Optimization of process parameters and kinetics of adsorption treatment of thallium-containing wastewater

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Abstract. Thallium is an extremely toxic metal and abundant in industrial wastewater but little studied. In order to understand the optimal adsorption kinetic parameters of the chelating resin containing hydrazine wastewater, we carried out adsorption experiments on the cerium-containing wastewater treated by chemical precipitation. In this chapter, the optimum adsorption conditions, adsorption model, dynamic adsorption curve and desorption and regeneration of adsorbent were determined at different pH, temperature, adsorption time and different adsorbent dosage. The results show that the removal rate of thallium wastewater by resin is up to 97.5% when the pH value is 9. The optimum adsorption temperature is 30 and the adsorption reached equilibrium at 80 min. The adsorption process is consistent with Lagergren quasi-second-order adsorption and Langmuir isotherm model. The regenerative properties of the resin show that the resin adsorption rates still reach 95.8% after repeat use for six times. In summary, the chelating resin has good adsorption and reusability to the thallium-containing wastewater

1. Introduction

With the development of modern society inevitably produced various types of contaminants ,such as organic compounds[1, 2], dyes[3-5], and heavy metals[6-8], which have caused widely concern for water pollution because of their acute toxicity, non-degradability and bioaccumulation. Thallium (Tl) is a rare heavy metal element with high toxicity[9-11], which is more toxic than lead, mercury, etc., and is equivalent to the toxicity of arsenic[12]. Tl is one of the priority pollutants listed in US Environmental Protection Agency as it has caused many accidents and occupational poisoning[13]. With the development of socials and the progress of industrial technology, Tl and its compounds have found an increasingly wide utilization in ore exploration, semiconductors, catalysis, etc.[14-17]. The pollution situation of Tl metal in the water environment is increasing year by year. If not treat in time, Tl metal will seriously threaten the water environment and human health[18]. The problem of Tl metal pollution has been increasingly valued by human beings.

At present, the main technologies for the treatment of Tl-containing wastewater have oxidation flocculation, chemical precipitation technique, ion exchange technique, solution extraction method, adsorption, etc. Adsorption is one of the most widely used methods. Other methods are basically in the phase of theoretical research, and there are few successful application examples[19]. Karatepe et al[20]used Chromosorb 105 resin to selectively adsorb the complexes of Tl contain chloride and iodine, but the enrichment effect of the technology was not ideal. Amin et al[21] fixed the Tl on the styrene divinylbenzene anion exchange resin, then measured the absorbance of Tl at 636 nm and 830 nm. The method has been successfully application to test Tl in water. Luo et al[22] used tributyl phosphate leaching resin as a column packing to separate the enriched water and Tl in wastewater. The research showed that in 5% (volume fraction) hydrochloric acid-nitric acid (1+3) solution, tributyl phosphate leaching resin could quantitative adsorption of trace Tl in water, rapid desorption with 2 g/L sulfurous acid and 2g/L ascorbic acid as desorbent, the recovery rate is between 96% and 102%. For industrial Tl-containing wastewater in our country, it is usually treated by chemical precipitation, but the post-treatment concentration is difficult to meet national standards. The limit value of Tl in environmental quality standards for surface water (GB 3838-2002) promulgated by China in 2002 is 0.0004 mg/L, the value of Tl is much lower than that of arsenic, lead, chromium and mercury[23]. Therefore, industrial wastewater needs to be treated in depth. In view of such refractory Tl-containing wastewater, the study is based on the existing advanced treatment process for Tl-containing wastewater. We need to find the optimal conditions for the adsorption of Tl by existing materials, and conducting adsorption kinetics research to optimize its process parameters.

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2. Experimental

2.1 Materials and reagents

Activated alumina (Tianjin Sailboat Chemical Reagent Technology Co., Ltd.), Chelating resin (Hunan Changsha Yuchi Chemical Co., Ltd.), Molecular sieve (Tianjin Kemiou Chemical Reagent Co., Ltd.)

The experimental wastewater was taken from the raw water and the secondary sedimentation tank effluent of the sewage treatment station of a zinc material co., Ltd. in Zhuzhou City, Hunan Province. The main pollutant of experimental wastewater was Tl, the concentration of Tl in the raw water was about 10 mg/L, and the concentration of Tl in the effluent of the second settling tank was about 10~30 μg/L. All drugs were analytical reagent.

