

Detection of silver(I) ion based on mixed surfactant-adsorbed CdS quantum dots

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Abstract Mixed cationic and anionic surfactants were adsorbed on cadmium sulfide quantum dots (CdS QDs) capped with mercaptoacetic acid. The CdS QDs can be extracted into acetonitrile with 98 % efficiency in a single step. Phase separation only occurs at a molar ratio of 1:1.5 between cationic and anionic surfactants. The surfactant-adsorbed QDs in acetonitrile solution display stronger and more stable photoluminescence than in water solution. The method was applied for determination of silver(I) ion based on its luminescence enhancement of the QDs. Under the optimum conditions, the relative fluorescence intensity is linearly proportional to the concentration of silver(I) ion in the range between 50 pmol L⁻¹ and 4 μmol L⁻¹, with a 20 pmol L⁻¹ detection limit. The relative standard deviation was 1.93 % for 9 replicate measurements of a 0.2 μmol L⁻¹ solution of Ag(I).

Keywords Cationic-anionic surfactant · Cadmium sulfide quantum dots · Silver

Introduction

There has been an increasing interest in quantum dots (QDs) in view of their optoelectronic properties that leads to extensive applications in optical devices and sensors for metal ions via the change of fluorescence induced by metal ions [1–6]. To date, various strategies have been developed for QDs synthesis in aqueous media; however, the major problem of the synthesis is the non-radiative decay channels on their

surface leading to a severe degradation in optoelectronic properties. Applying suitable protective coverings during their synthesis or addition of some organic additive such as thiol, carboxylic, amino, polymer groups can serve not only tuning the optoelectronic properties but also acting as stabilizing agent to prevent the interferences of QDs [1–6].

Surfactants have been widely used as the extractant because of their friendly-environment properties [7–9]. Moreover, surfactants are also used as stabilizing agents for QDs which provided the significant change of the photoluminescence (PL) properties of them [10–12]. There are literatures reveal that the size, structure, and stability of QDs can be controlled by the choice of suitable surfactants [10–12].

Silver (Ag) has been widely used as silver nanoparticle (AgNPs) in commercial products due to their antimicrobial and catalytic properties [13]. The importance of Ag determination is related to its toxicity. In humans and animals, Ag is absorbed by the gastrointestinal tract, skin, mucous and membranes, and chronic exposure to Ag is known to cause argyria and/or argyrosis [14]. A sensitive and simple technique is still required for Ag determination because it exists in environment at trace level. Recently, there are a number of reports using QDs for Ag detection [1–6]. For example, zinc sulfide quantum dots (ZnS QDs) doped with Mn(II) ions and modified surface with iminodiacetic acid (QDs-IDA) was successfully synthesized and applied for silver(I) ion (Ag⁺) detections [3]. QDs-IDA was selectively quenched by Ag⁺ in phosphate buffer solution (pH 7.3) and provided the detection limit of 0.26 μmol L⁻¹. The possible mechanism was the complex formation between the iminodiacetic acid of QDs-IDA and Ag⁺ which promoted photoinduced electron transfer. Mandal et al. [6] reported a simple and green synthetic method for thiolactac (TLA) capped ZnS QDs and used as the probe for Ag⁺ detection in the range of 0.5–1.0 μmol L⁻¹. Although there have been a number of publications that presented a sensitive QDs probes for Ag⁺, the applications

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of QDs for determination of Ag^+ in real sample matrices have not been reported in analytical area yet.

Since surfactants have the suitable properties for protection and improve the optical property of QDs and also the ability of them for extraction of trace analytes from complex matrices, the simple route to improve quality of CdS QDs using surfactant is possible. Good alternative way to improve the sensitivity and selectivity for Ag^+ determination by CdS QDs in real samples was also expected.

This research focuses on the analytical method using CdS QDs as a fluorescence probe for the determination of Ag^+ . Mixed cationic surfactant (tetradecyltrimethylammonium bromide; TTAB) and anionic surfactant (sodiumdodecyl sulfate; SDS) were used as the extractant of CdS QDs for its application on the determination of Ag^+ . Besides the role of extractant, mixed TTAB and SDS were also acted as co-stabilizing agents to enhance the fluorescence intensity of CdS QDs, thus increased the sensitivity for Ag^+ detection.

