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Spectroscopic studies of dye-surfactant interactions with the co-existence of heavy metal ions for foam fractionation

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Abstract

The interaction between a cationic dye Methylene Blue (MB) and an anionic surfactant sodium dodecyl sulfate (SDS) with the presence of Cd^{2+} was investigated spectrophotometrically in a certain concentration range. The spectrophotometric measurements of dye-metal ion-surfactant system were carried out. The results indicated that the SDS concentration had a significant influence on the dye spectrum, while the addition of Cd^{2+} hardly caused change of the maximum value of absorbance. According to this observation, we concluded that electrostatic and hydrophobic interaction between dye and surfactant occurred up to a certain level, and the homo-ions Cd^{2+} almost exerted no effect on the dye-surfactant complexation, establishing a theoretical foundation for simultaneous removal of organic dye and heavy metal using foam fractionation. Meanwhile, the effects of their interaction on foam performance were investigated. The results showed that the addition of Cd^{2+} favored the tendency to ameliorate foam properties just contrary to MB. The feasibility of foam separation for dye and heavy metal removal from simulated wastewater was also confirmed using a continuous foam fractionator. In the simultaneous removal process, with the initial SDS concentration ranging from 0.5 to 5.0 mmol/L, the maximum removal efficiencies of MB and Cd^{2+} were obtained as 99.69% and 99.61%, respectively. The enrichment ratios were reduced from 24.34 to 7.65 for MB and from 22.01 to 3.35 for Cd^{2+} .

Key words: foam fractionation; simultaneous removal; spectroscopic study; SDS; MB; Cd^{2+}

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Introduction

The removal of organic dyes and heavy metal ions is of particular interest in hazardous waste management due to their severe toxicity to living organisms. A large amount of the total world production of dyes is lost during the dyeing process and is released into the dyeing industry liquid effluents (Zollinger, 1992). Cd^{2+} is one of the harmful metal ions (Zeng et al., 2011; Zhang et al., 2011; Liu et al., 2011) found in dyeing industry wastewaters. Their high solubility in aquatic environments makes them accumulate in the food chain, which can damage the nervous system, kidneys and reproductive system (Mukherjee and Kabata-Pendias, 2007). As international environmental standards are becoming more stringent, these colored and poisoned wastewater need treatment before disposal.

Foam fractionation is based on the selective adsorption or attachment of materials onto the surfaces of gas bubbles rising through a solution (Prudich, 2007). It can be used to concentrate and remove dissolved materials, including surface active and non-surface active solutes from aqueous solutions. Frothers are utilized to enhance generation of fine bubbles and to stabilize the froth (Cho and Laskowski,

2002). This process offers many advantages such as low space and energy requirements; simple plant design, operation, and scale-up; and low capital and operating costs (Gerken et al., 2006; Lu et al., 2010). Furthermore, there is a strong attraction in the fact that foam fractionation has the potential to recover and recycle the heavy metal.

Dyes are mostly aromatic compounds containing chromophores, delocalized electron systems with conjugated double bonds, and auxochromes, electron withdrawing substituents that cause or intensify the color of the chromophore (Safavi et al., 2008; Bielska and Szymanowski, 2006). Although the interaction between dyes and surfactants has been reported previously (Simončič and Kováč, 1998; Bagha et al., 2007; Akbaş and Kartal, 2005; Ivšić et al., 2009), interaction between dyes and surfactants in aqueous solutions with the presence of heavy metal is of great importance in the theory and technology of foam fractionation. Especially for simultaneous removal of organic dyes and metal ions, the investigation would be particularly significant. This lack of information, coupled with the potential applications of foam fractionation for simultaneous removal have prompted us to investigate the interactions.

This study investigated the interaction between Methylene Blue (MB) and anionic surfactant, i.e. sodium dodecyl

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sulfate (SDS), in presence of Cd^{2+} . Dye-surfactant complex formation in a ternary mixture has been calculated. It is well known that ionic surfactants exert profound effects on conductivity and spectral behavior of dye molecules owing to interaction. Therefore, the analysis of UV-Vis spectrum of various solutions was explored first. Meanwhile, the effects of MB and Cd^{2+} on foam properties were investigated. The purpose of this work was to demonstrate the potential of anionic surfactants used in foam fractionation for simultaneous removal of dye and metal ions from analog composite wastewater. We have also discussed optimum concentration for surfactant used in the foam fractionation.

