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Rapid and Simple Synthesis of Graphene Quantum dots/Ag Nanocomposites and Its Application for Glucose Detection by Photoluminescence Spectroscopy

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***Abstract*— *A rapid, environmentally friendly and simple approach is conducted for the synthesis of graphene quantum dots (GQDs) using naturally available biomass materials such as lemon juice and ascorbic acid under supporting of microwave irridiation, at power 900 W and fast reaction time ~9 min. Besides, Ag nanoparticles were successfully combined with GQDs by a simple and easy to implement method, all solvents used here being deionized water with reaction temperature at 60^{o}C for 30 min to form GQDs/Ag nanocomposites. The synthesized GQDs/Ag nanocomposites were determined their properties, morphologies and compositions by: UV-vis, FTIR, XRD, TEM, and EDX. Results showed that the GQDs/Ag nanocomposites were spherical in shape, uniformly distribution, with an average particle size of GQDs and Ag nanoparticles (Ag NPs) respective being ~5-10 nm and ~10-25 nm. The percent (%) component of elements present in the synthesized GQDs/Ag nanocomposites sample were found to be composed mainly including: C (45.36%); O (39.04%); N (3.96%); and Ag (11.05%), respectively. Moreover, the synthesized GQDs/Ag nanocomposites have also applied in glucose detection by photoluminescence (PL) spectroscopy with an extremely low limit of detection (LOD) for glucose concentration being ~10 nM (~10^{-8} M). Based on experimental results, it indicated that GQDs/Ag sample is a potential and promising nanomaterial, which can be oriented and developed as nanomaterials to apply in many fields such as biomedical sensors, chemical sensors, etc*.

***Keywords*— *Graphene quantum dots/Ag nanocomposites (GQDs/Ag NCPs); photocatalytic activity; glucose; lemon juice; photoluminescence (PL) spectroscopy*.

I. INTRODUCTION

Photoluminescent carbonaceous dots have attracted increasing attention in the last decade due to the advantages of high photostability, high resistance against photobleaching compared to organic dyes and excellent biocompatibility compared to semiconductor quantum dots. Carbonaceous dots can be classified as nanodiamonds, carbon nanodots, or grapheme quantum dots (GQDs) according to their sp^2 and sp^3 hybridization composition [1]. Graphene quantum dots (GQDs), a promising carbon based luminescent material, have been attracted interesting of researchers due to their tune-able photoluminescence (PL) properties, low toxicity, hydrophilic nature, good biocompatibility and excellent photo-stability [2-6]. Thus, scientists have attented and focused on the synthesis of GQDs, inspired by their outstanding properties and potential applications. These unique properties enable their potential applications in biological, optoelectronic, energy or sensor related fields [7-13].

Commonly, GQDs are synthesized by cutting carbonic precursors, such as graphene, graphene oxide and carbon fibers, into smaller pieces by chemical oxidation, hydrothermal treatment or solvothermal treatment under harsh conditions which require the use of sulfuric acid, nitric acid or other strong oxidizers [2, 14, 15]. However, none of these methods are regarded as green and many drawbacks are unavoidable such as potential safety risks and environmental pollution (e.g, concentrated acids used so high such as sulfuric acid and/or nitric acid are necessary, toxic gases such as NO_2 and N_2O_4 may be generated during the synthesis process); high cost and complex fabrication process for the raw materials (i.e, graphene, graphene oxide, graphene nanosheets, graphite oxide). Therefore, developing eco-friendly alternative methods and finding new green precursors are desired.

Besides, many researchers have reported that graphene/semiconductor composite can improve the photocatalytic performance in terms of high pollutant absorption or significantly enhanced emission intensity on the photoluminescence (PL) spectroscopy signal, reduced the rate of charge carrier recombination, increasing the electron transport and also enhanced the light absorption band range [16, 17]. Thus, to further enhance its properties, noble metal nanoparticle also have been developed and combined to significantly improve the photocatalytic performance of nanocomposites materials (GQDs/Ag nanocomposites) as well as increase their light absorption ability in the visible region. This noble metal/composite can restrict the recombination of electron-hole pairs by efficient transport of photogenerated electron onto the noble metal [18].