Materials and methods

Chemicals and reagents

All reagents were of analytical-reagent grade and used without further purification. All aqueous solutions were prepared with deionized water with a resistivity of 18.2 M Ω cm (Millipore, France, www.millipore.com). Cadmium chloride ($\text{CdCl}_2 \cdot \text{H}_2\text{O}$) was purchased from Riedel-de Haën (Germany, www.sigmaaldrich.com). Mercaptoacetic acid (MAA) was obtained from Sigma Aldrich (USA, www.sigmaaldrich.com). Sodium sulfide ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$) and sodiumdodecyl sulfate (SDS) were obtained from BDH (England, www.bdhme.com). Salts of the different cations and anions were obtained from Carlo Erbar (France, www.carloerbareagents.com). Acetonitrile (ACN) and methanol (MeOH) were purchased from Lab-Scan (Thailand, www.rcilabscan.com). Tetradecyltrimethylammonium bromide (TTAB) was purchased from Fluka (USA, www.sigmaaldrich.com). Standard solutions of all surfactants, cationic and anionic ions were prepared daily by appropriate dilution of the stock solution with water.

Instrumentation

A Shimadzu RF-5301pc spectrofluorometer (Shimadzu, Japan, www.shimadzu.com) was used for recording the fluorescence spectra. All fluorescence spectra were obtained at room temperature using a xenon lamp as the excitation source equipped with a 1 cm quartz cell. Agilent 8453 UV-visible spectrophotometer (Agilent, USA, www.agilent.com) equipped with a 1 cm quartz cell was used for recording the

absorption spectra. FTIR/FT Raman spectrophotometer (Perkin-Elmer, USA, www.perkinelmer.com) and Tecnai G2 20 transmission electron microscope (FEI, USA, www.fei.com) were used to study the interaction of CdS QDs and surfactants.

Procedure

Synthesis of mercaptoacetic acid capped cadmium sulfide quantum dots

Water-soluble CdS QDs capped by MAA were synthesized following a method reported by Winter et al. [15] with some modifications (pH and stirring time). $\text{CdCl}_2 \cdot \text{H}_2\text{O}$ (3.7751 g, 18.75 mmol) was dissolved in 200 mL of water. Then, MAA 2.60 mL (37.50 mmol) was added into the solution under stirring and the solution pH was adjusted to 8.0 with 1 mol L^{-1} NaOH. After that, quickly added 10.00 mL of 1.4632 g $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ (18.75 mmol) aqueous solution to the solution with vigorous stirring and the solution gradually turned yellow-green color. The reaction mixture was stirred at 65 °C for 60 min and then aging at room temperature in air for 60 min without stirring.

Extraction of cadmium sulfide quantum dots by the mixed cationic and anionic surfactants

In the first step, 20.00 μL of CdS QDs solution (5.744 g L^{-1}) was added into 10.00 mL of water in a measuring cylinder with cap. Next, 200 μL of 2 % (w/v) TTAB was added into the aqueous solutions and vertical shook for 10 times. Then, 200 μL of 1 % (w/v) SDS was added into the mixture solution and shook for 60 s. After leaving at ambient temperature for 2 min, the surfactant rich phase (SRP) was

