



## Featured Letter

## Graphene oxide resorcinol hybrid material as fluorescent chemosensor for detection of cerium ion

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## ARTICLE INFO

## Article history:

Received 3 April 2018

Received in revised form 9 May 2018

Accepted 11 May 2018

## Keywords:

Carbon material

Resorcinol

Nanocomposite

Luminescence

Sensor

Cerium

## ABSTRACT

A new hybrid material, graphene oxide- Resorcinol (GO-R) is synthesized from graphite following the modified Hummers method, characterized and utilized as a fluorescent chemosensor to detect Cerium ion under aqueous conditions. The effect of pH, reversibility, time and the interference of other potentially competing ions have been studied. The sensing follows both the photo induced electron transfer and intramolecular charge transfer processes.

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## 1. Introduction

Graphene has been used to foray with many applications in chemical sensors [1]. The presence of various oxygenated functional groups (C–O, C=O and –COOH) and some oxidized sp<sup>3</sup> sites in graphene oxide (GO) contributes to the band gap of graphene and results in a unique photoluminescence emission pattern [2]. The control of the sp<sup>2</sup> domain in the sp<sup>3</sup> matrix of GO can tune the fluorescence intensity as well the emission wavelength [3]. Cerium's intrinsic toxicity, elicited research to selectively detect Ce<sup>3+</sup> ion in an aqueous solution using synthesized nano-sized fluorescent chemosensor of graphene quantum dots (GQDs) by Salehnia et al. [4,5]. Herein, we report a new reversible graphene oxide resorcinol hybrid material capable of detecting Ce<sup>3+</sup> ion through an intramolecular charge transfer (ICT) processes with a red shift in its emission studies.

## 2. Experimental

## 2.1. Materials and instruments

All solvents were purchased commercially from Merck & Co. Graphite and Resorcinol was purchased from Sigma-Aldrich. Fluor-

rescence measurements were taken with a Jasco FP-8200 spectrofluorometer equipped with quartz cuvettes of 1 cm path length.

## 2.2. Synthesis of graphene oxide-resorcinol nanocomposite (GO-R)

5 g of Graphite and other required precursors was used to prepare the GO by modified Hummers method [6]. 500 mg of prepared GO was sonicated with 100 ml DI water for 30 min to obtain a GO solution [7]. Similarly, resorcinol solution was prepared. The two solutions were mixed and transferred to a 250 ml RB flask in reflux condition for one day. The resulting solution was then dried in a hot air oven at 60 °C for one day to obtain the Graphene Oxide-Resorcinol Nanocomposite (GO-R).

## 2.3. Characterization

Scanning Electron Microscope (SEM) images was carried out on JEOL model JSM-6390 to characterize its morphology. X-Ray Diffraction (XRD) was carried out on SHIMADZU model XRD-6000 to analyse diffraction peaks. FT-IR studies was analysed with Shimadzu Prestige-20 IR Spectrometer. Absorption spectra were recorded on a Shimadzu UV-240 spectrophotometer.

## 3. Results and discussion

SEM micrographs in Fig. 1(a) shows the surface morphology of resorcinol as flakes and in Fig. 1(b) GO as sheets. However, in the

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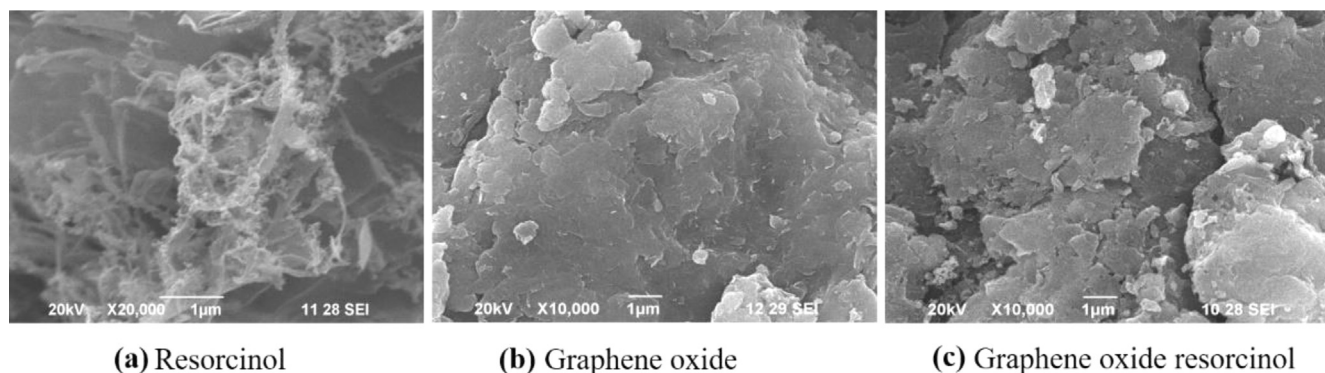


Fig. 1. SEM images of (a) Resorcinol, (b) GO and (c) GO-R.

case of GO-R, in Fig. 1(c) it is clearly observed that the Resorcinol is adsorbed on the surface of the Graphene Oxide sheets. Therefore, the SEM images show that the graphene-oxide is decorated with resorcinol molecules.

The XRD pattern shown in Fig. 2(c) of GO exhibited a strong peak at  $2\theta = 10.46^\circ$ , which corresponds to the presence of oxygen functionalities. The diffraction peak at  $2\theta = 43.27^\circ$ , indicates the short-range order in stacked graphene layers. The same diffraction peaks are observed in the case of the GO-R along with additional broad peaks of resorcinol around  $2\theta = 19.24^\circ$ . This shows that the resorcinol fluorophore is adsorbed on the surface of graphene oxide sheets in a dis-ordered manner.

