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Bing Han[a]; Zhaoliang Wu[a]; Hao Yin[a]; Tianshui Feng[a]
[a] Department of Bioengineering, Hebei University of Technology, Dingzi Gu, Hongqiao District, Tianjin, China

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Study on Streptomycin Sulfate Recovery by Batch Foam Separation

Bing Han, Zhaoliang Wu, Hao Yin, and Tianshui Feng
Department of Bioengineering, Hebei University of Technology, Dingzi Gu, Hongqiao District, Tianjin, China

The feasibility of foam separation as a technique was assessed for the recovery of streptomycin sulfate from the waste solution by using an anionic surfactant sodium dodecyl sulfate (SDS). The experimental parameters examined were SDS concentration, superficial gas velocity, initial pH, and liquid loading volume. The results showed that sodium dodecyl sulfate as the surfactant for foam separation had good foaming quality and could effectively concentrate streptomycin sulfate of the aqueous solution by technology of foam separation. The enrichment ratio and the recovery rate of streptomycin sulfate were 4.0 and 85%, respectively under the best operating conditions of sodium dodecyl sulfate concentration 0.4 g/L, superficial gas velocity 300 mL/min, liquid loading volume 300 mL and initial pH 6.0 when streptomycin sulfate concentration was 0.5 g/L.

Keywords foam separation; sodium dodecyl sulfate; streptomycin sulfate

INTRODUCTION

Streptomycin is an important antibiotic. It belongs to glucosamine-type and is applied to the medical industry. In recent years, its production in China has reached more than 1,000 tons per year (1). At present, the separation of streptomycin from the fermentation broth has been carried out by ion exchange (2), which contains adsorption, desorption, and regeneration of resin, So it causes losses of streptomycin dissolved in water with low concentration and at the same time leads to a large amount of wastewater, which is treated with difficulty.

Foam separation is a simple, low-cost, and environmentally-friendly method. It especially adapts to the enrichment of low-concentration solution and is used initially in mineral flotation and wastewater treatment (3–7). In recent years, it has been applied in bioseparation engineering, including the separation of proteins and vitamins, and desalination (8–14). Therefore in this paper, concentration of streptomycin sulfate will be studied by foam separation of aqueous streptomycin sulfate solution. Our aim is to assess the feasibility of foam separation as a technique for recovery and concentration of streptomycin sulfate from the waste solution.

In the process of ion-exchange, the adsorption quantity (Q) increases with the increase of the initial concentration (C_0) or the equilibrium concentration (C) of streptomycin sulfate. The reciprocals of C and Q have a good linear relationship, that is, its adsorption isotherm agrees with the Langmiur isotherm. The equation is $1/Q = 0.0103\,C^{-1} + 0.0008$ (15). The concentration of streptomycin sulfate in the waste solution is about 0.5 g/L and it will become higher through the foam separation. So the higher concentration solution can be applied to ion-exchange effectively and improve the separation efficiency.

The anionic surfactant sodium dodecyl sulfate [$CH_3(CH_2)_{11}OSO_3Na$] was used in this experiment and it could be ionized into $CH_3(CH_2)_{11}OSO_3^-$ and Na^+. Streptomycin sulfate could be ionized into $C_{21}H_{38}N_7O_{11}^{3+}$ and SO_4^{2-} in aqueous solution. When the foam separation began, $C_{21}H_{38}N_7O_{11}^{3+}$ combined with $CH_3(CH_2)_{11}OSO_3^-$ and formed the complex $[CH_3(CH_2)_{11}OSO_3^-]_3C_{21}H_{38}N_7O_{11}^{3+}$, which was absorbed to the interface of the gas-liquid and concentrated in the foam phase. The reaction could be written as:

$$3CH_3(CH_2)_{11}OSO_3^- + C_{21}H_{38}N_7O_{11}^{3+} \leftrightarrow [CH_3(CH_2)_{11}OSO_3]_3C_{21}H_{38}N_7O_{11}$$

For ion exchange, the weak acid cation resin (RCOONa) exchanged with the complex and adsorbed streptomycin. At the same time, the surfactant was regenerated. Streptomycin sulfate was desorbed with sulphuric acid in next process. Their reactions could be written as:

$$[CH_3(CH_2)_{11}OSO_3]_3C_{21}H_{38}N_7O_{11} + 3RCOONa \rightarrow (RCOO)_3C_{21}H_{38}N_7O_{11} + 3CH_3(CH_2)_{11}OSO_3Na\,2(RCOO)_3C_{21}H_{38}N_7O_{11} + 3H_2SO_4 \rightarrow 6RCOOH + (C_{21}H_{38}N_7O_{11})_2(SO_4)_3$$