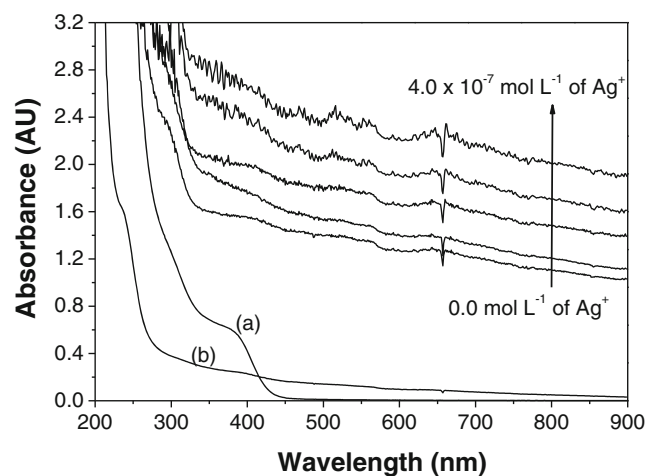
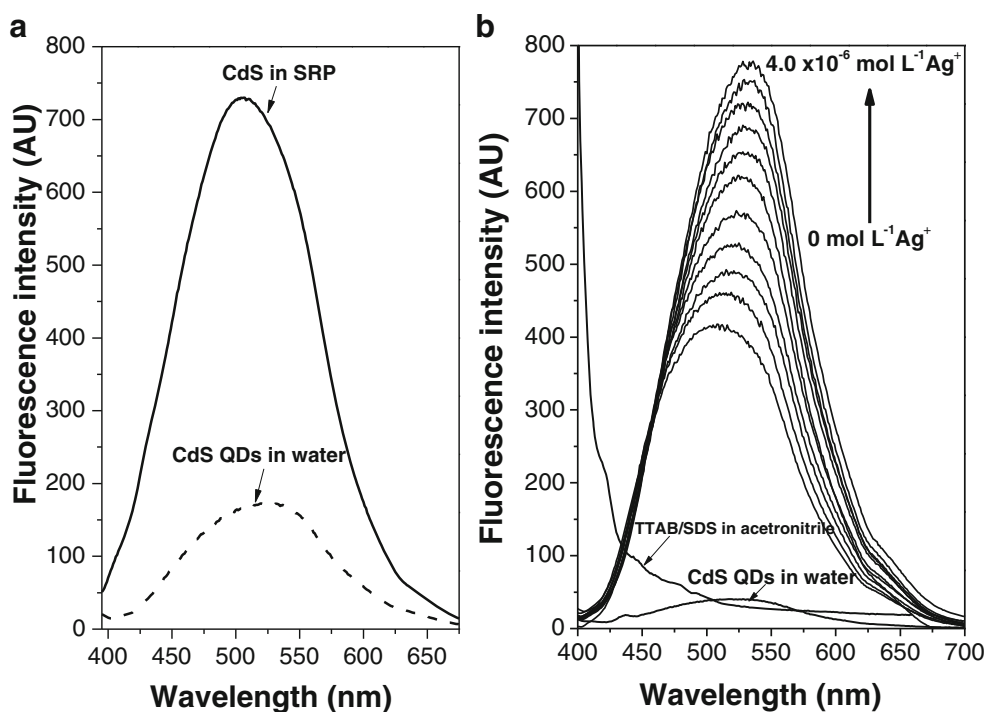


Fig. 1 Absorption spectra of CdS QDs (60 $\mu\text{g mL}^{-1}$) in the presence and absence of silver (I) ion after extraction by TTAB/SDS. The concentrations of Ag^+ were 0.0 to 4.0×10^{-7} mol L^{-1} **a** CdS QDs in water **b** TTAB/SDS in 2.0 mL of ACN (blank after extraction)

Fig. 2 Emission spectra of the CdS QDs **a** Fluorescence signal obtained from the determination of 30 μL of CdS QDs (5.744 g L^{-1}) after extraction by TTAB/SDS in 2.0 mL of ACN and CdS QDs at the same concentration in 10.0 mL aqueous solution **b** Fluorescence emission spectra of CdS QDs in the absence and presence of different silver (I) concentration; $\lambda_{\text{em}}=505 \text{ nm}$ ($\lambda_{\text{ex}}=370 \text{ nm}$) $0.116 \text{ mmol L}^{-1}$ CdS QDs



completely separated from aqueous phase which the floated yellow colloidal suspension was observed at the top of the solution. The aqueous solution was gently poured out into another container. SRP was then diluted with 2.0 mL ACN and finally measured the fluorescence (λ_{ex} ; 370 nm and λ_{em} ; 505 nm).

For Ag^+ determination, an aliquot of the Ag^+ standard solution was added into 10.00 mL water or sample before adding of CdS QDs into solution. The experimental procedure was performed in the same manner as mentioned above.

Results and discussion

Characterization of mecaptoacetic acid capped cadmium sulfide quantum dots

The UV–visible absorption and fluorescence spectra of CdS QDs in water and CdS QDs/TTAB/SDS in ACN are shown in Figs. 1 and 2, respectively. The absorption spectra of CdS QDs after extraction by TTAB and SDS (TTAB/SDS) was different from CdS QDs in water (Fig. 1a) and TTAB/SDS blank solution (Fig. 1b), confirming that the environment of CdS QDs particle was changed by TTAB/SDS molecules. The fluorescence spectrum of CdS QDs exhibits the maximum fluorescence intensity at 505 nm (Fig. 2a). As seen from Fig. 1(a), the absorption on set of CdS QDs located at $\approx 450 \text{ nm}$ which the particle size of the prepared CdS QDs is calculated to be about 2.69 nm [2], corresponding to TEM image (data not shown).

