Optical graphene quantum dots gas sensors: experimental study

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Abstract
We present a room temperature Graphene Quantum Dots (GQDs) based optical gas sensor for carbon dioxide gas detection. GQDs were prepared by a hydrothermal method and deposited on a quartz substrate using a drop-casting technique. The size of synthesized GQDs is in the range of 10 to 20 nm. GQDs films were characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), atomic force microscopy (AFM), photoluminescence (PL) and UV-Vis absorption spectroscopy. The gas sensing measurements were studied using optical absorbance changes of GQDs film upon exposure to different concentrations of CO2 gas. The as-prepared gas sensor showed a significant sensitivity with a partially reversible response to CO2 gas, indicating its great potential to pave a way toward a novel CO2 gas sensor.

1. Introduction

In the past years, gas sensors have been widely used in many applications including pollution monitoring [1], fire detection [2], leak detection [3], etc. Besides, carbon dioxide as one of the greenhouse gases emitted by fossil fuel combustion is essential to detect. CO2 has many applications in the food and beverage industry. Also, Global warming and ocean acidification are some of the CO2 gas emission disadvantages to the environment. Therefore the inappropriate amount of it could be harmful to the human body and the environment.

Graphene - the first and the smallest two-dimensional atomic crystal - attracted much attention to scientific research in the field of optoelectronics, used in many applications including photoluminescence bio-imaging [4], X-ray photon detection [5], photo-catalytic activities [6], photoconductive switching [7]. Graphene quantum dot (GQD) is a 0D nanomaterial that contains small pieces of few-layer graphene [8]. Owing to its unique properties, it has attracted much attention to scientific research in the field of optoelectronics, material science, etc. High optical absorption, tunable bandgap [9–13] and the large surface to volume ratio are some of these significant properties [14]. There are reports on using GQDs for solar cells [15], light-emitting diodes [16] and sensors [17–20]. In sensor devices, including electronic sensors [21], electrochemical sensors [22] and photoluminescence sensors [23], GQDs are being used as a sensitive part. It was also used as a dopant to improve the performance of the sensors [24].

The optical gas sensor is one of the most attractive sensing devices for using GQDs. Comparing with electrical gas sensors, optical gas sensors have some great advantages, including its inaction as an ignition source in gas leakage locations, not affected by electromagnetic interference and coverage of a wide area using optical fibers [25, 26]. In recent years good potential efficiency and high power detection have been reported for graphene-based optical gas sensors [27]. In this study, a GQD-based optical gas sensor was fabricated using a simple drop-casting deposition method. In addition, the simple hydrothermal method which has been used to synthesize Cu2O Nanowires [28] and 2D Nano-particles [29] was used to synthesize GQDs.

The changing of optical absorption intensity in the UV-Vis spectra of GQDs is a fingerprint that we employed to detect gas molecules. Due to variety of daily life requirements, our goal in this work is designing an optical gas sensor working at room temperatures. In [24] the comprehensive study on the effect of temperature has been performed.
We reported a good performance for CO$_2$ gas sensing which has a good agreement with our previous theoretical study [30]. Also, in our previous experimental work [31], the sensing study of water-soluble GQDs by analyzing the photoluminescence spectra of GQDs, diluted with carbonated water compare with deionized water was performed and significant results obtained which encouraged us to explore gas sensing properties of GQDs film using optical absorption spectroscopy. Different concentrations of CO$_2$ gas were examined to obtain the limit of detection. It is believed that the optical properties of GQDs can be implemented as an excellent gas sensing technique.

2. Experimental method

2.1. Materials

Graphite powder (C, ≥99.5%) was purchased from Merck. Potassium Sodium Tartrate (KNaC$_4$H$_4$O$_6$.4H$_2$O, ≥99%) was purchased from Sigma Aldrich and deionized water was used throughout the experiment.

