Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2019.23873

151 (2019) 264–272
May

Foam separation based Cr^{3+} collection optimization using cocamidopropyl betaine

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Received 22 August 2018; Accepted 27 January 2019

ABSTRACT

This study focuses on the application of cocamidopropyl betaine (CAPB) as a natural surfactant in the foam separation for recovery of Cr^{3+} ions. First, the critical micelle concentration, foaming capacity and stability of CAPB and other four kinds of chemical surfactants were compared, and the advantages of CAPB as foaming agent were demonstrated. Second, the effects of pH value, gas velocity, liquid loading volume, collection time and initial concentration of Cr^{3+} ion and CAPB concentration on the recovery percentage and enrichment ratio of Cr^{3+} were determined. Finally, an orthogonal test was designed to optimize the best harvest conditions for Cr^{3+} ion in foam separation process and the results are as follows: pH = 8, airflow rate of 150 mL/min, liquid volume of 150 mL, CAPB concentration of 0.19 g/L, Cr^{3+} concentration of 4 mg/L, collection time of 15 min. At this condition, the recovery rate of Cr^{3+} ion can reach to 89.64% and the enrichment ratio is about 5.28. And the influence of four parameters of foam separation to Cr^{3+} recovery is pH > airflow rate > liquid loading volume > CAPB concentration.

Keywords: Foam separation; Cr^{3+}; Cocamidopropyl betaine; Surfactants; Optimum conditions

1. Introduction

Normally, the toxicity of chemical elements depends on their valence states. As all other oxidation states are unstable in aqueous systems, chromium has either +3 or +6 oxidation states. If exposed to overdosed Cr, the standard of which are set by US EPA, the probability of cancer development is one per 10,000 of total exposed people, even though analyzes showed that more than 60% of the total chromium seems to be in less toxic Cr^{3+} forms[1,2]. Cr^{3+} is 0.1%–1% less toxic to biology than Cr^{6+} which is more readily transported in soils compared to Cr^{3+}. Besides, Cr^{3+} is an essential trace element for human, plant and animal metabolism [3]. In China, the national emission standard of Cr^{3+} in industrial sewage is 1–1.5 mg/L [4]. At present, there are many methods to deal with Cr^{3+} wastewater such as flocculation, chemical precipitation, electrolysis, nano-filtration membrane separation, ion exchange and so on [5–7].

However, those techniques have cumbersome equipment, complicated steps, high-cost and the possibility of second-contaminated. Thus, it is necessary to study an effective and environmentally-friendly separation means. Foam separation, as a new environmental protection separation technology, is suitable for the treatment of low concentration solution, and has the properties of high extraction rate, continuous operation, low investment energy consumption and high industrial application value [8]. Its principle is based on enriching or depleting surfactant solutions by adsorption onto a rising gas liquid interface as foam. In separation, gas is used to be injected into a solution

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and surfactant molecules adsorb onto the bubble surfaces because of favorable thermodynamics. Surfactant molecules aim to lower the surface tension between the gas phase and liquid phase [9], with the abilities of foaming, defoaming, emulsifying, antistatic and sterilizing [10]. In Wang's research [11], foam separation process was used to treat wastewater containing Cr^{3+}. The optimal conditions were determined and the cationic surfactant was applied. The results were: pH = 6.5, gas speed 400 ml/min and the dosage of the cationic surfactant were 50 mg/ml. The outcomes of continuous tests on a foam column showed that the Cr^{3+} can meet the national level-two wastewater discharge standard. Matsuoka [12] studied the removal of alkali metal ions (Li, K, Rb, Cs) by foam separation method with a kind of anionic surfactant (SDS). With the increase of ion radius, the removal rate grew (Li < K < Rb < Cs). This studied also proved that foam flotation could be an effective method to remove hazardous ions selectively form industry waste. Wang [13] used DSBA as a surfactant to separate Cu^{2+} and crystal violet by foam separation method. The optimal parameters of pH, superficial velocity and foam height were confirmed to achieve the best enrichment ratio (3.7 for Cu^{2+} and 3.6 for crystal violet) and remove percentage (96.5% for Cu^{2+} and 96.3% for crystal violet).

Cocamidopropyl betaine (CAPB), known as coconut oil foaming agent, is a compound gently extracted from mature coconut, without heating and chemical refining natural substances, and is an excellent nonionic surfactant [14]. It is also an amphoteric synthetic detergent which has been increasingly used in cosmetics and personal hygiene products [15]. Utilizing natural nontoxic foaming agent such as CAPB to carry on studying on separating heavy metals can avoid adding other hazardous chemical substances into the solution [16].