Effect of surfactants concentration

According to our preliminary study, it was found that the addition of TTAB at various concentrations could not extract CdS QDs (data not shown) whereas the phase separation was obtained when using mixed TTAB and SDS. Therefore, mixed TTAB and SDS were selected for the further experiments because they provided phase separation at room temperature.

From the results shown in Fig. S1 (Electronic Supplementary Material, ESM), concentration of surfactants has strongly influence on the extraction of CdS QDs. With increasing of surfactants amount, the fluorescence signal in SRP was increased which accompany to the decreasing of signal in

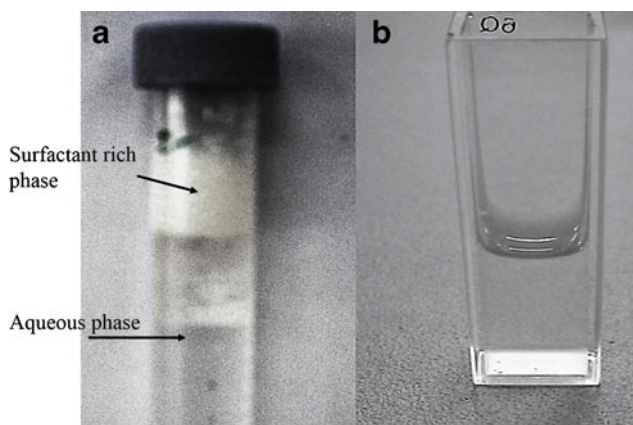


Fig. 3 **a** Photo of floatation phenomenon after the addition of TTAB/SDS under the optimum condition **b** the floated phase dilute in 2.0 mL of ACN

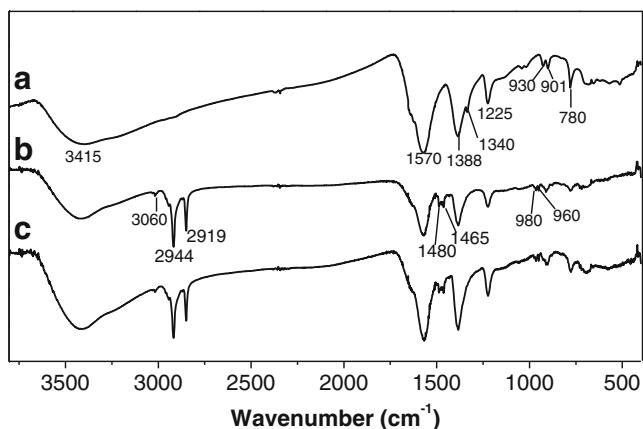


Fig. 4 IR spectra of CdS QDs **a** in aqueous solution **b** after extraction by TTAB/SDS **c** in the presence of silver (I) after extraction by TTAB/SDS

aqueous phase. It was found that $0.475 \mu\text{mol L}^{-1}$ TTAB and $0.690 \mu\text{mol L}^{-1}$ of SDS provided the highest yield of CdS QDs extraction. At this molar ratio for TTAB : SDS (1:1.5), the percentage extraction of CdS QDs was 98 %. However, the extraction yield decreased (data not shown) when the concentration of TTAB and SDS beyond this point. This phenomenon can be explained by the rearrangement of surfactant molecules (i.e. interparticle electrostatic double layer) at its high concentration on surface of CdS QDs which affected to the total charge density around CdS QDs and surfactant particles by changing their forms [7, 16].

Effect of organic solvent type and concentration

ACN and MeOH were investigated as the solvent to dissolve the obtained foam-like SRP (Fig. 3). ACN is superior to

MeOH, since ACN provided higher fluorescence intensity; therefore, the concentration of ACN in the range of 0.5–3.0 mL was further studied (data not shown). The fluorescence intensity of CdS QDs/TTAB/SDS was highest at 2.0 mL ACN, after that it was dramatically decreased. To compromise between the dilution of surfactants which resulted in the changing of their forms [16] and the fluorescence signal, ACN 2.0 mL was chosen.