2.2. Synthesis of GQDs

GQDs were synthesized using a hydrothermal process of graphite intercalation compounds (GICs) according to the well-known procedure. [31, 32]. Graphite mixed and ground with potassium sodium tartrate (organic salt) at a mass fraction ratio of 10:1 (such as 2 g graphite with 20 g potassium sodium tartrate). Then the mixture was reacting in a Teflon lined (50 ml) autoclave at 240$^\circ$C for 24 h to manufacture Graphene intercalation compounds (GICs). The autoclave was locked inside the glove box with an argon gas atmosphere, before putting it inside the furnace. This will further reduce the oxidation rate during the hydrothermal process. Then GICs were dissolved in water. As a result of this action, GICs were exfoliated in water because of the abrupt reduction between GICs and water [32]. So the mentioned GQDs are produced. Finally, dialysis tubing and filtration process are used to remove remaining salt and categorize GQDs by size respectively. Figure 1 shows a schematic of the synthesis process.

2.3. Sensing experiments

In order to fabricate the sensing device, 0.1 mg ml$^{-1}$ of the GQDs water solution was drop-casted on quartz cell. Then they were dried in a vacuum condition. After drying the GQDs solution, the sample was placed in a quartz chamber and they all together were placed in a UV-Vis spectrofluorometer. A gas-mixing system was used to generate different concentrations of CO$_2$ gas. During gas injection into the quartz chamber, the changing of absorption spectra for GQDs film was recorded. The intensity of the absorption spectrum in a typical wavelength is the fundamental factor of measurement and used as a sensing main parameter. 260 and 310 nm were the wavelengths that we tested for gas sensing experiments. Also, all tests were carried out at room temperature. The specified wavelengths of 260 and 310 nm are related to typical UV-Vis optical absorption peaks of GQDs, caused by the $\pi-\pi^*$ transition of C–C bonds and n-$\pi^*$ transition of C=O bonds respectively [32, 33].

2.4. Characterization

Scanning electron microscope images have been provided by with mira3 Tescan (TESCAN Orsay holding, Czech Republic). Atomic force microscopy (AFM) measurements were carried out on a Nanosurf Mobile S (Nanosurf AG, Switzerland) and the roughness parameters obtained by WSxM software [34]. Transmission electron microscopy (TEM) images were obtained by using a Zeiss LEO 906 (Carl Zeiss Microscopy GmbH, Germany). U-V-Vis absorption spectra were recorded on a Shimadzu UV-2450 UV-Visible spectrophotometer (Shimadzu Corporation, Japan). Photoluminescence spectra were performed with a JASCO FP-6200 spectrofluorometer (JASCO Inc, United States).
3. Results and discussion

The transmission electron microscopy (TEM) image of GQDs is shown in figure 2(a). From the figure, due to transparency and pale condition of GQDs, it concludes that they are few-layered. It is also observed that GQDs are aggregated and most of the GQDs centralized in one area. Probably, due to the synthesizing method that is free of surfactants, concentrated aggregation happened. Moreover, the angled shape of edge sides is proof of the low oxidation rate and the formation of fewer functional groups on synthesized GQDs. The low oxidation rate in hydrothermally synthesized GQDs also reported in our previous work using UV-Vis absorption spectroscopy \[31\] and probably it increases the aggregation possibility.

Figure 2(b) shows the TEM image of non-dialyzed GQDs. From the figure, GQDs are homogeneously dispersed and the aggregation rate is less than dialyzed GQDs (figure 2(a)). Therefore, one can conclude that to prevent GQDs aggregation, the presence of ions is being necessary. Typically to avoid aggregation phenomenon, the surfactants were used which makes the synthesis process difficult and complicate \[35\]. Also, the average size of non-dialyzed GQDs (20 nm >) is less than dialyzed GQDs (20 to 50 nm), maybe due to loss of smaller sizes resulting from the dialysis process.

Furthermore, the PL analysis also confirmed using the non-dialyzed GQDs as a better prototype. Figure 3 presents PL spectra of non-dialyzed GQDs deposited on a quartz substrate compare with water solution GQDs in the same excitation wavelength (310 nm). The luminescence intensity of non-dialyzed GQDs deposition was slightly less than that of the solution due to aggregation effects. However, for dialyzed GQDs deposited on a quartz substrate, no significant luminescence was observed. Therefore, according to the lower aggregation rate and ineffectiveness of ions in optical properties, we used non-dialyzed GQDs for the gas sensing process.

