

Study on Desorbing Sulfur Dioxide from Citrate Solution by Ultrasonification*

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Abstract Using a sonochemical reactor designed by the authors, the process of removing sulfur dioxide from citrate solution simulating the flue gas desulfurization was studied. The influence of ultrasonic frequency, ultrasonic power, reaction temperature, stirring speed, inert gases, initial concentration of sulfur dioxide and concentration of citrate on the efficiency of sulfur dioxide desorption, the stability of citrate solution and the concentration of sulfate radical was examined systematically. By comparing the desorption of sulfur dioxide with and without ultrasonification, it was concluded that (1) lower ultrasonic frequency results in a better degassing efficiency; (2) the use of ultrasonification in desorbing sulfur dioxide from citrate solution improves the desorbing efficiency in some conditions, without changing the essence of chemical reactions; (3) sparging inert gas into the liquid can lower the viscosity of solution and the cavitating threshold, and raise the desorption efficiency. These results demonstrate a technical way for deep desorption of sulfur dioxide and provide the fundamental data for future industrial disposal of sulfur dioxide.

Keywords flue gas desulfurization, desorption efficiency, ultrasonic degassing, citrate, acoustic cavitation

1 INTRODUCTION

According to the physical mechanisms and the purposes of application, ultrasonic wave (frequency above 2×10^4 Hz) can be roughly classified into two kinds: examining ultrasound and power ultrasound. Through the propagation in the medium, dispersion, absorption and wave-form change *etc.*, the examining ultrasound can extract the information of the inherent features or inner structure and thus achieve the purpose of examination. The power ultrasound can change the property and state of the material through its energy and interaction with the material, by the powerful and strong ultrasonic wave and vibration. Currently, efficient and economical power ultrasonic devices are widely available. This promotes the progress in sonochemistry, which studies the application of ultrasonics in chemical and metallurgical engineering[1—3], owing to the special physical and chemical effects caused by the ultrasonic cavitation in liquid medium. Due to its virtue of low consumption and little pollution, it gains wide application in some fields like metallurgy, chemical engineering and environmental protection[4—7].

Using citrate for desulfurization is a new method of removing sulfur dioxide from flue gas, which is developed by US Bureau of Mines[8]. In this approach to recycle the absorbent of citrate solution and exploit the sulfur dioxide, the solution must be desorbed. At present, the desorption is achieved mainly by steam heating and microwave heating[9,10]. Although these methods can make the desorption ratio above 90%, they have some shortcomings. For the steam heating, the desorption temperature is around 100°C, and the efficiency of heat utilization is low. For the microwave heating, it has higher requirement

for the filler and the prevention of human harm by microwave is very stringent.

As a new method, the ultrasonic degassing has been extensively studied. The ultrasonic technique was utilized to dispose waste water of high-concentration ammonia and nitrogen, and the ultrasonic radiation and the air blow were combined to increase the degassing rate by 17%—164%[11]. Degassing was effected by directly introducing the ultrasonic wave produced by a sonar pole into the metal solution and the mechanism of ultrasonic degassing was addressed[12]. The results showed that the ultrasonic degassing rate was proportional to the ultrasound strength. High-frequency sonochemical reactor was also used to study the degassing of oxygen from the solution under the atmospheric pressure and the constant temperature[13].

An ultrasonic reactor is used to introduce ultrasonic wave into the diverse weakly acidic solution systems and the process of desorbing sulfur dioxide from the citrate solution is investigated in this work. The steady cavitation by ultrasonic wave is utilized to degas the dissolved sulfur dioxide in the absorbent of citrate solution. In this case the fine cavitation bubbles grow into big bubbles and rise to the liquid surface, sulfur dioxide is desorbed and the absorbent can be recycled. The influence of ultrasonic frequency, ultrasonic power, reaction temperature, stirring speed, inert gases, initial concentration of sulfur dioxide and concentration of citrate on the efficiency of sulfur dioxide desorption was examined systematically. The citrate desorption of sulfur dioxide has increasingly gained attention owing to its good economic benefit[14—16]. The presented results suggest a technical way for the deep desorption of sulfur dioxide and also provide the fundamental data for future industrial

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disposal of sulfur dioxide.

