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Sensitive measurement of silver ions in environmental water samples integrating magnetic ion-imprinted solid phase extraction and carbon dot fluorescent sensor

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ABSTRACT

Increasing use of silver in various fields has caused Ag^+ pollution in water environment, taking great threats to people's health. As a consequence, establishing rapid and reliable methods for sensitive determination of Ag^+ is of great significance. Fluorescent (FL) sensors based on carbon dots (CDs), an excellent carbonaceous nanomaterial with strong and stable fluorescence, have absorbed extensive attentions in analysis of pollutants due to its advantages of carbon sources being readily available, low cost, easy operation and fast response. Moreover, ion-imprinting is a better way to increase the selectivity of the proposed method. Present work described an effective method for the sensitive measurement of silver ion in water samples in combination with magnetic ion-imprinted solid phase extraction and CDs based fluorescent sensor, which took full advantages of easy separation and high enrichment of magnetic solid phase extraction, high selectivity of ion-imprinting technology, and sensitivity and rapid response of fluorescent sensor from CDs. Sulfur-doped CDs derived from dithizone and magnetic ion-imprinted nanomaterial were prepared, and characterized with Fourier transform infrared spectroscopy and transmission electron microscope, *etc*. Magnetic Ag^+ imprinted nanomaterial based solid phase extraction was employed for separating and enriching Ag^+ from water samples. The significant parameters were optimized in detail. Under the optimal conditions, the proposed method provided good linearity in the range of 0.01–0.4 µmol/L and low detection limit of 3 nmol/L. The reliability of the proposed method was validated with real water samples, and the results demonstrated that the proposed method was simple, robust, selective and sensitive detection tool for Ag^+ in real water samples.

Silver, one of the metals used earlier by people, is widely employed in various fields including jewelry, photography, electroplating and catalysis [1–5], *etc.* With the development of economy, requirement of silver is daily on the increase. However, the increasing consumption of silver in industrial activities was accompanied by large quantities of silver ion pollution discharging into environmental water [6–8]. Silver ions in water are biologically active and rapidly associated with mammalian, eukaryotic cell membranes, proteins, amino acid residues, free anions and receptors. Then Ag^+-protein complexes can be formed and spread throughout human body, seriously affecting liver and kidney health. Long-term exposure to Ag^+ can lead to the deposition of Ag^+-protein complexes or metabolites in tissues, resulting in irreversible bluish-gray color of skin, eyes and hair [9–12]. In consideration of human health, it is of great significance to establish rapid and efficient methods for determination of Ag^+ in environmental water samples.

There have been many methods toward detection of Ag^+, such as electrochemical analysis [13], inductively coupled plasma-mass spectrometry (ICP-MS) [14], flame atomic absorption spectrometry (FAAS) [15], surface enhanced Raman scattering (SERS) [16] and fluorescence sensing [17,18]. Among them, fluorescence sensing has been more and more widely used due to its advantages of simplicity, easy operation, high sensitivity and rapidity. Correspondingly, various fluorescent materials including organic molecules, metal nanoclusters as well as semiconductor quantum dots (QDs) were designed and fabricated, which were extensively utilized to develop sensors for sensing Ag^+. In recent years, CDs, a newly emerging carbonaceous fluorescent nanomaterial, have attracted many researchers' attention due to its distinct features of excellent water solubility, good biocompatibility, less toxicity and superior photostability. In addition, it can be easily synthesized through burning, electrochemical oxidation, ultrasonication, microwave or

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hydrothermal treatment [19–22]. Since it was firstly introduced in 2004, lots of researches on CDs in various fields have popped up all the time [23–27]. Especially in analytical field, CDs based sensors or probes play significant roles. Chen's group proposed highly photoluminescent nitrogen-doped carbon quantum dots as a green fluorescence probe for determination of myricetin. The probe exhibited superior stability in high-salt conditions, ultra sensitivity and practicability for analysis of myricetin in spiked vine tea sample [28]. Deng's team synthesized aldehyde-functionalized dual-emissive CDs for ratiometric fluorescence detection of bisulfite in food samples. Due to the nucleophilic addition reaction between HSO_3^- and aldehyde groups on CDs, the fluorescence transforms from green to deep-blue [29]. Xu *et al.* summarized recent advances and sensing applications of CDs in detecting organic molecules and metal ions, many excellent sensing applications of CDs have been provided [30].