1 Materials and methods

1.1 Specimen preparation

SDS was supplied by Tianjin Fuchen Chemical Factory (China) with 99% purity. MB was purchased from Tianjin DaMao Chemical Agent Company (China) and used as received. The feed solution of Cd^{2+} was prepared by dissolving $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ which was obtained from Shanghai Tingxin Chemical Factory (China). All chemicals were of analytical agent grade and were used without further purification. Distilled water was used as a solvent in all runs. The pH of measured solution was not adjusted and it was in the range 5.0–7.0 in all runs.

1.2 UV-Visible spectroscopy

The visible absorption spectra of solutions were recorded at 25°C on a Varian Cary 1E UV-Visible Spectrophotometer using 1 cm cells. The spectrometer was located in an air-conditioned room and samples were allowed to adjust to the room temperature before measurement.

1.3 Foam properties

Oscillation method was used in the experiment. A graduated cylinder with stopper of 100 mL was loaded with 10 mL solution, kept shaking for 1 min at a speed of 120 times/min. The foam height when stop vibrating and the time for bubble collapse in half were as the initial height and half-life. Foam height and half-life were measured to represent the foamability and foam stability. MB and SDS solutions with the absence or presence of Cd^{2+} were freshly prepared prior to each determination.

1.4 Foam fractionation process

Figure 1 shows the apparatus used to fractionate the dyes and metal ions. The foaming-draining system was composed of a glass column with an internal diameter of 14 cm and a 190 cm height. After steady-state conditions, samples of the feed solution and the effluent were collected and analyzed for MB and Cd^{2+} concentrations. The sample chamber temperature was kept constant at room temperature (25–27°C).

1.5 Measurement and analysis

The concentration of MB was measured with Shimadzu UV-2550 (P/N206-55501-93) spectrophotometer.

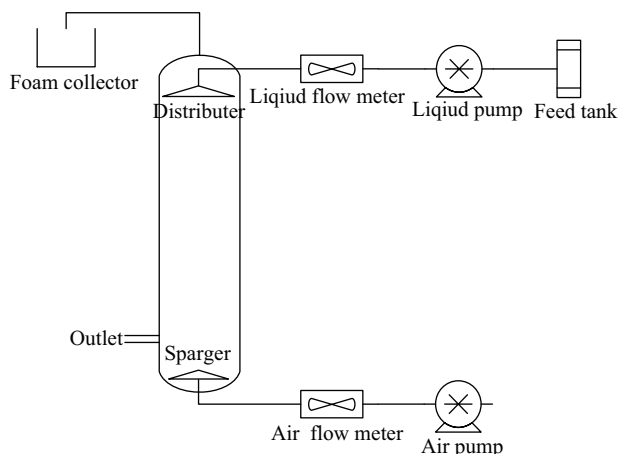


Fig. 1 Experimental apparatus for foam fractionation.

The concentration of cadmium ions was analyzed by atomic adsorption spectroscopy (Perkin-Elmer, Model AAnalyst 700). The separation efficiencies of the contaminants were evaluated using the removal fraction (R) and the enrichment ratio (E) as shown below:

$$R = \frac{C_i - C_e}{C_i} \times 100\% \quad (1)$$

$$E = \frac{C_f}{C_i} \quad (2)$$

where, C_i (mmol/L) and C_e (mmol/L) are MB or Cd^{2+} concentrations in the influent and effluent streams, respectively, and C_f (mmol/L) is MB or Cd^{2+} concentrations in the collapsed foam solution.

2 Results and discussion

2.1 Spectral characteristics of dye-surfactant system

Surfactants are able to affect the electronic absorption spectra of solution of many dyes (Akbaş and Taner, 2009). Figure 2 shows the variation of absorbance of MB with various concentration of SDS at 25°C. The cationic dye MB in aqueous solution (0.03 mmol/L) exhibited a maximum absorption band at 663 nm. The interaction