Effect of pH buffer solution and salt additive

The effect of pH in the range of 4.6–7.0 (using 50 mM of acetate and Tris-HCl buffer solution) on the extraction of CdS QDs by TTAB/SDS was studied (data not shown). It was found that the addition of buffer solution resulted in decreasing of the SRP fraction. Therefore, the effect of salt additive was also studied. It was found that the addition of NaCl, Na_2SO_4 , Na_2CO_3 , K_2HPO_4 and $\text{KNO}_3 \geq 0.1 \%$ (w/v) decreased the extraction efficiency which it was obviously observed that the separation of two phases was not completed (data not shown). The mechanism of the phase separation in this system can be explained in terms of the chemical potential of electrolyte and counter ions around CdS QDs and surfactants. The interface between these two coexisting phases acts as a semipermeable membrane [17]. In CdS QDs-surfactants phase, two types of ions are present: the charged electrolyte molecules and the accompanying counterions. The space between the charged micelles is filled with electrolyte and the counterions which released by the surfactant molecules. Therefore, the exchange of counterions between SRP and the electrolyte media can be occur [18]. Since cationic or anionic ions from salts were added into this media,

Fig. 5 Plot of I/I_0 value against silver(I) ion concentration at low concentration (**a**) and the fluorescence intensity of the CdS QDs in the presence of silver(I) ion after extraction by TTAB/SDS that correspond to the calibration curve (**b**)

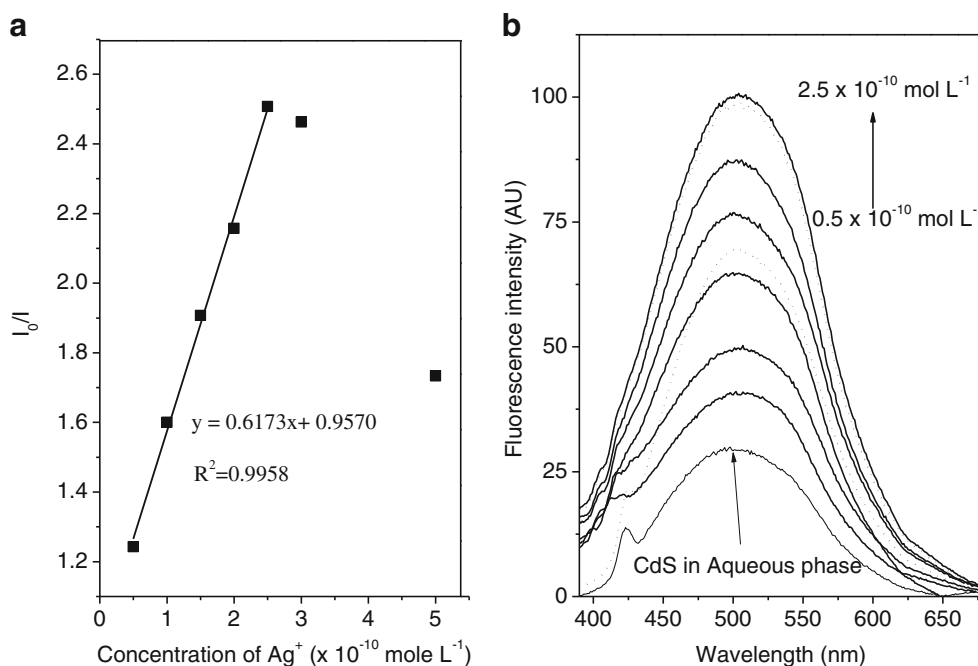


Table 1 Calibration curve parameters of silver(I) ion determination using CdS QDs in different media

Silver(I) ion determination condition	Slope	Linear regression	Linear range ($\mu\text{mol L}^{-1}$)
CdS + Ag in Aqueous solution	0.161	0.999	0.25–2.50
CdS + Ag in 2 % (w/v) TTAB solution	3.032	0.998	0.25–1.00
CdS + Ag in 1 % (w/v) SDS solution	0.07	0.996	0.25–1.50
CdS + Ag (SRP) in ACN	0.154	0.997	0.02–4.00

the equilibrium of the exchange ions between counterions at surfactants around CdS QDs surface and electrolyte phase was interrupted as mentioned in previous work [19]. From these results, the further experiments of the CdS QDs extraction by TTAB/SDS were then performed under non-ionic condition without buffer solutions and salt additives.