2.2 Characterization and analysis

Determination of Tl in wastewater by ICP Mass Spectrometer

2.3 Adsorption experiments

In this thesis, Tl-containing wastewater was used as a target pollutant to study the adsorption performance and optimization of process parameters of chelating resin. A series of adsorption experiments were carried out by adding a certain amount of chelating resin (5g) into 100 mL Tl-containing wastewater in a shaker (12h) at 30°C. The influence of Tl-containing wastewater pH on the adsorption was carried out from pH 6.0 to 11.0. The adsorption experiments had been determined at different temperatures (20, 25, 30, 35, 40, 45 and 50°C). The instantaneous equilibrium concentration of the filtrate was determined at different adsorption periods (10, 20, .30, 40, 50, 60, 70, 80, 90 and 100 min). The influence of initial Tl-containing wastewater concentration on the adsorption was carried out.

2.4 Desorption experiments

After adsorption, the chelating resin was washed three times with distilled water and dried, then desorption with a certain concentration of hydrochloric acid. Determined the concentration of Tl in the desorbed solution and calculated the desorption. The chelating resin can be reused in adsorption tests.

2.5 Dynamic adsorption

Weighing a certain amount of chelating resin into the adsorption column and wetting it with ultrapure water. Continuously adding wastewater to the adsorption column with a constant flow rate at room temperature (25°C). Then, at intervals, the solution penetrating the chelating resin was collected and detected until the concentration of Tl in the effluent was the same as the concentration of Tl in the original solution. At this point, the dynamic adsorption had reached saturation. The model diagram is as follows:

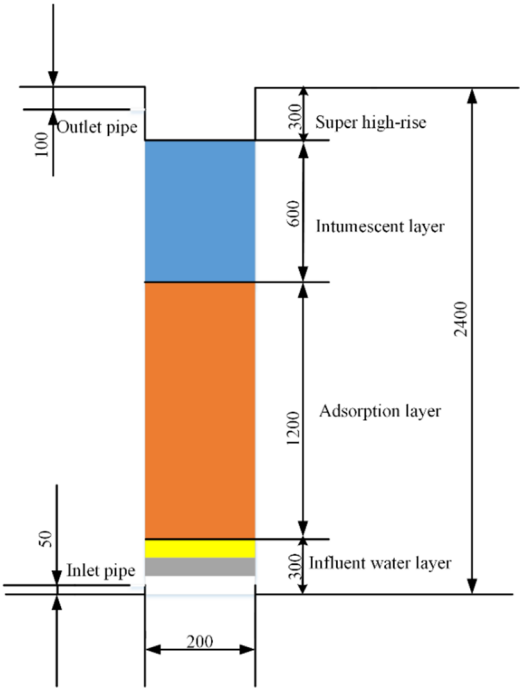

Fig.1 Model diagram

3. Results and discussion

3.1 Adsorption tests

3.1.1 Effect of pH

The adsorption tests on chelating resin were conducted in different pH range from 6.0 to 11.0, and the results were shown in Fig. 2. As we known, different metals have different optimum pH values of adsorption[24]. The values of pH not only affects the surface charges and state of the functional groups on the surface of adsorbents, but also affects the species of metal ions in solution[25, 26].The values of pH has a great influence on the adsorption performance of the adsorbent. From the Fig. 2, we can know that at relatively low pH values of 6.0 and 9.0, the removal efficiency of Tl in water samples is below 90%. When the values of pH increased from 9.0 to 9.5, the removal efficiency of Tl is up to about 97.5%. However, the removal efficiency of Tl has decreased at the pH values greater than 9.5. The phenomenon indicates that the greater the acidity of the wastewater, the Tl on adsorbent will compete with the H^+ in the water to reduce the adsorption capacity of the resin on the Tl. As the pH increased, the acid effect coefficient gradually decreases, which is more favorable for the adsorption of the resin to the Tl. At a higher pH, the hydrolysis reaction of Al^{3+} and Fe^{3+} in the wastewater will produce precipitation, which effect the adsorption capacity of the resin. So chelating resin can be used at a pH values about 9.0 for treating Tl-containing wastewater. The following experiments were all carried out under the conditions of pH=9.0.