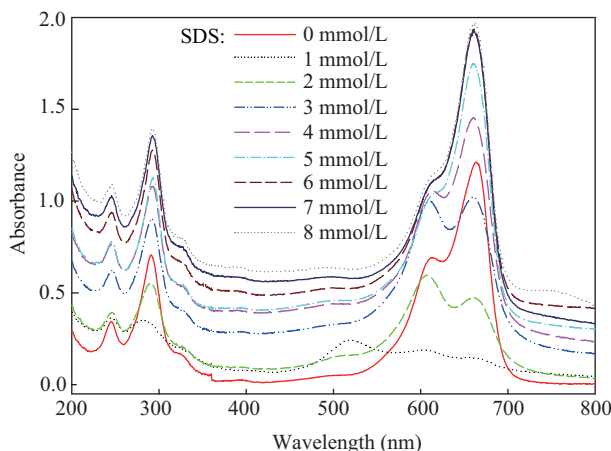


Fig. 2 Spectral changes in dye spectrum as a function of surfactant concentration. SDS: sodium dodecyl sulfate; Methylene Blue (MB) 0.03 mmol/L.

of dye with surfactants ($\text{SDS} < 4.0 \text{ mmol/L}$) resulted in an initial sharp decrease of the maximum absorption spectra and the appearance of a new absorption band on the spectrum of the dye. This suggests that dye-surfactant interactions exist in the studied solutions. These spectral changes can be attributed to the formation of a complex between dye and surfactant molecules, which is a consequence of mutual influences of long-range and short-range interactions (Kartal and Akba, 2005). Ionic associate may impact upon the benzene ring conjugated system, followed by an absorption reduction and hypochromic effect. While surfactant concentration increased, an increase in absorbance was observed. This behaviour can be explained as the monomeric, dimeric and trimeric conversion. This can be represent as dye-surfactant complex + micelle \leftrightarrow micellized dye. The micellized dye could increase the maximum absorbance, as showed in Fig. 3. Then with increasing surfactant concentration the absorbance values reached the limiting value. This case indicated that nearly all dye molecules were compartmentalized into micelles (Basu et al., 2005; Shakir et al., 2010), and increment of surfactants failed to cause any spectral change.

MB is an example of a molecule showing strong solvent dependent ground and excited state properties. In the ground state the molecule has a charge separated electronic structure (Raj et al., 2007). The auxochromous group being connected with chromophore group has an influence on

λ_{max} of chromophore absorption band and molar extinction coefficient through p- π conjugative effect, resulting in hyperchromicity. The presence of a chromophore in dye molecule enables aggregation to be followed by UV-Vis spectroscopy (Khana and Sarwar, 2006). As seen in Fig. 3, the changes in the absorption spectrum of dye with increasing concentrations were observed and similar spectral changes were found for the solutions of different surfactant concentrations separately. Double characteristic absorption peaks can be clearly seen from the absorption spectrum of MB solutions, 663 nm for the monomer while 610 nm for the dimer. With the MB concentration increasing, absorption peaks of monomer and dimer increased in varying degrees, on the contrary, the ratio of monomer and dimer's absorbance peak R (A_{663}/A_{610}) kept decreasing. This represented that the characteristic absorption peak of monomer was inhibited to some extent. The absorption spectra of aqueous dye solution exhibited a characteristic peak of 610 nm. But the addition of 4.0 and 6.0 mmol/L surfactant resulted increase of the absorbance with a well-defined absorption peak at 663 nm. This was probably due to that SDS concentrations can progressively affect R (A_{663}/A_{610}).

2.2 Spectral characteristics of dye -metal ion-surfactant system

Figure 4 depicts the effect of Cd^{2+} concentration on the value of absorbance of MB in the SDS solutions. According