Herein, we develop a green and efficient approach for the synthesis of GQDs using lemon juice as a precursor and ascorbic acid as a reducing agent under supporting of microwave irradiation at power 900 W for 9 min. In addition, these synthesized GQDs have been successfully combined

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with silver nanoparticles (Ag NPs) by a rapid, simple and environmentally friendly method at 60^{o}C for 30 min. Moreover, the synthesized GQDs/Ag nanocomposites have also been applied in detection of glucose by photoluminescence (PL) spectroscopy.

II. Materials and Methods

a. Materials

Silver nitrate ($AgNO_3$, 99%) and glucose ($C_6H_{12}O_6$, 99.5%) were purchased from Merck (Sigma-Aldrich). Sodium hydrogen phosphate (Na_2HPO_4, 99%), Potassium dihydrogen phosphate (KH_2PO_4, 99%), Ascorbic acid ($C_6H_8O_6$, 99.7%), Urea (CH_4N_2O, 99%), Postassium chloride (KCl 99%), and Sodium chloride (NaCl 99%) were bought from Hemidia, India. Hydrochloric acid (HCl 99%) was purchased from XiLong, China. Lemon was purchased at supermarket of Can Tho Coopmart, Viet Nam. All solutions were prepared with deionized water from a MilliQ system.

b. Syntheis of graphene quantum dots (GQDs)

Stirring the mixture of 4 mL of lemon juice and 5 mL of deioned water (DI H_2O) to obtain a homogeneous solution. Then, 0.18 g of urea was completely dissolved into the prepared solution by sonication for 10 min. Precursor solution were heated at a power level of 900 W under supporting of microwave irradiation. After that, dark brown solid was allowed to cool down at room temperature. Pre-product was centrifuged for 15 min and washed several times with DI H_2O to remove the insoluble solids. Finally, synthesized GQDs were re-diffused in 5 mL of DI H_2O and stored at 8°C for next steps.

c. Synthesis of GQDs/Ag nanocomposites (GQDs/Ag NCPs)

In typically, 0.5 mL of $AgNO_3$ salt solution (2 M) was added into the prepared-GQDs solution (5 mL) and heated to 60^{o}C for 5 min. After that, 0.25 mL of ascorbic acid (2 M) was added to the mixture and stirred at 60°C for 30 min. Finally, the product was collected by centrifugated, and washed with DI H_2O for several times and diffused in 5 mL of DI H_2O for further characterization.

d. Investigating photocatalytic activity of GQDs/Ag nanocomposites for glucose detection by photoluminescence (PL) spectroscopy

Preparation of PBS solution

Dissolving of (4 g) NaCl, (0.1 g) KCl; (0.72 g) Na_2HPO_4 and (0.12 g) KH_2PO_4 in 400 mL of deioned water (DI H_2O). Then, the solution was kept in 500 mL Volumetric Flask and adjusted the pH to about ~7.4. Finally, DI H_2O was carefully added to the mark to make 500 mL of PBS buffer solution.

Preparation of glucose solution

0.018 g of glucose and the prepared buffer solution were carefully added to a 100 mL volumetric flask so that the volume of the solution reached the mark. Stirring the mixture to obtain a homogenous glucose (10^{-3} M) solution. Glucose solution (10^{-3} M) is diluted in different proportions to get glucose solution with different concentrations: 10^{-5} M, 10^{-7} M, 10^{-8} M, 10^{-9} M and 10^{-10} M, respectively.

Preparation of GQDs/Ag nanocomposites in the presence of glucose with various concentrations

200 L of glucose solution with different concentrations (10^{-3} M, 10^{-5} M, 10^{-7} M, 10^{-8} M, 10^{-9} M, 10^{-10} M) was added into 1 mL of GQDs/Ag nanocomposites and 2 mL of DI H_2O. After that, the mixture was stirred vigorous in 10 min at room temperature. The prepared samples were analyzed and obtained their Fluorescence emission intensity on the photoluminescence (PL) spectroscopy with various intensities respectively.

