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One-step enrichment and chemiluminescence detection of sodium dodecyl benzene sulfonate in river water using Mg–Al–carbonate layered double hydroxides

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In this work, Mg–Al CO_3-layered double hydroxides (LDHs) were used as adsorbent materials for sodium dodecyl benzene sulfonate (SDBS) in aqueous solutions, the enriched SDBS can be directly detected by IO_4^-–H_2O_2 chemiluminescence (CL) system. The commonly existing cations cannot be enriched by Mg–Al CO_3-LDHs due to the structurally positively charged layers of LDHs, while other adsorbed anionic interferents had no effect on the IO_4^-–H_2O_2 CL reaction. The corresponding linear regression equation was established in the range of 0.1–10 μM for SDBS. The detection limit at a signal-to-noise (S/N) ratio of 3 for SDBS was 0.08 μM. The relative standard deviation (RSD) for nine repeated measurements of 0.5 μM SDBS was 2.6%. This proposed method has been successfully applied to the determination of SDBS in river water samples. To the best of our knowledge, we have first time coupled the high enrichment capacity of LDHs towards anions with CL detection for analytes.

1. Introduction

As the main ingredient of laundry detergents, anionic surfactants are extensively used in cosmetics, dyeing of fabrics in textile industry and flotation applications [1–3]. Over the past few decades, widespread and indiscriminate use of surfactants in household and personal cleansing products has led to water pollution [4]. Currently, sodium dodecyl benzene sulfonate (SDBS) is one of the most popularly used anionic surfactants in industrial cleaner formulations [5]. It enters the environment primarily through wastewater, causing great masses of foams in river water with long-term contamination effects on account of its difficult biodegradability. In addition, acting as a solvent, SDBS can transfer some organic pollutants (e.g., petroleum products, oil and pesticides) to water [6]. Therefore, it is becoming extremely urgent and significant to establish an accurate method for the determination of SDBS in river water. The classic analytical techniques are currently employed to determine SDBS, including spectrophotometric method [7], fluorescent method [8,9], gas chromatography coupled with flame ionization detector (GC-FID) [10], gas chromatography coupled with mass spectrometry (GC–MS) [11], and capillary electrophoresis coupled with mass spectrometry (CE-MS) [12].

Chemiluminescence (CL) method is becoming a promising technique with reliability, fast response, cheap instrument, simple operation and high sensitivity [13–15]. To the best of our knowledge, there is only one report on CL determination for SDBS [16], which has demonstrated the feasibility of CL detection for SDBS based on SDBS-enhanced the IO_4^-–H_2O_2 CL reaction. However, this method suffered from the low selectivity due to serious interference from other surfactants, including anionic surfactant sodium dodecyl sulfonate (SDS), cationic surfactant cetyltrimethylammonium bromide (CTAB), and nonionic surfactants Triton-X 100/Tween 40. These disadvantages resulted in that the SDBS-enhanced the IO_4^-–H_2O_2 CL reaction can only be used for the determination of SDBS in laundry detergents not in real samples (e.g., river water) [16]. Therefore, considerable attention has been devoted to the improvement of CL detection selectivity for SDBS by virtue of distinct adsorption technique.

Layered double hydroxides (LDHs), also known as hydrotalcite-like compounds, are an important class of synthetic anionic layered clays with structurally positively charged layers and interlayer balancing anions [17–19]. The general formula is $[M^{2+}_{1-x}M^{3+}_{x}(OH)_2]^{x+}[X^{m-}_{x/m}]\cdot nH_2O$, where M^{2+} and M^{3+} are divalent and trivalent metal cations that occupy octahedral positions in the brucite-like layer, and X^{m-} is structural balance anions. LDHs have been considered as novel class of green adsorbents in environment purification owing to their nontoxicity, layer positive charge, high anion-exchange capacity, large surface area, and water resistant structure [20–24]. Particularly, it has

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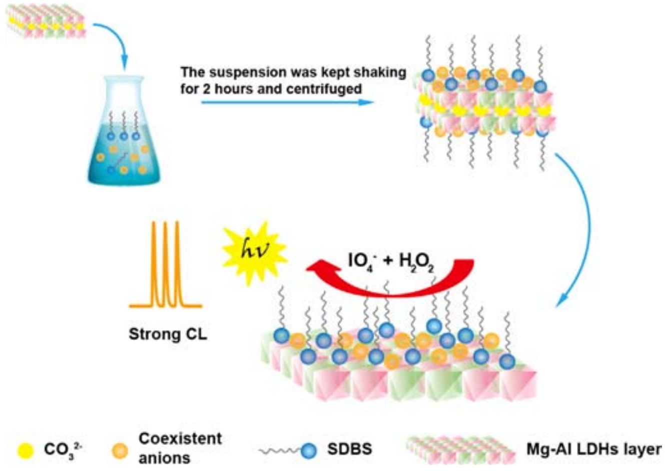

