done by a glass processing machine. The multimode optical fiber tip is dip-coated with GO and annealed at 70 °C to enhance the binding of the nanomaterials to the silica fiber. FESEM, Raman microscopy and XRD analyses are performed to micro-characterize the GO thin films. The morphology of the GO is observed to be in sheets forms. The reflectance response of the GO coated fiber tip is compared with the uncoated tip. The measurements are taken using a spectrophotometer in the optical wavelength range of 550–720 nm. The reflectance response of the GO coated fiber tip reduced proportionally, upon exposure to ethanol with concentration range of 5–80%. The dynamic response of the developed sensor showed strong reversibility and repeatability when it is exposed to ethanol with concentrations of 5%, 20% and 40% in distilled water. At room temperature, the sensor shows fast response and recovery as low as 19 and 25 s, respectively.


## 1. Introduction

Graphene oxide (GO) consists of carbon bonded with oxide functional groups [1]. GO are widely used for the improvement of electrochemical sensors [2]. It is an attractive material due to the ability of detecting different types of chemicals such as ethanol and benzene [3]. Excellent transparency, high surface-to-volume ratio and high thermal conductivity as compared to other semiconductors are some of the unique optical and physical properties of GO [4–7].

Electrical transducers for ethanol sensing have been widely explored. Weng et. al [8] and Bairiu et. al [9] developed amperometry ethanol sensors with sensitivity of 3.08 $\mu A\ \mu M^{-1}\ cm^{-2}$ with sputtered Ni/Pt/Ti as the sensing layer and 7.8 $mA\ mM^{-1}\ cm^{-2}$ on silicon nanowires with palladium–nickel electrode, respectively. Although electrical sensors are relatively low in cost and offer high sensitivity, optical fiber sensors are currently attracting considerable interest due to their exceptional characteristics such as immunity to electromagnetic interference, temperature and large bandwidth compared to electrical transducing platforms [10–12]. Optical fiber has also low attenuation of 3.5 dB/km compared to electronic transducers system that have very high loss and need to be compensated with amplifiers.

One of the most suitable optical transducing platforms for sensing applications is tapered optical fiber. Tapered optical fiber is found to be more sensitive as compared to the conventional fiber due to the manner of light propagation in its core. Larger fraction of the optical power propagates outside the tapered fiber core as compared to the normal fiber core and thus, allowing the interaction of the light with the sensing layer [13]. However, the integration of GO with tapered optical fiber as a chemical sensor is yet to be fully explored. It is anticipated that GO with its high surface-to-volume ratio when integrated with tapered fiber will lead to the enhancement in the device sensitivity [14].

One of the chemicals frequently utilized in the industries such as biomedical, chemical and food is ethanol [15–18]. Ethanol

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sensors are also widely deployed for health applications such as breath analyzer. However, most of the ethanol sensors are based on electrical domain. Only a few studies have been reported on ethanol sensing using optical domain [19].

The development of optical sensors coated with GO is still in their maturing stage. One of the advantages of these types of sensors is room temperature operation thus reduced power consumption and complex circuitry. Therefore, there are exciting opportunities in the investigation of the sensing properties of GO nanostructured thin film deposited onto tapered optical fiber tip. This paper presents performance analysis of ethanol sensor based on tapered multi-mode optical fiber (MMF) tip coated with GO via a dip-coating technique. The microcharacterization results of the GO thin film are investigated via field emission scanning electron microscopy (FESEM), Raman spectroscopy, and X-ray diffraction (XRD). The dynamic responses of the developed fiber sensor with and without GO towards different concentrations of ethanol (5–80%) are also reported.

## 2. Experimental

The synthesis of GO is done using the simplified Hummers' method [20] with ultrasonic agitation after chemical oxidation of graphite. The obtained GO solid is suspended in water with the concentration of 0.5 mg/ml. The GO solution is used to coat the nanostructured thin film onto optical substrates via the dip-coating technique. The dip-coating of GO onto tapered fiber tip and glass substrate is done using a PTL-MMB01 dip coater machine. The film is produced using dipping and withdrawal speed of 200 mm/min. Prior to the deposition, the substrates are annealed at 70 °C to enhance the binding of the nanomaterial to the tip [21]. The dip-coating process was repeated three times. After that, the samples are dried in the air for 1 h and then heated up in the oven for 10 min at 70 °C to improve the film adhesion [22]. The films were deposited onto fiber tip and quartz substrates for ethanol sensing investigation and micro-characterization, respectively. Table 1 summarizes the preparation parameters of the GO thin films.