In present study, CAPB was used as foaming agent and trapping agent to explore the effects of various factors including the pH, airflow rate, loading volume, concentration of CAPB and collecting time on foam separation and recovery of Cr^{3+}. Based on this, the recycling conditions of natural foaming agent coconut oil extract for Cr^{3+} were optimized, which provided important experimental basis for the effective separation of Cr^{3+} in wastewater.

2. Material and methods

2.1. Equipment and reagents

The foam column was made of polymethyl methacrylate tube with the length of 1.5 m and inner diameter of 50 mm and a schematic diagram of foam separation experimental device diagram is illustrated in Fig. 1. Six surfactants including CAPB, cetrimonium bromide (CTAB), sodium dodecyl sulfate (DBS), sodium dodecyl benzene sulfonate (SDBS), 10-decyl polyglycoside (C10APG) were used to compare their surface tensions and foaming stabilities. At the same time, pH value can be regulated by sodium hydroxide and hydrochloric to make a series of pH gradients. Also, chromic sulfate was regarded as material which needs to be separated.

2.2. Experiment procedures

2.2.1. Foaming ability and foaming stability test

The foaming ability and foam stability testing device is shown in Fig. 1. For each test, one of five kinds of surfactant was poured into a glass tube, with the concentration of 0.1 g/L and total volume of 200 mL. Then, open air compressed bottled, regulate the pressure and flow meter into an appropriate value to make sure the airflow rate was 100 mL/min. Recording the first 1 min and measure its initial foam height, which can be regarded as a significant ability evaluation. After that, record the height of foam in 0.5, 1, 1.5, 2, 2.5, 3 and 3.5 min respectively, which, then divided by initial foam height, were actual degrees of foaming ability and stability. Each experiment was repeated at the same temperature for three times, after which mean values were obtained.

2.2.2. Calculation of Cr^{3+} separation efficiency

Nonionic surfactant CAPB worked as foaming agent as well as Cr^{3+} trapping agent. In alkaline conditions, $Cr(OH)_3$ generated by Cr^{3+} ion binding with OH^- adsorbed on the gas-liquid interface. After concentrating in foam phase,

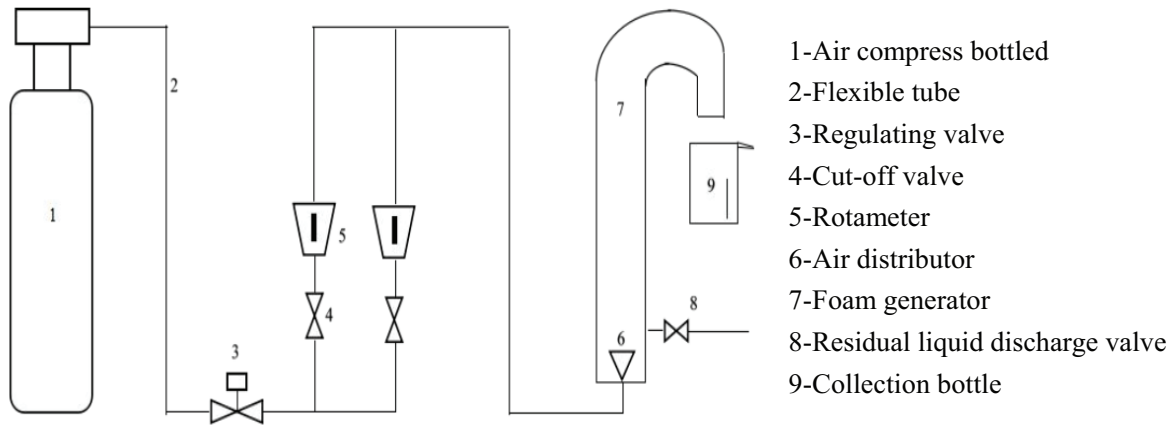

Fig. 1. Schematic diagram of foam separation experimental device diagram.

precipitate discharged with airflow, concentrating in foam phase, achieving recovery and separation progress [17]. Eqs. (1) and (2) indicate the enrichment ratio and recovery percentage respectively and are listed as follows [18]:

$$\text{Enrichment ratio-}(E) = \frac{C_f}{C_0} \tag{1}$$

$$\text{Recovery percentage-}(R) = \frac{C_f \times V_f}{C_0 \times V_0} \tag{2}$$

Note that C_0 and C_f represent the concentration of Cr^{3+} in raw material solutions and collected foam liquids, V_0 and V_f indicate the total volume of raw material solutions and collected foam liquids (deformed).