The SEM image of GQDs sensing films is shown in figure 4. It is observed that GQDs are homogeneously dispersed on a quartz substrate using the drop-casting method. Also, the observed porosity on the surface of the sample indicates that the sample is qualified for sensing application. Because gas molecule diffused more easily through the porous structure and its reaction to sensing films occurs easily \[36, 37\]. From the figure, the particle diameter was in the range of 10 to 20 nm in lateral size.

AFM image of GQDs sensing films represented in figure 5 to demonstrate more information about surface topology. The average surface roughness of the film is estimated to be about 20 nm which results in a high-quality gas sensing performance \[38–40\]. Consequently, these results are in good agreement with SEM images.

Figure 6(a) shows the response-recovery curves of GQDs based optical gas sensor at the absorption wavelength of 260 nm during the air and pure CO\(_2\) gas exposure. From the figure, it is clear that relative optical absorbance increases when the sensor is exposed to CO\(_2\) and recovers near the baseline when it is exposed to air. Also, the response-recovery curves of the sensor at the absorption wavelength of 310 nm show a greater increase by CO\(_2\) gas exposure (figure 6(b)). Increasing of optical absorbance in longer wavelengths is also shown in the
UV-Vis absorption spectra of GQDs exposed to air and pure CO₂ gas (inset of figure 6), reported in reference [30].

Carbon dioxide is approximately an inert gas, which is in some cases acts as an oxidizing agent and in some other cases acts as a reducing agent [41–43]. Therefore, both of these cases may occur depending on the type and molecular structure of the sensing material. GQDs that were synthesized under an air-free hydrothermal condition had few oxygen-containing functional groups on the edge sides and the basal plane of GQDs. Therefore the potential reaction with GQDs and introducing of oxygen-containing functional groups on GQDs is high. As a result of this, carbon dioxide increases the formation of oxygen-containing bonding on a basal plane and edge sides of GQDs. Consequently, this increases the absorption of light by increasing the bonds (C=O bonding) [32]. So the optical absorption intensity gradually increases through the CO₂ gas injection.

Figure 6 also shows different cycles of response-recovery curves at the absorption wavelength of 310 nm for 1000, 500, 300 and 100 ppm of CO₂ gas. For all testing cycles, relative optical absorbance rises once CO₂ is

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**Figure 3.** The PL spectra of non-dialyzed GQDs deposited on a quartz substrate and GQDs water solution.

**Figure 4.** The SEM image of non-dialyzed GQDs film.
exposed and returned to the near of its initial state by removing CO2 gas out of the chamber. In 100 ppm of CO2 gas, which was the lowest concentration that has been tested, the GQDs also showed good sensitivity. This dynamic test was also repeated at 260 nm and the same results are observed. Dynamic tests for nitrogen gas were also investigated, and the GQDs gas sensor didn’t show much sensitivity to it [30]. During the experiments, to eliminate the impact of oxygen gas in the air, we used Argon as a recovery gas but it didn’t show much impact on sensor recovery. So the tests has been done with air. The effect of CO2 gas and the type of reaction (oxidation or reduction) with GQD is still unknown. Accordingly the effect of air containing a lot of oxygen- which recovers the effect of CO2 gas is also unclear.

Based on our analysis, we found disordered reversibility behavior for different tests. But in general, for most of the samples partly week reversibility was observed which indicates the formation of some chemical bonds between the gas molecule and GQDs that sensor wasn’t fully recovered by using of air gas.

The gas sensor response S is defined using the following equations:

\[ S(\%) = \frac{I_{\text{gas}} - I_{\text{air}}}{I_{\text{air}}} \times 100 \]  

where \( I_{\text{air}} \) and \( I_{\text{gas}} \) are the optical absorption intensity of GQDs in the air and CO2 gas exposure respectively.

