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Mercaptoacetic Acid Capped Cadmium Sulfide Quantum Dots as Novel Fluorescence Sensors for Determination of Cetirizine

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In a one-step process using low-priced materials, water-soluble mercaptoacetic acid-capped cadmium sulfide quantum dots (MAA-CdS QDs) were synthesized. The mercaptoacetic acid was used as a stabilizing agent and surface modifier in the aqueous solution. The synthesis conditions were optimized to discover the effects of sodium sulfide concentration and pH values on the optical properties of cadmium sulfide quantum dots. Transmission electron microscopy (TEM), ultraviolet-visible absorption spectroscopy (UV-Vis), Fourier transform infrared (FT-IR) spectroscopy, and photoluminescence emission spectroscopy were used for studying the as-prepared quantum dots. This nanosized probe was used for selective and fast determination of cetirizine. The fluorescence emission intensity of mercaptoacetic acid-capped cadmium sulfide quantum dots with excitation/emission peaks at 335/500 nm was quenched by cetirizine, effectively. The significant factors in the detection of cetirizine were examined, and the optimum conditions were recorded. Linear fluorescence intensity response of Mercaptoacetic acid capped cadmium sulfide quantum dots with cetirizine concentration is proportional in the concentration range of 1.60×10^{-10} to 1.16×10^{-9} M under optimum conditions. The detection limit of this nanosensor was 6.48×10^{-11} M, and its correlation coefficient was 0.9904. In the presence of other drugs and amino acids, the fluorescence emission intensity of the MAA-CdS QDs probe was investigated to define the sensor's selectivity.

Keywords: Cadmium sulfide quantum dots, Mercaptoacetic acid, Fluorescence, Cetirizine

INTRODUCTION

In the determination field, nanomaterials have attracted increasing attention for their typical optical properties. Semiconductor QDs are individual nanoparticles. In the small dimensions, different behaviors of the nanomaterials cause unprecedented capabilities in the scientific and technical applications of the QDs. QD nanoparticles have unique optical characteristics such as high luminescence efficiency, size-dependent emission wavelengths, and good photostability [1-3]. QDs have narrow emission spectra [4]. Therefore, they exhibit better optical characteristics than organic fluorophores [5-8]. These advantages of the QDs make them ideal fluorescent indicators in medicine [9-12], biology [13,14], sensing [15], technology [16,17], and determination of necessary analytes [18,19]. Since the photophysical properties of QDs depend on their size, controlling the size is an essential factor. The QDs with larger sizes have a smaller bandgap and lower energy levels. The wavelength and the emission color of the nanoparticles changes by varying the size of the QDs [20,21]. Among QDs, cadmium sulfide QDs (CdS QDs) have the most negligible excitation Bohr radius (2.4 nm) and the highest bandgap (2.4 eV) [22,23]. Many studies have focused on the development of a new method to synthesize high-quality QDs with high fluorescence intensity [24]. Since the electron-hole recombination process depends on the interaction of the analyte and the surface of QDs, the fluorescence of QDs dramatically depends on their surface

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functional groups and states [25]. Modifying the QDs leads to the tenability of the fluorescence emission wavelength, the excitation and emission intensities enhancement, and the development of the photostability and selectivity, which depends on the type of the capping agent [26]. QDs are usually modified with hydrophilic stabilizing functional agents such as l-cysteine, mercaptopropionic acid, and thioglycerol [27-31]. Therefore, a simple procedure was provided for functionalizing the surface of the QDs to prepare sensitive and selective fluorescence sensors. The polar -COOH group of mercaptoacetic acid (MAA) leads to water solubility and biocompatibility of the QDs.