Though CDs based fluorescence detection is simple and rapid, the sensitivity generally needs to be improved. Especially, pollutants are present mostly at trace level in environment samples, taking great difficulty to direct detection. Appropriate pretreatment technology can effectively extract target analytes from aqueous samples. Recently, many extraction technologies have been explored and established. Among them, magnetic solid phase extraction (MSPE) is a favorable sample-process technology due to its merits such as easy separation, high enrichment and rapidness. In order to improve the selectivity of the developing method, molecularly imprinted or ion-imprinted process is often optioned. Hence magnetic molecularly or ion imprinted polymers (MMIPs or MIIPs) based magnetic solid phase extraction have attracted more attention due to the features of selective recognition and excellent adsorption capacity [31,32]. Moreover, the magnetism of MMIPs or MIIPs endows them with rapid separation from complicated samples with the aid of an external magnet without centrifugation or filtration. Chen *et al.* prepared magnetic molecularly imprinted polymers (Fe_3O_4@MIPs) for extracting and separating chlorogenic acid from the complex matrix. The established method provided good limit of detection (LOD) (0.03 µg/mL) [33]. In Wang's work, Fe_3O_4 nanoparticles are subjected to chemically etching to reduce the overall mass and then employed as carriers to prepare a novel type of lightweight daisy-like magnetic molecularly imprinted polymers (LD-MMIPs) adopting template immobilization strategy [34]. The daisy-like LD-MMIPs exhibited strong magnetic responsiveness, well crystallinity, fast binding kinetics, high adsorption amount, and excellent selectivity for the detection of 17β-estradiol. Yang's team proposed a novel platform of surface molecularly imprinted magnetic metal-organic frameworks (mMOFs@MIPs) based molecularly imprinted electrochemical sensors (MIECS) for the recognition of oxytetracycline (OTC). MIECS combined the merits of MIPs and electrochemical sensing, which not only retained the specific recognition ability of MIPs, but also combined the fast response and excellent sensitivity of electrochemical sensing, improving the speed and accuracy of target detection [35]. Zhou *et al.* successfully synthesized a regenerable ion-imprinted magnetic biocomposite (IIMB) by using *Serratia marcescens* and carboxymethyl chitosan (CMC) as functional carriers for detection of Pb^{2+}. Due to the principle that Pb^{2+} can be specifically and strongly adsorbed on IIMB, a simple method for detection of Pb^{2+} was established by coupling IIMB with flame atomic absorption spectrometry (IIMB-FAAS) [36]. Najafi *et al.* prepared a novel ion imprinted polymer (IIP) based on *N*-(pyridin-2-ylmethyl) ethenamine, and the IIP was coated on Fe_3O_4 nanoparticles for synthesizing the target adsorbent, which was successfully applied for determination of low levels of Hg(II) ions in various fish samples [37].

In this work, hollow Fe_3O_4 were employed as the carriers to prepare MIIPs with large specific surface area. Then the MIIPs were used as the adsorbent for magnetic solid phase extraction of Ag^+ from environment water samples. CDs derived from dithizone were prepared as fluorescence sensing sensor for detecting Ag^+. Fig. 1 demonstrates the enrichment and detection principles with MIIP based on MSPE and fluorescent probe with CDs. Factors affecting the process of MSPE and fluorescence detection were optimized. Under optimal conditions, the proposed method was evaluated with real water samples.

Reagents used and synthetic procedure of hollow Fe_3O_4 and MIIPs can be seen in Supporting information [38,39]. In order to analysis Ag^+ in environmental water samples, dithizone containing sulfur element was chosen as the precursor for synthesizing CDs. The exact steps were as follows: 10 mL deionized water containing 0.25 g dithizone were poured into Teflon-lined autoclave. After heating at 200 °C for 4 h, CDs solution was obtained. Then the CDs were purified with dialysis bag (1000 Da of cut-off molecular weight) for further use as fluorescent sensor.