2.2.3. Cr^{3+} analysis methods

The AA-6860 type flame atomic absorption spectrophotometer was used to measure the concentration of trace Cr^{3+} in the range of 0–2.5 mg/L. Flame atomic absorption spectrophotometric utilizes air-acetylene to determine metal elements with the precision of approximate 1%. The heating elements are atomized in flame atomizer to a ground state atomic vapor, which can absorb the characteristic radiation transmitted by hollow cathode lamp selectively. Within a certain range of concentration, the absorption intensity is proportional to the content of elements in solution. After foam collected for a certain period of time, residual liquid was sampled from the bottom of the column. All test samples were required to be filtered in advance before using the atomic absorption spectrophotometric to determine their concentrations. However, the concentrations of Cr^{3+} foam at the top of column were calculated through the material balance method.

2.2.4. Surface tension test

Surface tension is a considerably important characteristic of the surfactant, representing surface activity of the surfactant. While, the critical micelle concentration (CMC) is the exact concentration when micelles form, above which monomers and micelles form in dynamic equilibrium [19]. Before reaching the CMC, then surface tension alters strongly with the concentration of the surfactant. However, after arriving the CMC, the surface tension remains constantly or changes slightly. Generally, by measuring the surface tension of a concentration series through a tensiometer, the CMC is determined [20,21]. First, the solution of five kinds of surfactants was prepared in 6–7 concentration gradients for each surfactant, making sure the theoretical CMC was included. Next, use surface tension meter to measure all the surface tension of solutions, each surfactant gradient was tested tripled. After all results obtained, the average value of surface tensions and the average temperatures were calculated. Eventually, the graph of the numerical number of average surface tension versus average surface tension was plotted, from which CMC values of every surfactant were easily observed.

2.2.5. Single factor experimental procedures

(1) Prepare a certain concentration of six hydrated chromium sulfate ($Cr_2(SO_4)_3 \cdot 6H_2O$) solution and CAPB solution, mixed thoroughly before use.
(2) Using 6 and 1 mol/L NaOH solution, 6 and 1 mol/L HCI solution to make desirable pH value.
(3) Certain volume of mixed solution was poured into the air float glass tube shown in Fig. 1, and opens the air valve. Then, adjust the airflow rate according to the experimental conditions, and use the beaker to collect foam samples.
(4) After collecting samples for a period of time, open the discharge valve to collect about 8 mL residual liquid for Cr^{3+} concentration measurement and measure the amount of residual liquid volume. The collected condensed foam need to be sealed by plastic film, defoamed for overnight. At the next day, measure the liquid volume. Each experiment was done triple and then error analyzes were handled.
(5) The residual liquid was detected by atomic absorption spectrophotometer, and the content of Cr^{3+} can be recorded.
(6) According to the data obtained, the values of recovery percentage (R) and enrichment ratio (E) were calculated and plotted as well as the effect level vs. foam volume, residual liquid volume as well as residual liquid concentration.

2.2.6. Orthogonal experiments

After determining the collecting time and the investigated Cr^{3+}concentration, select several points approximating the best pH value, the airflow rate, the liquid volume and CAPB concentration, on which the orthogonal table of four factors and three levels was based. Finally, through intuitive analysis of experiment results, the degree of influence of Cr^{3+} to the optimum technological conditions were obtained and verified.

3. Results and discussion

3.1. Surface tension and CMC of surfactants

Five surfactants' interfacial tension results as shown in Fig. 2(a) as well as five types of surfactant CMC values and their corresponding interfacial tension are illustrated in Table 1, from which the conclusion can be drawn that the surface tension reducing ability of five materials were C10APG > CAPB > SDBS > CTAB > DBS. Generally, surface tension reducing ability closely related to foaming properties. Compared with other nonionic surfactants (C10APG), the surface tension reducing ability of nonionic surfactant CAPB was considerably weaker, while better than chemical ionic experiment groups, with much lower concentration in need. This indicated great advantages of CAPB in the foam ability and using amount. In addition, when the nonionic surfactant dissolved in water, it cannot become disassociated, thus having a high hydrophobicity [22], which correlates to its CMC tested value [23]. Such kind of surfactant can effectively reduce the volume of foam liquid, at the same time, improve the enrichment ratio under the required recovery percentage.

3.2. Foaming ability and foaming stability comparison

As illustrated in Fig. 2(b), the comparison of the foaming abilities of five kinds of surfactant was C10APG > CAPB > SDBS > CTAB >DBS, which in accordance with the surface tension reducing capacity. However, they formed diverse final foam heights, due to variable foaming stabilities. During the first 3.5 min, the heights of CAPB and CTAB increased linearly, with the appearance of bubble growth trend, meaning the stability of CAPB and CTAB were the best. For SDBS and C10APG, their data were slightly unstable in the last 1 min but the overall characters were good. While the foaming capability of DBS compared with the previous surfactants was significantly worst, and its rising speed became slightly slower than former at 1.5 min, with an unexpected de-foaming phenomenon.