Cetirizine (CTZ), named as 2-[2-[4-[(4-chlorophenyl) phenylmethyl]piperazin-1-yl]ethoxy] acetic acid, a non-sedative histamine H1-receptor antagonist used for the treatment of seasonal rhinitis and chronic urticaria or pruritus of allergic origin (Fig. 1) [32]. CTZ is commonly prescribed to patients with allergic diseases such as allergic rhinitis and chronic urticarial, which reduces the symptoms of allergic asthma. CTZ is approved for use in two-year-olds, older children, and adults. The function of CTZ in allergic asthma symptoms may be due to the contractionary effects of histamine on the lungs. Various expensive and complicated methods have been reported for CTZ measurement, which includes gas chromatography, high-performance liquid chromatography (HPLC) with ultraviolet absorption (UV), and mass spectrometry detection [33-42]. This research developed a rapid, simple, and highly selective method for measuring CTZ based on the fluorimetric method. For the sensitive and selective determination of CTZ, the CdS QDs were synthesized using mercaptoacetic acid as the stabilizer and surface modifier. MAA-CdS QDs have a selective and rapid response to CTZ compared to other drugs.

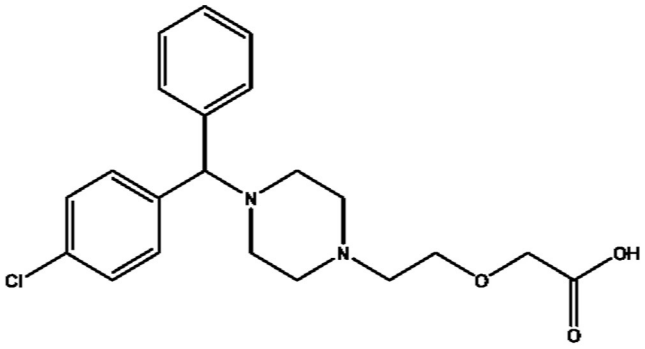

Fig. 1. The structure of Cetirizine.

EXPERIMENTAL

Materials

Cadmium nitrate tetrahydrate ($Cd(NO_3)_2.4H_2O$), sodium sulfide pentahydrate ($Na_2S.5H_2O$), potassium dihydrogen phosphate (KH_2PO_4), and di-potassium hydrogen phosphate (K_2HPO_4) were purchased from Merck (Germany). Cetirizine, amino acids, and other drugs were of analytical grade. 0.01 M CTZ standard solution was prepared daily using a proper amount of CTZ drug. The low-concentration solutions of the CTZ were obtained by diluting the stock solution.

Instruments

Ultraviolet-visible absorption spectra were measured by a computerized WPA-Biowave II. Fluorescence spectra were recorded with a JASCO-FP-6500PC spectrofluorimeter. A Metrohm 827 pH meter was used to measure the pH of the solution. The morphology of QDs was performed by transmission electron microscopy using a Philips-CM 30 model with a voltage of 100 kV. The Fourier-transform infrared (FT-IR) spectrum of the QDs was recorded using a nexus 670 Thermo Nicolet spectrometer with a KBr disk.

Preparation Procedure for MAA-CdS QDs

The preparation of CdS QDs functionalized with mercaptoacetic acid was performed using a fast method. First, 0.1 M NaOH was added to a 40mL solution containing 9.0×10^{-4} M $CdNO_3.4H_2O$ and 9.2×10^{-3} M MAA to obtain the pH = 9. After purging the solution with pure nitrogen for 30 min, 10 ml of a 4×10^{-3} M Na_2S solution was added drop by drop. Then, the mixture was reacted under pure nitrogen for 30 min.

General Procedure for Determination of CTZ

Generally, 1.5 ml of QDs solution, 0.5 ml of phosphate buffer solution (pH = 5.0), and an appropriate concentration of CTZ solution were added to a quartz cell. The volume of the solution reached 3 ml with deionized water, and the

fluorescence spectra were recorded after 5 min at the excitation wavelength of 335 nm at room temperature.

Real Sample Preparation

The urine samples of healthy volunteers were spiked with different concentrations of CTZ standard solution. The samples were centrifuged at 10000 rpm for 10 min and diluted up to 10 times with deionized water. The solution was used after the filtrations by 0.22 µm membrane.

