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# Thermodynamics of $NO_3^-$ adsorption on different kinds of anion exchange resins

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The thermodynamic behavior of $NO_3^-$ adsorption from water on 201×7(717), Amberlite IRA 402 and Amberlite IRA 900, strong-base anion-exchange resin, were researched. After reaching equilibrium about adsorption of $NO_3^-$ on three resins, adsorption capacity and adsorption efficiency were analyzed. Freundlich and Langmuir isotherm model were fitted for the behavior of resin adsorbing $NO_3^-$. The thermodynamic functions of adsorption process were calculated. The results indicate that the adsorption effect of $NO_3^-$ for IRA 402 resin is the best, 717 is the second, and IRA 900 is the last, but there are an insignificant difference. Adsorption efficiency was very high for adsorption of 5-20 mg N/L $NO_3^-$ on three resins, which greater than 96%, but slightly lower than adsorption efficiency of the dynamic ion exchange column. Whereas the ion exchange resins have certain saturation capacity for the exchange of ions. The Langmuir equation could well describe the resins adsorbing $NO_3^-$. The adsorption process was spontaneous for Gibbs free energy change ($\Delta G$) value was negative. The enthalpy change ($\Delta H$) and entropy change ($\Delta S$) were positive, which indicate that adsorption of $NO_3^-$ is endothermic and entropy increasing process. Therefore increasing temperature is conducive to the more complete and faster adsorption.

**Key words:** $NO_3^-$, Anion exchange resin, Adsorption isotherm model, Thermodynamics.

## INTRODUCTION

Concentrated urban population, relatively low urban wastewater treatment rate, industrial waste water and exhaust, as well as excessive fertilizer of agricultural production, which result in nitrate pollution in water body [1]. High levels of nitrate in water can cause met-hemoglobin hematic disease. Furthermore, nitrate is reduced to carcinogenic nitrosamines, which make a serious threat to human health [2]. Therefore, to study how to remove nitrate in water and to protect the water ecological environment, maintaining the healthy development of the people and water in harmony, have certain theoretical and practical significance [3-4-5-6-7].

Compared with the other method, ion exchange technology due to its simplicity, effectiveness and good selectivity, easy recovery of inorganic ions and the cost is relatively low, which is more suitable for applied to remove the nitrate nitrogen in water [8-9-10-11]. Moreover, new preparation research based on ion exchange adsorption technology and how to improve the efficiency of ion exchange resin, which caused global attention increasingly [12-13-14-15-16].

Strong basic anion exchange resin, 201 x 7 (717), has been widely used in research on nitrate nitrogen adsorption [1, 2]. So we chose two kinds of new type of strong basic anion exchange resin, IRA 402 and IRA 900, produced by American Rohm Haas Company, and attempted to study $NO_3^-$ adsorption thermodynamics experiments of the three resins, expecting to find the ideal of $NO_3^-$ selective adsorption resin.

## MATERIALS AND METHODS

### *Experimental materials*

*Experimental reagents*. 1 mol/L HCl and 1 mol/L NaOH solution were acid and alkaline detergent respectively. 5.0, 10.0, 15.0, 20.0 mg N/L $KNO_3$ solution were nitrate simulation wastewater.

Strong basic anion exchange resin, 201 x 7 (717) (sand, 1 mm in diameter, yellow, easy to conglutinate things), was produced by Shantou Xilong Chemical Plant, Guangdong Province. Strong basic anion exchange resin, AMBERLITE IRA 402 (granular, fine, canary yellow, has certain adhesivity), and IRA 900 (bead-like structure, 1.5 mm in diameter, white, not easy to conglutinate things) were all produced by American Rohm Haas Company. Performance parameters of these three resins were shown in Table 1.

*The pretreatment of ion exchange resin*. Took certain amount (2 g) of resin in a 150 ml conical flask, poured 1 mol/L HCl solution inside the conical flask, which liquid level was above the resin 2-3 cm and the resin was soaked for 2-4 hours. After soaking by dilute hydrochloric acid solution, the resin was rinsed repeatedly with distilled water until the pH to neutral. Then poured 1 mol/L NaOH solution inside the conical flask, and operation method was the same as the first and

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second step. Finally, use 1 mol/L HCl solution soaking the resin for 2 hours, again washed the resin with distilled water until the pH to neutral.