3.3. Single factor effect in Cr^{3+} collection process

3.3.1. pH

First, the experiment of foam separation and recovery of Cr^{3+} ion was carried out when the concentration of CAPB in solution was 0.1 g/L, the concentration of Cr (III) was 3 mg/L, the volume of liquid was 200 mL, the airflow rate was 300 mL/min, and the collecting time was 20 min, the solution pH was adjusted to 1, 3, 5, 7, 9, 11, 13, respectively.

The results (Figs. 3(a) and (b)) illustrated that when pH was equal to 9, Cr^{3+} recovery rate was up to 68.1%, while the enrichment ratio ranking highest when pH was equal to 7, 8.64. While pH was 9, the Cr^{3+} concentration in residual liquid was less than 1 mg/L. This conclusion correlated to Cr^{3+} ion charge properties. Within alkaline range, $Cr(OH)_3$ precipitated massively, which, in some degree, made contributions to Cr^{3+} adsorption on the bubble surface. By continuously bubbling air into the solution, it eventually discharged with the bubble flow [13]. Thus, in this pH value (pH equal to 9), even though CAPB would carry a large amount of water to leave, resulting in a light decrease in enrichment ratio, it was still an optimal condition in the single factor experiment, considering both effects of enrichment and recovery process.

Table 1
Interfacial tension and CMC of five surfactants

Number	Type of surfactant	Interfacial tension (mN/m)	CMC (g/L)
1	C10APG	30.155	0.4
2	SDBS	35.222	0.45
3	DBS	48.737	1.0
4	CTAB	39.194	1.5
5	CAPB	31.812	0.20

3.3.2. Airflow rate

Second, the experiment was conducted while the concentration of CAPB in solution was 0.1 g/L, concentration of Cr (III) was 3 mg/L, the volume of loading volume was equal to 200 mL, pH was 9, and the collecting time was equal to 20 min, the adjusted airflow rate was 50, 100, 150, 200, 300, 400, 500, 600, and 700 mL/min.

Cr^{3+} enrichment ratio dropped along with the increase of airflow rate, reaching the highest value of 12.45 at minimum gas velocity (50 mL/min). After changing the airflow rate at 150 mL/min, the recovery rate decreased from its maximum data of 76.33%. The reason why enrichment ratio changed reversely as the changing of airflow rate is that while, the foam speed rose faster, the foam phase drainage velocity decreased slower, leading to the water content between the gap in bubble phase going up, the collected liquid volume increased, so that the Cr^{3+} enrichment ratio decreased gradually. And, in low gas velocity, CAPB produced relatively small bubble, resulting in foam layer unstable and lax, so that Cr^{3+} cannot effectively be taken away from the foam solution phase, leading to a considerably small recovery rate. When gas velocity reached a certain value, foam produced by CAPB started to be thick and have large specific surface, separating Cr^{3+} efficiently. But if the gas velocity increased continuously, the diameter of bubble became larger, the specific surface reduced, the resident time was short, making Cr^{3+} cannot be

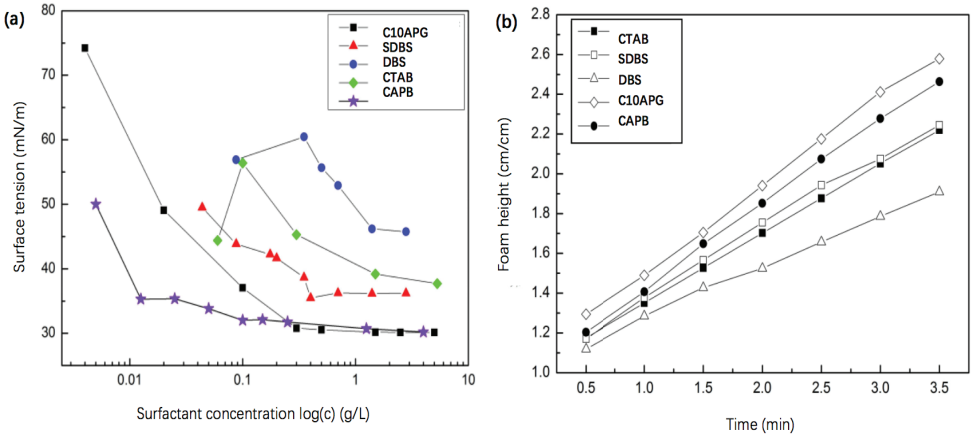

Fig. 2. (a) Different concentrations of the corresponding surface tension of five surfactants; and (b) Foaming ability and stability of five surfactants.