Phase separation mechanism

According to UV-visible absorption, fluorescence and TEM results, the morphology, particle size and optical properties of QDs were significantly changed which can be concluded that TTAB/SDS were adsorbed on CdS QDs surface. Moreover, IR spectrum of the QDs after the extraction by TTAB/SDS (Fig. 4b) shows new peaks different from IR spectrum obtained from CdS QDs (Fig. 4a) which related to the surfactants at 3,060, 2,944, 2,919, 1,480, 1,465 980 and 960 cm^{-1} [20]. This observation may due to the interactions between oppositely charged surfactants (TTAB and SDS) such as the electrostatic interactions reinforced by hydrophobic interactions, ion-pair association and complex coacervate formation [21]. Therefore, the phase separation mechanism in this work can be explained on the basis of binary combination of oppositely charged ionic surfactant at instantaneous micelle formation process. Since the appearance of turbidity is consequence of the formation of insoluble layer on surface of CdS QDs owing to the strong electrostatic interactions between anionic charge ($-\text{COO}^-$) of MAA and cationic polar head group ($-\text{N}^+(\text{CH}_3)_3$) of TTAB [22]. However, the hydrophobicity acquired is not enough for phase separation. According to the quality of synthesized CdS QDs,

the defect sites were always found on the surface of QDs (cation dangling bond); therefore, SDS was added to associate the phase separation by electrostatically adsorbed the defected sites on CdS QDs surface with anionic polar head group ($-\text{SO}_3^-$) and increase in CdS QDs hydrophobicity [10–12].

Moreover, the stability of CdS QDs in surfactants after extraction was also studied (data not shown). The fluorescence intensity of CdS QDs in surfactants was increased up to around 870 and level off on the 30th days, after that gradually decreased to the beginning point on the 55th days. This phenomenon can support the hypothesis that TTAB and SDS were electrostatically adsorbed on CdS QDs surface, resulting in the fluorescence intensity significantly increased.

Detection of silver(I) ion

Surprisingly, the fluorescence intensity of CdS QDs (Fig. 2a) increased markedly by the presence of TTAB/SDS (solid line) around 7 times when compared with the aqueous solution of CdS QDs (dash line). From this result, the effect of various important metal ions (Fe^{3+} , Co^{2+} , Cd^{2+} , Ni^{2+} , Hg^{2+} , Cu^{2+} , Zn^{2+} , Pb^{2+} , Au^{3+} and Ag^+) on the fluorescence intensity of the extracted CdS QDs was investigated. Among these, selective luminescence enhancement with Ag^+ was found to be predominant (Fig. 2b). While, Au^{3+} and other divalent cations which can be electrostatically adsorbed on the surface of CdS QDs [23] quenched the fluorescence intensity of CdS QDs. Therefore, the present method was investigated for Ag^+ detection. Figure 2b shows the fluorescence spectra obtained from the determination of Ag^+ in the range of 0.0–4.0 $\mu\text{mol L}^{-1}$. The standard deviation for 9 replicate measurements of

Table 2 Comparison of silver determination by quantum dots

Quantum dots	Sample	Linear range	Detection limit	Reference
L-cysteine capped CdS	–	20 nmol L^{-1} –1.0 $\mu\text{mol L}^{-1}$	5.0 nmol L^{-1}	[1]
L-cysteine capped CdS	–	0.125–5.0 $\mu\text{mol L}^{-1}$	20 nmol L^{-1}	[2]
Mn(II) doped iminodiacetic acid capped ZnS	–	0.5–4.5 $\mu\text{mol L}^{-1}$	0.26 $\mu\text{mol L}^{-1}$	[3]
Citrate capped CdSe	–	1.7–18 $\mu\text{mol L}^{-1}$	–	[4]
Mercaptoethanol capped PbS	–	100 $\mu\text{mol L}^{-1}$ –3 mmol L^{-1}	sub- $\mu\text{mol L}^{-1}$	[5]
Thiolactic acid capped ZnS	–	0.5–1.0 $\mu\text{mol L}^{-1}$	–	[6]
Mercaptoacetic acid capped CdS extracted by TTAB/SDS	Tap water and film developing solvent wastewater	50 pmol L^{-1} –4.0 $\mu\text{mol L}^{-1}$	20 pmol L^{-1}	This work