### *Experimental apparatus*

The analyses of nitrate-nitrogen were performed using the Shimadzu 2550 ultraviolet-visible spectrophotometer (Shimadzu, Japan) and the Dionex ICS-1000 ion chromatography equipment (Dionex, USA).

### *Experimental method*

The static adsorption method was used for the need to study the thermodynamic behavior of resin adsorption $NO_3^-$ at different temperature. 100 ml $KNO_3$ solution of different concentrations (5.0, 10.0, 15.0, 20.0, 30.0, 20.0, 30.0 mg N/L) was poured into seven conical flasks containing resins with a volume of 150 ml. Then these conical flasks were fixed in the water-bathing constant temperature vibrator, and set different temperature (25 ºC, 35 ºC, 45 ºC) vibrating for a period of time. After fully exchange adsorption reaching equilibrium (according to the actual experimental conditions), ultraviolet spectrophotometry was used to determine residual nitrate nitrogen concentration in the solution [17]. Adsorption capacity, adsorption efficiency and the residual nitrate nitrogen concentration after adsorption equilibrium on resins for different initial concentrations of $KNO_3$ solution at different temperature were calculated. Freundlich and Langmuir isotherm model were fitted for the behavior of resin adsorbing $NO_3^-$. The thermodynamic function of adsorption process was calculated.

The computation formula of adsorption capacity and adsorption efficiency are as follows.

$$q_e = \frac{(c_0 - c_e) \times V}{G} \tag{1}$$

$$E = \frac{c_0 - c_e}{c_0} \times 100\% \tag{2}$$

Where $q_e$ is equilibrium adsorption capacity (mg/g); $c_0$ is initial concentration of nitrate nitrogen (mg/L) ; $c_e$ is nitrate nitrogen equilibrium concentration after adsorption (mg/L) ; $V$ is volume of solution (L) ; $G$ is resin quality (g) ; $E$ is adsorption efficiency (%).

## RESULTS AND DISCUSSION

### *Adsorption capacity and adsorption efficiency for resin adsorption of different initial concentration of $NO_3^-$*

Adsorption capacity and adsorption efficiency for resin adsorption of different initial concentration of $NO_3^-$ at different temperature were shown in table 2. Adsorption of 5-20 mg N/L $NO_3^-$ on resins had high efficiency, which was greater than 96%. Adsorption capacity had increased gradually. Compared with dynamic adsorption in the ion exchange column, adsorption efficiency in this experiment was slightly lower [18] It illustrate contact area of dynamic adsorption is greater than that static adsorption, and large area is conducive for resin to fully contact with the liquid phase. As the initial concentration of $NO_3^-$ solution increasing to 30 mg N/L, adsorption efficiency showed the decrease tendency and equilibrium adsorption capacity changed little. Which indicate the ion exchange resin have certain saturation capacity on the exchange of ions. The $NO_3^-$ equilibrium adsorption capacities for the three kinds of resin were all 1.55 mg/g approximately. With the increase of temperature, so did the $NO_3^-$ equilibrium adsorption capacity and adsorption efficiency for resin. It show that temperature rising is helpful to the resin adsorption, but the influence of temperature is not significant (Table 2).

From Table 2, data showed $NO_3^-$ adsorption effect for IRA 402 resin was the best, 717 was the second, and IRA 900 was the last. At 45 ºC, for the initial concentration of 5 mg/L of nitrate nitrogen, the adsorption efficiency was as high as 99.7% for IRA 402, 717 was 99.4%, and IRA 900 was 99.2%. At 25 ºC, adsorption efficiency for 717, IRA 402 and IRA 900 are 99.0%, 99.3% and 98.6% individually. It show that these three kinds of resin can be very good selective adsorption of $NO_3^-$

**Table 1.** Performance parameters of the three kinds of resin.

| Resin type | Moisture content/% | Total exchange capacity/ (mmol/g) | Functional group | Wet superficial density/ (g/mL) | Granularity (0.315~ 1.25mm) /% | Factory pattern |
|---|---|---|---|---|---|---|
| 717 | 42~48 | 3.6 | [—N($CH_3$)$_3$] | 0.66~0.75 | ≥95 | chloro-type |
| IRA 402 | 50~56 | 3.8 | [—N($CH_3$)$_3$] | 0.66~0.73 | ≥95 | chloro-type |
| IRA 900 | 50~60 | 4.0 | [══N══] | 0.65~0.73 | ≥95 | chloro-type |