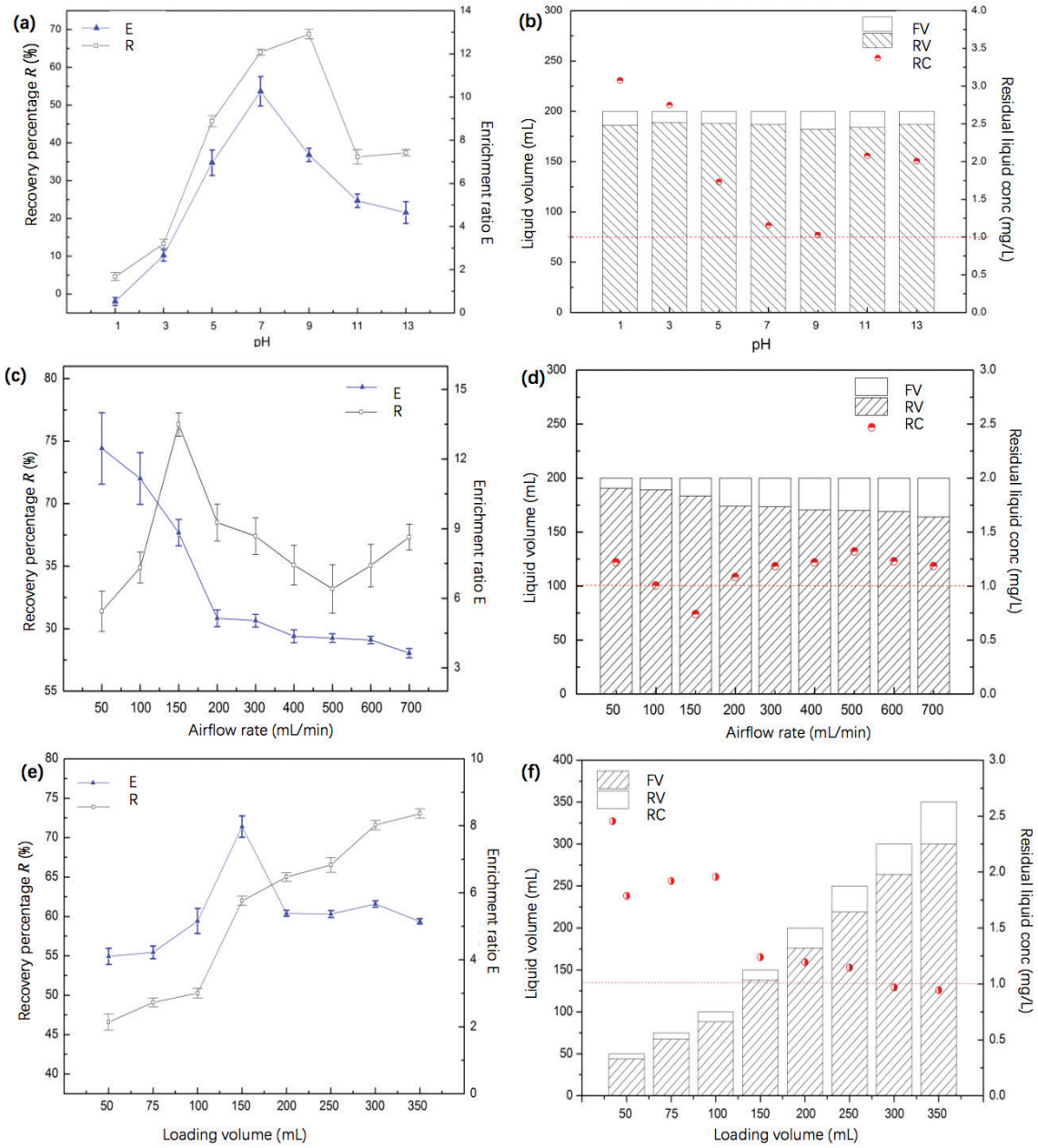

Fig. 3. (a), (c), (e) respectively represent the effects of pH, airflow rate, loading volume on recovery percentage and enrichment ratio; (b), (d), (f) separately suggest the effects of pH, airflow rate, loading volume on separation efficiency and the volume of foam. FV, RV and RC represent for foam volume, residual liquid volume as well as residual liquid concentration, respectively.

timely carried out. Overall, the single factor optimal airflow rate was 150 mL/min. The experimental results are shown in Figs. 3(c) and (d).

3.3.3. Loading volume

Third, experiment was carried out at 0.1 g/L of CAPB, 3 mg/L of Cr (III), 300 ml/min of airflow rate, pH at 9, 20 min of collecting time, and the loading volume of 50, 75, 100, 150, 200, 250, 300, and 350 mL.

