Graphene quantum dots as efficient, metal-free, visible-light-active photocatalysts

Jiechao Ge[†], Minhuan Lan[†], Weimin Liu^{*}, Qingyan Jia, Liang Guo, Bingjiang Zhou, Xiangmin Meng, Guangle Niu and Pengfei Wang

ABSTRACT This paper reports on new applications of water-dispersible graphene quantum dots (GQDs) that we recently developed. The prepared GQDs not only show broad absorption in the visible spectrum from 400 to 700 nm, but can also serve as smart photosensitizers with high singlet oxygen (¹O₂) production under visible-light irradiation (≥420 nm). We showed that the prepared GQDs can potentially be used as a metal-free, visible-light-active, sensitized photocatalyst via energy transfer mechanism, in which the light energy was converted by GQDs to produce ¹O₂, which can kill nearby microorganisms and degrade organic dyes.

Keywords graphene quantum dots, photocatalysts, metal-free, visible-light-active

Metal-free photocatalysts may have the advantages of nontoxicity and good processability, and are gradually becoming an important class of catalysts [1–3]. Up to now, some metal-free visible-light-active photocatalysts such as organic dyes [4], crystalline silicon [5], selenium [6], red phosphorus [7], α-sulfur [8], and boron carbides [9] have been discovered. However, more efforts should be made to explore other metal-free photocatalysts.

Recently, metal-free nanocarbon photocatalysts have attracted increasing interest owing to their advantages such as low cost, environmental friendliness, high selectivity, and long-term stability under mild conditions [10–13]. Among them, graphene quantum dots (GQDs), a new kind of carbon nanomaterials, have been suggested as an emerging replacement for the conventional II–VI semiconductor quantum dots applied in biomedical fields because of their unique properties such as high water solubility, surface modification flexibility, low toxicity, excellent biocompatibility, and high photostability [14–20]. Interestingly, in addition to their excellent photoluminescent properties, it has been recently demonstrated that laser-ablation-produced GQDs with the surfaces passivated by polyethylene

glycol are able to both quench and produce reactive oxygen species (ROS) in cell-free conditions [21]. These results suggest that GQDs may be a potential candidate for environmental, energy-related applications [22,23]. For example, Ristic *et al.* [24] reported that GQDs could be used as blue-light-active sensitized photocatalysts to kill microorganisms. Unfortunately, most of the GQDs reported thus far usually exhibit their absorption bands in the UV region (<400 nm); as a result, the photocatalytic activity in the visible region observed for these GQDs is far from exciting; hence, scaffolding (e.g., TiO₂, Cu₂O, Ag₃PO₄, ZnO, or Pt) is required to achieve higher photocatalytic activity [17,25]. Although recently, some impressive advances have been achieved in the synthesis of S- or N-doped GQDs as visible-light-active photocatalysts via a charge transfer mechanism [26,27], GQD photocatalysts based on an energy transfer mechanism are still limited. Therefore, developing an effective method to produce GQDs as visible-light-active photocatalysts via an energy transfer mechanism is highly challenging.

It is well known that highly promising approaches for the synthesis of carbon materials are often based on the use of well-defined molecular precursors [28,29]. Very recently, a novel approach to synthesize some water-dispersible GQDs by using well-defined conjugated polymer molecular precursors was developed by our group [30–33]. Among them, one of the as-prepared GQDs could produce ¹O₂ via a multistate sensitization (MSS) process, resulting in a quantum yield of over 1.3 (the highest ever reported in photosensitizers), and also exhibits a broad absorption band covering the entire visible region [31]. Because of water-dispersibility, broad absorption in the visible spectrum (400–700 nm), and high ¹O₂ production, as shown in Scheme 1, this type of GQD can be used as an efficient, metal-free, visible-light-active photocatalyst based on an

Key Laboratory of Photochemical Conversion and Optoelectronic Materials and CityU-CAS Joint Laboratory of Functional Materials and Devices, Technical Institute of Physics and Chemistry, Chinese Academy of Sciences, Beijing 100190, China

[†] These two authors contributed equally to this work.

^{*} Corresponding author (email: wmliu@mail.ipc.ac.cn)

Scheme 1 Schematic illustration of GQDs as an efficient, metal-free, visible-light-active photocatalyst for degradation of organic dyes and killing bacteria.

energy transfer mechanism in the photodegradation of organic dyes and to kill both Gram-negative and Gram-positive bacteria in water.