Fig. 1. Schematic illustration for the enriched SDBS on the external surface of Mg–Al CO_3-LDHs-enhanced IO_4^-–H_2O_2 CL system.

been reported that Mg–Al CO_3-LDHs can be employed as adsorbents to remove SDBS from aqueous solutions [25], making it possible to develop a new method for the determination of SDBS after SDBS is enriched with Mg–Al CO_3-LDHs.

We recently demonstrated that the adsorbed SDBS on the external surface of SDBS-intercalated LDHs could significantly enhance the CL from the IO_4^-–H_2O_2 reaction due to the disappearance of electrostatic repulsion between SDBS and anionic CL reactants, resulting in concentrating of anionic CL reactants on the adsorbed surfactant layers of SDBS-intercalated LDHs and higher CL efficiency in LDHs microenvironment [26]. In this study, we utilized carbonate in the LDHs instead of SDBS used in our previous work [26] based on the following two causes: firstly, the adsorbed SDBS on the surface of the SDBS-intercalated LDHs were difficultly removed, leading to high background CL; secondly, the CO_3-intercalated LDHs are prepared easily and other anions cannot enter the interlayer due to the greater affinity of carbonate for LDH layers [27]. Interestingly, the enriched SDBS at the surface of Mg–Al CO_3-LDHs can be selectively detected by IO_4^-–H_2O_2 CL system (Fig. 1). The potentially interfering cations were not able to be enriched by Mg–Al CO_3-LDHs in river water due to the native characteristics of LDHs; while the enriched other anionic interferents had no effect on the IO_4^-–H_2O_2 CL reaction. Under the optimum experimental conditions, this proposed method was successfully used for the determination of SDBS in the river water samples. To the best of our knowledge, this is the first report that the high enrichment capacity of LDHs towards anions is coupled with CL detection for anionic compounds.

2. Experimental

2.1. Reagents

All the reagents were analytical grade and used as received without further purification. $Mg(NO_3)_2 \cdot 6H_2O$, $Al(NO_3)_3 \cdot 9H_2O$, Na_2CO_3, NaOH, and chloroform were purchased from Beijing Chemical Reagent Company (Beijing, China). Cetyltrimethylammonium bromide (CTAB), sodium dodecyl sulfate (SDS), and Triton-X 100 were purchased from J&K Chemical Ltd. (Beijing, China). Sodium dodecyl benzene sulfonate (SDBS) and Methylene Blue (MB) were purchased from Tokyo Chemical Industry Co. Ltd. (Tokyo, Japan). All solutions were prepared with deionized water (Milli-Q, Millipore). Working solutions of 0.15 M H_2O_2 were prepared daily from 30% (*v*/*v*) H_2O_2 (Beijing Chemical Reagent Company, China). Working solutions of 0.25 M $NaIO_4$ were prepared by dissolving $NaIO_4$ (Beijing Chemical Reagent Company, China) in deionized water. A 0.01 M stock solution of SDBS was prepared by dissolving 0.0349 g SDBS in 10 mL of deionized water.

2.2. Preparation of Mg–Al CO_3-LDHs

In a typical experiment, Mg–Al CO_3-LDH colloidal suspension was synthesized by constant pH co-precipitation method. Solution A consisted of 0.04 mol $Mg(NO_3)_2 \cdot 6H_2O$ and 0.02 mol $Al(NO_3)_3 \cdot 9H_2O$, which were dissolved in 60 mL of deionized water with a Mg/Al molar ratio 2. Solution B was prepared dissolving NaOH (0.12 mol) and Na_2CO_3 (0.01 mol) in 60 mL of deionized water. The two solutions were added dropwise to a 250 mL flask under vigorous stirring maintaining about pH 10 at room temperature. The resulting white precipitate was aged for 24 h at 65 °C before it was separated by centrifugation and washed thoroughly with deionized water. Afterward, the colloidal suspension was stored at 4 °C until further use. Mg–Al CO_3-LDHs with Mg/Al molar ratio 3 and 4 were also synthesized by the same procedure.