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Address correspondence to Zhaoliang Wu, Department of Bioengineering, Hebei University of Technology, No. 8 Guangrong Road, Dingzi Gu, Tianjin, Hongqiao District 300130, China. E-mail: zhaoliangwu@163.com

MATERIALS AND METHODS

Materials

The materials used were all analytical grade and included streptomycin sulfate (obtained from Hebei Shengxue Dacheng Pharmaceutical Co., Ltd.), sodium dodecyl sulfate (SDS), sulphuric acid and sodium hydroxide (obtained from Tianjin Chemical Reagents Co., Ltd.), ammonium ferric sulfate (obtained from North Chemical Reagents Co., Ltd.). All of the above materials were used without further purification. Distilled water was used in all experiments.

Instruments

The instruments used included UV-spectrophotometer (752, Shanghai Exactitude Apparatus Company, China), pH meter (PHS-25, Shanghai Jingke Industrial Co., Ltd., China), rotameter (LZB-3WB, Tianjin meter factory, China), and air compressor (AC0-318, Tianjin Meter Factory, China).

Methods

Foam separation column was self-made. It was 800 mm in height and 40 mm in inside diameter and a porous gas distributor with a diameter of 20 μm was at the bottom.

All experiments were carried out at room temperature (25°C). Streptomycin sulfate solution mixed with sodium dodecyl sulfate was loaded into the column at the beginning of each experiment and foam was then generated by adjusting the superficial gas velocity. The experiments were run until foam ceased flowing from the outlet of the column.

At the end of the experiments, streptomycin sulfate concentration in the collapsed foam solution and the residual solution in the column were measured (16). The maximum absorption peak of streptomycin sulfate was at 220 nm and a good linear in the range from 12 mg/L to 48 mg/L was found. The standard curve equation was $A = 0.01682c - 0.011$, $R = 0.9998$. The enrichment ratio (E) and the recovery rate (R) were used to characterize the foam separation.

$$E = \frac{C_f}{C_i}$$

$$R(\%) = \frac{C_i V_i - C_r V_r}{C_i V_i} \times 100\%$$

where C_i and C_r were the streptomycin sulfate concentrations in the initial and residual solution, respectively, and C_f was the streptomycin sulfate centration in the collapsed foam solution. V_i and V_r were the volume of the initial and residual solutions, respectively.

RESULTS AND DISCUSSION

Single Factor Experiments

Effect of SDS Concentrations

The effect of SDS concentrations from 0.1 to 0.5 g/L was conducted with streptomycin sulfate concentration 0.5 g/L, superficial gas velocity 100 mL/min, liquid loading volume 300 mL and initial pH 7.0. The results of effect of SDS concentrations on E and R were shown in Fig. 1.

The results of Fig. 1 showed that the recovery rate of streptomycin sulfate increased and the enrichment ratio decreased gradually with the increase of the SDS concentration. This could be explained by the following. With the increase of the SDS concentration, more streptomycin molecules could combine with $CH_3(CH_2)_{11}OSO_3^-$, be adsorbed on the bubbles and so the recovery rate of streptomycin sulfate increased when the concentration of the surfactant SDS was less than the critical micelle concentration (CMC) (17). The foam with a higher surfactant concentration was characterized by smaller and more stable bubbles (18) and had higher liquid holdup (19,20), and so the enrichment ratio of streptomycin decreased. Therefore, the appropriate concentration of SDS was 0.3 g/L.

Effect of Superficial Gas Velocity

SDS concentration and streptomycin sulfate concentration were 0.3 g/L and 0.5 g/L, respectively and the liquid loading volume was 300 mL and initial pH was 7.0. Superficial gas velocity was adjusted between 100 and 300 mL/min. The results of the effect of superficial gas velocity on E and R were shown in Fig. 2.

With the increase of the superficial gas velocity, the recovery rate of streptomycin sulfate increased and the enrichment ratio decreased gradually. When the superficial gas velocity was low, the bubbles rose slowly and had long residence time in the foam phase. There was enough time for the foam phase to discharge liquid and so the liquid holdup at the top of the column was small. Therefore,

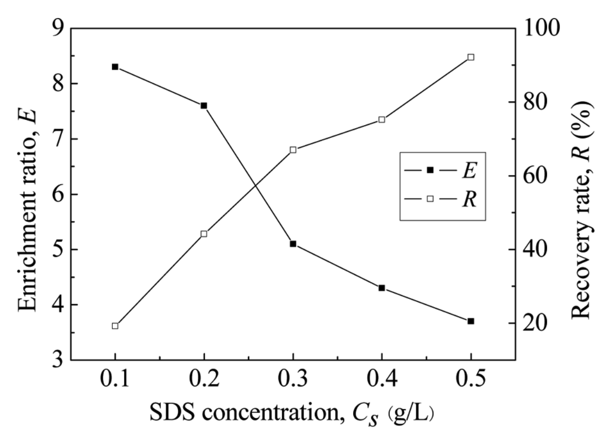

FIG. 1. Effect of SDS concentrations on *E* and *R*.