2 EXPERIMENTAL

2.1 Experiment equipment and materials

The sonochemical reactor with adjustable reaction temperature and variable ultrasonic power was designed by ourselves and manufactured by Engineering Division of Ultrasonic Technique, Xiangda Co. (Shanxi, China)[17]. The design of the reactor took the factors of material, shape, ultrasonic frequency, power and the control of the system temperature into consideration. The sonochemical reactor was made of acid- and erosion-resistant 304L stainless steel with low sonic resistance and good thermal conductivity. Finally the reactor with the dimension of $\phi 180\text{mm} \times 200\text{mm}$ was designed, equipped with ultrasonic generator ACQ-300 and 3 porcelain energy translators HNC-4SS-3060 ($\phi 25\text{mm} \times 35\text{mm}$) made by Hainertec Co. Ltd (Suzhou, China) as shown in Fig.1. Three porcelain energy translators are tightly fixed underneath the bottom of the sonochemical reactor. And the schematic of the sonochemical reaction setup is shown in Fig.2. The frequency of the ultrasound is 40kHz and its power is between 20W to 320W. The main parts used in the experiment and their properties are listed in Table 1, where the propeller of the magnetic mixer is an airscrew with a diameter of 80mm. When the depth of the liquid in the reactor is 10cm, good efficiency of stirring is obtained with the propeller installed 2cm above the bottom.

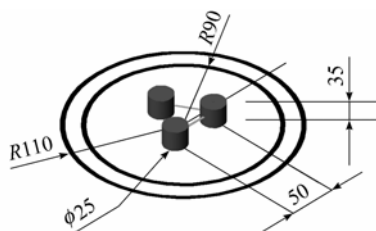


Figure 1 Ultrasonic converters on the reactor bottom

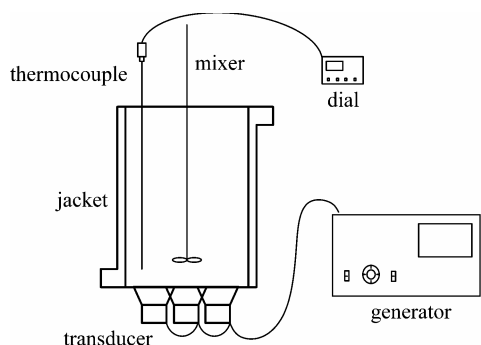


Figure 2 Diagram of sonochemical reactor

Experimental gas is the simulated refining flue gas, which is a mixture of air from a compressor and SO_2 from a steel container by using a high-precision gas-distribution system. The fraction of sulfur dioxide in the gas is in the range of 0.001 to 0.01 and the oxygen content is always $>20\%$. The present study is focused on the performance of SO_2 -desorption under ultrasonic action from SO_2 -absorbed citrate solution.

2.2 Experimental procedure

The prepared citric acid-sodium citrate buffer solution ($T = 25^\circ\text{C}$, $c = 0.25\text{mol}\cdot\text{L}^{-1}$, $0.5\text{mol}\cdot\text{L}^{-1}$, $0.75\text{mol}\cdot\text{L}^{-1}$, $1.0\text{mol}\cdot\text{L}^{-1}$, $1.25\text{mol}\cdot\text{L}^{-1}$, $1.5\text{mol}\cdot\text{L}^{-1}$) was allowed to absorb sulfur dioxide to a certain concentration to provide the raw solution for testing the performance of the ultrasonic desorption reactor. In each experimental run, 2.2L of absorbant solution is filled into the degassing reactor. Then start temperature-control system to increase the liquid temperature to the set value. Start the ultrasonic system and the mixer to carry on the experiment of ultrasonic desorption, and monitor the concentration of sulfur dioxide in the solution. The concentrations of citrate and sulfate radicals are also determined after typical experimental runs.

When examining the influence of different gases on the desorption efficiency of SO_2 , argon or air was sparged through a plastic tube with diameter of 20 mm, which is 15mm above the bottom and at the side wall of the reactor. The amount of sparging argon or air was chosen to be $0.5\text{L}\cdot\text{min}^{-1}$. When discussing the relation of argon flow rate and the desorption efficiency of SO_2 , argon flux was taken as $0.1\text{L}\cdot\text{min}^{-1}$, $0.2\text{L}\cdot\text{min}^{-1}$, $0.4\text{L}\cdot\text{min}^{-1}$, $0.6\text{L}\cdot\text{min}^{-1}$ and $0.8\text{L}\cdot\text{min}^{-1}$, respectively.