2.3. Enrichment of SDBS in aqueous solutions

The enrichment procedure was carried out at room temperature (25 °C). In stoppered conical flasks, 20 mL aqueous SDBS solution in the concentration range from 0.1 μM to 10 μM were added into 0.2 mL Mg–Al CO_3-LDH colloidal solution (0.04 g/mL). The mixed suspension kept shaking for 2 h under gentle stirring. After that, the sediment obtained by centrifugation was diluted with 2 mL deionized water for the next CL detection. The concentration of SDBS in the supernatant was determined by fluorescence spectrophotometry, calculating the adsorption percentage from the following equation: $R(\%)=(C_0-C_e)/C_0$, where R is the adsorption percentage of SDBS (%), C_0 refers to the initial concentration of SDBS, C_e refers to the concentration of SDBS in the supernatant (mg/L).

2.4. Apparatus

Powder X-ray diffraction (XRD) measurements were performed on a Bruker (Germany) D8 ADVANCE X-ray diffractometer equipped with graphite-monochromatized Cu/Kα radiation ($\lambda=1.5406$ Å). The 2θ angle of the diffractometer was stepped from 5° to 75° at a scan rate of 0.02°/s. The Fourier-transform infrared (FT-IR) spectra were obtained with a Nicolet 6700 FT-IR (Thermo, America) using the KBr disk technique. Zeta potentials were measured using the Zetasizer 3000HS nanogranularity analyzer (Malvern Instruments) with the water RI of 1.33, the water dielectric constant of 78.55 and the water viscosity of 0.8872 cP. Fluorescence intensity was detected by F-7000 fluorescence spectrophotometer (Hitachi, Tokyo, Japan) at a slit of 10 nm with a scanning rate of 1200 nm/min. The CL detection was conducted on Biophysics Chemiluminescence (BPCL) luminescence analyzer (Institute of Biophysics, Chinese Academy of Science, Beijing, China).

2.5. CL measurements

The CL detection was conducted using the flow injection analysis (FIA) system depicted in Fig. 2. $NaIO_4$ (0.25 M) was used as the carrier stream, the adsorbed SDBS on the external surface of CO_3-LDHs was injected into it through a 50 μL loop-valve injector, which then mixed with H_2O_2 (0.15 M) through a three-way piece. The flow rates of $NaIO_4$ and H_2O_2 were 2.0 mL/min and 2.5 mL/min, respectively. The CL signals were detected when the mixed stream reached the detection cell, which was adjacent to a photomultiplier tube (PMT). The produced PMT signals were processed by the BPCL analyzer and imported to the computer for data acquisition. The work voltage of

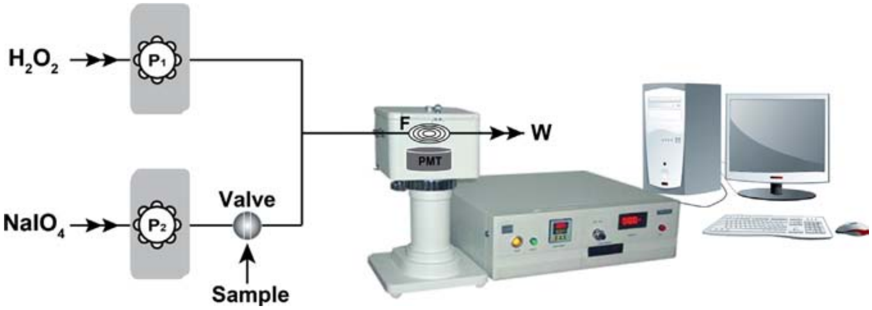

Fig. 2. Schematic diagram of CL detection coupled with flow injection system. P_1 and P_2, peristaltic pumps; Valve, six-way valve with 50 μL loop injector; F, flow cell; PMT, photomultiplier tube (−1000 V); W, Waste. Experimental conditions: the flow rates of P_1 and P_2 were 2.5 mL/min and 2.0 mL/min, respectively; the concentration of H_2O_2 and $NaIO_4$ were 0.15 M and 0.25 M, respectively.