The loading volume had a great impact on both the recovery rate of Cr^{3+} and the enrichment ratio, recovery rate peaked at 72.56% when loading volume was 350 mL, while the liquid volume is 150 mL, the enrichment ratio arrived maximum (7.93). The reason may because in the small loading volume, the height of liquid level was low, the residence time of air bubbles remaining in the liquid phase was relatively short, the gas phase and liquid phase did not contact thoroughly. So that the Cr^{3+} ion cannot be effectively adsorbed, making recovery efficiency declined. As for enrichment ratio, when the liquid volume becomes larger than a certain value, a large number of Cr^{3+} adsorbed on the bubble by two phases fully contacting. At the same time, enrichment specific value deteriorated due to water carried

out by a lot of air. Put two sides into consideration, the best loading volume was designed to be 150 mL. The results are shown in Figs. 3(e) and (f).

3.3.4. Concentration of CAPB

Next, respectively make the concentrations of CAPB were 0.05, 0.075, 0.1, 0.125, 0.15, 0.175, and 0.2 g/L, at the same time, keep the Cr (III) concentration 3 mg/L, the airflow rate 300 mL/min, the loading volume 200 mL, the pH at 9, and the collecting time 20 min.

As indicated in Figs. 4(a) and (b), Cr^{3+} recovery percentage increased first and then decreased with the ascending of CAPB concentration, while enrichment ratio came down gradually. So that, at the CAPB concentration of 0.175 g/L, the maximum recovery rate of 82.21% reached, however, the highest enrichment ratio was 11.66 under the 0.05 g/L of CAPB. This was mainly because with the increasing of CAPB