MIIPs (45 mg) were mixed with 60 mL aqueous samples containing 0.2 µmol/L Ag^+. Then the mixture was blended at 25 °C for 45 min in an oscillator. When reaching adsorption equilibrium, Ag^+ was eluted from MIIPs by 6 mL 0.5 µmol/L sodium diethyldithiocarbamate (DDTC) in 10 min in four elution cycles. In one elution cycle, 1.5 mL eluent and 2.5 min were used. After dried with N_2 gas, 1.2 mL CDs solution (0.4 µg/mL) was added, completely mixed and transferred into quartz cuvette for fluorescence detection. Meanwhile, mixture of MIIPs and water samples without targets were also analyzed with fluorescence detection after MSPE procedure.

Transmission electron microscope (TEM) was employed for observing morphology of CDs and MIIPs. As shown in Figs. 2A and B, particle sizes of the prepared CDs were in the range of 1.2–2.4 nm and highest fluorescence intensity of CDs can be obtained with excitation wavelength of 265 nm. The high resolution transmission electron microscope (HRTEM) image reveals that lattice fringe of the prepared CDs is 0.21 nm, which belongs to (100) plane of graphite (Fig. S1 in Supporting information) [40]. Morphologies of hollow Fe_3O_4 microspheres and MIIPs were shown in Figs. 2C and D respectively. From Fig. 2C, it can be seen that the prepared hollow Fe_3O_4 microspheres were regular spherical. Compared with Fig. 2C, there was a thin layer of polymer covering the surface of hollow Fe_3O_4 microspheres in Fig. 2D, which clearly demonstrated the successful synthesis of MIIPs. As shown in Fig. 2E, C 1s spectra of CDs can be divided into two peaks at 284.1 and 285.4 eV, which contributed to C=C and C–N/C-N. N 1s spectra located at 399.3 eV can be assigned to N–C. The peaks at 532.9 and 531.4 eV in O 1s spectra correspond to C–OH and C=O, respectively. In Sp2 spectra of CDs, four peaks at 161.2, 163.4, 164.3, 167.8 eV appeared, indicating the presence of N-S, S $2p_{3/2}$, S $2p_{1/2}$ and oxidized S. In addition, X-ray diffraction (XRD) analysis of hollow Fe_3O_4 and MMIPs were conducted and the results were shown in Fig. S2 (Supporting information). It can be seen that diffraction peaks of the hollow Fe_3O_4 and MMIPs were consistent with the standard Fe_3O_4 JCPDS No. 19–0629, indicating the successful synthesis of Fe_3O_4 and demonstrating that modification of molecularly imprinted polymer did not change the crystal structure of hollow Fe_3O_4.

Generally, some factors may affect the extraction and detection performance in the method development, which should be paid more attention. In extraction procedure, factors including adsorbent amount, adsorption time, types of eluents, elution time, eluent volume, pH of sample, NaCl amount should be focused. In detection stage, CDs concentration and pH should be investigated.

Adsorbent amount is one of the critical factors affecting the extraction efficiency. A small quantity of adsorbent may lead to poor extraction efficiency due to the fact that there are not enough active adsorption sites for adsorbing metal ions. Excessive adsorbent is not necessary and will result in waste [41]. Thus, the dosage of adsorbent was optimized. It can be seen from Fig. 3A, the recovery

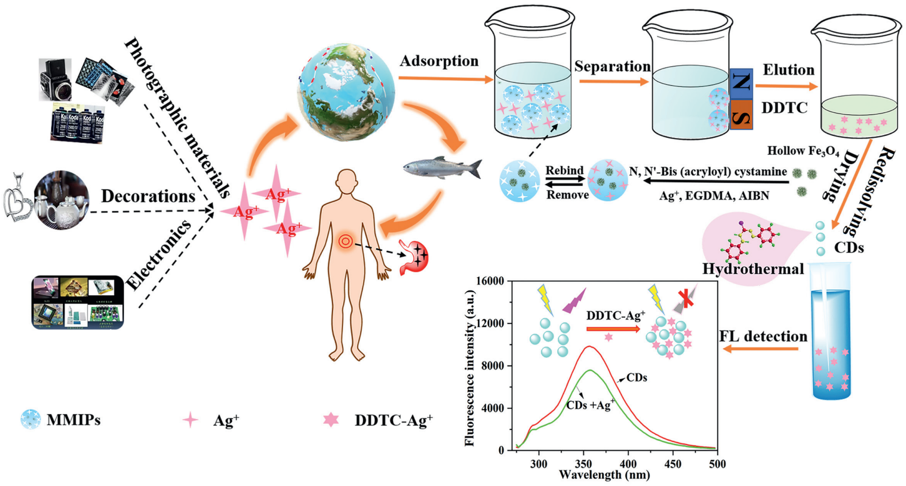

Fig. 1. Schematic diagram of the fluorescent probe from CDs and MIIP based MSPE for detection of Ag^+.