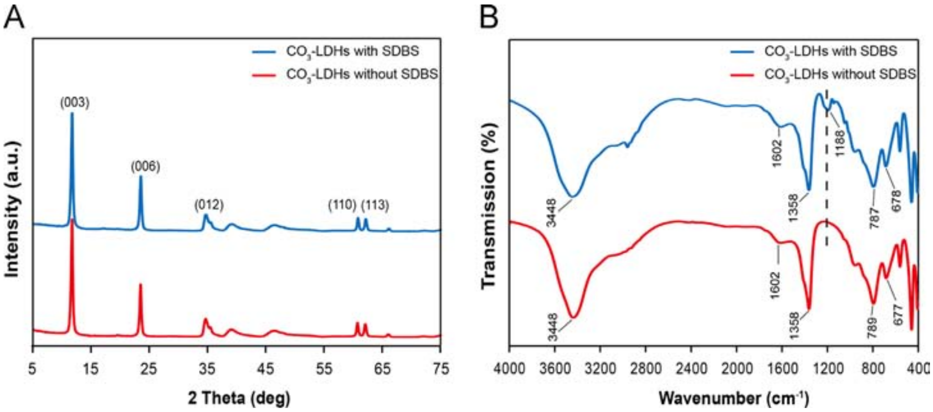

Fig. 3. (A) Powder XRD patterns and (B) FTIR spectra of Mg–Al CO_3-LDHs before (red, lower curve) and after (blue, upper curve) they were mixed with the aqueous SDBS solution. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article).

the BPCL analyzer was set at −1000 V, and a data integration time of 0.1 s was used for recording CL emission.

2.6. Sample preparation

River water samples from the Xiaoyue River and Xiba River (Chaoyang District, Beijing, China) were collected in a 250 mL polyethylene bottle. The collected samples were filtered through medium-speed qualitative filter papers in order to remove some biological and insoluble impurities in environmental water. After that, adequate water samples were immediately analyzed by the proposed and standard methods.

3. Result and discussion

3.1. Enrichment of SDBS on the surface of Mg–Al CO_3-LDHs

The powder XRD measurements (Fig. 3A) of Mg–Al CO_3-LDHs were performed before and after they were mixed with the aqueous SDBS solution. The original Mg–Al CO_3-LDHs showed the typical XRD pattern of the CO_3-LDHs. After enrichment experiments, there was no change in the interlayer distance of Mg–Al CO_3-LDHs, meaning that SDBS anions were adsorbed on the surface of Mg–Al CO_3-LDHs.

Fig. 3B showed the FT-IR spectra of Mg–Al CO_3-LDHs before and after the enrichment, respectively. The results showed that the original Mg–Al CO_3-LDHs exhibited the characteristic absorption bands centered at ~3448 cm^{-1} due to the stretching mode ν_{O-H} of hydration water molecules and OH-groups of the brucite-like layers, centered at 1602 cm^{-1} corresponding to the H_2O water bending mode δ_{O-H} of the interlayer water molecules. Furthermore, the strong peak at 1358 cm^{-1} was attributed to the characteristic of carbonate vibration. The bands in the range of 500–800 cm^{-1} could be assigned to metal–oxygen lattice vibration, suggesting the formation of Mg–Al hydroxide layers [28,29]. After the enrichment, the presence of SDBS in the Mg–Al CO_3-LDHs was confirmed by the new band referent to the symmetric stretching vibration of S=O at 1188 cm^{-1} [25]. These results further confirmed that the SDBS anions were adsorbed on the external surfaces of Mg–Al CO_3-LDHs.

Finally, the zeta potential of Mg–Al CO_3-LDHs by adding different concentrations of SDBS was measured in order to figure out the configuration on the exterior surfaces of the LDH particles. In the absence of SDBS, Mg–Al CO_3-LDHs had a relatively high positive zeta potential (36.6 mV). Upon the addition of SDBS, the zeta potential was obviously dropped to 26.4 mV at around 10 μM SDBS. Based on the above results, we could speculate that SDBS anions electrostatically adsorb on the surface of Mg–Al CO_3-LDHs as a monolayer [13].