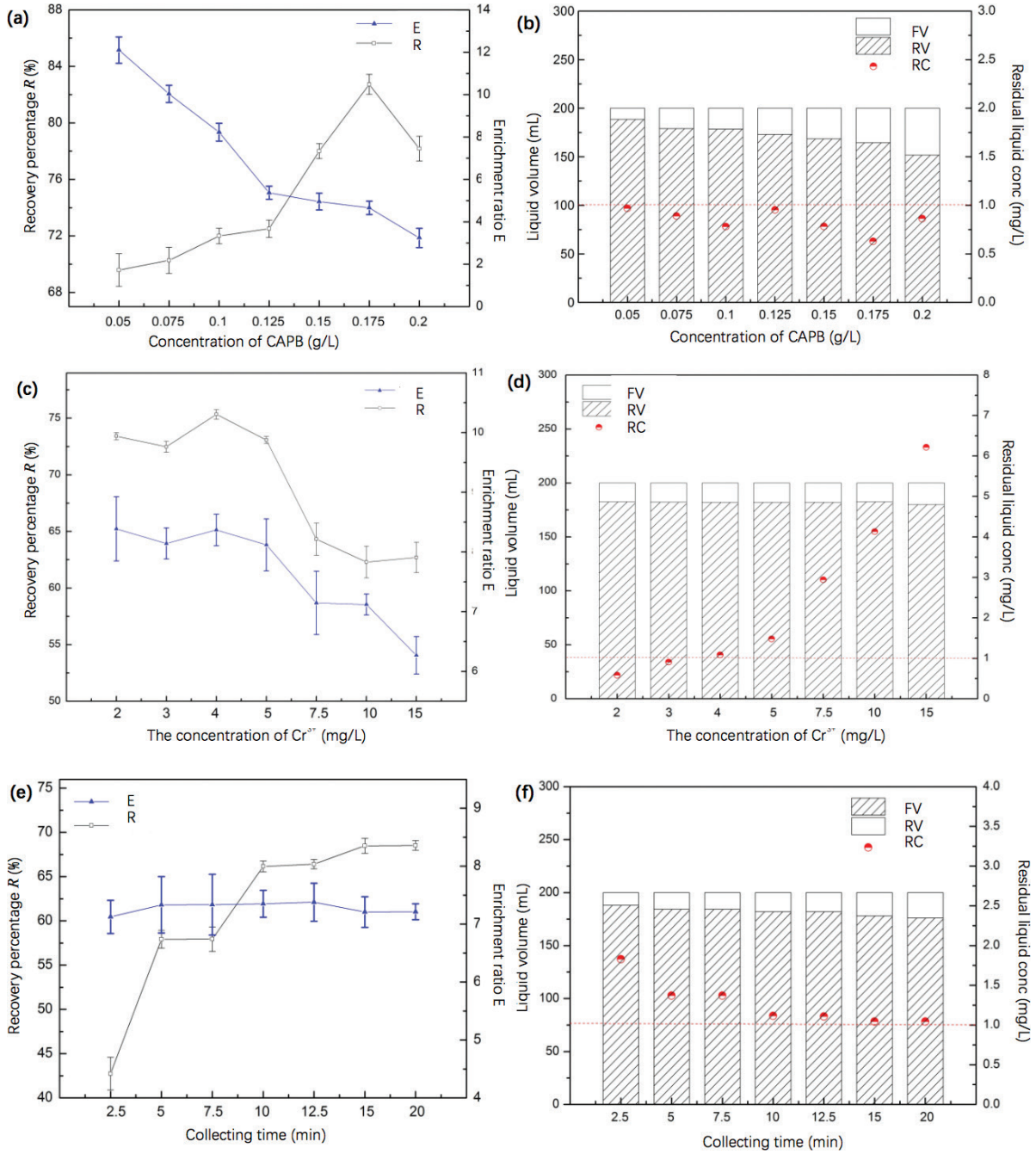

Fig. 4. (a), (c), (e) respectively represent the effects of CAPB concentration, initial concentration of Cr^{3+}, collecting time on recovery percentage and enrichment ratio; (b), (d), (f) separately suggest the effects of CAPB concentration, initial concentration of Cr^{3+} and collecting time on separation efficiency and the volume of foam. FV, RV and RC represent for foam volume, residual liquid volume as well as residual liquid concentration, respectively.

concentration, foam amount and stability increased as well, removing more and more Cr^{3+}ions. As the concentration of CAPB approaching the CMC of 0.2 g/L, CAPB easily reunited and cannot combine with Cr^{3+}, thus declining recovery efficiency. In addition, due to the increase of CAPB concentration can produce a large amount of foam to clip away more water, so the enrichment ratio continuously dropped. Considering comprehensively the effects of CAPB concentration on both Cr^{3+} enrichment ratio and recovery percentage, the optimal conditions of single factor CAPB decided to be 0.175 g/L.

3.3.5. Initial concentration of Cr^{3+}

Then, the Cr^{3+} ion experiment of foam separation and recovery under the situation while the concentration of CAPB in solution was 0.1 g/L, the airflow rate was 300 mL/min, the loading volume was 200 mL, the pH was 9, the collection time was equal to 20 min, the concentrations of Cr (III) were 2, 3, 4, 5, 7.5, 10, and 15 mg/L.

As shown in Figs. 4(c) and (e), with the growing of the initial Cr^{3+} concentration, the recovery percentage and enrichment ratio were gradually decreased. However, there was slight difference between them before the Cr^{3+} concentration was less than 7.5 mg/l. When the Cr^{3+} concentration was 4 mg/L, there was the maximum recovery rate of 75.27% as well as the enrichment ratio of 8.09. That was to say, the proportion of Cr^{3+} uniting with CAPB was significantly stable. If the molar amount of Cr^{3+} was much more than the limited binding number CAPB as high Cr^{3+} concentration, recovery value came down. At the same time, enrichment ratio declining in high Cr^{3+} concentration can be explained by constant foam volume. Above all, the concentration of Cr^{3+} in single factor optimal conditions decided in 4 mg/L.

3.3.6. Loading volume

Finally, when the concentration of solution Cr (III) was 3 mg/L, CAPB concentration was 0.1 g/L, airflow rate was 300 ml/min, loading volume was 200 mL, pH was 9, the collecting time was 2.5, 5, 7.5, 10, 12.5, 15 and 20 min, the process of foam separation and recovery of Cr^{3+} was carried out.

With the increasing of collecting time, recovery rate of Cr^{3+} reached 69.12% in the 20 min, while the changes of enrichment ratio maintained at between 6.4–6.9. With the development of separation process conducted longer, the more foam collected, so more Cr^{3+} was taken out of. At approximate 15–20 min, when the bubble basically no longer produced, the recovery rate changed slightly. Enrichment ratio fundamentally maintaining at a certain level on behalf of CAPB binding with Cr^{3+} ratio for a fixed value. Thus, the single factor experiment optimal collecting time was 15 min. Details are shown in Figs. 4(e) and (f).

The single factor experiment results were as follows: pH = 9, airflow rate 150 mL/min, loading volume 250 mL, CAPB concentration of 0.175 g/L, Cr^{3+} concentration of 4 mg/L, collecting time of 15 min. After verification, the recovery rate of Cr^{3+} ion can reach to 78.46%, the enrichment ratio was about 4.35, and residual liquid concentration was 0.788 mg/L.

3.4. Orthogonal experiments

The results were obtained by the single factor experiment, and the orthogonal test plan was designed by $L_9 3^4$ (in Table 2) and the detail results (in Table 3). The recovery rates in Table 3 were statistically analyzed, the results can be seen in Fig. 5.

