A “bottle-around-ship” method to encapsulated carbon nitride and CdTe quantum dots in ZIF-8 as the dual emission fluorescent probe for detection of mercury (II) ion

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Abstract
A facile and efficient “bottle-around-ship” approach for preparing the ratiometric fluorescent probe has been developed by encapsulating the red-colored fluorescence CdTe quantum dots (QDs) and blue-colored fluorescence graphitic carbon nitride quantum dots (g-CNQDs) into the zeolitic imidazolate metal–organic frameworks (ZIF-8) in one step. At a single excitation of 360 nm, the obtained probe ZIF-8@g-CNQD/CdTe shows the dual-emission peaked at 450 and 633 nm, respectively. The red emission of CdTe QDs is selectively quenched by the Hg2+, whereas the blue fluorescence of g-CNQDs as an internal reference is insensitive, resulting in an apparent color transformation from pink to blue for special recognition of Hg2+. By this approach, the relative fluorescence intensity ratio \( \frac{F_{633}}{F_{450}} \) decreased linearly with increasing Hg2+ concentration in the 0.2–3.5 μM range with a low limit of detection (LOD) of ~ 46 nM. Therefore, we demonstrate that this “bottle-around-ship” process provides a new strategy for the construction of ratiometric fluorescent Hg2+ probes with good simplicity, high efficiency, and excellent stabilities. Moreover, the obtained Hg2+ fluorescent probe shows good results in the detection of actual samples.

Keywords Metal–organic frameworks · Ratiometric fluorescence · Mercury (II) ion · Graphitic carbon nitride quantum dots

Introduction
As we all know, Hg2+ is a highly toxic pollutant, which widely exists in various environmental media and food chains (especially fish) due to the wide distribution of mercury in nature (common in air, water and soil). Hg2+ can be easily absorbed by the skin, respiratory tract and digestive tract, and at the same time accumulate in the body, eventually causing permanent damage to the nervous system, digestive system and kidneys [1–3]. Therefore, detection of Hg2+ is undoubtedly an important step in environmental and health monitoring. The traditional Hg2+ detection method uses a variety of approaches, including cold vapor atomic absorption spectrometry (AAS) [4], atomic emission spectrometry (AES) [5], cold vapor atomic fluorescence spectrometry (AFS) [6], inductively coupled plasma-mass spectrometry (ICP-MS) [7], etc. Although these methods have high sensitivity and low limit of detection, they usually require complex sample pre-treated and professional technical operations, resulting in time-consuming, and high-cost detection. Other arising techniques such as electrochemistry [8], surface-enhanced Raman scattering [9] fluorescent and colorimetric methods [10] also have been applied. Among them, different types of fluorescent sensor approaches for the detection of Hg2+ have been developed, including organic small molecules, polymers, liposomes, DNAzymes, proteins, oligonucleotides, inorganic nanomaterials, etc. [11, 12]. The development of highly sensitive and selective fluorescence sensors for Hg2+ has been of particular interest for a long time with the advantages of simple operation and low price.

Till now, there have been a large number of reports on Hg2+ ions’ detection through fluorescence approach [13, 14]. Nevertheless, fluorescence probes based on changes in signal emission intensity may be influenced by instrument performance, environmental conditions and fluorescent molecular concentration. As ratiometric fluorescent sensors that compare the fluorescence intensity at two different wavelengths before and after with analyte recognition, they are receiving

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more and more attention [15]. Compared with the single-emission sensors, the signals change of the ratio fluorescent sensors combined with the dual-wavelength intensity ratio are independent of the probe concentration, environmental influence and excitation light intensity fluctuation, so the quantitative accuracy and sensitivity are effectively improved. They consist of response/indicator signals and reference signals to build a stable analysis system through the use of self-calibration and the sensitivity and selectivity of the response signal are usually required as indicators, while the inert response signal is used as a reference signal in the system, which helps to achieve a stable ratio constant. Ratiometric fluorescence sensing technology has been regarded as an effective strategy for specific recognition and anti-interference sensing of target analytes. Moreover, the ratiometric assay can also cause color changes to facilitate visual detection [16, 17].