2.3 Analysis method

2.3.1 Analysis of the concentration of citric radical

The content of citric radical is analyzed by the method of mass analysis[18]. In the buffer solution of acetic acid and sodium acetate with $\text{pH}=5-6$, the citrate acid radical interacts with Pb^{2+} in the lead acetate solution to form white precipitate of lead citrate. Then after filtration and cleansing of the precipitate, the content of citrate acid radical can be measured through mass analysis after drying under temperature of 140°C .

2.3.2 Analysis of sulfur dioxide and sulfate radical in the solution

The concentration of sulfur dioxide in the solution can be analyzed by the iodometric method. For the analysis of sulfate radical, it first needs to boil the

Table 1 Main parts of experimental apparatus

No.	Name	Model	Manufacturer
1	reinforcement motor stirrer	JJ-1 (160 W)	Guohua Instrument Plant, Changzhou, Sichuan, China
2	ultrasonic generator	ACQ-300	Xiangda Ultrasonic Technical Project Division, Shaanxi
3	ultrasonic reactor	$\phi 180\text{mm} \times 200\text{mm}$	Xiangda Ultrasonic Technical Project Division, Shaanxi
4	mesa pH/ISE test instrument	828	Orion Research, Inc

solution to remove the interference in low concentration of hydrochloric acid, and then carry on the analysis with the BaSO₄ mass method.

For this experimental system, the desorption efficiency is used to express the percent desorption of the absorbed sulfur dioxide:

$$\eta = \frac{C_1 - C_2}{C_1} \times 100\%$$

where c_1 is the concentration of sulfur dioxide in the citrate solution before desorption (mol·L⁻¹) and c_2 is the concentration of sulfur dioxide in the citrate solution after desorption (mol·L⁻¹).

3 RESULTS AND DISCUSSIONS

3.1 Influence of the solution quality on the desorption efficiency

3.1.1 Influence of different gases on the desorption efficiency

Figure 3 shows the influence of different gases on the sulfur dioxide desorption efficiency. The change of the sulfur dioxide desorption efficiency with the time is investigated in three cases: using argon gas, using air and not using any at all. Here, the reaction temperature is 50°C, the stirring speed is 300r·min⁻¹, the initial concentration of the sulfur dioxide is 120g·L⁻¹, the concentration of citrate is 1.0mol·L⁻¹, pH at 4.5, the liquid depth is 10cm, the ultrasonic frequency is 40kHz and the power is 180W.

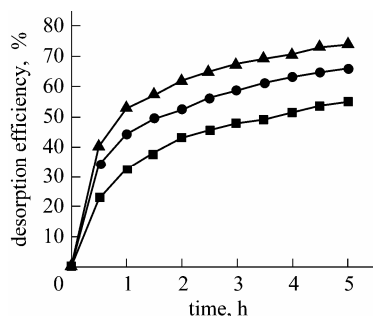


Figure 3 The influence of different gases on the sulfur dioxide desorption efficiency ($c_1 = 120\text{g}\cdot\text{L}^{-1}$, sparging gas $0.5\text{L}\cdot\text{min}^{-1}$)
 ■ no gas; ● air; ▲ argon

From Fig.3 the sulfur dioxide desorption efficiency with the use of inert gas is found to be higher than that without the use of inert gas. With the inert gas in the solution, the cavitation threshold is lowered and thus makes the cavitation easier. At the same time, the heat conduction rate of argon gas is lower than the air, which leads to a higher temperature for the breaking up of cavitation bubbles and thus it is easier to produce the sulfur dioxide cavitation cores, make the cavitation easier and propitious to the desorption of the sulphur dioxide.

Here argon flux is small because of its role played is different from that of gas in distilling tower. The relation of the argon flux and the desorption efficiency of SO₂ is shown in Fig.4., suggesting that high-flux argon leads to higher desorption efficiency.