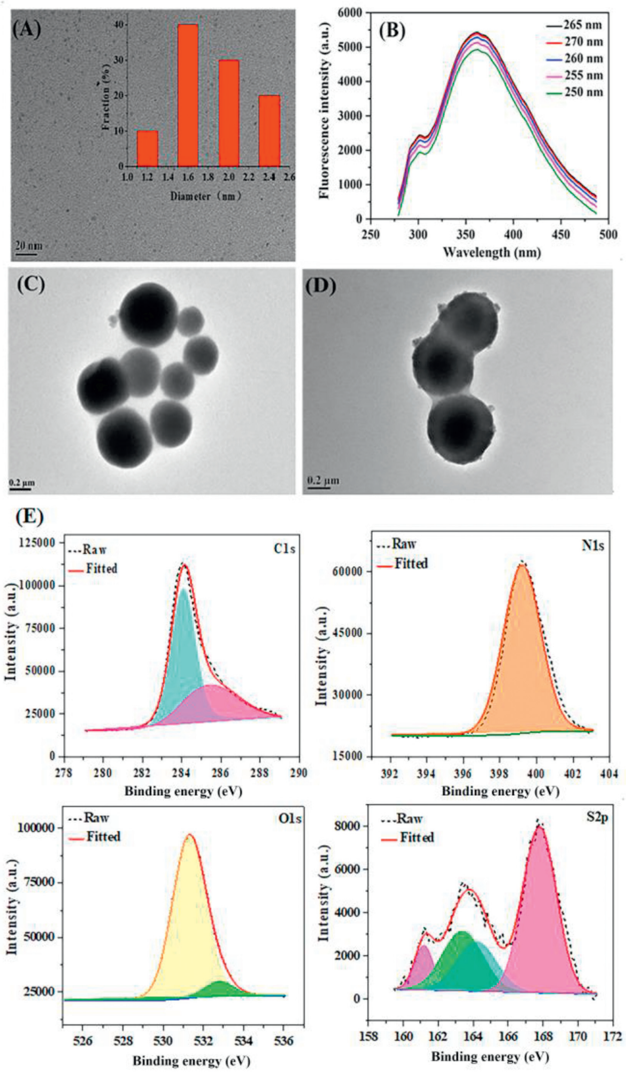

Fig. 2. Characterization of CDs and MIIPs. (A) TEM images and size distribution histogram of CDs. (B) Fluorescent spectra of CDs at various excitation wavelengths. (C) TEM images of hollow Fe_3O_4. (D) TEM images of MIIPs. (E) C 1s, N 1s, O 1s, S 2p X-ray photoelectron spectroscopy (XPS) spectra of CDs.