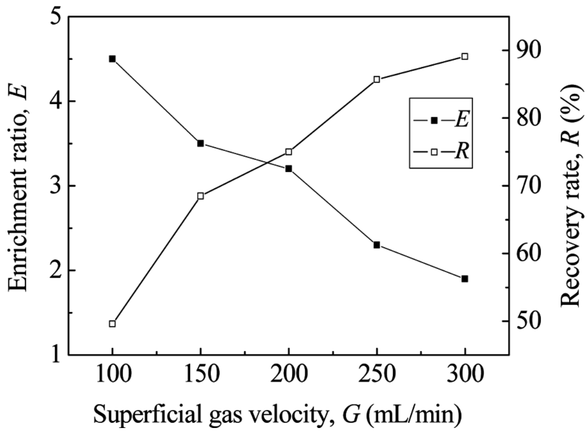

FIG. 2. Effect of superficial gas velocity on E and R.

the enrichment ratio of streptomycin sulfate increased. With the increase of superficial gas velocity, the bubble production would increase. The lower residual time of bubbles in the foam phase and the lower drainage velocity of the interstitial water caused by gravity had resulted in a higher content of water in foamate (9). So, the recovery rate increased and the enrichment ratio of streptomycin sulfate decreased. The appropriate superficial gas velocity was 200 mL/min.

Effect of Liquid Loading Volume

SDS concentration and streptomycin sulfate concentration were 0.3 g/L and 0.5 g/L, respectively, initial pH was 7.0 and superficial gas velocity was 200 mL/min. The liquid loading volume was changed between 100 and 500 mL. The results of the effect of the liquid loading volume on E and R were shown in Fig. 3.

As the liquid loading volume increased, the recovery rate, and the enrichment ratio of streptomycin sulfate changed conversely. When the liquid loading volume was small, the height of the foam phase was high and an increase in the foam height led to a longer foam residence time, which allowed more drainage of the liquid in the films (21). So the enrichment ratio of streptomycin sulfate was higher because of the dryer foam. Large liquid loading volume led to the foam's short time staying in the foam phase and a large amount of the foamate, and so the recovery rate increased with the increase of the liquid loading volume. The appropriate liquid loading volume was 300 mL.

Effect of Initial pH

SDS concentration and streptomycin sulfate concentration were 0.3 and 0.5 g/L, respectively, the liquid loading volume and superficial gas velocity were 300 mL and 200 mL/min, respectively. The initial pH of the solution was adjusted between 5.0 and 7.0. The results of effect of initial pH on E and R were shown in Fig. 4.

Figure 4 showed that with the increase of the initial pH, the enrichment ratio and the recovery rate all increased first and then decreased. At $pH > 6.0$, the positive charge density of streptomycin sulfate decreased gradually with an increase of the initial pH, subsequently less amount of streptomycin–surfactant complex was formed, so the enrichment ratio and the recovery ratio decreased. At $pH \leq 6.0$, a larger fraction of streptomycin^{3+} was available, SDS tended to complex with streptomycin^{2+} and not to streptomycin^{3+}, so the enrichment ratio and the recovery ratio decreased sharply (11). In addition, the competitive interaction of SDS by a great deal of H^+ with streptomycin^{3+} and streptomycin^{2+} at lower pH could be used to explain this phenomenon. Therefore, the appropriate initial pH was 6.0.

Orthogonal Experiments

Methods

Four factors were selected (A, initial pH values; B, SDS concentration Cs; C, gas flow rate G; D, liquid loading

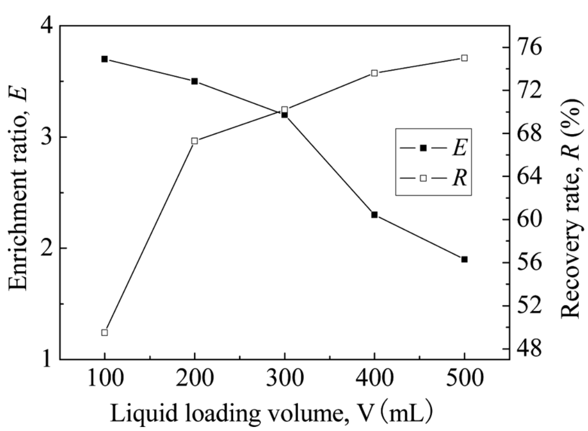

FIG. 3. Effect of liquid loading volume on E and R.