At shorter wavelengths, the level of change in optical absorption intensity during air and gas exposure was lower than longer wavelengths (inset of figure 6). Consequently sensing response in shorter wavelengths was lower than longer wavelengths. The calculated response in the wavelength of 310 nm for 1000 ppm of CO2 gas was about 50% and for the concentration of 500, 300 and 100 ppm it was about 40%, 8%, and 4% respectively. For a wavelength of 260 nm, the response was far less than the obtained amount for 310 nm.

![AFM 3D image of non-dialyzed GQDs film](image)

**Figure 5.** The AFM 3D image of non-dialyzed GQDs film (the inset shows the AFM measurement parameters obtained by WSxM software).

![Response-recovery curve of GQD gas sensor](image)

**Figure 6.** The response-recovery curve of GQD gas sensor at (a) 260 nm and (b) 310 nm (the inset shows the absorption spectra of GQD exposed to air and CO2).
Several samples were prepared and were tested many times. Repeatable results in terms of relative optical absorption changes were obtained in these experiments. Based on the results, average recovery time and average response time were calculated. The response and recovery time is defined as the time to reach 90% of relative optical absorption in the final state, respectively after the target gas exposed and removed. The average response time and recovery time were 106 and 150 s respectively, which is less than previously reported results for Graphene-based optical gas sensors [27]. Moreover, the amounts of changes were better than previously reported results by Wlodaeski and et al [27]. The sensor made by Wlodaeski’s group showed a maximum response of 0.4% for 10000 ppm of Hydrogen gas. This is while the Graphene quantum dot gas sensor showed a response about 50% for 1000 ppm of CO2 gas at the absorption wavelength of 310 nm. The minimum response time of the sensor, reported by Wlodarski et al was about 2 min for 100 ppm of H2 gas exposure, for GQDs gas sensor this value is about 100 s at 100 ppm of CO2 gas exposure.

However, the lowest concentration of gas that has tested for the GQD gas sensor is less than that reported by Wlodarski’s group. Also, the number of gases in this report compared with the work reported by Wlodarski et al was fewer, which requires a further investigation in the future.

Also, since CO2 is approximately inert gas [41], this was a great challenge to check the sensing properties of GQDs. Because, if the gas sensor can easily detect the approximately inert CO2 gas, it is likely easier to detect strong oxidizing gases such as NO2 or strong reducing gases such as H2 and CO. Hence, the lack of using various gases in this paper does not mean that the sensor was unable to detect other gases or doesn’t have selectivity.

Based on the previous works reported by other researchers [19, 44, 45], GQD gas sensors are sensitive to oxygen gas. Especially at higher humidity, the oxidation rate of these sensors increases. Therefore, they can even be used as humidity sensors [45]. GQDs has also been studied for sensing of NH3, CO [19], etc these gases can also be tested for GQD optical gas sensor in future studies.

The relationship between CO2 gas concentration and relative optical absorption can be expressed by an equation as: [46].

\[
A = A_0 \exp \left( (\alpha N)^\gamma \right)
\]

where \(A_0\) is initial optical absorption, \(A\) is optical absorption at the concentration of \(N\), \(\alpha\) and \(\gamma\) are constants. Thus, it can say that \(\log(\log(A/A_0))\) is equal to \(\log(N)\). Accordingly, a particular type of calibration curve is obtained using equation (2), which is shown in figure 7. From the figure, the changes in the concentration of 500 and 1000 ppm reach its saturation state and they show approximately the same amount. The curve has an exponential shape and describes the effect of CO2 gas concentrations on spectral change rate in the gas sensor.

4. Conclusion

In this article, we report a new optical gas sensor based on GQDs, prepared by a hydrothermal method using graphite intercalation compounds. Both synthesizing and sensor fabrication methods are very cheap and easy to prepare due to the lack of multiple chemicals and using the simple drop-casting method. SEM and TEM results indicated that the size of GQDs is in the range of 10 to 20 nm and the film aggregation rate is low. The AFM analysis demonstrated GQDs film is reasonable for sensing applications because of high surface roughness rate.
GQDs sensing films showed a significant optical absorption change upon exposure to CO2 gas at room temperature and were in good agreement with our previous theoretical investigation. The high and partly reversible response observed, demonstrating its potential application as an optical gas sensor for detecting CO2 gas.

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