The exclusive optical properties of quantum dots (QDs), including high photostability, great photoluminescence efficiency, strong resistance to photobleaching, sharp emission profile and size-dependent emissive wavelength, cause them extremely attractive fluorophore in the field of fluorescence sensing [18]. The excellent optical properties of quantum dots make them the best choice for the preparation of ratiometric dual emission fluorescent probes, which can integrate two kinds of quantum dots into one probe. For instance, Jiang et al. developed a ratiometric fluorescent probe for the detection of copper ions by covalently linking carboxy-modified cadmium telluride QDs (CdTe QDs) complexed with amino-functionalized carbon nanodots [19]. In Qian’s work, graphene quantum dots as reaction sites are self-assembled electrostatically onto silica spheres wrapped with red-emitting CdTe QDs that provide a built-in correction for environmental effects to construct fluorescent sensors, which can be applied to visualize the detection of mercury(II) ions [20]. The construction of ratiometric fluorescent probes usually involves tedious multi-step preparation and sophisticated coupling or chemical modification processes. Therefore, obtaining a suitable fluorescence ratiometric system remains a difficult task, including the development of a simple ratiometric method for the determination of Hg²⁺.

Because of the high porosity and surface area, easy preparation, good chemical robustness and prominent thermal stability, ZIF-8 as zeolitic imidazole-based metal–organic frameworks (MOFs) have been considered as an ideal vehicle to incorporate with types of functional materials for the great potential in catalysis, gas storage, and adsorption [21]. Furthermore, it has been used to encapsulate fluorescent nanoparticles such as carbon QDs [22], graphene QDs [23], and copper nanoclusters [24] to enhance their stability, sensitivity and selectivity for target detection. In this work, the dual emission fluorescent probe ZIF-8@g-CNQD/CdTe was developed as a facile and efficient fluorescent probe to sensitively detect Hg²⁺. The red-emitting CdTe QDs and blue-emitting graphitic carbon nitride QDs (g-CNQDs) were simultaneously embedded into the ZIF-8 via the “bottle-around-ship” approach [25] by directly mixing with the precursors of ZIF-8 (Scheme 1). Dual emission was observed from ZIF-8@g-CNQD/CdTe at 450 and 633 nm under a single wavelength excitation at 360 nm. In the presence of Hg²⁺, the luminescence of CdTe QDs (633 nm) is dynamically quenched, while the blue light emission of g-CNQDs (450 nm) remains. An obvious fluorescent color change from pink to blue can be observed by the naked eye under a UV lamp. As a ratiometric and colorimetric fluorescent sensor, the probe displays high sensitivity and selectivity. The application of the dual emission probe for recognizing Hg²⁺ in real water sample obtained from laboratory tap water was investigated. The research provides a new technology for the construction of dual emission ratiometric fluorescence sensors.
**Experimental**

**Materials**

CdCl$_2$·2.5H$_2$O (99.99%), tellurium powder (99.99%), 2-methylimidazole (98%), sodium borohydride (NaBH$_4$ ≥ 96.0%), 3-mercaptopropionic acid (MPA, ≥ 98%), 4-(2-hydroxyethyl)-1-piperazinethanesulfonic acid (HEPES) and poly(vinylpyrrolidone) (PVP, MW = 58,000) were supplied by Aladdin (Shanghai, China). Urea (H$_2$NCONH$_2$, 99%), sodium citrate (C$_6$H$_5$Na$_3$O$_7$·2H$_2$O, ≥ 99.0%), HgCl$_2$, CaCl$_2$·2H$_2$O, BaCl$_2$·2H$_2$O, MgCl$_2$·6H$_2$O, SrCl$_2$·6H$_2$O, MnCl$_2$·4H$_2$O, CoCl$_2$·6H$_2$O, CuCl$_2$·2H$_2$O and Pb(NO$_3$)$_2$ were purchased from Sinopharm Chemical Reagent Co. Ltd (China). All other reagents were of analytical grade. Milli-Q water (18.2 MΩ cm) was used in all the related procedures.

**Experimental measurements**

UV–Vis absorption spectroscopy measurements were performed using a Shimadzu UV-2550 spectrophotometer. Fluorescence experiments were performed by an Agilent Cary Eclipse (USA) fluorescence spectrometer with the emission and excitation slit widths were both 10 nm. The analysis of powder X-ray diffraction (XRD) spectrum was performed on a Cu-Kα X-ray diffractometer (Rigaku D/max2500 PC; Japan). Fourier Transform infrared spectrum was taken on a PerkinElmer Spectrum Two spectroscope. The morphologies were characterized with a JSM-7900F scanning electron microscopy (SEM, JEOL; Japan) and a 2100F transmission electron microscope (TEM, JEOL; Japan).

**Preparation of g-CNQDs**

The fluorescent carbon nitride dots (g-CNQDs) were obtained according to a previously reported method with some modifications [26]. Urea and sodium citrate with a molar ratio of 6:1 were ground in an agate mortar. After ground for 45 min, the powders were transferred into a Teflon autoclave, sealed, and reacted at 180 °C for 2 h. The resultant yellowish product was collected by washing three times with ethanol by centrifugation for 24 h in the dark, and then dispersed in 2 mL of methanol for further use.

