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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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ABSTRACT

In this work, Mg–Al CO₃-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 IO₄[−]–H₂O₂ chemiluminescence (CL) system. The commonly existing cations cannot be enriched by Mg–Al CO₃-LDHs due to the structurally positively charged layers of LDHs, while other adsorbed anionic interferences had no effect on the IO₄[−]–H₂O₂ 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.

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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 IO₄[−]–H₂O₂ 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 IO₄[−]–H₂O₂ 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 [M₁²⁺_{1-x}M₂³⁺_x(OH)₂]^{x+}[X^{m−}_{x/m}][−]·nH₂O, where M²⁺ and M³⁺ 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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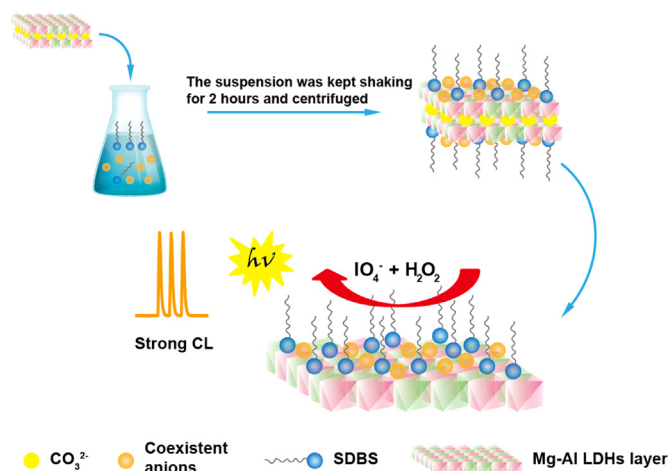


Fig. 1. Schematic illustration for the enriched SDBS on the external surface of Mg–Al CO_3 -LDHs-enhanced IO_4^- – H_2O_2 CL system.

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. $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 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 $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 0.02 mol $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 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 $\text{Cu}/\text{K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). 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

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