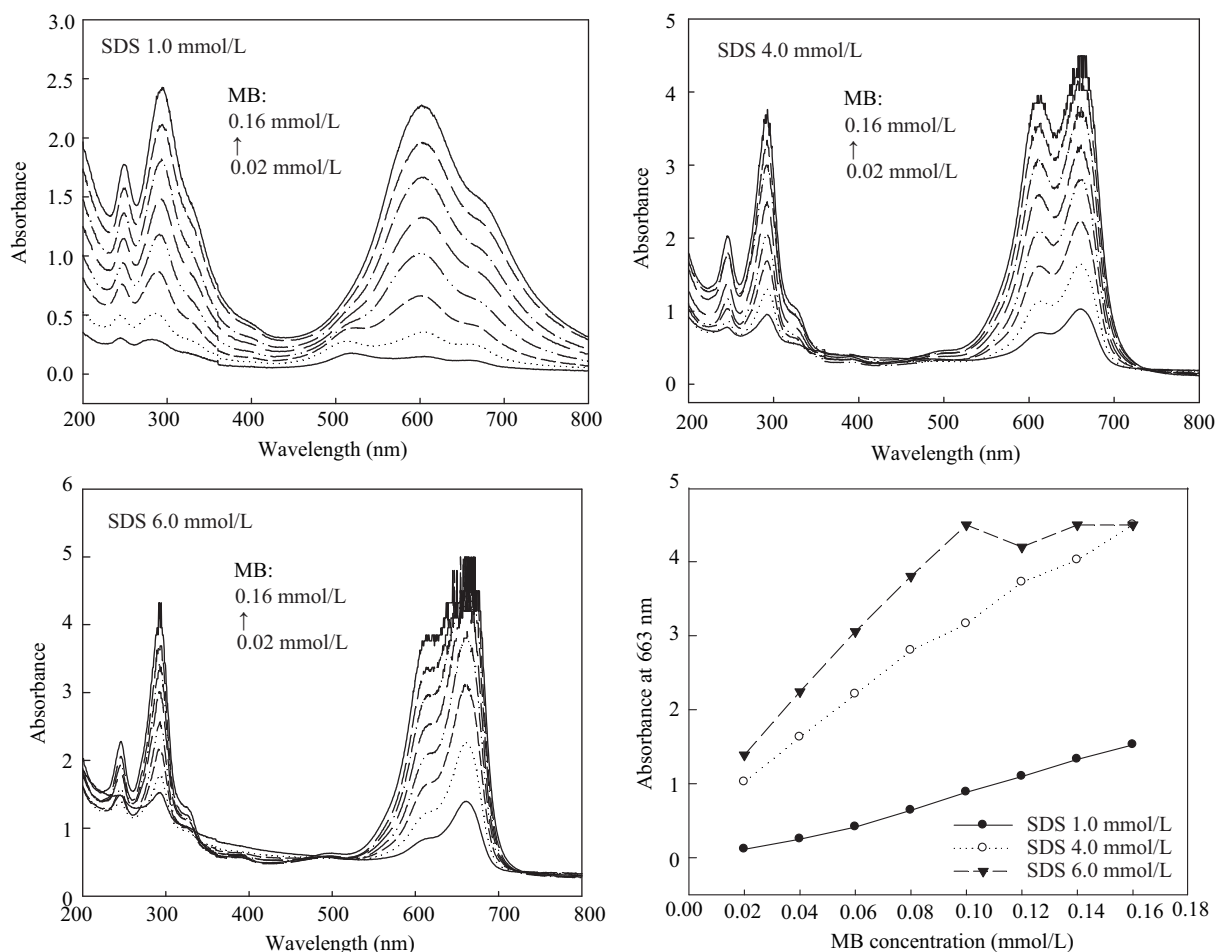


Fig. 3 UV-Vis spectra of MB-SDS system obtained at room temperature. MB: 0.02, 0.04, 0.06, 0.08, 0.10, 0.12, 0.14, and 0.16 mmol/L.

to the theory of organic spectral analysis, the characteristic absorption peak of benzene ring is at 272 nm, while the other absorption peak is caused by a large conjugate chromophore system formed by the azo structure with both ends of the benzene ring connected with amidocyanogen and sulfonic acid substituent group. As can be seen in both systems, the values of absorbance of dye did not vary enormously with increasing Cd^{2+} concentration. The

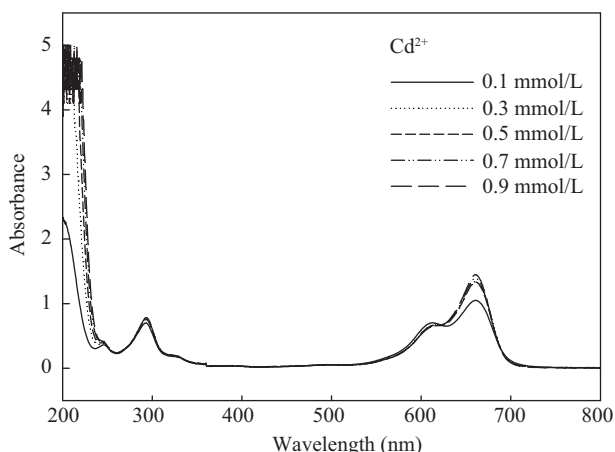


Fig. 4 Spectral changes in MB spectrum as a function of Cd^{2+} concentration.