**Preparation of CdTe QDs**

The aqueous red-emitting CdTe QDs were synthesized according to the previous report with some modifications [27]. NaHTe as the precursor of CdTe QDs was prepared by adding 47.2 mg (1.25 mmol) NaBH$_4$ to a flask containing 64.2 mg (0.5 mmol) tellurium powder and 5 mL Milli-Q water under nitrogen atmosphere in an ice bath. The reaction was stirred for several hours until all tellurium powder was dissolved and leaving a pink mixture. 0.1144 g (0.5 mmol) of CdCl$_2$·2.5H$_2$O and 100 μL (2.5 mmol) of MPA were dissolved in 50 mL deionized water followed by adjusting pH to 11 with 1 M NaOH under vigorous stirring. After the mixture has been deaerated under bubbling N$_2$ for at least 30 min, 2.8 mL of the prepared freshly NaHTe solution was quickly injected under vigorous stirring. The solution was refluxed at 100 °C under open-air conditions for 48 h to obtain red CdTe QDs. Finally, the prepared CdTe QDs were purified by centrifugation with ethanol and redispersed in 30 mL pure water for further use.

**Preparation of PVP-stabilized CdTe QDs**

1.00 mL of PVP methanolic solution (5%, Mw = 58,000) was mixed with 10 mL of CdTe QDs solution and the pH of the solution was then adjusted to 9.0 with 1.0 M NaOH. After the resulting mixture was stirred at room temperature for 24 h in the dark, PVP-stabilized CdTe QD was precipitated with acetone and collected by centrifugation, the product was washed with chloroform and cyclohexane for several times to remove excessive free PVP, and the purified QDs were dispersed in 2 mL of methanol for further use.

**Preparation of ZIF-8@g-CNQD/CdTe dual-emission probe**

ZIF-8@g-CNQD/CdTe probe was fabricated by a one-pot method. First, 164.2 mg of 2-methylimidazole was dispersed in 5 mL of methanol, after that 200 μL of g-CNQDs (76 mg mL$^{-1}$) aqueous solution and 20 μL of PVP-stability CdTe QDs solution were added under stirring for 3 min at room temperature. Second, 5 mL methanol solution of Zn(NO$_3$)$_2$·6H$_2$O (148.75 mg) was mixed and kept stirring for 5 min. Subsequently, the mixture was allowed to react at room temperature for 24 h without stirring. Finally, ZIF-8@g-CNQD/CdTe composites were collected by centrifugation, washed with methanol, and vacuum-dried overnight (50 °C). The product was dispersed in 2 mL ultrapure water to obtain ZIF-8@g-CNQD/CdTe suspension and stored at 4 °C for further use.

**Fluorescence assay for Hg$^{2+}$**

The Hg$^{2+}$ sensing assay was performed by using mercury chloride (HgCl$_2$) aqueous solution. An excitation wavelength of 360 nm was used to excite ZIF-8@g-CNQD/CdTe, and the emission spectrum was recorded from 400 to 800 nm. The photos were taken with a digital camera in a dark box under 365 nm UV light. All the experiments for Hg$^{2+}$ detection were performed at room temperature. All fluorescence measurements were performed in 10 mM HEPES–NaOH buffer (pH 8.0).
In a typical process, 0.2 mL of ZIF-8@g-CNQD/CdTe was first added to 0.3 mL of HEPES buffer (10 mM, pH 8.0) and mixed well. Then, a serial of Hg$^{2+}$ solutions with different concentrations was added to the above solution to ensure the final concentration range from 0 to 10 μM and HEPES buffer was used to maintain the final volume of the mixture at 2 mL. The mixture was incubated at room temperature for 5 min before measuring the emission spectra of these reaction solutions. To assess the selectivity of the dual-emission probe to Hg$^{2+}$, the ratio of the $F_{633}/F_{450}$ fluorescent intensity of ZIF-8@g-CNQD/CdTe exposed to other metal ions, such as Mg$^{2+}$, Ca$^{2+}$, Ba$^{2+}$, Sr$^{2+}$, Mn$^{2+}$, Cd$^{2+}$, Pb$^{2+}$, Co$^{2+}$ and Cu$^{2+}$ was studied under the same conditions.