The details of the GQD synthesis and X-ray diffraction (XRD), transmission electron microscopy (TEM), and X-ray photoelectron spectroscopy (XPS) characterization are described in Supplementary information 1 and our previous report [31]. In this letter, the atomic force microscopy (AFM) images of the GQDs (Figs 1a and b) indicate that most of the vertical heights are less than 3 nm, suggesting that most of the samples consist of at most nine graphene layers [34]. The dynamic light scattering (DLS) analysis further indicates that the GQDs have diameters of 4.5–8.5 nm (Fig. 1c). This result is similar to that obtained from TEM characterization because the hydrodynamic diameter is usually slightly larger than the dry-state diameter [35]. In the ^{13}C nuclear magnetic resonance (NMR) spectrum (Fig. 1d), signals in the range of 30–85 ppm, which correspond to aliphatic (sp^3) carbon atoms, and signals from 100–185 ppm, which are indicative of sp^2 carbon atoms, were observed [36]. In addition, we also examined the surface charge of the GQDs by measuring the ζ -potential, which was $+28.8 \pm 2.3$ mV (Fig. S1).

To demonstrate the feasibility of GQDs for the photodegradation of organic dyes, we initially investigated the photodegradation of rhodamine B (RhB, 1.0×10^{-5} mol L^{-1}) under different GQD concentrations (Fig. S2). The results show that the photodegradation efficiency clearly depends

strongly on the concentration of GQDs. The decolorization rate of RhB was observed to increase upon increasing the concentration of GQDs, reaching a maximum value at $[\text{GQDs}] = 0.1$ mg mL^{-1} . Beyond this point, further increasing the concentration of GQDs led to a lower decolorization rate, suggesting that the favorable concentration of GQDs for standard conditions should be 0.1 mg mL^{-1} . This phenomenon may be ascribed to the fact that once the concentration of GQDs was increased up to 0.2 mg mL^{-1} , the high absorbance exceeded 2 optical density, which was obviously different from the presented absorption spectrum (Fig. S3). Like the traditional photosensitizers, a high concentration of GQDs can result in inner-filter effects giving non-uniform radiation distribution within the sample volume leading to lower ROS generation [37], and thus the occurrence of lower decolorization rates. Fig. 2a shows the absorption spectra of an aqueous solution of RhB (1.0×10^{-5} mol L^{-1} , 20 mL), which was exposed to visible light for various durations in the presence of GQDs (0.1 mg mL^{-1}). The absorption peaks corresponding to the RhB molecule, such as the sharp peak at 553 nm, diminish gradually as the exposure time increased before completely disappearing after approximately 12 min, which indicates the complete decolorization of RhB. The color change sequence of the sample during this process is shown in the insert of Fig. 2a, from which it is clear that the intense pink color of the starting solution gradually disappeared upon increasing the exposure time to 12 min. The mineralization behavior

Figure 1 Characterization of GQDs. (a) and (b) AFM images. (c) DLS analysis. (d) ^{13}C NMR.

Figure 2 Organic dye photodegradation. (a) Absorption spectra of a solution of RhB (initial concentration: $1.0 \times 10^{-5} \text{ mol L}^{-1}$, 20 mL) in the presence of GQDs (2 mg) under exposure to visible light. (b) Photodegradation of RhB ($1.0 \times 10^{-5} \text{ mol L}^{-1}$, 20 mL) under different conditions. (c) Absorption spectra of a solution of hydroxycoumarin (initial concentration: $2.0 \times 10^{-5} \text{ mol L}^{-1}$, 20 mL) in the presence of GQDs (2 mg) under exposure to visible light. (d) Irradiation-time dependence of the relative concentration C/C_0 of the RhB dye in solution over GQDs during repeated photodegradation experiments under visible light.

of RhB over GQDs was also examined. As shown in Fig. S4, the mineralization rate of RhB is found to be 47.0%; however, the photodegradation rate of RhB is nearly 100%, indicating that the mineralization rate is slower than the decolorization rate of dyes. In other words, the chromophores of the dye molecules are destroyed, but not completely mineralized to inorganic molecules.

The destruction of the conjugated chromophore structure was confirmed by gas chromatography-mass spectrometry (GC/MS). Several small organic compounds with low molecular weights were extracted and identified as degradation intermediates by mass spectrometry, utilizing the commercial library (NIST2) for structural assignments (Table 1). Among the several degradation products, most components were substituted benzenes and small alkanes, indicating the cleavage of RhB, followed by further degradation to produce small organic molecules. The significant reduction in total organic carbon suggests the formation of volatile organic compounds among the reaction intermediates, such as CO_2 [38].