RESULTS AND DISCUSSION

Characterization

The UV-Vis absorption and fluorescence emission spectra of the synthesized MAA-CdS QDs are shown in Fig. 2. According to the figure, the absorption peak of the MAA-capped CdS nanoparticles is located at 305 nm. The maximum fluorescence emission spectra of the MAA-CdS QD is at the wavelength of 500 nm when excited at 335 nm (Fig. 2). As illustrated in Fig. 3, the TEM film of MAA capped CdS QDs confirmed the size of the nanoparticles in the range of 18 to 20 nm with the spherical shape.

Figure 4 shows the Fourier-transform infrared spectroscopy (FTIR) spectra of MAA and MAA-CdS QDs. IR spectra of both MAA-CdS QDs and free MAA showed peaks for carboxyl and carbonyl groups. The peak for S-H (2645.25, 2565.23 cm^{-1}) vibration disappeared in the IR spectrum of MAA-CdS QDs, and that was the result of covalent bonding between thiols of mercaptoacetic acid and Cd atom on the surface of quantum dots. The results are summarized in table S1.

Effect of Sodium Sulfide Concentration on the Synthesis of MAA-CdS QDs

The effect of Na_2S concentration on the synthesis of surface-modified cadmium sulfide quantum dots with mercaptoacetic acid was investigated. Changes in the intensity and symmetry of the fluorescence emission were observed as the sodium sulfide concentration changed. The fluorescence spectra of the nanoparticles at different concentrations of sodium sulfide are shown in Fig. 5. According to Fig. 5, the concentration 8×10^{-4} M was selected as the optimum concentration.

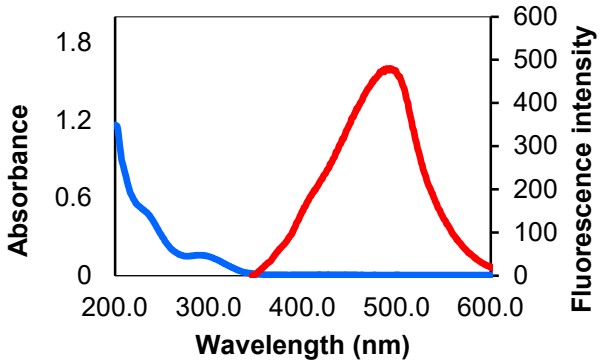

Fig. 2. The absorption and fluorescence emission spectra of MAA-CdS QDs.

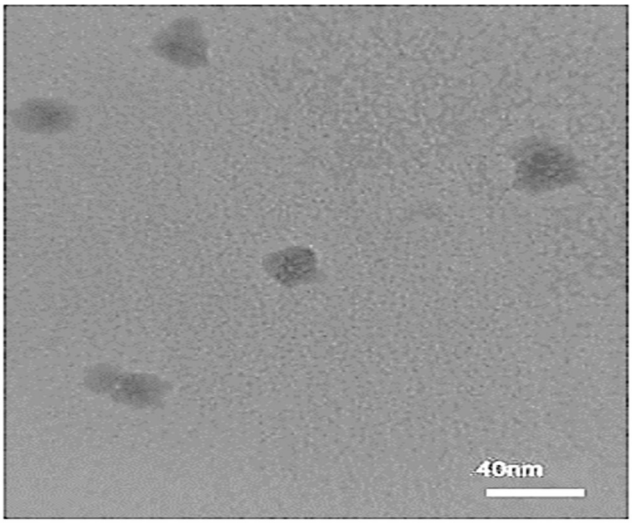

Fig. 3. Transmission electron microscopy (TEM) image of MAA-CdS QDs.

Effect of pH on the Synthesis of MAA-CdS QDs

The impact of pH on the synthesis of nanoparticles was investigated to inspect the quality of cadmium sulfide quantum dots modified by mercaptoacetic acid. As illustrated in Fig. 6A, pH = 9 was chosen as the optimum pH because of its higher symmetric fluorescence peak and better fluorescence intensity. This pH improves the peak symmetry and increases the intensity of the fluorescence emission. To Tangible the effect of pH, the emitted fluorescence from QDs that were synthesized at different pH levels, against the UV lamp is shown in Fig. 6B.