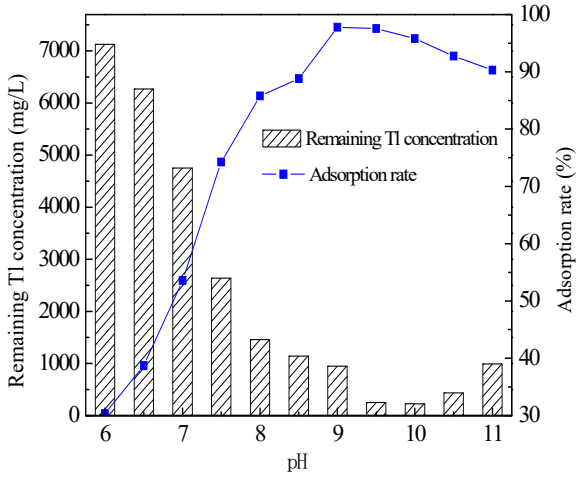

Fig.2 Effect of wastewater pH on adsorption of Tl by chelating resin (5 g) at 30°C

3.1.2 Effect of temperature

In this paper, the adsorption effect of chelating resin on Tl in wastewater was tested at different temperature, the results were shown in Fig. 3. The adsorption efficiency of resin to Tl increased with increasing temperature from 20°C to 30°C. Results indicated that the adsorption process was difficult to reach equilibrium quickly at a lower temperature. The increase in adsorption capacities of the adsorbent at higher temperature may be attributed to the enlargement of pore size or specific surface area[27]. When further increasing temperature to 50°C, the adsorption efficiency instead to decreased. This indicates that the adsorption may be an exothermic process. The adsorption capacity of the resin decreases as increasing the temperature after reaching the adsorption equilibrium. Fig. 3 displays that the optimum temperature for the adsorption of resin on Tl is 30°C.

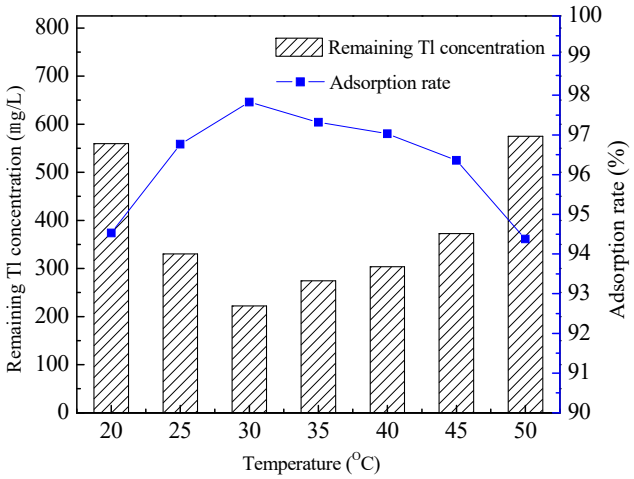

Fig. 3 Effect of temperature on adsorption of Tl by chelating resin (5 g)

3.1.3 Adsorption kinetics

The effect of contact time on the adsorption efficiency of Tl was investigated with the contact time from 0 to 100 min. It is obvious that the adsorption rate of Tl by chelating resin is very rapid and the adsorption reaches equilibrium within 80 min (Fig. 4). The amount of adsorption on Tl increases dramatically at a lower contact time. The adsorption process of resin on heavy metal ions in wastewater is a liquid-solid adsorption process.

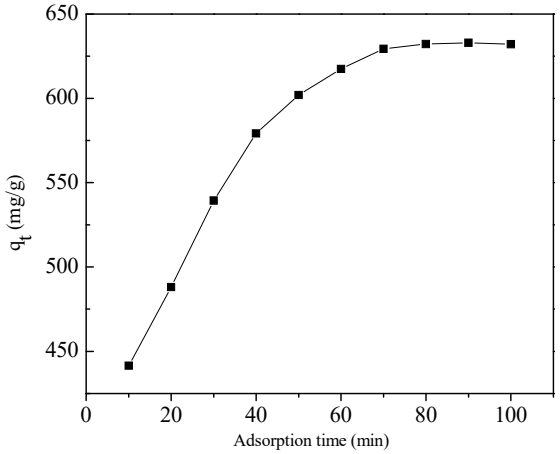

Fig. 4 Effect of adsorption time on adsorption capacity

In order to study the mechanism of liquid-solid adsorption, our experiments propose two kinetic models (Lagergren quasi-first-order kinetic models and Lagergren quasi-second-order kinetic models), the equations of the kinetic models abovementioned are described as follows [28-30]:

Lagergren quasi-first-order kinetic models

$$\ln(q_e - q_t) = \ln(q_e) - K_1 t \quad (3\text{-}1)$$

Lagergren quasi-second-order kinetic models

$$\frac{t}{q_t} = \frac{1}{K_2 {q_e}^2} + \frac{1}{q_e} \quad (3\text{-}2)$$

Where, q_e is the amount of adsorption (μg/g) at the equilibrium of adsorption. q_t is the amount of adsorption (μg/g) at any instant of time t (min) K_1 (min^{-1}) and K_2 (g/(μg·min) are the rate constant of Lagergren quasi-first-order adsorption and Lagergren quasi-second-order adsorption, respectively.

Linear regression fitting was performed on the two kinetic models according to experimental data. The results were shown in Fig. 5 and equation are displayed below[31, 32]:

Lagergren quasi-first-order kinetic models

$$\ln(q_e - q_t) = 6.655 - 0.07659t \quad (3\text{-}3)$$

R^2=0.9498

Lagergren quasi-second-order kinetic models

$$\frac{t}{q_t} = 0.00988 + 0.00147 \quad (3\text{-}4)$$

R^2=0.9996

We can see from Fig. 5 that the resin adsorption progress has a good linear regression effect on both kinetic models. According to the fitting results of the above two models, the process of resin adsorption is more in line with Lagergren quasi-second-order kinetic model according to the comparison of correlation coefficients (R^2). From the

result, the adsorption progress of Tl onto the absorbents proceeded by chemisorption mechanism[33].

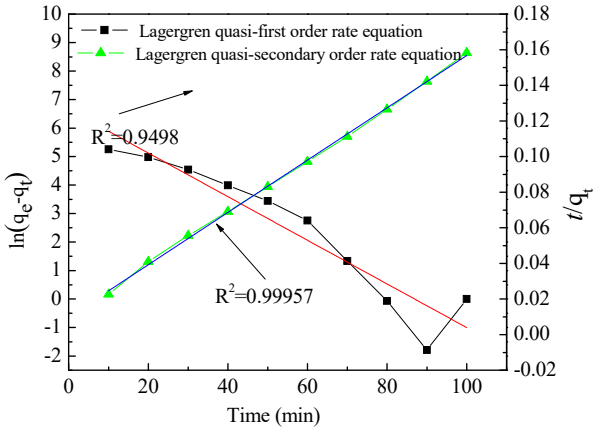

Fig. 5 Linear fitting of Lagergren kinetic models

3.1.4 Adsorption isotherm

In order to study the adsorption isotherms of Tl on resin, the adsorption of Tl at different initial concentrations was studied. Results were shown in Fig. 6. In this study, two common adsorption isotherm models (Langmuir and Freundlich models) were used to investigate the interaction of Tl and surface of adsorbent by the test data at adsorption equilibrium.

Langmuir adsorption isotherm model

Langmuir isotherm is based on the assumption that the adsorption sites on the adsorbent surface are limited and evenly distributed. The equation is displayed as follows[31, 34]:

$$\frac{c_e}{q_e} = \frac{1}{q_0 b} + \frac{c_e}{q_0} \quad (3\text{-}5)$$

Where, q_e and c_e are absorption amount (μg/g) and concentration of Tl (μg/L) at adsorption equilibrium respectively, and q_0 and b represent the maximum adsorption capacity of adsorbents (μg/g) and the Langmuir adsorption constant (L/μg), respectively. The values of q_0 and b can be calculated from the slope and intercept of the linear plot of c_e/q_e against c_e (Fig. 7).

Freundlich adsorption isotherm model

Freundlich isotherm model is used to describe the adsorption behavior that occurs on the heterogeneous surface[35]. The expression for Freundlich isotherm model is described in the following form:

$$\ln q_e = \ln K_F + 1/n\, c_e \quad (3\text{-}6)$$

Where, q_e and c_e are absorption amount (μg/g) and concentration of Tl (μg/L) at adsorption equilibrium respectively, K_F is Freundlich isotherm constant, n is another constant in Freundlich isotherm model to indicate how favorable the adsorption progress. If the value of $1/n$ is between 0 and 1, which indicated that the adsorption process is in favor of the reaction conditions; If the value of $1/n$ greater than 1, it is un-favorable; If the value of $1/n$ is approximately equal to 1 then the process is homogeneous[36]. K_F and n can be obtained from the intercept and the slope of the linear plot of $\ln q_e$ versus $\ln c_e$ (Fig. 7).