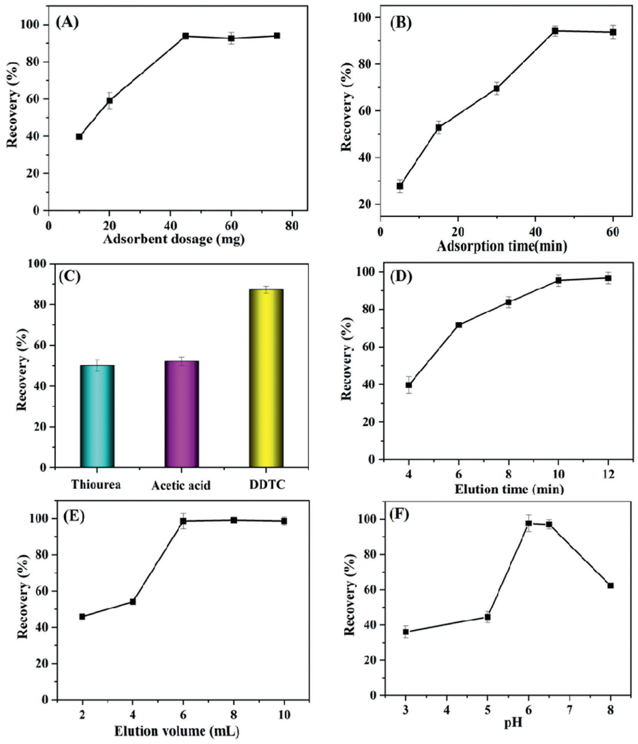

Fig. 3. (A) Influence of adsorbent amount on recovery. (B) Effect of adsorption time on recovery. (C) Effect of different eluents on recovery. (D) Influence of elution time on recovery. (E) Effect of elution volume on recovery. (F) Effect of sample pH on recovery of Ag^+. Experimental conditions: spiked level, 0.2 μmol/L; sample volume, 60 mL; adsorbent dosage, 45 mg; methanol eluent; adsorption time, 45 min; elution volume, 6 mL; elution time, 10 min; pH, not adjusted; When each parameter was optimized, other parameters remain unchanged, and the optimal value was selected for subsequent optimization.

of Ag^+ increased companied with the increase of adsorbent from 10 mg to 45 mg and then kept almost constant, which indicated that 45 mg MIIPs was enough to adsorb Ag^+ in sample solutions. Usually, it needs a certain time for achieving the best extraction performance. In order to save time, effect of adsorption time on recovery was investigated in the range of 5–60 min. Fig. 3B demonstrates that the extraction efficiency of Ag^+ increased with prolonging time, and adsorption equilibrium reached at 45 min. Continuing increase of time resulted in almost unchanged extraction

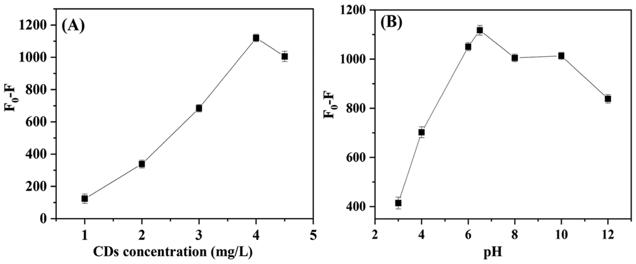

Fig. 4. (A) Effect of CDs concentration on detection of Ag^+. (B) Effect of pH on detection of Ag^+.

efficiency. In the extraction system, eluent is another key parameter for achieving best extraction efficiency. Appropriate eluent cannot only accelerate the elution process but also improve the extraction efficiency. According to reported literatures, there are many reagents such as chelating agents, organic acids and other reagents being used for elution of heavy metals [42]. In this work, thiourea, DDTC–Na and acetic acid were tested for eluting Ag^+. The results were shown in Fig. 3C, which showed that thiourea and acetic acid resulted in similar elution efficiency, and DDTC–Na obtained much higher elution efficiency than thiourea or acetic acid. Hence, DDTC–Na was used in subsequent experiments. In the elution process, Ag^+ was desorbed from MIIPs through several circles of continuous elution. Each circle contained elution with 1.5 mL eluent for 2.5 min. The effects of elution time and eluent volume were exhibited in Figs. 3D and E. It was apparent that the recovery of Ag^+ increased with the increase of eluent volume and elution time. When eluent volume and elution time reached 6 mL and 10 min, the elution efficiency of Ag^+ was highest and then kept almost constant. Hence eluent volume and elution time were set at 6 mL and 10 min, respectively. In MSPE procedure, sample pH always takes great effect on extraction recovery. Especially, MIIPs materials may be destroyed and which would bring negative effect on the extraction under acidic condition. As shown in Fig. 3F, when pH value was lower than pH 5, the extraction efficiency was very poor. This may be contributed to the acid intolerance of magnetic materials. In addition, protonation of the amino groups and sulfur containing groups of MIIPs also occurred under strong acidic condition, which resist the Ag^+ interaction with binding sites [41,43]. When the solution pH changed from 5 to 6, the recovery increased obviously and then kept in high level with pH increase to pH 6.5. When solution pH continuously increased from 6.5 to 8, the recovery conspicuously decreased. This may be explained by the fact that the precipitation of Ag^+ as hydroxide occurred at higher pH [44,45]. So, pH 6.5 was optioned in further experiments.