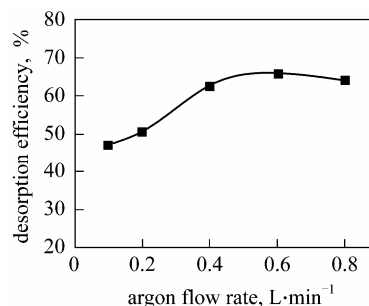


Figure 4 The influence of argon flow rate on the sulfur dioxide desorption efficiency ($c_1 = 120\text{g}\cdot\text{L}^{-1}$, sparging time = 5h)

When the argon flux is $0.8\text{L}\cdot\text{min}^{-1}$, the desorption efficiency is enhanced by 15% compared with argon flux of $0.1\text{L}\cdot\text{min}^{-1}$. However, when argon flux exceeds $0.4\text{L}\cdot\text{min}^{-1}$, the desorption efficiency remains basically unchanged. Therefore, it is suitable to choose argon flux as $0.5\text{L}\cdot\text{min}^{-1}$.

3.1.2 Influence of the initial concentration of sulfur dioxide on the desorption efficiency

The experiment is also conducted under the following conditions: reaction time of 5h and others are the same as in Section 3.1.1. Please note that no gas sparging was used in subsequent experiments. Results in Fig.5 suggest that the desorption efficiency increases as the initial concentration of sulfur dioxide increases. But when the initial concentration of sulfur dioxide is above $100\text{g}\cdot\text{L}^{-1}$, the increase of the desorption fraction is slow. The reason is that with a higher initial concentration of sulfur dioxide, the beginning pH value of the solution is lower and thus makes desorption easier. At the same time, with a higher initial concentration of sulfur dioxide, the cavitation threshold value gets lower and makes it easier to produce cavitation bubbles, and thus accelerate the desorption. At the time of 5h, the remained sulfur dioxide in the solution has approximately the same concentration around $25\text{g}\cdot\text{L}^{-1}$. The concentration of citrate radical and the sulfate radical did not change during the desorption.

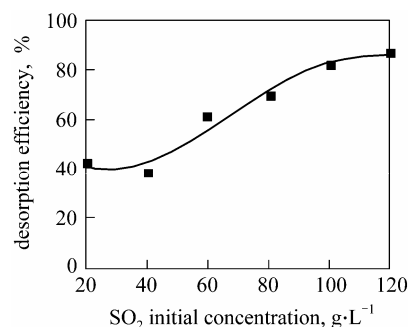


Figure 5 The influence of initial sulfur dioxide concentration on desorption efficiency

3.1.3 Influence of the concentration of citric radical on the desorption efficiency

Figure 6 is the relationship between the sulfur

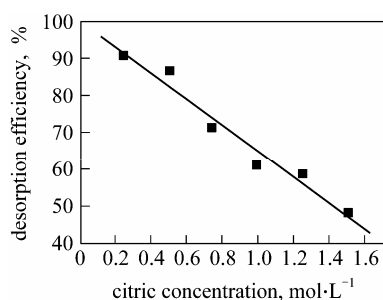


Figure 6 The influence of citric concentration on sulfur dioxide desorption efficiency

dioxide desorption efficiency and the citrate concentration under the conditions that the beginning concentration of sulfur dioxide is $120\text{g}\cdot\text{L}^{-1}$, reaction time of 5h and others are the same as in Section 3.1.1. It is observed that with the same initial sulfur dioxide concentration, the desorption efficiency is lower for higher citrate concentration. When the concentration of citrate is $0.25\text{mol}\cdot\text{L}^{-1}$ and the reaction time is 5h, the sulfur dioxide desorption can amount to 91%. This is because for the same initial sulfur dioxide concentration, higher citrate concentration leads to a higher pH value and higher viscosity of solution as well as a higher ultrasonic wave cavitation threshold[19], and thus it is more difficult to create cavitation bubbles. But when the citrate concentration is higher, the sulfur dioxide absorbing capacity is greater. Therefore, the absorbing capacity and the desorption efficiency should be well compromised when determining a reasonable citrate concentration.

3.2 Influence of operation parameters on desorption efficiency

3.2.1 Influence of reaction time of ultrasound

Figure 7 shows the dependence of the sulfur dioxide desorption efficiency on the reaction time with or without ultrasound. It is obtained under the conditions that reaction temperature is 50°C , stirring speed $300\text{r}\cdot\text{min}^{-1}$, the initial sulfur dioxide concentration $120\text{g}\cdot\text{L}^{-1}$, citrate concentration $1.0\text{mol}\cdot\text{L}^{-1}$, pH at 4.5, and the liquid depth is 10cm.