The adsorption parameters are listed in Table 1. It is obviously that the correlation coefficient (R^2) of Langmuir isotherm model is much higher than that of Freundlich isotherm model, which indicates that the Langmuir model describes the adsorption process better. Furthermore, the adsorption of Tl is homogeneous. After the adsorbate is adsorbed on the surface of the adsorbent, the adsorbate has no interaction with each other. At the same time, the adsorption process is not affected by the adsorption capacity. The adsorption mechanism of Tl by the resin is that Tl can be exchanged with cations in the resin. Due to the unique structural characteristics of the resin, large exchangeable space spacing, there is no interaction between Tl ions after exchange. Meanwhile, the maximum adsorption capacity of Tl is lower than the maximum exchangeable amount of resin, which is consistent with the basic assumptions in the Langmuir adsorption model[37].

Table 1 The adsorption parameters according to Langmuir and Freundlich adsorption isotherm model

Adsorption isotherm models	R^2	Model parameters	Linear equation
Langmuir	0.9688	q_0=636.94μg/L; b=0.01032	$\frac{c_e}{q_e}$=0.15214+0.00157c_e
Freundlich	0.8888	$1/n$=0.00848; $\ln K_F$=4.56771	$\ln q_e$=4.56771+0.00848c_e

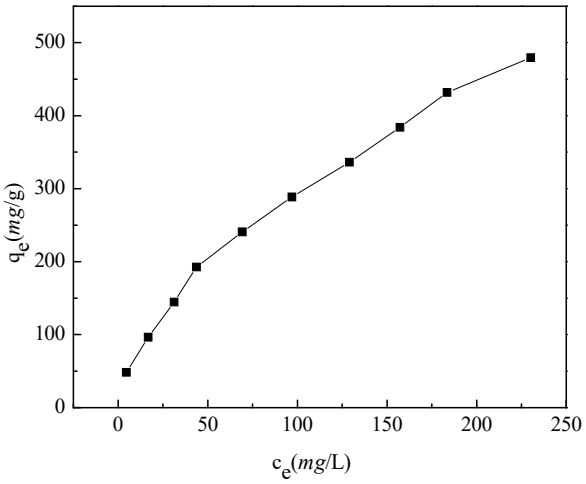

Fig. 6 Resin adsorption Tl adsorption isotherms

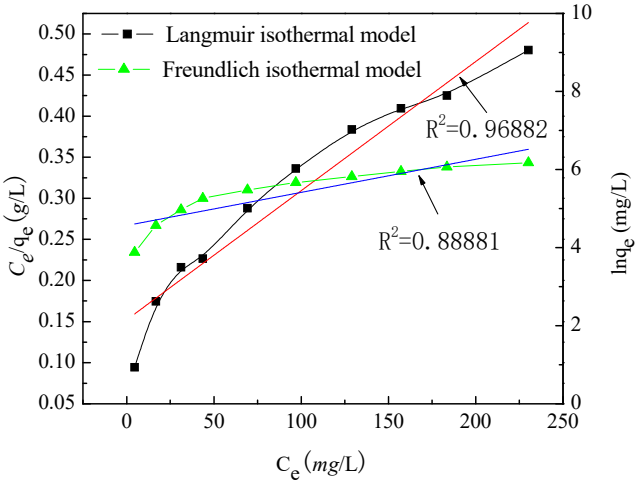

Fig. 7 Linear fitting of Langmuir and Freundlich isotherm models

3.2 Desorption tests

The raw water (12133.64 μg/L, 2 L) was adsorbed with 500 g chelating resin for 6h, and the concentration of Tl after adsorption is 43.62 μg/L. Therefore, the amount of Tl adsorbed by the resin was 48.36 μg/g.

3.2.1 Effect of desorbent concentration

5%, 10%, 15%, 20% hydrochloric acid (60 mL) was added to 4 parts of the resin (30 g) after adsorbing raw water, and test the sample after shaking for 4h. The results were shown in Fig. 8 and Table 2. We can obviously see that the desorption effect is basically unchanged between 60% and 67%. It may be that the resin adsorption does not reach saturation. Desorption process reached equilibrium state after added the desorbent, the desorption rate cannot be increased even if the desorbent solution concentration is increased. Excessive concentration of the desorbent solution may destroy the structure of the resin, decrease the adsorption capacity and difficult to cleaning and operating. Therefore, the optimum desorbent concentration in the subsequent static desorption is 5%.