sudden increase at 200 nm was mainly caused by nitrate ions introduced by the dilution of $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$. As shown in Fig. 5, the absorbance of MB changed in similar manner with various SDS and Cd^{2+} concentrations. The change in the value of absorbance of dye is a measure for the extent of interaction. From these spectra, the areas were corrected for dilution and plotted as a function of SDS concentration. It was evident that the UV absorption intensity of MB at 663 nm increased regularly with the variation of SDS concentration. However, in the three separate systems as illustrated in Fig. 5, hypsochromic shifting emerged with the increased Cd^{2+} concentration. This can be interpreted as higher metal ion concentration presumably results in a stronger interaction with the dyes stabilizing the excited state and leading to a lower energy separation from the ground state. Overall, the absorption value changing slightly demonstrated that the addition of Cd^{2+} almost did not affect the interaction between MB and SDS. It's mainly because that SDS possesses a strong polar group. In aqueous solution they dissociate into ions and therefore only electrostatic attractive interaction can appear between metal ions and hydrophilic head groups of surfactants. However, the aggregation of oppositely-charged dyes with surfactants is also strongly dependent on noncoulombic interactions. The short-range dispersive

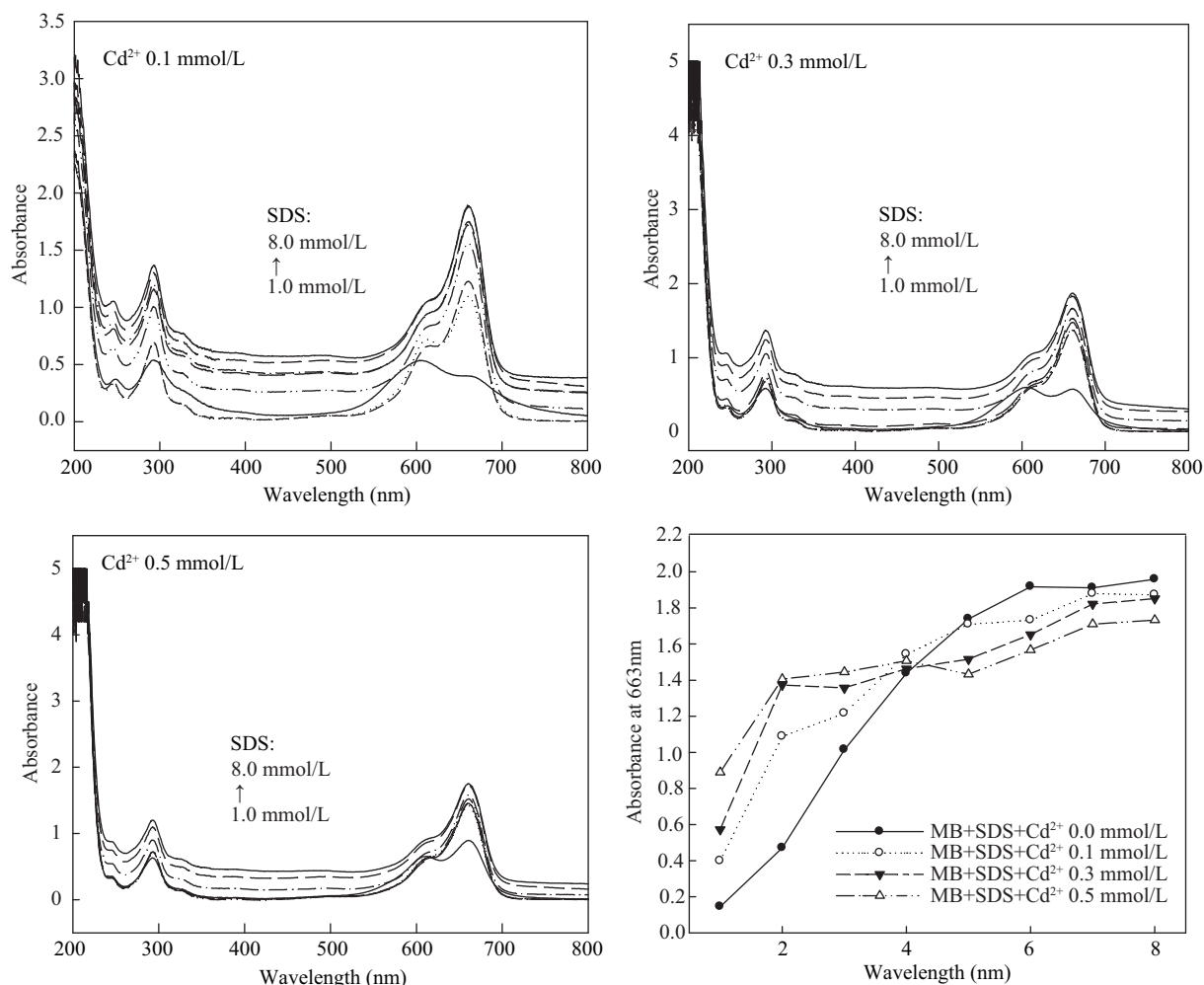


Fig. 5 Changes of MB spectrum with increasing SDS concentrations in presence of Cd^{2+} at room temperature. SDS: 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0 and 8.0 mmol/L, MB: 0.03 mmol/L.

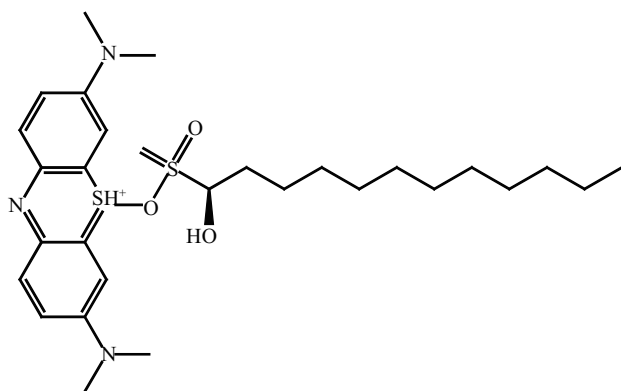


Fig. 6 Proposed structure of SDS-MB ionic associate.