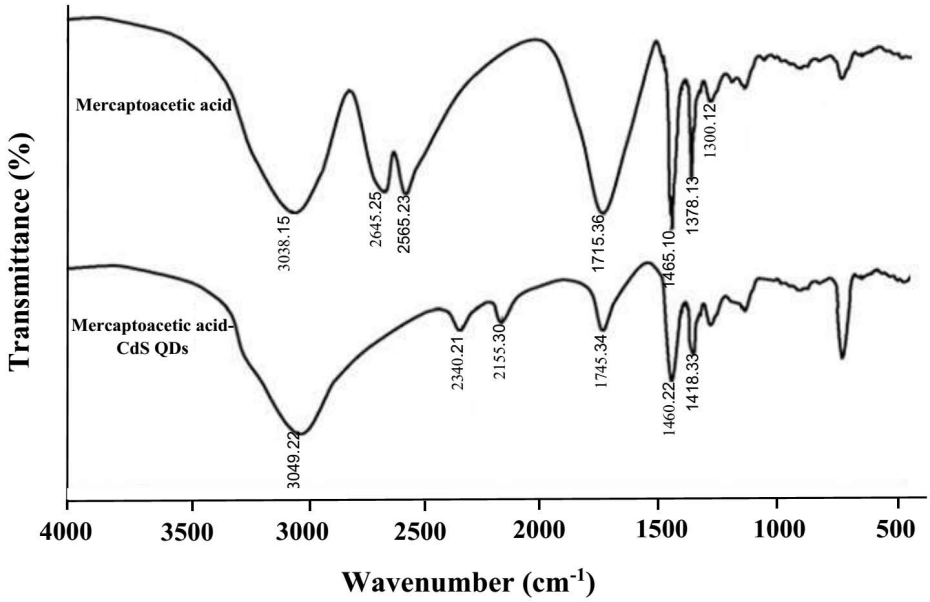

Fig. 4. The Fourier-transform infrared spectroscopy spectra of MAA and MAA-CdS QDs.

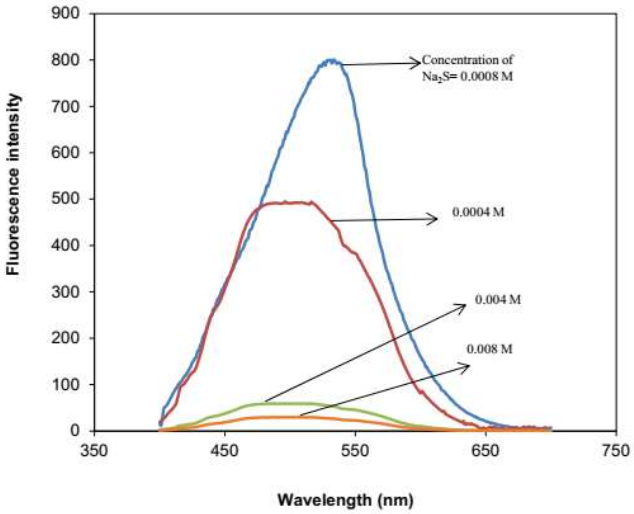

Fig. 5. The fluorescence emission spectra of the as-prepared MAA-CdS QDs with different concentrations of Na_2S (0.0008 to 0.08 M). The excitation wavelength was 335 nm.