Figs. 4A and B exhibited the effect of CDs concentration and pH value of the solution on detection of Ag^+. It was obviously that F_0-F increased accompanied with the increase of CDs concentration from 1 mg/L to 4 mg/L and then had slightly decrease with the continuous growth of CDs concentration. Then F_0-F presented a certain decline with the continuous increase of CDs concentration. This may be contributed to the self-quenching effect of CDs. Fig. 4B exhibits the effect of pH on detection of Ag^+, and it could be found that the quenching effect decreased significantly in strong acid environment, resulting in poor detection sensitivity. Under weak alkaline environment, quenching effect resulted in slight decrease. According to the results that better detection efficiency can be obtained at pH 5–8, pH 6.5 (pH of pure water) was chosen in further study.

Ag^+ was quantified in this work based on the principle that fluorescence intensity of CDs was quenched in the presence of Ag^+. In order to furtherly explore the sensing mechanism, some related characterizations were conducted. As shown in Fig. 5A, compared

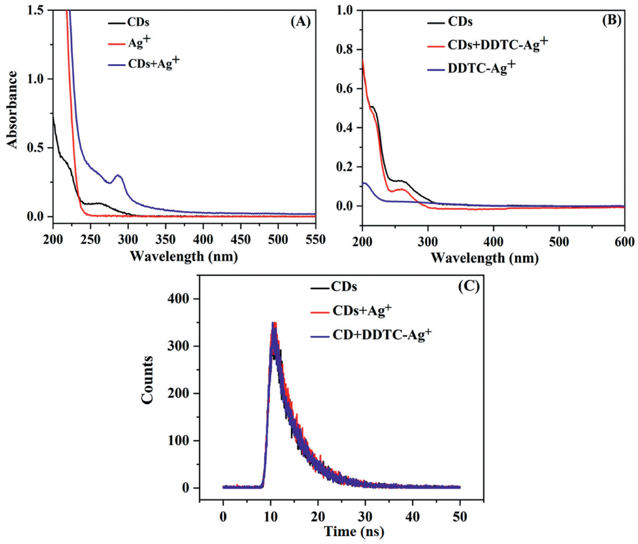

Fig. 5. (A, B) UV–vis spectra of CDs, Ag^+, mixture of CDs + Ag^+, CDs + DDTC-Ag^+. (C) Lifetimes of CDs, mixtures of CDs + Ag^+ and CDs + DDTC-Ag^+.

with ultraviolet–visible (UV–vis) spectra of CDs, there was a new peak appearing in UV–vis spectrum of CDs + Ag^+, indicating that CDs-Ag^+ complex may be formed. However, after the MSPE procedure, DDTC-Ag^+ formed and which was eluted from the MIIPs adsorbents. In the detection process, DDTC-Ag^+ quenched the fluorescence intensity of CDs.

The sensing mechanism of DDTC-Ag^+ was explored. According to Fig. 5B, addition of DDTC-Ag^+ weakened the absorption of CDs and new peak at 287 nm in UV–vis spectra of CDs-Ag^+ disappeared, which indicated that a ternary system of CDs-DDTC-Ag^+ may be formed and resulted in the quenching of CDs. As shown in Fig. 5C, life time of CDs (4.69 ns) was almost kept unchanged when Ag^+ (4.69 ns) or DDTC-Ag^+ (4.72 ns) were added. All the results indicated that the sensing mechanism may be contributed to static quenching [46].

Sensitivity is an important index to evaluate the performance of analytical methods. As shown in Fig. 6A, fluorescence intensity of the proposed CDs was significantly quenched in the presence of DDTC-Ag^+ with Ag^+ concentration of 0–400 nmol/L, and good linearity with correlation coefficient of $R^2 = 0.997$ was obtained (Fig. 6B). Limit of detection (LOD) toward Ag^+ was calculated based on the algorithm formula of 3σ/slope (σ refers to the standard deviation of blank signals (n=15)). According to the results of 15 times repeated experiments for magnetic solid phase extraction and detection of blank samples, LOD of Ag^+ was 3 nmol/L. Compared with other reported literatures on CDs based fluorescence sensor for detection of Ag^+ (Table S1 in Supporting information), LOD of this work was much lower. This can be contributed to the utilization of MIIP based MSPE before CDs based FL detection. Cayuela's group proposed CDs hydrogels-based FL sensor for analysis of Ag^+ with LODs of 4.6 µmol/L [47]. Liao's team established S, N-doped CDs as FL sensor for detection of Ag^+ with LOD of 0.4 µmol/L [48]. Zhao and coworkers synthesized graphene quantum dots for determination of Ag^+, and the obtained LOD was 0.25 µmol/L [49]. Xie's crew proposed TGA-CdTe QD as sensor for FL detection of Ag^+ with LOD of 25.3 nmol/L [50]. Additionally, Fig. 6C compares the results of MIIPs and MNIPs for adsorption of Ag^+. MNIPs exhibited inferior performance toward Ag^+. In order to evaluate the selectivity of the proposed method, other heavy metal ions with five-time higher concentrations were added in the system of CDs-Ag^+ mixture. As shown in Fig. 6D, the effect of other heavy metal ions on the re-