Table 3 Analytical results of samples and recovery ($n=3$)

Concentration added ($\mu\text{mol L}^{-1}$)	Concentration found ($\mu\text{mol L}^{-1}$)		Recovery (%)	
	Tap water	Film developing solvent wastewater	Tap water	Film developing solvent wastewater
0.05	0.048	0.045	96 (± 1.0)	89 (± 1.7)
0.10	0.097	0.092	97 (± 1.0)	92 (± 0.6)
2.00	1.950	1.920	98 (± 0.6)	96 (± 1.0)

$0.2 \mu\text{mol L}^{-1} \text{Ag}^+$ was 1.93 % and the detection limit which was calculated following the 3σ IUPAC criteria obtained from calibration curve in Fig. 5 was 20 pmol L^{-1} .

According to the fluorescence spectra (Fig. 2) of the QDs after the extraction by TTAB/SDS, the fluorescence intensity increased by increasing of Ag^+ concentration with a gradual red shifted (Fig. 2b) and then levels off which can be described by a Langmuir-type binding isotherm. Therefore, we speculated that the mechanism to explain the interaction between Ag^+ and CdS QDs in TTAB/SDS are the formation of Ag^+ and MAA complex on surface of QDs which corresponding to the mechanism as mentioned in previous works [1, 2].

The analytical characteristics of the optimized method including regression equation and linear ranges for Ag^+ measurement in different media solution were also investigated (Table 1). The dynamic range of using TTAB/SDS extraction is wider up to $4 \mu\text{mol L}^{-1}$ indicating the enhancement of the detection. The results reveal that the linear range obtained from SRP phase was higher than in aqueous phase and pure SDS or TTAB for 2 orders of magnitude. From the analytical performance when compared with the other works, it can be concluded that the present method is much more selective for Ag^+ than most of current used and recently prescribed QDs methods as shown in Table 2 in term of linear range of detection, limit of detection and also its application in real sample.

Selectivity

To evaluate the selectivity of this method, the influence of some potentially interfering ions on the fluorescence intensity of CdS QDs/TTAB/SDS was investigated using $20 \mu\text{mol L}^{-1}$ of Ag^+ . The tolerance limit was defined as the concentration of the added ion that caused less than 5 % relative error. The results indicate that Au^{3+} was interference at $5 \mu\text{mol L}^{-1}$, this may due to the effective binding of Au^{3+} with CdS QDs [24]. Common anionic and cationic ions (Na^+ , K^+ , Cl^- and Br^-) interfere when its concentration is higher than 100 mmol L^{-1} . While, CO_3^{2-} , NO_3^- , PO_4^{3-} , SO_4^{2-} and CH_3COO^- significantly interfered the extraction of CdS QDs and Ag^+ by TTAB/SDS when their concentrations are higher than 10 mmol L^{-1} . These results may be relate to the counterions effect around the CdS QDs surfaces. For metal ions including Co^{2+} , Fe^{3+} , Mn^{2+} , Zn^{2+} , Cd^{2+} , Pb^{2+} ,

Ni^{2+} even concentrations of 10 mmol L^{-1} did not interfere the determination of Ag^+ .

Analytical application

This method was applied to determine Ag^+ in tap water and film developing solvent wastewater which was obtained from the local hospital in Khon Kaen province, Thailand. Because of the interferences in film developing solvent wastewater have a fluorescence signal which interfering the determination of CdS QDs, the optimum condition was filtered the sample with filter paper No.1 and then diluted the sample (100 times) before spiked the Ag^+ standard solution. The obtained recoveries (Table 3) were satisfied.

Conclusion

This research demonstrates the mixed cationic surfactant (TTAB) and anionic surfactant (SDS) for the extraction and enrichment of CdS QDs and Ag^+ from aqueous phase. The phase separation mechanism can be explained by the electrostatic adsorption of TTAB and SDS on surface of CdS QDs. The mixed TTAB and SDS was not only used as the extractant, but also acted as co-stabilizing agents to enhance the fluorescence intensity of CdS QDs. Therefore, the present method has high selectivity and sensitivity for the detection of Ag^+ at trace level (20 pmol L^{-1}).

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