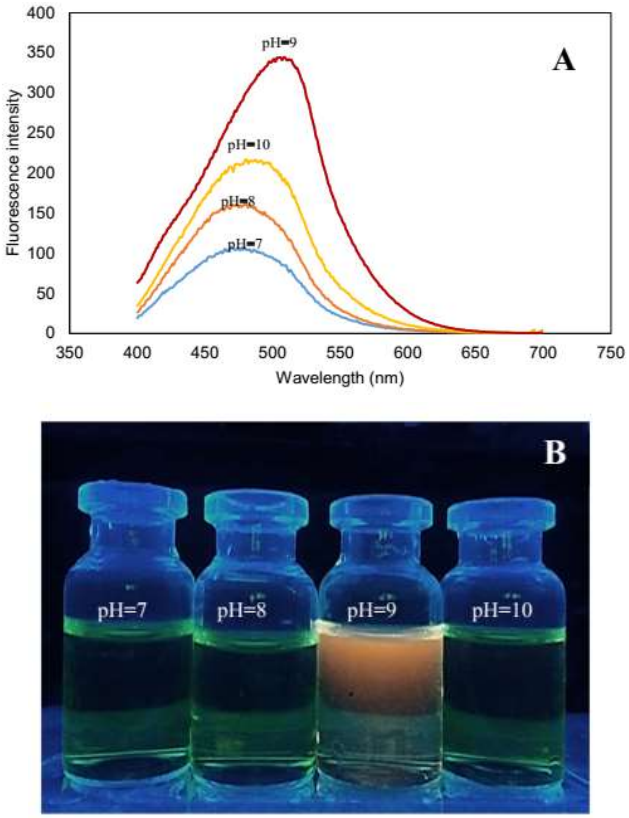

Fig. 6. The fluorescence spectra of MAA-CdS QDs, synthesized at different pHs (A), and the fluorescence intensity of the prepared MAA-CdS QDs at different pHs against UV lamp (B). (Excitation wavelength = 335 nm).

Evaluation of MAA-CdS QDs Shelf Life Stability

After optimizing the synthesis conditions, to investigate the possibility of long-term use of QDs and to stop the gradual and spontaneous deterioration of the cadmium sulfide modified surface with mercaptoacetic acid, MAA-

CdS QDs were transferred to a dark glass container and kept in the fridge for two months. A comparison of the fluorescence emission intensity of these nanoparticles after the elapsed time with the freshly prepared solution indicated the stability of the prepared nanoparticle solution without aggregation or discoloration.

Evaluation of the Fluorescence Emission Stability of MAA-CdS QDs

To investigate the stability of the QDs, the fluorescence emission of these nanoparticles was recorded every 5 min for 35 min. According to Fig. S1, after 35 min, the fluorescence intensities of the QDs were constant, indicating the high stability of these nanoparticles.

Identification and Determination of Cetirizine by MAA-CdS QDs

In this section, MAA-CdS QDs were used as high-selectivity fluorometric sensors for the measurement of CTZ. By examining the changes in the fluorescence intensity, it was found that the interaction between the QDs and CTZ leads to a decrease in the fluorescence emission intensity. Therefore, the proposed system can be considered a new method for the sensitive and selective fluorometry measurement of CTZ in an aqueous solution. Fig. S2 shows the fluorescence spectra of MAA-CdS QDs before and after adding CTZ.

The effect of pH on the Interaction of Cetirizine with MAA-CdS QDs

To obtain the optimized measurement condition, the impact of different pH levels on the identification and measurement of CTZ was investigated by adjusting the pH of the solution containing QDs at a pH range of 3-10 using 0.1 M phosphate buffer. The optimal pH=5 was selected due to the maximum interaction between MMA-CdS QDs and CTZ in this pH (Fig. S3).

The Influence of Interaction Time of Cetirizine with MAA-CdS QDs

The effect of time on the measurement of CTZ by MAA-CdS QDs for determining the optimal incubation time was investigated. The results showed that the highest interaction of cetirizine with the QDs occurs after 5 min. After 5 min, the fluorescence intensity was stable for about half an hour (Fig. S4). Therefore, to obtain repeatable and acceptable results in further research, a minimum of 5 min was taken to measure the fluorescence after drug interaction.

The Selectivity of the Sensor

The effect of different drugs, amino acids, and ions was studied to show the selectivity of the MAA-CdS QDs (Fig. S5). The results showed that this nanosensor was sensitive to CTZ, while other drugs, amino acids, and ions have little effect on the fluorescence intensity of QDs. The MAA-CdS QDs can determine CTZ as a highly sensitive and selective nanoprobe.

Comparing the Proposed Method with other Methods

The performances of different methods for the determination of CTZ are reported in Table 1. According to Table 1, the proposed method has a lower detection limit response compared with other methods. The presented method is very selective, simple, and inexpensive. The detection limit was calculated at 6.48×10^{-11} M based on the 3Sb/m criterion.