3.2. Enriched SDBS on the surface of Mg–Al CO_3-LDHs-enhanced IO_4^-–H_2O_2 CL

The Mg/Al molar ratios directly affected the adsorption capacity of SDBS on the surface of Mg–Al CO_3-LDHs [30]. In this study, the effect of Mg/Al molar ratios of Mg–Al CO_3-LDHs between 2 and 4 on the adsorption percentage of SDBS and the CL intensity was investigated, respectively (Fig. 4A). The results showed that the CL intensity of Mg–Al CO_3-LDHs with Mg/Al molar ratio 2 was the strongest, and the higher Mg/Al ratios resulted in a decrease of the CL intensity. Meanwhile, the adsorption percentage was increased with a decrease in the Mg/Al molar ratios from 4 to 2. It is easily understandable that the higher charge density in the layers of Mg–Al CO_3-LDHs with a Mg/Al ratio of 2 can result in the stronger

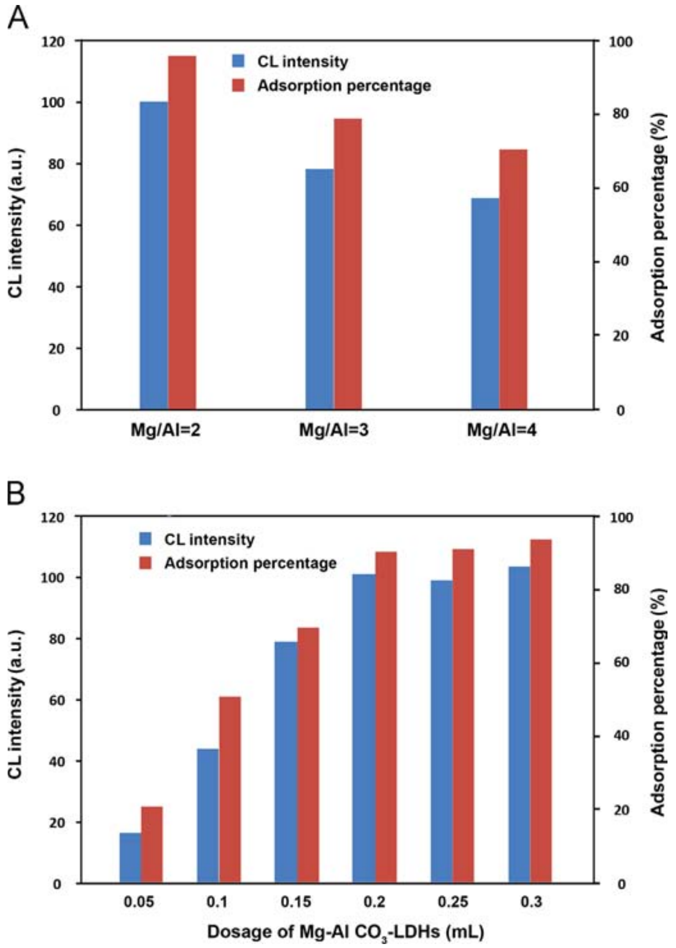

Fig. 4. (A) Effect of different Mg/Al molar ratios on the IO_4^-–H_2O_2 CL intensity (blue, left) and adsorption percentage of 10 μM SDBS (red, right); B) Effect of the dosage of Mg–Al CO_3-LDHs on the IO_4^-–H_2O_2 CL intensity (blue, left) and adsorption percentage for 10 μM SDBS (red, right). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article).

anion exchange capacity [31]. In addition, the as-prepared LDHs with Mg/Al molar ratio of 2 owned lower pH than those of 3 and 4, even when the initial pH was carefully adjusted to the same [32]. It was reported that the solid surface became more positively charged when the pH of the aqueous phase was reduced, due to the adsorption of protons from the solution onto charged sites, leading to an increase in the adsorption of anions [32,33].

3.3. Optimization conditions for CL system

In general, the dosage of Mg–Al CO_3-LDHs greatly influenced the adsorption amounts of SDBS [34], leading to large variations in CL intensity. A series of dosage of 0.04 g/mL Mg–Al CO_3-LDHs in the range 0.05–0.3 mL was investigated to affect the adsorption amounts of 10 μM SDBS, and the results were shown in Fig. 4B. The maximum adsorption was obtained at an adsorbent dosage of 0.2 mL, and then the adsorption percentage remained constant up to 0.3 mL. Correspondingly, the CL intensity was also enhanced with the increase of the adsorption percentage. Therefore, it is realizable that the relationship between CL intensity and different concentrations of SDBS enriched can be utilized for the determination of SDBS.

The enrichment of SDBS on the surface of Mg–Al CO_3-LDHs depends on the adsorption time. Herein, the adsorption time was optimized in the range of 10–150 min. The results showed that the strongest CL intensity was observed at 120 min, and then the CL intensity remained constant up to 150 min, meaning the occurrence of adsorption equilibrium. Therefore, 120 min adsorption time was chosen in further experiments.