e. Characterization of synthesized GQDs/Ag nanocomposites

The GQDs/Ag NPs synthesis was observed by recording the absorbance spectra between 200 and 900 nm on the UV-vis spectrophotometer (Thermo Scientific Evolution 60S UV-Vis spectrophotometer, USA). X-ray diffraction (XRD) was performed on a D8-Advance machine (Bruker, Germany) in the 2θ range of 10°-80°. The Fourier transform infrared (FT-IR) spectra were obtained by Perkin Elmer Frontier MIR/NIR (Perkin Elmer, USA) was conducted in KBr pellet at room temperature in the range of 4000-400 cm^{-1}. Transmission electron microscopy (TEM) characterization was performed on a Jem1010 device (Joel Company, Japan). Chemical properties and constituent components were analyzed via Energy-dispersive X-ray spectroscopy (EDX H-7593, Horiba, England). Photoluminescence spectra (PL) was recorded at 360 nm using FluoroMax-4 spectrofluorometer (Horiba Jobin Yvon, France).

III. Results and Discussion

3.1 Characterization and morphology of GQDs/Ag nanocomposites

Figure 1 shows the UV-vis spectra result of GQDs/Ag nanocomposites with various volumes of $AgNO_3$ salt solution. It is shown that the absorption intensity of different GQDs/Ag nanocomposites samples is similar. GQDs/Ag nanocomposites with 500 L of $AgNO_3$ salt solution presented the highest absorption intensity compared to other samples –shown in Figure 1(c). In addition, this sample shows a smooth, clear and sharp absorption peak indicating optical absorption and electronic conversion, easier energy conversion. It was predicted that GQDs and silver nanoparticles (Ag NPs) are uniformly dispersed in GQDs/Ag solution. The absorption peak intensity at 360 nm showed that in the solution of GQDs/Ag nanocomposites, silver nanoparticles (AgNPs) are formed with high concentration [19-21]. While the GQDs/Ag solution was not added any reducing agent and no Ag_2O precipitate was observed when $AgNO_3$ was added. Simultaneously, the maximum absorption peak at 335 nm of GQDs was became an absorption edge with low absorption intensity in GQDs/Ag nanocomposites sample. This proved that the concentration of free GQDs in the solution of GQDs/Ag nanocomposites was significantly reduced. This proves that the functional groups -NH-, -COOH, -CHO and -NH_2 of GQDs nanoparticles have adsorbed and reduced Ag+ ions into silver nanoparticles (Ag NPs) which attached around the GQDs nanoparticles' surface. Thus, GQDs act as both a

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reducing agent and a stabilizer for silver nanoparticles (Ag NPs) in this work.

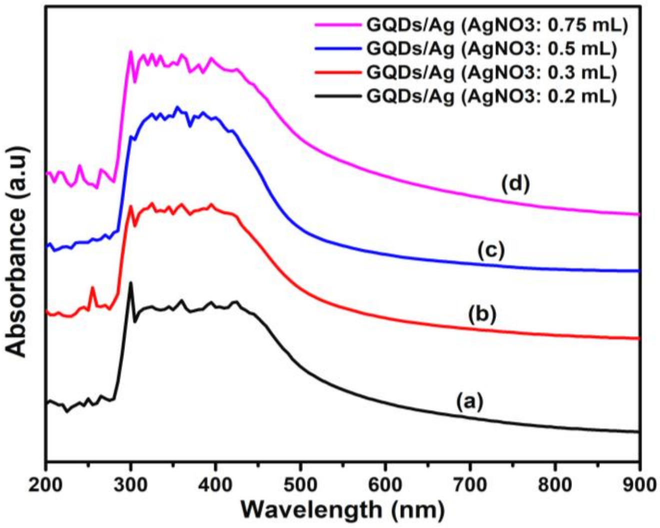

Figure 1. UV-vis spectra of GQDs/Ag nanocomposites with various volumes of $AgNO_3$ salt solution: (a) 200 μL; (b) 300 μL; (c) 500 μL; and (d) 750 μL, respectively.

Figure 2 shows the FTIR spectra of the samples of interest. The absorption band of the GQDs sample – see Figure 2(a) at 3450-3380 cm^{-1} was attributed to the hydroxyl (-OH) groups (stretching mode) [20]. Besides, the GQDs showed absorption of stretching vibration –CH group at 2347 cm^{-1} and stretching vibration of -C=O group in the range at 1717-1665 cm^{-1}. The vibration band at 1450-1414 cm^{-1} are corresponding to -C=C bonds. In addition, in the oscillation region from 1200-1190 cm^{-1} are the characteristic stretching vibration of the -C-OH groups. The transformation of chemical groups in GQDs when adding Ag NPs was shown in Figure 2(b), the broad absorption band ~625 cm^{-1} was enhanced in the presence of Ag-O. Based-on the result, it confirmed that the synthesized nanomaterial were GQDs and GQDs/Ag nanocomposites because their FTIR spectra results were completely consistent with previous studies [22, 23].