forces or hydrophobic interactions may align the hydrocarbon chain of the surfactant with the hydrophobic moiety of the dye. For the interaction between ionic surfactants and ionic dye, both electrostatic and hydrophobic forces are important. Different principal forces bring on little impact on the formation of SDS-MB complex even in presence of Cd^{2+} , which has provided a theoretical basis for the advanced treatment of wastewater containing both dyes and heavy metals.

SDS molecular mainly existed as disordered monomer in the low concentration. The negatively charged SDS monomers thus can easily form complex with cationic MB monomers due to the electrostatic interaction. The complex shown in Fig. 6 incorporated the electronegative sulfate groups and the positively charged thiophenol groups as ligands. The interaction presumably accounted for this preference for surfactant-dye complex formation at the position proposed.

2.3 Foam properties

Foamability and foam stability analysis were performed with SDS concentrations ranging from 1.0 to 8.0 mmol/L in SDS, SDS+MB, SDS+ Cd^{2+} and SDS+MB+ Cd^{2+} aqueous systems. A comparative study has been done on the foam properties of these four systems at the same experimental conditions. Oscillation method was adopted without adjusting PH and temperature. As shown in Fig. 7, the foamability of SDS increased with increasing SDS initial concentration since the surface tension was reduced by adding more surfactant resulting in more foam forming. But little change in foaming properties can be seen once close to the critical micelle concentration (about 8.0 mmol/L). This could be attributed to the fact that surfactants possess weak surface adsorptive capacity and large gradient in surface tension reduction before critical micelle concentration. And a constant additional of surfactants may significantly increase the surface activity, which results in a raise of foam volume and stability obviously. However, once exceeded a limitation, the foam height and half-life tend towards stability due to the minor potential to reduce surface tension.