The concentrations of $NaIO_4$ and H_2O_2 were key parameters for the $NaIO_4$–H_2O_2 CL reaction. We investigated the effect of $NaIO_4$ concentration (0.05–0.25 M) on the CL intensity, and found that the CL intensity increased with an increase in $NaIO_4$ concentration. However, higher concentrations than 0.25 M $NaIO_4$ are difficultly soluble in water at room temperature. Therefore, 0.25 M $NaIO_4$ was selected as the optimum one. On the other hand, the concentration of H_2O_2 was examined in the range of 0.02–0.2 M. In this work, H_2O_2 with a concentration higher than 0.15 M can produce many gaseous bubbles. Therefore, 0.15 M H_2O_2 was utilized in subsequent studies.

The flow rates of pump 1 (P_1) for H_2O_2 and pump 2 (P_2) for $NaIO_4$ in Fig. 2 were optimized in the range of 1.0–3.0 mL/min. The optimal values for P_1 and P_2 were 2.5 and 2.0 mL/min, respectively.

3.4. Analytical performances

Under the optimum conditions employed in the present study, different concentrations of the aqueous SDBS solutions were tested using the proposed approach shown in Fig. 2. It was found that the calibration curve was linear from 0.1–10 μM for SDBS (Fig. 5). The regression equation was $y=9.1058x+8.7654$ ($R^2=0.9936$), where y is the relative CL intensity and x is the concentration of SDBS (Fig. 5, inset). The detection limit of SDBS (S/N=3) was 0.08 μM. The relative standard deviation for nine repeated measurements of 0.5 μM SDBS was 2.6%.

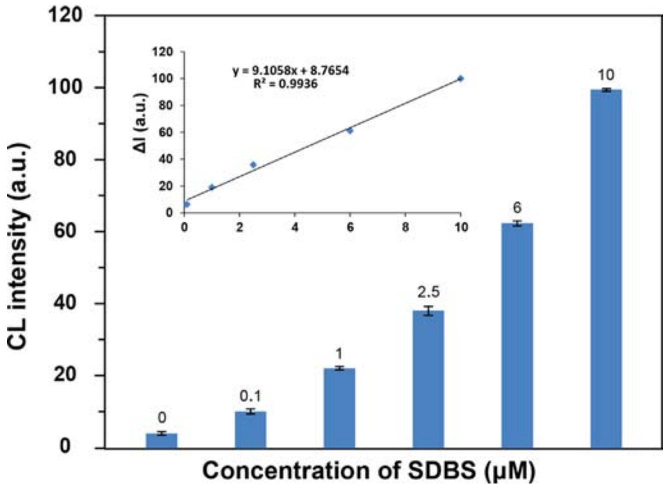

Fig. 5. CL intensity of the IO_4^-–H_2O_2 CL system by different concentrations of SDBS. Inset: the calibration curve for SDBS.

Table 1
The tolerable concentration of some interfering species to 10 μM SDBS.

Tolerance/M	Interfering ions
3×10^{-4}	CO_3^{2-}, SO_4^{2-}, PO_4^{3-}, Mn^{2+}
5×10^{-4}	SDS, CTAB, Triton-X 100
1×10^{-3}	Zn^{2+}, Fe^{3+}
3×10^{-3}	K^+, Ba^{2+}, Ca^{2+}, NH_4^+
5×10^{-3}	Al^{3+}, Mg^{2+}
2×10^{-2}	Na^+, Cl^-

Table 2
Assay results of SDBS in river water samples.

Sample	Standard method (μM)[a]	Present method (μM)[a]	Added (μM)	Found (μM)[a]	Recovery (%)[a]
Xiaoyue River	5.7 ± 0.21	5.5 ± 0.23	6.0	11.7 ± 0.11	103.3 ± 1.33
			8.0	13.3 ± 0.18	97.5 ± 2.88
Xiba River	4.6 ± 0.16	4.4 ± 0.15	4.0	8.3 ± 0.10	97.5 ± 3.00
			5.0	9.5 ± 0.05	102.0 ± 1.60

[a] Mean ± SD of three measurements.