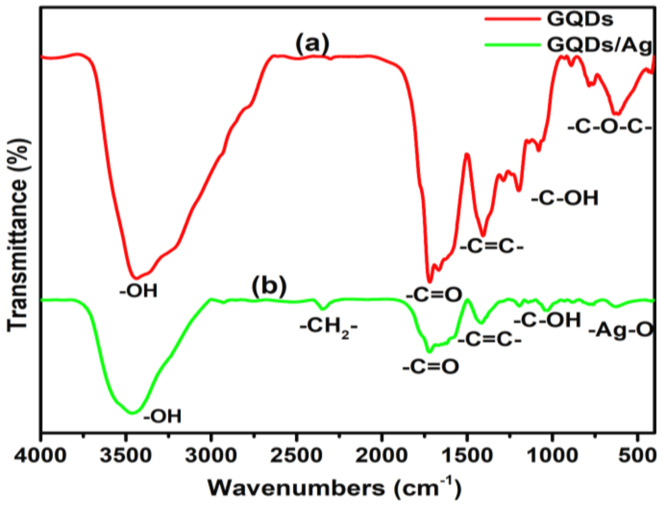

Figure 2. FTIR spectra of (a) GQDs and (b) GQDs/Ag nanocomposites, respectively.

As shown in Figure 3, the X-ray diffraction (XRD) results of samples obtained correspond to the GQDs and GQDs/Ag nanomaterials, respectively. Typical XRD profiles of the prepared GQDs are shown in Figure 3(a). As can be seen, there is an amorphous diffraction peak at an angle of 2θ = 26^o corresponding to the (002) plane of the sp^2 hybridization of graphene is characteristic diffraction peak of the GQDs [22]. Based on the XRD diffraction pattern, the synthesized GQDs are mainly form in the amorphous structure – see in Figure 3(a). Meanwhile, the result of XRD diffraction analysis of the GQDs/Ag nanocomposites (Figure 3(b)) showed that the characteristic diffraction peaks of Ag NPs were identified at four clear peaks with 2θ values of 38.14^o; 44.37^o; 64.45^o; and 77.57^o corresponding to the crystal planes (111); (200); (220); and (311) of standard Ag (JPCDS Card No.04-0783). The obtained results show that the synthesized GQDs/Ag nanocomposites samples still have the presence of amorphous structure from GQDs while the characteristic diffraction peaks of the nano-silver crystal structure appeared [23]. Therefore, it confirmed that GQDs/Ag nanocomposites have the presence of silver nanoparticles (Ag NPs) to form nanocomposites material as expected.

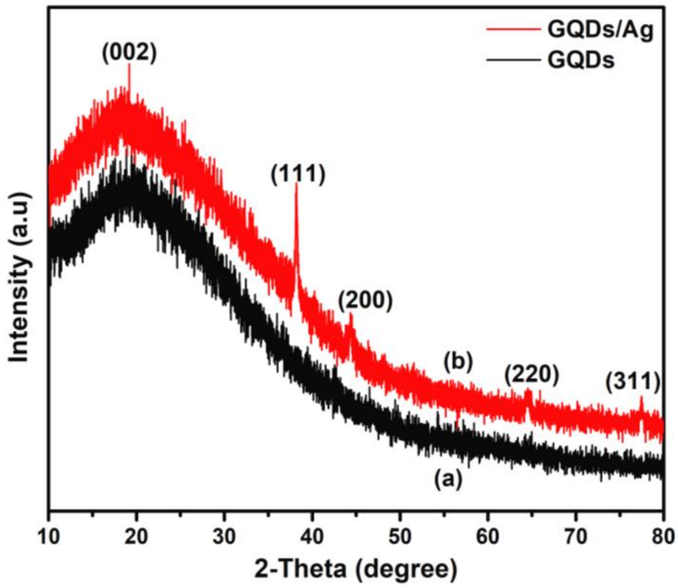

Figure 3. XRD patterns of (a) GQDs and (b) GQDs/Ag nanocomposites, respectively.