A further comparative experiment was carried out to investigate the visible-light-induced catalytic activity. The solution of RhB was subjected to a series of experimental conditions, as follows: 1) without catalyst, visible light, and air saturated; 2) with GQDs (2 mg), in the dark, and air saturated; 3) with Degussa P25 titania (2 mg), visible light, and air saturated; 4) with GQDs (2 mg), visible light, and

air saturated; and 5) with GQDs (2 mg), visible light, and O_2 saturated. The absorption characteristic of RhB at 553 nm was chosen as the monitored parameter for the photocatalytic degradation process. The results are illustrated in Fig. 2b. The blank test (RhB without any catalyst) indicated that photolysis was negligible. Only 3% of RhB was decomposed after 60 min of irradiation under visible light. These results show that the extent of photolysis of RhB is extremely low in the absence of a photocatalyst (curve 1). A slight decrease in the concentration of RhB took place in the presence of GQDs (curve 2) in the dark, which may be mainly ascribed to the adsorption of RhB on the GQDs. In contrast, using commercial P25, 20% RhB degradation was observed after 60 min of irradiation (curve 3). The sensitization of the TiO_2 catalyst by dye molecule absorption at high wavelengths likely played a role in this oxidation [39]. The results of a typical GQD run, however, show a marked increase in dye degradation, which is up to 100% after approximately 12 min of irradiation (curve 4). This result indicates that the GQDs are comparatively more active than the traditional catalysts under visible-light irradiation. Furthermore, as can be seen from the data in curve 5 (O_2 saturated), the exposure of the RhB solution to visible light in the presence of GQDs with O_2 saturated conditions results in the complete decolorization of the dye in approximately 9 min, indicating that GQDs with O_2 saturation show an activity greater than that of air-saturated GQDs. This dif-

Table 1 Identification of the small molecular intermediates of RhB by GC/MS

Product	Retention time (min)	Molecular weight (g mol^{-1})	Formula	Structure
1	4.69	116	$\text{C}_6\text{H}_{12}\text{O}_2$
2	7.67	122	$\text{C}_7\text{H}_6\text{O}_2$
3	8.80	146	$\text{C}_6\text{H}_{10}\text{O}_4$
4	9.02	180	$\text{C}_9\text{H}_8\text{O}_4$
5	9.45	180	$\text{C}_9\text{H}_8\text{O}_4$
6	11.47	204	$\text{C}_{13}\text{H}_{16}\text{O}_2$

ference in the photocatalytic activities between air-saturated and O_2 -saturated GQDs can be explained by the higher 1O_2 generation of the latter.

To obtain further evidence that the degradation of RhB is due to the visible-light-active GQDs, we selected hydroxycoumarin, which absorbs in the UV region, as a probe molecule to investigate the photocatalytic activity of GQDs. As shown in Fig. 2c, the absorption peaks corresponding to the hydroxycoumarin molecule, such as the sharp peak at 363 nm, diminish gradually as the exposure time increases to 32 min, suggesting that the photoactivity of GQDs derives from absorbing the energy from visible light rather than receiving the energy absorbed by the dye in the visible region. Therefore, the robust activity of GQDs is ascribed to the combined improvement of visible-light absorption and ultra-high 1O_2 generation. For a photocatalyst to be useful, it should be stable under repeated applications. The N-doped TiO_2 and sulfide photocatalysts sometimes suffer from instability under repeated use [40]. To test the repeatability of RhB bleaching on as-prepared GQDs, we carried out the bleaching experiment 12 times. As shown in Fig. 2d, the RhB dye is quickly bleached after every injection of the RhB solution, and the GQD photocatalysts are stable under repeated applications with nearly constant photodecomposition rates.

On the basis of the results, we suggest a possible mechanistic pathway based on the MSS process for singlet oxygen generation [31]. As shown in Fig. 3, the reaction begins when the GQDs absorb visible light and reach the singlet excited state $^1GQDs^*$, where different processes could occur: (1) $^1GQDs^*$ return from S_1 to the ground state of GQDs (S_1-S_0) to give a fluorescence emission; (2) 1O_2 is generated through either a conventional pathway (ET from T_1) or the ET from the S_1 to 3O_2 pathway, owing to both ΔE_{ST} (the energy gap between S_1 and T_1) and ΔE_{TS} (the energy gap

between T_1 and S_0) being larger than the formation energy of 1O_2 (22.5 kcal mol $^{-1}$). As a result, small organic molecules and CO_2 can be produced by the reaction between RhB and 1O_2 .

As we all know, it is important to evaluate the toxicity of GQDs for further biomedical applications. HeLa cells were chosen for these experiments, and an MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide) assay was used to estimate the cytotoxicity of GQDs. The results shown in Fig. 4a indicate that HeLa cells are more resistant to GQDs even under the high concentration of 150 $\mu\text{g mL}^{-1}$. Thus, this concentration range of GQDs can be used in the bacterial test.