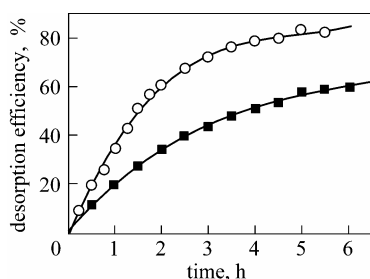


Figure 7 Relationship between desorption efficiency and reaction time
■ no ultrasonic; ○ ultrasonic

Figure 7 suggests that the ultrasonic wave can significantly accelerate and promote the degassing process. For a reaction time of 5h, the use of ultra-

sound can enhance the sulfur dioxide desorbing efficiency by about 25%. In the first 2h, the sulfur dioxide desorption rate grows linearly with time and can reach 60%. But in the subsequent 3h, the desorbing rate grows slowly with time. The reason for such behaviors is that at the beginning period the concentration of HSO_3^- is higher and the pH is lower, which makes the sulfur dioxide desorption easier and the desorption belongs to the physical desorption.

3.2.2 Influence of temperature

As the temperature goes up, the solubility of sulfur dioxide becomes lower and sulfur dioxide can be easily desorbed from the solution. For the reaction time of 5h and other conditions as the same in Section 3.2.1, it is observed from Fig.8 that without the use of ultrasonic wave, the sulfur dioxide desorption rate goes up linearly with the increase of temperature because of lower viscosity of the solution. With the use of ultrasonic wave, the desorption efficiency increases with the temperature for temperature below 50°C and reach the maximal value at 50°C , and then decrease with the temperature. This behavior can be understood as follows. With the increase of the temperature, the number of cavitation bubbles increases rapidly, and then, with the action of stirring, the number of sulfur dioxide bubbles escaping from the solution also increases. But as the temperature continues to go up, saturation of cavitation bubbles is rapidly achieved. When the temperature is sufficiently high, the rise of steam pressure and the decrease of surface tension as well as the screening effect and the increasing break up rate of the cavitation bubbles will cause the cavitation activity to get lower and make the sulfur dioxide bubbles collapse before ascending to the surface of liquid[20].

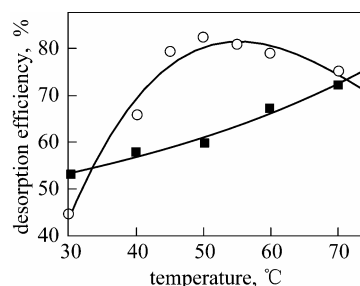


Figure 8 Relationship between desorption efficiency and temperature
■ no ultrasonic; ○ ultrasonic

3.2.3 Influence of the ultrasonic frequency

With the same conditions as in Section 3.2.1, the sulfur dioxide desorbing experiment is conducted with different ultrasonic frequency (20kHz, 40kHz, 60kHz). The results in Fig.9 show that the sulfur dioxide desorption efficiency gets lower as the frequency gets higher. As the frequency goes up, the time of ultrasonic wave inflation phase becomes shorter and the cavitation cores do not have enough time to grow up to cavitation bubbles. Therefore, a lower frequency results in a better ultrasonic desorption. On the other hand, for the cavitation under a higher frequency, the

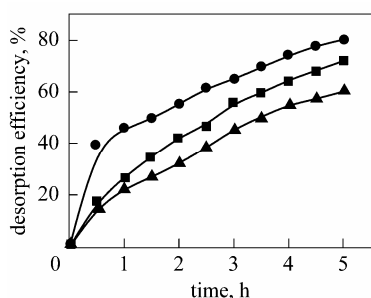


Figure 9 Relationship between desorption efficiency and different ultrasonic frequency

● 20kHz; ■ 40kHz; ▲ 60kHz

threshold of cavitation is increased. But the ultrasonic waves with high frequency are more rapidly dissipated in the solution and thus, in order to obtain a same sonochemical effect, more energy consumption is needed for the high frequency ultrasonic waves. So the ultrasonic with low frequency is beneficial in the aspect of energy consumption.