The morphology of the GQDs/Ag nanocomposites was studied by transmission electron microscopy (TEM). As shown in Figure 4, GQDs/Ag nanocomposites are uniformly distributed. The synthesized GQDs/Ag nanocomposites have the spherical structure with an average particle size ~5-25 nm. Herein, the white nanoparticles are probably GQDs with a small particle size ~5-10 nm, which is consistent with the particle size of GQDs according to the theory of graphene quantum dots (GQDs). The black nanoparticles are probably silver nanoparticles (Ag NPs) with an average particle size ~10-25 nm. The density/content of silver nanoparticles (Ag NPs) is less than the component of GQDs nanoparticles present in the synthesized GQDs/Ag nanocomposites samples.

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This result was completely consistent with previous studies [23].

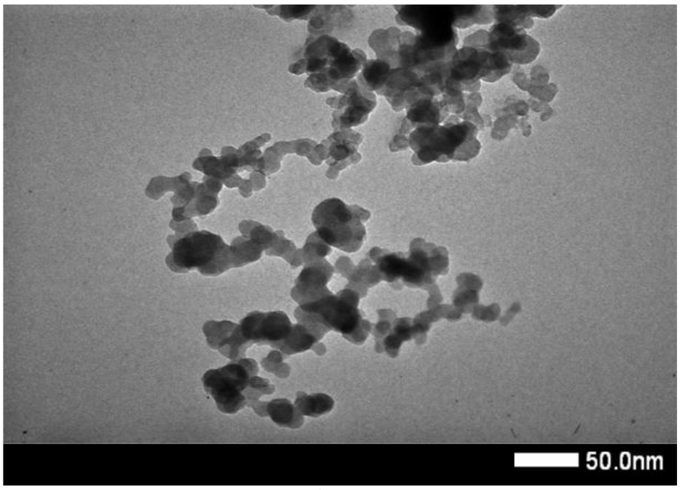

Figure 4. TEM image of GQDs/Ag nanocomposites.

EDX analysis was performed to check the percent (%) component of all elements of the synthesized GQDs/Ag nanocomposites specimen. Figure 5 shows that the GQDs/Ag nanocomposites correspond to the weight composition of elements including: C (45,36%); O (39.04%); Ag (11.05%); N (3.96%) and no other peak for any other element has been found. The obtained results show the presence of C, O, N from the GQDs and the typical content of silver nanoparticles (Ag NPs) in the GQDs/Ag nanocomposites sample. Therefore, this once again confirmed that GQDs/Ag nanomaterials have been successfully synthesized.

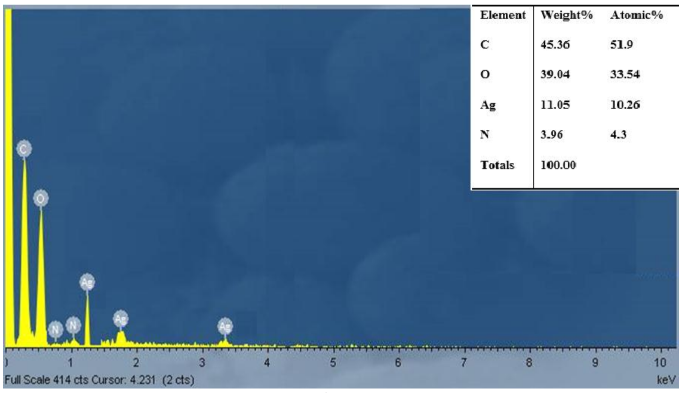

Figure 5. EDX spectra of GQDs/Ag nanocomposites.

3.2 Testing photocatalytic activity of GQDs/Ag nanocomposites for glucose detection by photoluminescence (PL) spectroscopy

Depending on the different concentrations of glucose, the GQDs/Ag nanocomposites samples have different interactions leading to significant intensity changes on the emission signal of PL spectrum- see in Figure 6. The greater the presence of glucose (the higher concentration), the higher the emission intensity of GQDs/Ag/glucose with luminescent signal. Besides, the glucose concentration was gradually decreased, leading to decreasing its signal intensity on the PL spectrum. And the intensity signal on the PL spectrum or "fluorescence quenching", begins to occur when the glucose concentration is too low. When the extremely low glucose concentration at 10^{-8} M, the emission intensity signal of GQDs/Ag nanocomposites in the presence of glucose still is detected on the PL spectrum. And this value is determined as the limit of detection (LOD) for detecting of glucose concentration.