The antibacterial behavior of the prepared GQDs was tested following the procedure described in the experimental section (Supplementary information). The effective performance of the GQDs as an antimicrobial reagent is clearly established by the agar plate assay. Initially, we evaluated the antibacterial activity of the GQDs towards Gram-negative *Escherichia coli* (*E. coli*) under visible-light irradiation for 10 min, as shown in Fig. 4b (top panel). The number of viable colonies on the plates decreased significantly as the concentration of GQDs increased, and at the concentration of 150 $\mu\text{g mL}^{-1}$, complete inhibition of microbial growth was achieved. The number of surviving *E. coli* was estimated by counting the exact number of colonies of each of the respective samples, and the statistical results suggest that the antibacterial activity of GQDs against *E. coli* is concentration-dependent, as shown in Fig. 4c. *E. coli* viability of 40% was observed in the presence of 30 $\mu\text{g mL}^{-1}$ of GQDs, which decreased with increasing GQD concentration, and dropped to approximately 5% with the solution containing 150 $\mu\text{g mL}^{-1}$ of GQDs. To further evaluate the antimicrobial activity of GQDs against Gram-positive bacteria, we compared these results with those of *Staphylo-*

Figure 3 Possible mechanism of photosensitized degradation of dyes over GQDs under visible-light irradiation.

Figure 4 Antibacterial activities of GQDs. (a) Dark cytotoxicity of GQDs on HeLa cells. (b) Photographs of colonies of *E. coli* and *S. aureus* incubated on agar plates obtained from different concentrations of GQDs under irradiation for 10 min. Quantitative evaluation of the antimicrobial ability of the GQDs against Gram-negative *E. coli* (c) and Gram-positive *S. aureus* (d) by counting the colonies grown on agar plates. Data are shown as mean $\pm$ standard deviation ($n = 3$).

coccus aureus (*S. aureus*) under the same conditions. It was obvious that the GQDs exhibited higher efficiency against Gram-positive *S. aureus* than against Gram-negative *E. coli* from the fact that fewer colonies could be observed on the plates in the presence of GQDs at the same mass concentration (Fig. 4b, bottom panel). The statistical results in Fig. 4d also show 90% Gram-positive *S. aureus* death at $30 \mu\text{g mL}^{-1}$ of GQDs, corroborating the conclusion mentioned above more clearly. This can be attributed to the different structures of Gram-positive and Gram-negative bacteria, because Gram-negative bacteria possess an additional outer membrane, which can protect the inner layer from attacks by chemicals or radicals [41]. In contrast, no bacteria death was observed in the dark with $200 \mu\text{g mL}^{-1}$ of GQDs (Fig. S5). The high antibacterial activity of GQDs can be attributed to the high $^1\text{O}_2$ -generation efficiency of GQDs.

In summary, GQDs were first used as metal-free, visible-light-active photocatalysts based on an energy transfer mechanism for photodegradation of organic dyes and inactivation of bacteria. The GQDs show higher efficiency in decomposing RhB under visible-light irradiation. In addition, GQDs are extremely efficient in killing both Gram-negative *E. coli* and Gram-positive *S. aureus* in water even at lower concentrations. Furthermore, the GQDs are photostable during the photodegradation process. In view

of the above, GQDs can be a promising candidate in the photodegradation and disinfection fields.

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- Author contributions** Wang P supervised the project. Ge J, Lan M, and Liu W synthesized the GQDs, designed and carried out the experiments, analyzed the data, and wrote the manuscript. Zhou B and Guo L helped with the evaluation of the cytotoxicity of the GQDs. Niu G, Jia Q, and Meng X contributed to the AFM, TEM, and HRTEM measurements and analysis of the data.
- Conflict of interest** The authors declare that they have no conflict of interest.
- Supplementary information** Experimental details and supporting data are available in the online version of the paper.

Weimin Liu is an associate professor at the Technical Institute of Physics and Chemistry, Chinese Academy of Sciences (TIPC, CAS). She received her PhD degree in organic chemistry from TIPC in 2008 and then joined the TIPC. Her current research interests focus on the design of organic or inorganic luminescent materials and their application in biosensing and bioimaging.

Jiechao Ge is currently an associate professor at the TIPC, CAS. He received his PhD degree from Shandong Normal University in 2008 under the guidance of Prof. Bo Tang. During 2008 and 2009, he joined the group of Prof. Harry Dorn and worked as a postdoctoral researcher at Virginia Polytechnic Institute & State University. He joined TIPC in 2010. His research interests focus on nanoparticles as phototheranostics and photocatalysts.

可见光响应的高效石墨烯量子点光催化剂

葛介超, 蓝敏换, 刘卫敏, 贾庆岩, 郭亮, 周炳江, 孟祥敏, 牛广乐, 汪鹏飞

摘要 本文提供了石墨烯量子点的应用新领域. 在此前的研究中, 我们发现以聚噻吩衍生物 (PT2) 为前驱物制备的石墨烯量子点在太阳光的照射下可高效产生单线态氧. 本文进一步研究发现, 此类石墨烯量子点在模拟太阳光的照射下 (大于420 nm) 可以快速多次降解水中有机污染物和高效灭杀细菌, 表明石墨烯量子点在环境保护领域拥有巨大的应用潜力.