**Table 2.** Adsorption efficiency and adsorption capacity for three resins adsorption of different initial concentration of $NO_3^-$ at different temperature.

| Temperature (°C) | Initial concentration (mg/L) | 717 | | IRA 402 | | IRA 900 | |
|---|---|---|---|---|---|---|---|
| | | E (%) | $q_e$ (mg/g) | E (%) | $q_e$ (mg/g) | E (%) | $q_e$ (mg/g) |
| 25 | 5 | 99.0 | 0.25 | 99.3 | 0.25 | 98.6 | 0.25 |
| | 10 | 98.3 | 0.49 | 98.9 | 0.49 | 97.9 | 0.49 |
| | 15 | 97.4 | 0.73 | 97.9 | 0.73 | 97.0 | 0.73 |
| | 20 | 97.2 | 0.97 | 97.6 | 0.98 | 96.1 | 0.96 |
| | 30 | 90.2 | 1.35 | 90.8 | 1.36 | 89.3 | 1.34 |
| | 40 | 75.7 | 1.51 | 77.9 | 1.56 | 73.5 | 1.47 |
| | 50 | 63.7 | 1.59 | 63.8 | 1.59 | 60.1 | 1.50 |
| 35 | 5 | 99.3 | 0.25 | 99.5 | 0.25 | 99.0 | 0.25 |
| | 10 | 98.6 | 0.49 | 98.8 | 0.49 | 98.1 | 0.49 |
| | 15 | 97.9 | 0.73 | 98.2 | 0.74 | 97.6 | 0.73 |
| | 20 | 97.5 | 0.98 | 97.7 | 0.98 | 97.0 | 0.97 |
| | 30 | 90.6 | 1.36 | 90.9 | 1.36 | 90.0 | 1.35 |
| | 40 | 77.4 | 1.55 | 78.2 | 1.56 | 75.1 | 1.50 |
| | 50 | 63.8 | 1.60 | 64.2 | 1.61 | 60.9 | 1.52 |
| 45 | 5 | 99.4 | 0.25 | 99.7 | 0.25 | 99.2 | 0.25 |
| | 10 | 99.0 | 0.49 | 99.0 | 0.49 | 98.3 | 0.49 |
| | 15 | 98.6 | 0.74 | 98.7 | 0.74 | 98.0 | 0.74 |
| | 20 | 97.7 | 0.98 | 98.2 | 0.98 | 97.3 | 0.97 |
| | 30 | 90.7 | 1.36 | 91.3 | 1.37 | 90.4 | 1.36 |
| | 40 | 77.6 | 1.55 | 80.0 | 1.60 | 75.5 | 1.51 |
| | 50 | 63.9 | 1.60 | 64.8 | 1.62 | 62.0 | 1.55 |

### *Isothermal adsorption model for resin adsorption of $NO_3^-$*

In the process of ion exchange adsorption, adsorption isotherm can be used for analysis of adsorption behavior for adsorbent and the specific adsorbate [19]. According to the previous experiences, the resin adsorption capacity ($q_e$) depends on adsorption equilibrium concentration of adsorbate ion ($c_e$) in the liquid phase and the temperature (T) under the specific conditions. When the temperature is constant, the function equation between $q_e$ and $c_e$ is adsorption isotherm. Langmuir and Freundlich isothermal adsorption model are commonly used as common adsorption isotherms to describe adsorption equilibrium between solid and liquid phase [20]. Langmuir adsorption isotherm model can predict resin maximum adsorption capacity, who assumes that adsorption process occurs on the surface of the adsorbent, and has the same affinity on adsorbate. Adsorbate transfer into the adsorbent surface and form into monolayer adsorption, without considering inhomogeneous adsorption [21]. Freundlich model is the liquid-solid empirical adsorption equation, considering inhomogeneous adsorption. However, it cannot predict the situation of adsorption balance, and have no actual physical meaning, is believed as purely empirical [22].