GQDs/Ag nanocomposites with various concentrations of glucose were investigated by photomispectroscopy which recorded with an excitation wavelength at 360 nm. In the presence of glucose, the luminescence intensity of GQDs/Ag nanocomposites had the emission (luminescence) wavelength at 530 nm. The obtained results showed that the amount of glucose with a concentration of 10^{-3} M showed excellent interaction with GQDs/Ag and the luminescence occured strongly with high intensity. While the amount of glucose in the solution was 10^{-9} M, the luminescence intensity of this sample did not change compared to GQDs/Ag nanocomposites sample without glucose. In addition, with glucose concentration at 10^{-10} M, the fluorescence quenching is occurred. Therefore, the minimum emission intensity of GQDs/Ag nanocomposites for glucose detection obtained on the PL spectrum is respective with glucose concentration at 10^{-8} M. As a result, it can be seen that the limit of glucose detection (LOD) of GQDs/Ag nanocomposites material on the PL spectroscopy achieved at extremely low glucose concentration (10^{-8} M).

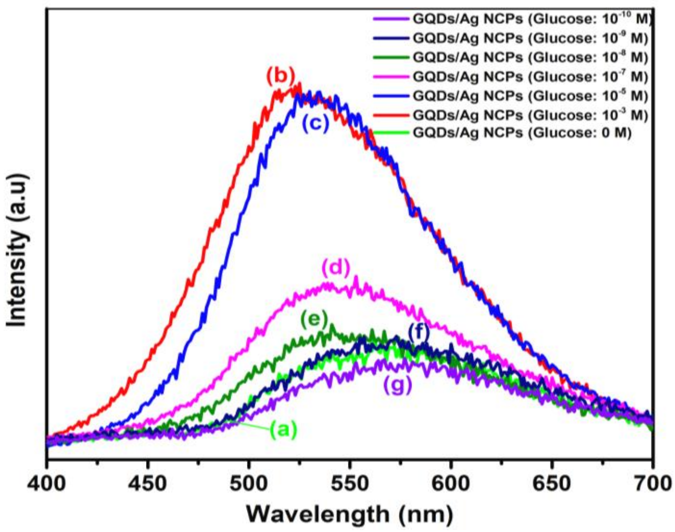

Figure 6. Photoluminescence (PL) spectroscopy of GQDs/Ag nanocomposites with various glucose concentrations at the excitation wavelength of 360 nm: (a) 0 M; (b) 10^{-3} M; (c) 10^{-5} M; (d) 10^{-7} M; (e) 10^{-8} M; (f) 10^{-9} M; and (g) 10^{-10} M, respectively.

IV. CONCLUSIONS

Graphene Quantum Dots (GQDs) have been prepared by green chemistry method (easy to implement, fast response time, environmentally friendly) using lemon juice as a precursor and under microwave irradiation supporting. In addition, GQDs/Ag nanocomposites have been successfully developed and synthesized via a simple chemistry method using ascorbic acid as a reducing agent at 60°C for 30 min. Results obtained the GQDs/Ag nanocomposites were spherical shape with an average particle size of GQDs and Ag

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nanoparticles (Ag NPs) respective being ~5-10 nm and ~10-25 nm. The percent (%) composition of elements present in GQDs/Ag nanocomposites corresponds to: C (45.36%); O (39.04%), N (3.96%); and Ag (11.05%). Moreover, GQDs/Ag nanocomposites were applied to detect for the presence of glucose with the extremly low detection concentration at LOD value being 10^{-8} M with the excitation wavelength of 360 nm (UV) and corresponding emission wavelength at 530 nm (visible light). Therefore, the synthesized GQDs/Ag nanocomposites can be oriented for development as a nanomaterial used in biomedical sensors, chemical sensors, etc., to detect glucose concentration in blood or detect fluorescent molecules at extremely low concentration for the early diagnosis and treatment of serious diseases.

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