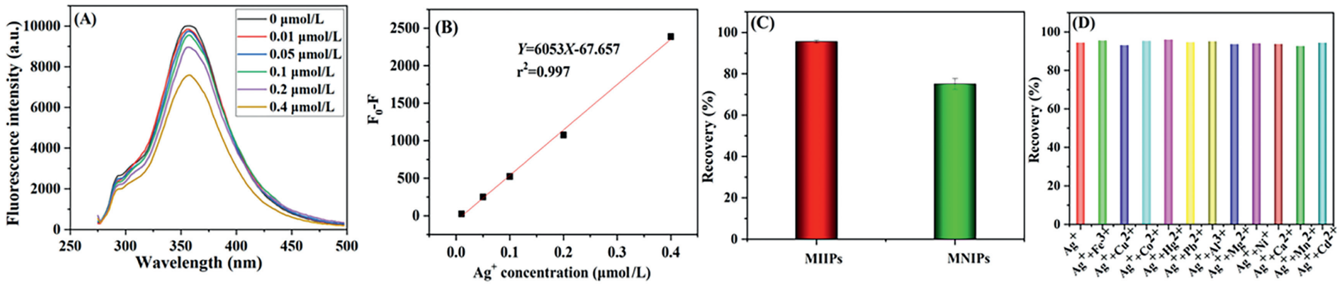

Fig. 6. (A) Fluorescence spectra of CDs in the presence of Ag^+ with different concentrations. (B) Linear plots of the fluorescent probe. (C) Comparison of MIIPs and MNIPs toward Ag^+. (D) Selectivity of MIIPs (n=3, RSDs < 2.7%).

covery of Ag^+ was slightly, indicating the good selectivity of this proposed method.

Feasibility in practical applications is a key factor for evaluating reliability of the proposed method. In order to realize the evaluation, three real water samples were analyzed. Reclaimed water was collected from the campus of China University of Petroleum (Beijing). The other two water samples were collected from Qikongqiao flower sea and Binhe Park in Changping (Beijing). Before analysis, all the water samples were filtered through 0.22 μm filter membrane. Table S2 (Supporting information) exhibited the analytical results of real water samples. It can be seen that satisfactory recoveries in the range of 94.0%–96.0% and lower relative standard deviations (RSDs) of 1.9%–3.9% were achieved at different spiked levels. All the results indicated that the proposed CDs based fluorescence sensor combined with MIIPs based MSPE for determination of Ag^+ in real water samples was reliable and valuable.

In conclusion, CDs containing sulfur element was successfully synthesized with dithizone as the carbon precursor for fluorescent sensing of Ag^+. Ag^+ MIIPs exhibited high selectivity and enrichment performance for Ag^+ from water samples. The combination of CDs based fluorescent sensing and MIIPs based MSPE greatly improved the sensitivity and selectivity of the established method. This may be contributed to the integration of double excellent selectivity of CDs derived sensor and MIIPs, and high enrichment capacity of MIIPs. The introduce of MSPE lead to the rapid separation and enrichment of Ag^+ from water samples and lower LOD of 3 nmol/L due to the good adsorption performance of MIIPs. The practicality was validated with real water samples, and good recoveries in the range of 94.0%–96.0% were obtained. These results demonstrated that the established method earned a promising application in detecting Ag^+ and provided new approaches for establishing methods toward other pollutants in environmental waters.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2023.108693.

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