**Application in real sample**

The water sample was obtained from tap water in our laboratory. First, the solution was filtered twice using ordinary qualitative filter paper and 0.45 μm NYLON filters to remove excess solid impurities and suspended matter, then the pH of the water sample was adjusted to 8.0 by adding HEPES buffer solution, and finally different concentrations of mercury ions (II) were added to the prepared water sample for the determination of mercury ions using the standard addition method.

**Results and discussion**

**Preparation and characterization of ZIF-8@g-CNQD/CdTe**

In this work, ZIF-8@g-CNQD/CdTe probe was simple and effective obtained via a simple one-pot synthesis by mixing the g-CNQDs and PVP-stabilized CdTe QDs with the precursors of ZIF-8 (Scheme 1). This ZIF-8@g-CNQD/CdTe probe shows the dual-emission peaked at 450 and 610 nm which could be a ratiometric fluorescent sensor for Hg$^{2+}$ detection. The g-CNQDs have good photostability and chemical inertness in the presence of Hg$^{2+}$ and thus can be selected as a reference signal for ratiometric fluorescent probes. Meanwhile, the red fluorescence of PVP-stabilized CdTe QDs is effectively quenched by Hg$^{2+}$ and thus can serve as a reaction site for Hg$^{2+}$.

The morphological structure of ZIF-8@g-CNQD/CdTe was characterized by scanning electron microscope (SEM) and the transmission electron microscope (TEM) techniques (Fig. 1). The size of the resulting ZIF-8@g-CNQD/CdTe is mainly distributed in the range of 30–50 nm as determined by the TEM image. SEM image shows the uniform hexagonal for particles. The powder X-ray diffraction (XRD) measurements of both ZIF-8 and ZIF-8@g-CNQD/CdTe

![Fig. 1](image)

**Fig. 1** a The SEM image and b TEM image of the as-prepared ZIF-8@g-CNQD/CdTe sample

![Fig. 2](image)

**Fig. 2** XRD patterns of the simulated ZIF-8 (blue), the as-prepared ZIF-8 (red) and ZIF-8@g-CNQD/CdTe (black)
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exhibit identical patterns to that of the simulated patterns of ZIF-8 (Fig. 2), implying that the ZIF-8 was successfully prepared in the presence of the g-CNQD and PVP-stabilized CdTe QDs. The FTIR spectrum of ZIF-8@g-CNQD/CdTe is depicted in Fig. 3 that shown the same characteristic peaks with ZIF-8. This can clearly demonstrate that both the g-CNQD and PVP-stabilized CdTe QDs are well coated in ZIF-8 and the basic framework structure of ZIF-8 is retained [28].

The prepared carbon nitride quantum dots (g-CNQDs) have a strong fluorescence emission peak at 450 nm under 360 nm excitation, and the aqueous solution of g-CNQDs shows bright blue fluorescence under 365 nm UV irradiation (Fig. 4). Furthermore, as shown in Fig. 5, the ZIF-8@g-CNQD/CdTe appeared two distinct fluorescence peaks centered at 450 and 633 nm, respectively. The 450 nm peak should be due to the encapsulated g-CNQDs. The profile of the normalized fluorescent characteristic emission peak at 450 nm of ZIF-8@g-CNQD/CdTe is almost the same as that of g-CNQDs, suggesting that the fluorescent characteristics of g-CNQDs in ZIF-8 have not been changed. It has been reported that the functionalization of nanoparticle surfaces with polyvinylpyrrolidone (PVP) can facilitate the crystallization of ZIF-8 [29]. Here PVP-stabilized CdTe QDs were used for the construction of the probe. The CdTe QDs,
PVP-stabilized CdTe QDs and ZIF-8@g-CNQD/CdTe have almost identical emission peaks of CdTe QDs at 633 nm under 360 nm excitation, indicating that PVP surfactant and ZIF-8 shell have no significant effect on the fluorescence spectra of CdTe QDs.

**Ratiometric fluorescent detection of Hg²⁺**

To excite the g-CNQDs and CdTe QDs simultaneously, the excitation wavelength for the ratiometric fluorescent probe is at 360 nm. To examine the effect of pH of the reaction solution on ZIF-8@g-CNQD/CdTe, the fluorescence intensity in the pH range from 5.0 to 12.0 was measured. According to the result in Fig. 6, the intensity at 450 nm hardly changes with pH, while the intensity at 633 nm is tend to be unstable at different pH values; therefore, the ratio of $F_{633}/F_{450}$ is higher under neutral or alkaline conditions than that of acid conditions [30]. The maximum ($F_{633}/F_{450}$) value was obtained when the pH was 8.0, so the pH 8.0 was selected as the optimum pH value for further studies.