Table 2 Effect of desorbent concentration on desorption rate

Tl content in resin (μg/g)	The concentration of desorbent (%)	Desorption content of Tl (μg)	Desorption rate (%)
483.6	5	964.64	66.49
	10	865.63	59.67

15	947.21	65.29
20	948.85	65.40

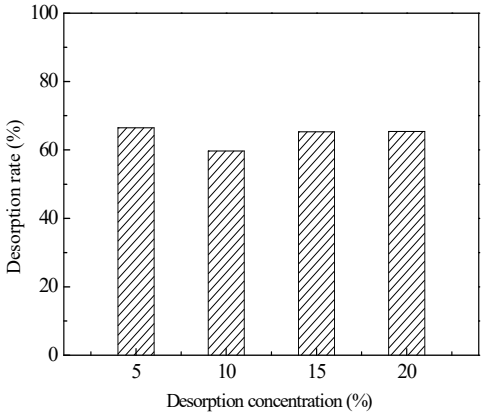

Fig. 8 Effect of desorption concentration on desorption rate

3.2.2 Effect of solid-liquid ratio of adsorbent and desorbent

30, 60, 90, 120 mL hydrochloric acid (5%) was added to 4 parts of the resin (30 g) after adsorbing raw water, and determined the sample after shaking for 4h. Results were shown in Fig. 9 and Table 3. As the liquid-solid ratio increased, the desorption rate of Tl in the resin increased from 50.09% to 81.72%. The desorption process was promoted by the increase of the concentration difference between adsorbent and Tl. Therefore, the liquid-solid ratio should be increased as much as possible to make desorption more complete.

Table 3 Effect of solid-liquid ratio of adsorbent and desorbent on desorption

Tl content in resin (μg/g)	Solid-liquid ratio of adsorbent to desorbent	Desorption content of Tl (μg)	Desorption rate (%)
483.6	1:1	726.66	50.09
	1:2	943.88	65.06
	1:3	1128.50	77.78
	1:4	1185.63	81.72

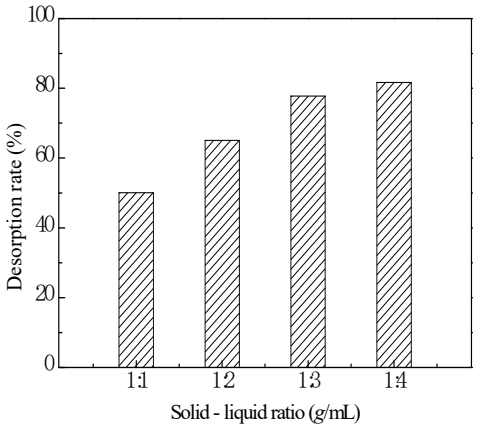

Fig. 9 Effect of solid-liquid ratio of adsorbent and desorbent on desorption

3.2.3 Effect of desorption time

Added 5% hydrochloric acid (60 mL) to the resin after the adsorption of raw water, and test the sample after shaking for 4, 8, 12, 16, 20 h respectively. The results were shown in Fig. 10 and table 4. As the desorption time is extended, the desorption rate is not significantly improved and remains at 64%-68%. Since the desorption process reached equilibrium within 4 h, there was no significant change in the desorption rate with the continued extension of the desorption time.

Table 4 Effect of desorption time on desorption

Tl content in resin (μg/g)	Desorption time (h)	Desorption content of Tl (μg)	Desorption rate (%)
483.6	4	996.63	68.69
	8	929.22	64.05
	12	1000.49	68.96
	16	956.63	65.94
	20	948.91	65.41