A standard multi-mode silica fiber with core and cladding diameter of 62.5 μm and 125 μm respectively is used in this experiment. The fabrication of the tapered fiber is done with a glass processing workstation (Vytran GPX-3000, USA) shown in Fig. 1. Initially, the polymeric coating of the bare optical fiber for several centimeters is removed using a fiber stripper and then cleaned with alcohol. The optical fiber is then placed on the glass processing workstation with the area to be tapered is just above the filament. The two end of the fiber are fixed to the fiber holding block. This machine operates by pulling the two ends of the fiber while it is heated by the filament. The power of the filament heater was set to 38 W. The fiber holding block controls the pulling distance from both ends of the fiber. The dimensions of the fiber are configured according to the taper parameters as shown in Fig. 2. Tapered fibers with waist diameter of 50 μm, waist

**Table 1**
Preparation parameters of GO thin film.

| Preparation parameters | |
|---|---|
| Annealing temperature (°C) | 70 |
| Dip coater reaction time (m) | 18 |
| GO concentration (mg/ml) | 0.5 |
| Type of substrate | Multimode fiber (MMF) & quartz |
| MMF tip diameter (μm) | 50 |
| Surface area of the tapered fiber tip ($m^2$) | $7.869 \times 10^{-7}$ |

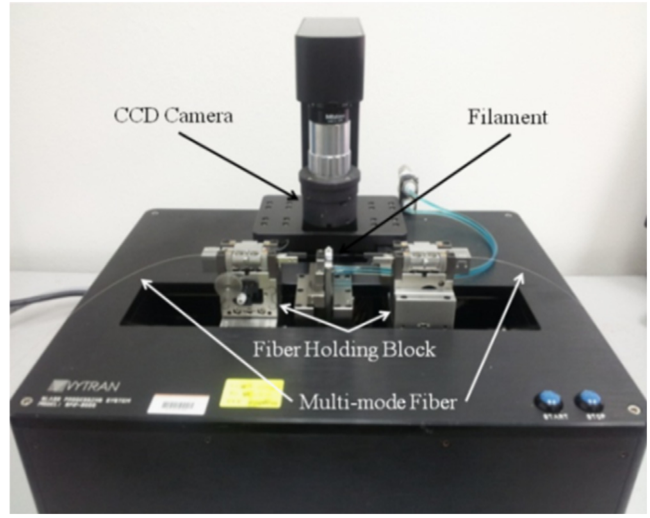


**Fig. 1.** Optical fiber tapering system.

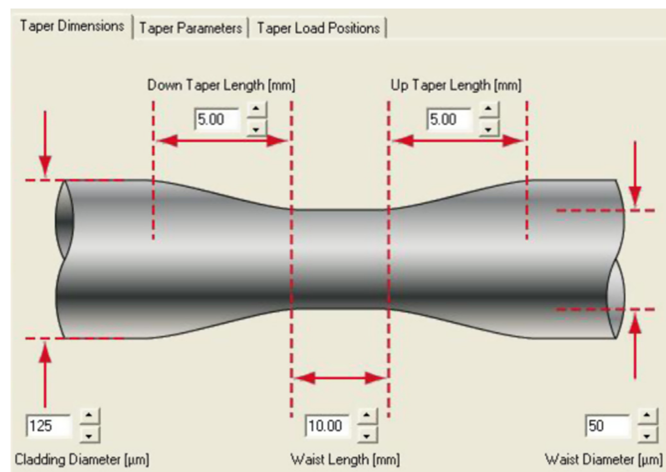


**Fig. 2.** Tapered fiber parameters.

length of 10 mm and down/up taper length of 5 mm are fabricated. The tapered fiber is then cleaved at the middle to achieve a tapered tip of 5 mm length to be coated with the nanomaterials.

The ethanol sensing setup consists of a tungsten halogen light source (HL-2000, Ocean Optics, USA) with wavelength emission ranging from 360 to 2500 nm and a spectrophotometer (USB4000-VIS-NIR, Ocean Optics, USA) with spectral response from 200–1100 nm as shown in Fig. 3. The tapered optical multi-mode fiber tip coated with GO thin film is connected to 1 × 2 coupler (50:50 coupling ratio). The coupler was connected to the light source and the spectrophotometer using optical cables. Reflectance measurement is performed by transmitting the optical signal into the standard optical fiber. The reflected signal from the fiber tip is collected by the spectrophotometer. Subsequently, the response from the developed sensor was processed by the computer via SpectraSuite software.

## 3. Results and discussion

### 3.1. Tapered optical fiber properties

Fig. 4 shows the image of the tapered optical fiber taken by the glass processing system CCD camera. The waist diameter of the fiber decreases linearly from 125 μm to 50 μm. The inset shows