Table 2
Factors and levels of orthogonal experiments

Code	pH	Air flow rate (mL/min)	Loading volume (mL)	Concentration of CAPB (g/L)
1	8	125	175	0.16
2	8.5	150	200	0.175
3	9	175	225	0.19

Table 3
Plan of orthogonal experiments

Factor	pH	Air flow rate (ml/min)	Loading volume (ml)	Concentration of CAPB (mg/L)	Recovery percentage (*R*%)
1	8	125	175	0.16	81.95
2	8	150	200	0.175	89.98
3	8	175	225	0.19	80.02
4	8.5	125	200	0.19	76.09
5	8.5	150	225	0.16	77.43
6	8.5	175	175	0.175	66.73
7	9	125	225	0.175	71.15
8	9	150	175	0.19	74.78
9	9	175	200	0.16	71.51
*K*1	83.983	76.597	74.487	74.963	
*K*2	73.417	80.73	79.193	76.153	
*K*3	72.680	72.753	76.4	76.963	
R	11.303	7.977	4.706	0.81	

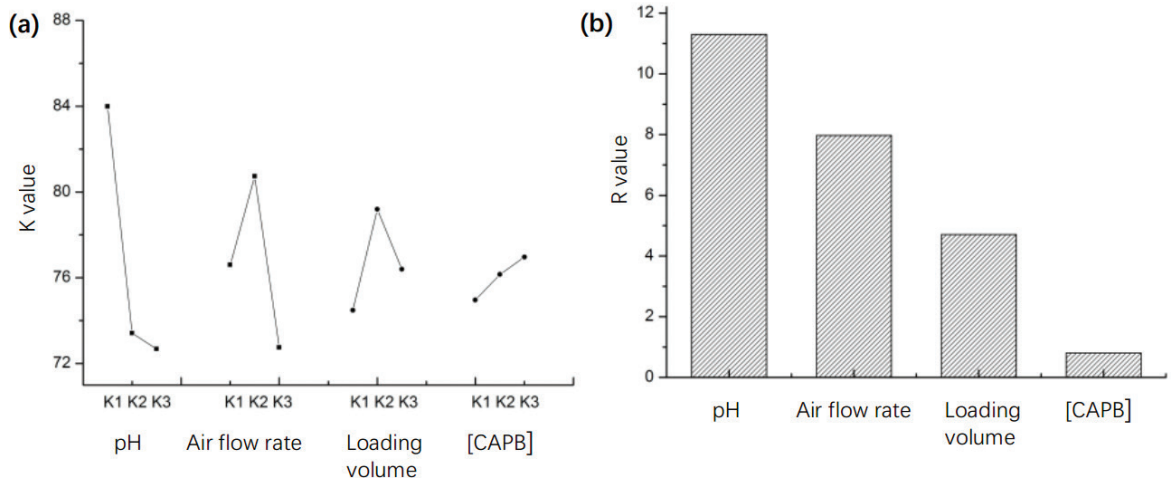

Fig. 5. Result analyzes of orthogonal experiments.

The distribution of *K* value and *R* value are shown in Fig. 5(a) and (b), respectively where *K*1, *K*2 and *K*3 represent the mean value of the experimental results corresponding to the equivalent levels of each factor, and *R* represents the extreme difference of each horizontal mean level.

As drove in the *K* value distribution, the optimal experimental conditions were pH = 8, airflow rate 150 mL/min, loading volume of 200 mL, the concentration of CAPB of 0.19 g/L. In this situation, the recovery rate of Cr^{3+}, the enrichment ratio and the residual liquid concentration were 89.98%, 5.29, as well as 0.483 mg/L respectively. Triple the process under optimal technology condition, finally conclude the average recovery rate was 89.64%, the concentration ratio was 5.28, and the residual liquid concentration was 0.499 mg/L. It was a remarkable improvement when compared with the results of single factor experiment in which the recovery rate and enrichment ratio was 78.46% and 4.35 respectively. By the *R* value distribution, the influence of four parameters of foam separation on Cr^{3+} recovery was pH > airflow rate > liquid loading volume > CAPB concentration.

4. Conclusion and future work

This paper explored the feasibility of CAPB as a natural surfactant for foam separation on recovery of trace Cr^{3+} ions. Eventually, the results are as follows: pH = 8, airflow rate 150 mL/min, liquid volume 150 mL, CAPB concentration of 0.19 g/L, Cr^{3+} concentration of 4 mg/L, collecting time of 15 min. At this condition, the recovery rate of Cr^{3+} ion can reach to 89.64%, the enrichment ratio is about 5.28, and most importantly, residual liquid concentration is 0.499 mg/L, which is below industrial water discharge standard. Such result proves CAPB foam separation process reliable.

It is practical to combine natural surfactant as CAPB with foam separation, which can ensure the Cr^{3+} separation process environmental-friendly, safely, efficiently. In the future, research in foam separation technology for the recovery of other substances needs more exploration.

Acknowledgements

The authors gratefully acknowledge the financial support by Fundamental Research Funds for Technology Planning Project of Xiamen City, China (No. 3502Z20183016), the Central Universities (No. 20720170027), National Natural Science Foundation Youth Fund of China (No. 21406185) and National Natural Science Foundation of China (No.21736009).

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