Effects of Capping Agents on the Selectivity of MAA-CdS QDs

To examine the effect of surface functionalize groups on fluorescence quenching of MAA-CdS QDs, CdS QDs capped with cysteine, citrate, and mercaptoethanol were synthesized with the same procedure that the MAA-CdS QDs were synthesized. When the same concentration of CTZ was added to the four synthesized QDs, the fluorescence intensity of citrate-CdS, cysteine-CdS, and mercaptoethanol-CdS QDs did not change remarkably at the excitation wavelength of 335 nm, but the fluorescence intensity of MAA-CdS QDs quenched by CTZ at the same excitation wavelength, significantly (Fig. S6).

Analytical Performance

Figure 7a shows fluorescence quenching of the MAA-CdS QDs when different concentrations of CTZ were added. A linear graph of analytical signal changes $(I_0\text{-}I)/I_0$ against the concentration of CTZ has been shown in Fig. 7b.

Table 1. Comparisons of Various Methods for Detection of CTZ

Methods	Linear range (M)	Detection limits (M)	Ref.
Reversed-phase high-pressure liquid chromatography (RP-HPLC)	7.7×10^{-5}-1.8×10^{-4}	7.4×10^{-8}	[40]
High-pressure liquid chromatography (HPLC)	5.1×10^{-4}-2×10^{-3}	2.5×10^{-7}	[41]
Reversed-phase high-pressure liquid chromatography (RP-HPLC)	3.2×10^{-6}-2.5×10^{-5}	2.5×10^{-7}	[42]
Square wave voltammetric (SWV)	1×10^{-6}-2×10^{-5}	1.8×10^{-8}	[43]
Square wave voltammetric (SWV)	5×10^{-7}-1×10^{-5}	1.6×10^{-7}	[44]
Differential pulse voltammetry	6.7×10^{-8}-5.4×10^{-7}	1.6×10^{-8}	[45]
Capillary zone electrophoresis (CZE)	5.1×10^{-6}-1.2×10^{-4}	1.5×10^{-6}	[36]
Differential pulse adsorptive stripping voltammetry (DPAdSV)	4×10^{-8}-4.8×10^{-7}	8×10^{-9}	[46]
Cyclic voltammetric (CV)	5×10^{-7}-1×10^{-5}	7.07×10^{-8}	[47]
Cyclic voltammetric (CV)	1.9×10^{-7}-1.9×10^{-4}	5.8×10^{-8}	[48]
Cyclic voltammetric (CV)	2×10^{-5}-1×10^{-4}	4.3×10^{-6}	[49]
Spectrophotometric	5.1×10^{-6}-5.1×10^{-5}	2.8×10^{-7}	[50]
This method (Fluorescence)	1.6×10^{-10}-1.16×10^{-9}	6.48×10^{-11}	