3.5. Interference study

To demonstrate the selectivity of the developed method for the detection of SDBS in real samples, a mixed solution of 10 μM SDBS and each interference species with different concentrations was analyzed by the proposed method. A compound was considered as non-interfering if the analytical CL signal variation was less than 5% when compared to the analytical signal obtained in the absence of the referred compound. The results were summarized in Table 1. The influences of monovalent inorganic anions on the CL intensity can be ignored, because SDBS anions have stronger affinity for LDH layers [30,35]. In addition, 30-fold CO_3^{2-}, SO_4^{2-}, PO_4^{3-} and Mn^{2+}, 50-fold SDS, CTAB, Triton-X 100, 100-fold Zn^{2+}, Fe^{3+}, 300-fold K^+, Ba^{2+}, Ca^{2+}, NH_4^+, 500-fold Al^{3+}, Mg^{2+}, and 2000-fold Na^+, Cl^- at a relative mass ratio regarding SDBS did not interfere. Note that the maximum concentration of Cl^- in real water samples is 88 mg/L [36]. Therefore, it can be concluded that the proposed method showed high selectivity towards SDBS in real samples.

3.6. River water samples

The advantages of this work included the enrichment of SDBS on the surface of Mg–Al CO_3-LDHs and CL detection for SDBS with high selectivity and sensitivity. Currently, the existence of SDBS is not only in wastewater but also in river water samples. However, the concentration of SDBS in wastewater is 400 mg/L or so; on the contrary, river water samples contain low SDBS concentration ($\leq$5.0 mg/L) [37]. Therefore, we would evaluate the applicability and reliability of the present method by determining SDBS in river water samples. The samples were filtrated through membrane. Assay results of SDBS in river water samples and recoveries were summarized in Table 2. The recoveries for SDBS in the spiked samples ranged from 97.5% to 103.3%. These results clearly demonstrated that the proposed method exhibited higher accuracy and selectivity than the current standard MB method [38]. Therefore, the proposed method is promising for the analysis of SDBS in river water samples at trace levels for environment control. In addition, the analysis time and characteristics of the present method were compared with the standard method in Fig. 6. The standard method is simply described as follows: the filtered water sample of 50 mL was placed in a separating funnel and mixed with 5.0 mL of a 0.1 mM MB solution and 5.0 mL chloroform in turn. After then, the mixture was shaken for 1.0 min, followed by the phase separation between chloroform and water. The extraction procedure was repeated three times and all the obtained chloroform phases were collected. After filtration, the absorbance of the chloroform solution was measured at 654 nm.

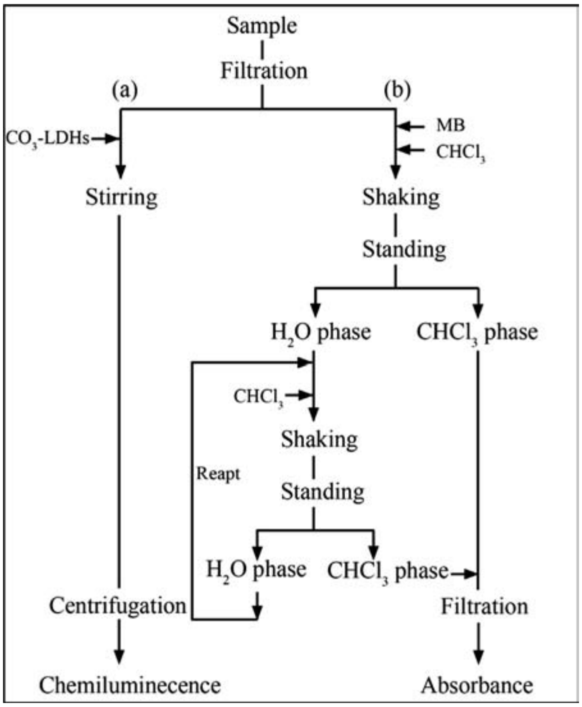

Fig. 6. Experimental procedures of the present method (a) and the standard method (b) for the determination of SDBS in water samples.

4. Conclusions

In summary, we have developed a novel method for the determination of SDBS in river water samples, combining the adsorption capacity of Mg–Al CO_3-LDHs with the CL detection of high sensitivity. Mg–Al CO_3-LDHs can provide a green method for one-step enrichment of SDBS without the help of any organic solvent. Although various concentrations of possible interfering anions and cations were dozens-fold higher than the SDBS level, there were still unsaturated sites for SDBS adsorption due to the high surface area of Mg–Al CO_3-LDHs. The proposed method is green, simple, and convenient. Our next aim is to cultivate an automated on-line trace-enrichment method with the CL detection by means of decreasing the equilibrium time within the scale of minutes, which is underway in our lab.

Acknowledgments

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