Foam is a disperse system composed of air bubbles, fluid walls, and plateau borders. In Fig. 7, the addition of dye or metal ion can significantly change the bubble performance. It indicates that the properties of foam are determined by

not only the physicochemical properties of the foaming solution, but also the interaction of the foam with the ambient surroundings. Comparison of the foam height and half-life curves of the SDS-only and SDS+ Cd^{2+} systems revealed that the presence of a small fraction of Cd^{2+} (0.1 mmol/L) in the SDS system produced a significant melioration in foam properties. This was ascribed to that the monomers, micelles and adsorption layers of SDS were all charged and double electrical layers existed around the surfactant ions. Therefore, the electrostatic repulsion of surface-ion hydrophilic head in the surface of single-molecule adsorption layer was weakened when anti-ion Cd^{2+} was added in the solution, meanwhile further reducing critical micelle concentration and the equilibrium surface tension of the solution. Foaming ability is mainly related to surface tension of the solution and surface activity of the surfactant. The lower surface tension of the solution, the higher surface activity of the solute and the easier foam is formed (Zhang et al., 2010). Moreover, direct visualization of the foam after the measurement revealed that the SDS+ Cd^{2+} system formed a dry foam layer which was highly stable with a very thin lamella. This implied that the presence of small concentrations of Cd^{2+} in the SDS could increase the stability of the foam film against rupture. In the SDS+MB system, on the other hand, the foam height and half-life showed a steady decrease compared with SDS only system (Fig. 7). The MB-containing system showed a tendency to form wet foam in comparison to other systems, with noticeably lower value of foamability and foam stability. As a surfactant is dye-substantive when it carries a charge opposite to that of the dye and, as a result, complexes between surfactant ions and dye ions are formed (Kert and Simončič 2008). The adsorption layer at the air-water interface can be considered as an additional phase having properties which are decisive for the stability of foams (Fruhner et al., 2000). When the organic dye is connected with surfactant via electrostatic interaction, reactive groups of surfactant are complex or hidden, followed by a decrease of hydrophilicity, foamability and foam stability (Qiu et al., 2005).

2.4 Optimization of surfactant concentration

The concentration of surfactant (SDS) in the feed solution has been found to significantly influence the performance of foam fractionation. As observed from Fig. 8, results obtained at MB 0.03 mmol/L and Cd^{2+} 0.1 mmol/L indicted an increase in the collector dose resulted in a reduction in the enrichment ratio as well as removal fraction of both MB and Cd^{2+} . It was caused by lower surface tension and higher surface liquid viscosity which caused the rate of film drainage decreasing (Qu et al., 2008). As shown from the experimental consequence, the MB removal fraction increased up to a maximum value of 99.69%, which corresponded to an initial SDS concentration of 1.5 mmol/L, and the Cd^{2+} removal fraction yielded increase to 99.61%, which corresponded to the same SDS concentration. It indicates that foam fractionation is available for simultaneous removal of dye and heavy metal. Meanwhile, it is

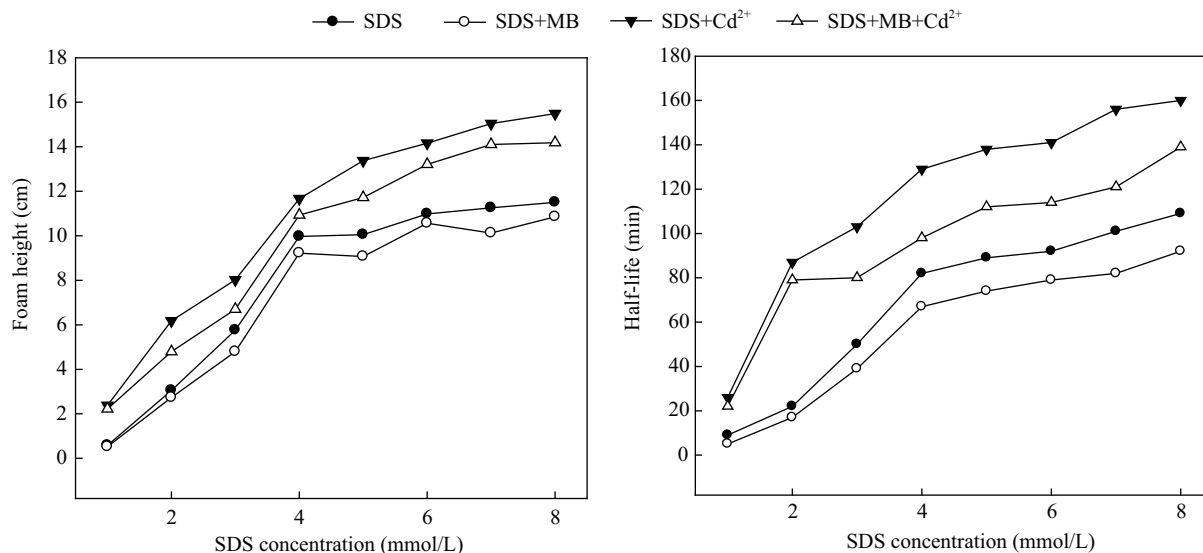


Fig. 7 Comparison of the foamability and foam stability of various aqueous solution. MB and Cd²⁺ concentrations were kept fixed at 0.03 and 0.1 mmol/L, respectively.