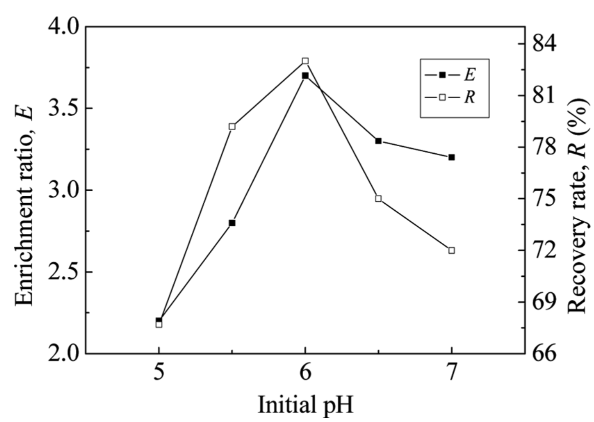

FIG. 4. Effect of initial pH on E and R.

TABLE 1
Factors and levels of orthogonal experiments

	Factor			
Level	A, pH	B, C_s (g/L)	C, G (mL/min)	D, V (mL)
1	5.5	0.2	100	200
2	6.0	0.3	200	300
3	6.5	0.4	300	400

TABLE 2
Results of the orthogonal experiments

No.	A	B	C	D	R	E
1	1	1	1	1	72.5	2.7
2	1	2	2	2	73.3	2.8
3	1	3	3	3	75.0	3.5
4	2	1	2	3	86.0	3.7
5	2	2	3	1	84.3	4.1
6	2	3	1	2	82.9	4.0
7	3	1	3	2	80.2	3.6
8	3	2	1	3	76.3	3.0
9	3	3	2	1	74.3	3.2

Subscript R refers to recovery rate; Subscript E refers to enrichment ratio.

volume V) for the orthogonal experiment and each factor had 3 levels. The experiment conditions were shown in Table 1 and the results were shown in Table 2.

Results and Analysis

The total evaluation index was used for analysis by statistical method. The results of orthogonal experiment and extreme difference analysis were presented in Table 3.

TABLE 3
Analysis of the experimental result (E)

Item	A, pH	B, C_s (g/L)	C, G (mL/min)	D, V (mL)
T_{1j}	9.0	10.0	9.7	10.0
T_{2j}	11.8	9.9	9.7	10.4
T_{3j}	9.8	10.7	11.2	10.2
M_{1j}	3.0	3.3	3.2	3.3
M_{2j}	3.9	3.3	3.2	3.5
M_{3j}	3.3	3.6	3.7	3.4
J_j	0.9	0.3	0.5	0.2

Subscript E refers to enrichment ratio; C_s refers to SDS concentrations; G refers to superficial gas velocity; V refers to liquid loading volume; T, M and J refer to the results of extreme analysis.

The results of Table 2 indicated that the maximum enrichment ratio and recovery ratio were 4.1 and 86%, respectively. The T, M, and J values were calculated and listed in Table 3. As seen from Table 3, it could be found that the influence to the mean recovery ratio of streptomycin sulfate decreased in the order: pH > superficial gas velocity > surfactant concentration > liquid volume. A concentration according to the results of extreme analysis. pH was found to be the most important factor controlling the separation of streptomycin sulfate. So the maximum enrichment ratio and recovery ratio were obtained when SDS concentration, the initial pH, superficial gas velocity and liquid loading volume were 0.4 g/L, 6.0, 300 mL/min, and 300 mL, respectively.

CONCLUSIONS

1. Sodium dodecyl sulfate as the surfactant for foam separation had good foaming quality. It could effectively concentrate streptomycin sulfate of the aqueous solution by technology of foam separation.
2. By using a single-stage batch foam separation process, the enrichment ratio, and the recovery rate of streptomycin sulfate were 4.0 and 85%, respectively under the best operating conditions of sodium dodecyl sulfate concentration 0.4 g/L, superficial gas velocity 300 mL/min, liquid loading volume 300 mL and initial pH 6.0 when streptomycin sulfate concentration was 0.5 g/L.

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