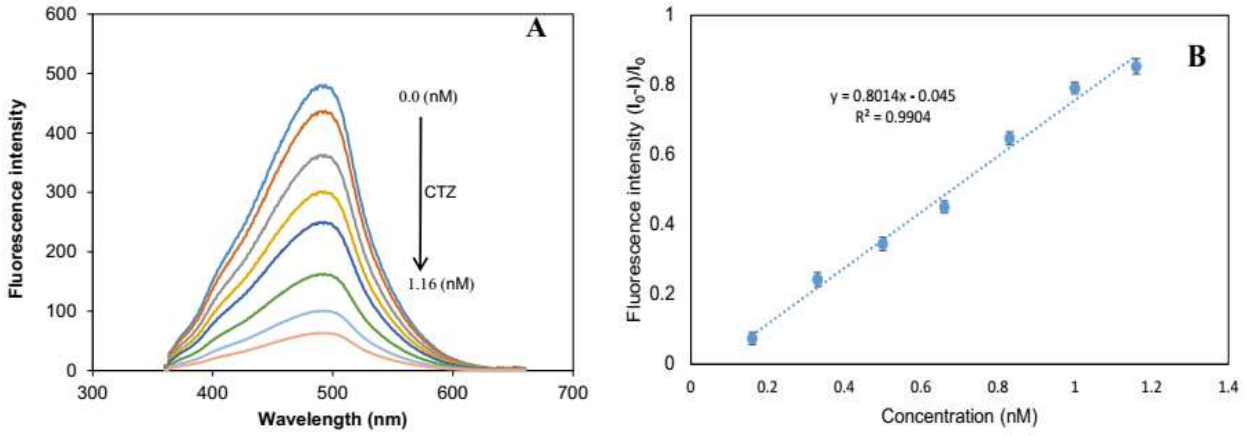

Fig. 7. Fluorescence emission spectra of MAA-CdS QDs in the presence of different concentrations of Cetirizine (a). The fluorescence signal changes of MAA-CdS QDs versus Cetirizine concentration (b). The limit of detection was estimated to be 6.48×10^{-11} M for cetirizine. (Excitation wavelength = 335 nm).

The plot showed a linear response to CTZ over the ranges from 1.6×10^{-10} to 1.16×10^{-9} M. Moreover, the LOD was estimated to be 6.48×10^{-11} M for CTZ. The related linear equation was $(I_0\text{-}I)/I_0 = 0.8014\ C - 0.045$, $R^2 = 0.9904$, where I_0 and I refer to the fluorescence intensity of MAA-CdS QDs in the absence and presence of CTZ, respectively. C is the concentration of the CTZ. The precision of the sensor was investigated by calculating inter-day and intraday relative standard deviation (RSD). The intraday (five repetitive determinations of 0.6 nM CTZ) and inter-day (the same samples in 5 days) RSD was calculated to be 2.7%, and 3.1%, respectively.

Fluorescence Sensing Mechanism

To study the possible mechanism for the fluorescence quenching by CTZ, the fluorescence emission spectra of QDs

Table 2. Determination of Cetirizine in Real Samples

Sample	Added (nM)	Found (nM) Mean ± SD (n = 3)	Recoveries (%)	RSD (n = 3)
Urine 1	0.0	ND[a]	-	-
	0.5	0.49 ± 0.17	98.0	3.5
	0.9	0.89 ± 0.23	98.8	2.6
Urine 2	0.0	ND[a]	-	-
	0.5	0.51 ± 0.11	102.0	2.3
	0.9	0.87 ± 0.27	96.6	3.2
Urine 3	0.0	ND[a]	-	-
	0.5	0.48 ± 0.12	96.0	2.5
	0.9	0.91 ± 0.28	101.1	3.1

Samples 1, 2, 3, and 4 are from the volunteer's human urine. [a]Not detected.

and the absorption spectra of CTZ were recorded. The fluorescence resonance energy transfer (FRET) mechanism is impossible because, according to Fig. S7a, there is no overlap between the fluorescence emission spectrum of QDs and the absorption band of CTZ. Conversely, since the UV-Vis spectrum of the CTZ is close to the absorption spectrum of the QDs, electron transfer from the conduction band of QDs to the lowest orbital of CTZ occurs (Fig. S7b). As a result, the mechanism of fluorescence quenching by CTZ is due to the electron transfer type.

Analysis of Real Samples

The validity and accuracy of the method were confirmed by analyzing the real samples from three healthy volunteers. A known amount of standard CTZ was added to the urine samples, and the samples were diluted with water and filtered. Table 2 shows the results. The recoveries ranged from 96% to 102%, with RSDs of 2.3 and 3.5%, which offers the reliability of the proposed method in this paper.

CONCLUSIONS

Water-soluble MAA-CdS QDs were successfully synthesized by a simple process, under optimized synthesis conditions. The synthesis solution pH and the concentration of Na_2S were optimized for synthesizing high fluorescence MAA-CdS QDs. The MAA was used as a modifier and stabilizer agent to improve the stability and selectivity of the QDs. The fluorescence intensity of MAA-CdS QDs was quenched after adding CTZ. This method exhibited an excellent sensitivity to CTZ compared to other drugs. In conclusion, this article proposed a photoluminescent nanoprobe that can provide excellent selectivity and sensitivity for fast and straightforward detection of trace CTZ in real samples.

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