3.2.4 Influence of ultrasonic power

Ultrasonic power is defined as the total actual sound energy radiated into the reaction system in unit time. Only when the ultrasonic power input into the solution is above the threshold value of cavitation can the cavitation effects occur. The ultrasonic degassing process utilizes mainly the steady cavitation. This kind of steady cavitation process can occur under the ultra-low sound strength, which means that under the continuous action of sound wave, the bubbles in the liquid keeps growing until rising to the surface of liquid and merging into the space. When the wave strength is too strong, the cavitation happens too rapidly and the bubbles collapse before rising to the liquid surface, and, as a result, the sulfur dioxide re-dissolves in the citrate solution. Therefore, it is necessary to choose a better ultrasonic power for the desorption of sulfur dioxide. Experiment for the reaction time of 5 h and other conditions being the same as in Section 3.2.1 was performed, using the input electric current to represent the ultrasonic power. The relationship between input electric current and the power in the citrate solution through the heat-measuring method was determined beforehand as shown in Table 2.

Table 2 Relationship between input current and ultrasonic power (40kHz)

Current, A	Power, W
0.6	22.4
0.8	45
1.0	89.3
1.2	136.9
1.4	177.2
1.6	280.7
1.8	314.3

The influence of the electric current on the desorption rate is shown in Fig.10, suggesting that the

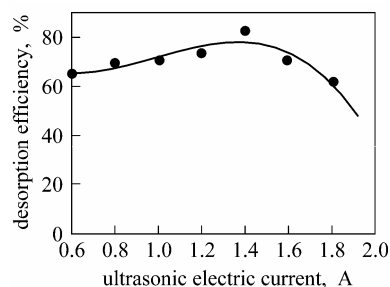


Figure 10 Relationship between desorption efficiency and ultrasonification current

desorbing rate increases with the electric current for the electric current below 1.4A and reaches the maximal value 83% at 1.4A. When the electric current is above 1.4A, the desorbing rate decreases with the electric current and becomes as low as 61.7% at 1.8A. Therefore, for the 40kHz frequency, the best electric current is 1.4A and it is mostly chosen in our experiments[21,22].

3.2.5 Influence of stirring speed

Figure 11 (the conditions are the same as in Section 3.2.1 except the stirring speed) suggests that the sulfur dioxide desorption efficiency increases with the stirring rate with or without the ultrasound. For the stirring rate above $250\text{r}\cdot\text{min}^{-1}$, such increase is slow. This means that stirring can speed up the gas diffusion and thus speed up the escape of the sulfur dioxide bubbles. Increasing the stirring rate can also enhance the cavitation effect[23]. Note that the influence of stirring rate on the sulfur dioxide desorption is independent of the use of the ultrasound. Under the same stirring rate, the use of ultrasound can enhance the desorption efficiency by 25%. So the ultrasound just accelerates the desorption, without changing the mechanism of the desorption process.

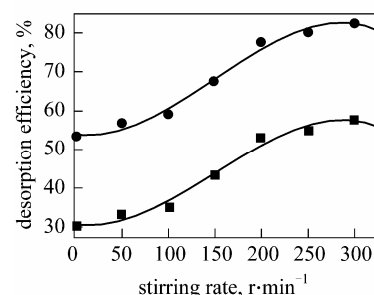


Figure 11 Relationship between desorption efficiency and stirring efficiency

■ no ultrasonic; ● ultrasonic

After using ultrasound 48h in above experiments, the chemical analysis was performed on the citrate solution, and neither the decomposition of the citrate nor the increase of sulfate radical in the absorbent was found.

4 CONCLUSIONS

From the study of desorbing sulfur dioxide from

citrate solution by ultrasound, it is found that the use of ultrasound in desorbing sulfur dioxide from citrate solution can improve the desorption efficiency under some conditions. Sparging minute argon can enhance cavitation action and make sulfur dioxide desorbed faster. With the same initial concentration of sulfur dioxide, desorption efficiency decreases linearly with the citric concentration. The desorption efficiency of sulfur dioxide increases with the initial concentration of sulfur dioxide. Lower ultrasonic frequency results in a better degassing efficiency. For a frequency of 40kHz for ultrasound, the best desorption temperature is 50°C, the electric current is 1.4A and the stirring rate is 250r·min⁻¹. In such a case, for citrate concentration of 1.0mol·L⁻¹ and the initial sulfur dioxide concentration of 120g·L⁻¹, a reaction of 5h results in desorption efficiency of 82%.

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