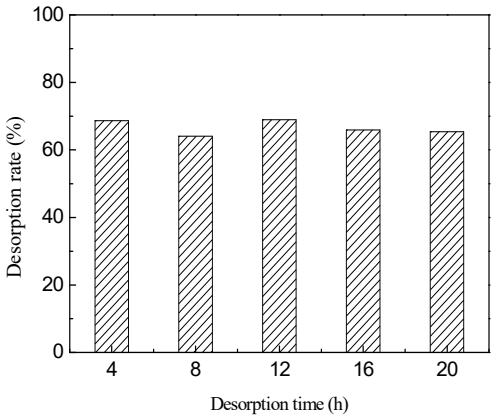

Fig. 10 Effect of desorption time on desorption

3.3 Resin regeneration properties study

Regeneration is an vital factor to assess the application performance of the adsorbent[38]. In order to investigate the regenerative properties of the resin, this paper studied the re-adsorption of used resin treated with hydrochloric acid on Tl-containing wastewater. The relationship between the times of adsorptions and the adsorption capacity and adsorption rate of the resin for Tl were shown in Fig. 11. It is found that the adsorption capacity and efficiency of the resin after repeated use for 6 times were still 612.6 μg/g and 95.8% respectively, indicating that the resin have good regenerability.

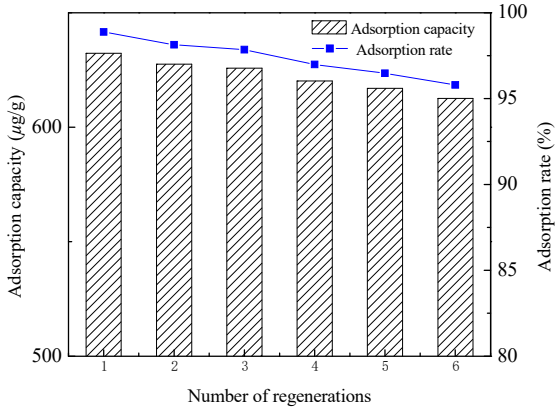

Fig. 11 The relationship between the number of regeneration and the adsorption capacity and adsorption rate

3.4 Dynamic adsorption study

Dynamic adsorption curve of chelating resin on Tl-containing wastewater were shown in Fig. 12. When the adsorption time is 25h, the concentration of Tl in the effluent from the adsorption column is exactly the same as the concentration of Tl in the original solution. At this point we can conclude that the adsorption of the resin has reached dynamic saturation. The amount of saturated adsorption can be calculated by the following formula:

$$q = \int_0^t Q\,(C_0 - C_t)dt = QC_0t - \int_0^t QC_t dt \quad (3\text{-}7)$$

Where, C_0 is the inlet concentration, C_t is the outlet concentration at t h, Q is wastewater flow, and the $\int_0^t QC_t dt$ is the area under the penetration curve when t h. It can be seen from the calculation that the cumulative adsorption capacity at the time of dynamic adsorption saturation (621.4μg/g) is slightly lower than that at the static adsorption saturation (636.94μg/g). Because of the adsorption heat and dynamic equilibrium, the temperature of the dynamic adsorption may rise, so the amount of dynamic adsorption is lower than that of static adsorption.

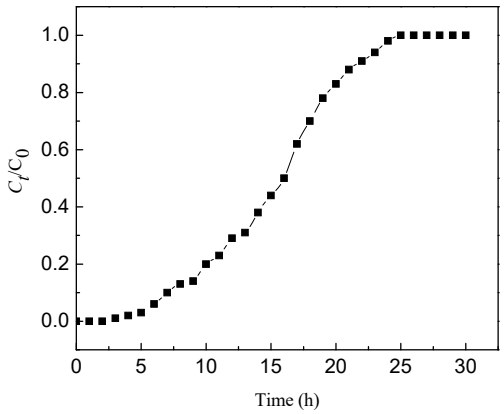

Fig. 12 The dynamic adsorption curve of resin on Tl

4. Conclusions

Based on the previous research on the treatment of Tl-containing wastewater, this work aims to optimize the process parameters by measuring the effects of pH, temperature, initial concentration of Tl and adsorption time on the adsorption performance of the resin. We can draw the following conclusions: The optimum pH and temperature of the adsorption process are 9 and 30°C; respectively.; the adsorption kinetics study shows that the adsorption of resin can reach the adsorption equilibrium at 80 min and consistent with the second-order kinetic adsorption model; the maximum adsorption capacity of the resin on Tl is 636.94 μg/g; the adsorption is better described by Langmuir isotherm model. After six reused cycles, the adsorption efficiency of resin on Tl was still at a high level (95.8%). This work could not only help us understand how the factors influence the adsorption behavior of resin onto Tl and determine the optimization process parameters, but also could provide a strong basis for the removal of heavy metals from wastewater.

Acknowledgements

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