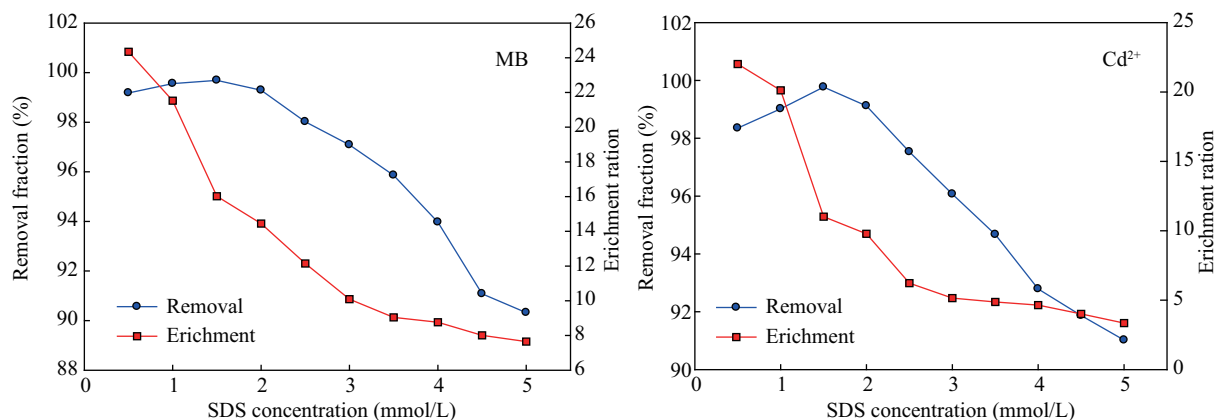


Fig. 8 Effect of SDS concentration on removal fraction and enrichment ratio of MB and Cd²⁺. MB: 0.03 mmol/L, Cd²⁺: 0.1 mmol/L.

apparent that foam fractionation should be used at lower surfactant concentrations, presumably due to the fact that once the surface is saturated, surplus surfactant in aqueous can form the micelles to solubilize MB and absorb Cd²⁺. But in the low concentration range, a relatively high surfactant concentration can result in a high foam production rate and a high mass transfer surface area available for dye and heavy metal ion adsorption, leading to higher removal. Figure 8 also shows that with an increase of surfactant concentration, the enrichment ratios were reduced from 24.34 to 7.65 for MB and from 22.01 to 3.35 for Cd²⁺. The contribution of adsorbed surfactant to enrichment ratio decreased since the amount of surfactant in the bulk liquid was larger. Further increase in surfactant concentration will significantly slow down the rate of drainage of the foam. As a consequence, liquid-keeping was increased, directly decreasing the enrichment ratio.

3 Conclusions

Spectrophotometric methods were used to investigate the interaction between the cationic dye and anionic surfactant with or without the co-existence of heavy metal ions. The spontaneity of the ion pair formation process was

confirmed by the absorbance values. In general, we found that the electrostatic and hydrophobic interaction between dye and surfactant depended strongly on surfactant's concentration. However, the addition of Cd²⁺ ions hardly imposed any significant influence on the dye spectrum. It represented that the electrostatic interaction between SDS and Cd²⁺ did not inhibit a formation of MB-SDS complex. A comparative study done on foam properties revealed that the presence of a small fraction of Cd²⁺ in the SDS system made a significant melioration in foam performance, just opposite to MB. Furthermore, experiment study was conducted with a continuous foam fractionator. In conclusion, foam fractionation is feasible to remove organic dye and heavy metal simultaneously by the anionic surfactants, i.e. SDS, with the negligible dosage of surfactants. The experimental results obtained in the present study would facilitate the design and optimization of surfactant concentrations for their better performance in foam fractionation.

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