The FT-IR spectrum in Fig. 2(b) shows Resorcinol having clear peaks for  $-\text{OH}$  at  $3215\text{ cm}^{-1}$  and  $\text{C}=\text{C}$  absorptions peaks at  $1617\text{ cm}^{-1}$ . The existence of a variety of oxygen functionalities in GO was confirmed at  $3379\text{ cm}^{-1}$  ( $\text{OH}$  stretching vibrations), small emerging peaks at  $1617\text{ cm}^{-1}$  (stretching vibrations from  $\text{C}=\text{O}$ ), and at  $1059\text{ cm}^{-1}$  ( $\text{C}-\text{O}$  stretching vibrations) [8]. The shifts in GO-R to  $1597\text{ cm}^{-1}$  are due to the possibility of the noncovalent interactions between the  $-\text{OH}$  groups of the resorcinol and the  $-\text{C}=\text{O}$  and  $-\text{OH}$  groups of the GO like hydrogen bonding. It further advocates the noncovalent functionalization process of the resorcinol molecules on GO. The interaction between the graphene oxide and resorcinol was investigated by the UV-Vis spectroscopy pattern as shown in Fig. 2(a). The absorption peak of the free resorcinol to bound GO-R has slightly broadened and a very small peak at  $272\text{ nm}$  has increased from 0.23 for resorcinol to 0.26 for GO. This is indicative of the  $\pi-\pi$  interactions between Resorcinol and Graphene oxide [9].

### 3.1. Selectivity of metal ions

As shown in Fig. 3(a) there was a significant enhancement of fluorescence for the receptor GO-R with an emission maximum at  $306\text{ nm}$  on addition of  $100\text{ equiv.}$  of  $\text{Ce}^{3+}$  to a solution of GO-R. Interestingly, there was a red shift of the fluorescence intensity to  $351\text{ nm}$  from  $306\text{ nm}$  with the enhancement. It produced minor changes for other metal ions in the fluorescence spectra [10]. Therefore, GO-R was highly selective towards  $\text{Ce}^{3+}$  ion when compared to other metal cations and can detect  $\text{Ce}^{3+}$  ions as a fluorescence “off-on” probe in  $\text{THF}-\text{H}_2\text{O}$  (1:1 v/v, HEPES  $50\text{ mM}$ ,  $\text{pH} = 7.4$ ) solution.

### 3.2. Effect of pH and time

The fluorescence intensity changes of GO-R and  $\text{GO-R}+\text{Ce}^{3+}$  in  $\text{THF}-\text{H}_2\text{O}$  solution (1:1 v/v) at different pH values ( $\lambda_{\text{ex}} = 273\text{ nm}$ ) shown in Fig. 3(e) were studied in order to explore its photo physical properties. The high fluorescence intensity at pH 5 and 10 for the free receptor may be credible to the protonation and deprotonation of the receptor at highly acidic and basic pH values, respectively [11]. The fluorescence intensity of  $\text{GO-R}+\text{Ce}^{3+}$  complex is relatively stable at a pH range from 7 to 9 with enhanced intensity. Hence, the physiological pH of 7.4 was chosen for all the experiments. In Fig. 3(f), changes in fluorescence intensities of GO-R to  $\text{Ce}^{3+}$  ( $\text{THF}-\text{H}_2\text{O}$ , 1:1 v/v, HEPES  $50\text{ mM}$ ,  $\text{pH} = 7.4$ ) against time showed a fluorescence intensity increase with a red shift reaching the maximum level of saturation in 2 min and remained steady for 50 min. This indicates the chemosensor GO-R selectively detect  $\text{Ce}^{3+}$  ion in a short span of time [12].

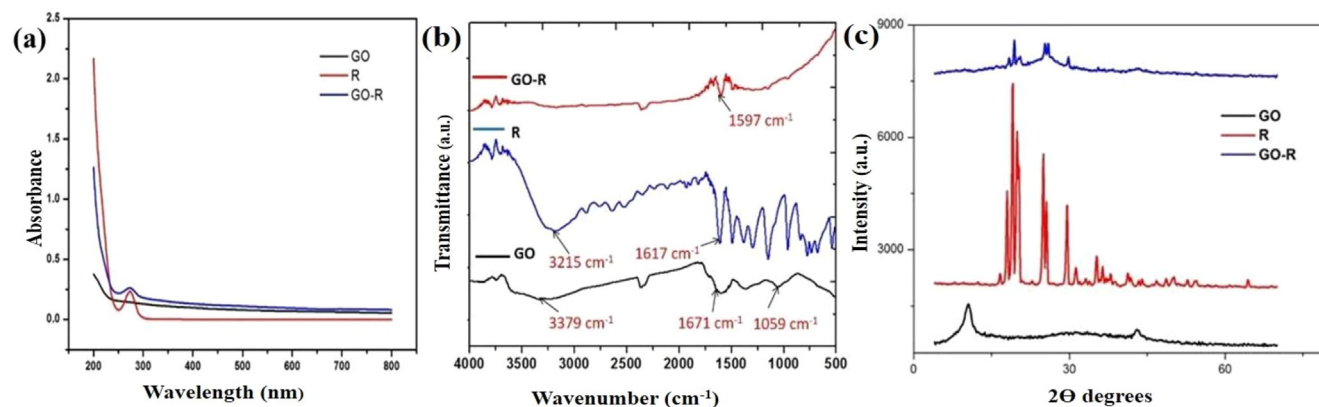


Fig. 2. (a) UV Spectra (b) IR Spectra and (c) XRD Spectra of GO, Resorcinol and GO-R nanocomposite.

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