Fluorescence spectra were recorded after treatment of the various concentrations of Hg²⁺ to estimate the fluorescence intensity ratio ($F_{633}/F_{450}$) to various concentrations of Hg²⁺ (Inset: a linear region). CIE chromaticity coordinates of ZIF-8@g-CNQD@CdTe in the presence of different concentrations of Hg²⁺ under the single excitation of 360 nm.
sensing capacity of the ZIF-8@g-CNQD/CdTe sensor. As shown in Fig. 7, with the addition of Hg²⁺ into the suspension of ZIF-8@g-CNQD/CdTe, the fluorescence peaks at 633 nm ascribed to the emission of CdTe QDs are decreased. By contrast, the fluorescence peak at 450 nm ascribed to the emission of g-CNQD remained constant. The ratios of \( F_{633}/F_{450} \) are closely associated with the degree of the impact of the ZIF-8@g-CNQD/CdTe caused by Hg²⁺. Therefore, the ratio of \( F_{633}/F_{450} \) can be used as an internal reference ratiometric fluorescence. In the range of Hg²⁺ concentration from 0 to 10 μM, the fluorescence intensity at 633 nm decreases substantially with the increasing of Hg²⁺. The fluorescence at 633 nm was completely quenched when the concentration of Hg²⁺ was greater than 10 μM, and the dual-emission fluorescent spectra no longer showed any change with increasing the concentration of Hg²⁺. A good linear relationship between the \( F_{633}/F_{450} \) ratio and the Hg²⁺ concentration was obtained at Hg²⁺ concentrations from 0.2 to 3.5 μM (Fig. 8B, inset), and the limit of detection (LOD) for Hg²⁺ was as low as 46 nM (signal-to-noise ratio of \( S/N = 3 \)).

The CIE chromaticity diagram shows a clear color change, as the concentration of Hg²⁺ increases the points on the coordinates gradually move from the red region to the blue region (Fig. 8C).

**Selectivity of the assay**

The specific selectivity of the ZIF-8@g-CNQD/CdTe sensing system to the target metal ions over other metal ions is crucial for a probe to evaluate the performance of the sensor.

As shown in Fig. 8, selectivity is an important parameter in assessing the performance of fluorescent probes, especially in real samples. Thus, the effect of potentially interfering ions (Mg²⁺, Ca²⁺, Ba²⁺, Sr²⁺, Mn²⁺, Cd²⁺, Pb²⁺, Co²⁺, Cu²⁺) on the relative fluorescence intensity of ZIF-8@g-CNQD/CdTe under the same conditions was investigated. We can find that Hg²⁺ (5 μM) displayed the strongest fluorescence quenching and other high concentrations of cations (50 μM) have no significant effect on the FL intensity. Besides, Cu²⁺ (10 μM) shows a slight reduction in the FL signal. It has been reported that Cu²⁺ has strong quenching effect on CdTe QDs [31]. But in this case, the quenching effect can be partly reduced with the hybrid ZIF-8 shell. These results revealed that the dual emission probe presented an excellent selectivity for Hg²⁺ sensing over the other metal ions.

**Practical application of ZIF-8@g-CNQD/CdTe in the real water sample**

To evaluate the general practicability of the ratiometric sensor, a standard addition method was performed on the tap water sample for our assay. Different amounts of Hg²⁺ were spiked and their corresponding fluorescence spectra were obtained. As listed in Table 1, the recoveries in actual samples with the relative standard deviations (RSD) were obtained. The results demonstrate that the dual emission probe is feasible in the real sample for Hg²⁺ detection.

**Conclusions**

In summary, we have demonstrated a facile and effective strategy for a dual-emission ratiometric fluorescence sensor by encapsulation of red-emissive CdTe QDs and blue-emissive g-CNQDs in ZIF-8. The method is robust, sensitive and selective by using dual signal response as signal readout. The red fluorescence of the CdTe QDs can be selectively quenched by Hg²⁺, while the blue fluorescence of the g-CNQDs is an internal reference, resulting in a distinguishable fluorescence color change from pink to blue upon the addition of Hg²⁺. On this basis, we have developed ZIF-8@g-CNQD/CdTe based fluorescence probe for the sensitive and selective detection of Hg²⁺ with a linear range from 0.2 to 3.5 μM and a LOD of 46 nM (\( S/N = 3 \)). This study
highlighted the potential of ZIF-8 as a support material for the construction of ratiometric fluorescence sensors and extends its application in the detection of contaminants in food and the environment.

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Declarations

Conflict of interest The authors declare that there is no conflict of interest associated with this research.

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