### *Adsorption isothermal equation*

(1) Freundlich adsorption isothermal model

$$q_e = k_f (c_e)^{\frac{1}{n}} \tag{3}$$

The logarithmic form is

$$\ln q_e = \ln k_f + \frac{1}{n} \ln c_e \tag{4}$$

where, $k_f$ and $1/n$ are constant related to the adsorption intensity and capacity; $q_e$ and $c_e$ is as in the above case.

(2) Langmuir adsorption isothermal model

$$\frac{1}{q_e} = \frac{1}{b} \times \frac{1}{c_e} + \frac{1}{q_m} \tag{5}$$

where $q_m$ is the maximum uptake value; $b$ is coefficient related to the adsorption capacity ($b=qm/k_l$) ; $k_l$ is Langmuir dissociation constant of adsorption equilibrium; $q_e$ and $c_e$ is as in the above case.

### *Adsorption isotherm model analysis*

According to equilibrium concentration of $NO_3^-$ in the solution and adsorption capacity after adsorption, drew the adsorption isotherm of $NO_3^-$ at different temperature (25 °C, 35 °C, 45 °C). Experimental data were respectively fitted with the Freundlich and Langmuir adsorption isotherm equation. Fitting results were shown in figure 1, 2, 3, and the fitting parameters were listed in table 3.

Figures 1~3 and Table 3 showed that adsorption isotherm of three kinds of resin at different temperature were coincide with the Langmuir adsorption model, and the correlation coefficient ($R^2$) were greater than 0.95. In compared, $R^2$ of Freundlich was around 0.89, slightly smaller than that of Langmuir. The adsorption thermodynamics experiment was conducted in 150 ml flask, and adsorbent were evenly lay on the flat surface, which conform to the adsorption conditions of Langmuir adsorption model. Langmuir adsorption model also shows that adsorption of $NO_3^-$ on resins will not always increase with the increment of initial concentration, but has its maximum uptake value, namely the saturation capacity. With temperature rising from 25 °C to 45 °C, qm increased and $k_l$ decreased in the adsorption process, which indicate that temperature is advantageous to the adsorption. At different temperature, Freundlich constant n was all greater than 1, showing that the three kinds of resin have strong adsorption ability for $KNO_3$ solution (Table 3).

*Analysis and calculation of thermodynamic function*

At the temperature of 25 °C (298k), 35 °C (308k) and 45 °C (318k), thermodynamic parameters for $NO_3^-$ adsorption on resins in water can be estimated based on the following equations.

*Gibbs change of adsorption process (ΔG).* Change of the Gibbs free energy ($\Delta G$) in the process of $NO_3^-$ adsorption on resins was estimated by Van't Hoff isothermal formula [23].

$$\Delta G = -RT \ln K_0 \tag{6}$$

Where, $R$ is gas constant, and the value is 8.314 J/(mol.K). $T$ is temperature, and the unit is K. $K_0$ is the adsorption equilibrium constant, which is equal to $K_f$ as estimated by Freundlich equation fitting parameters [23]. And $K_0$ is equal to $1/K_l$ and $b/q_m$ as estimated by Langmuir equation fitting parameters [24]. We used Langmuir equation parameters to calculate $K_0$ (Table 4)

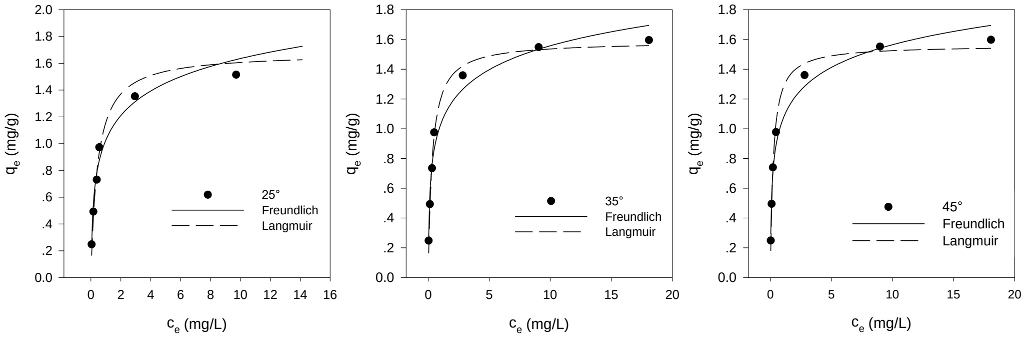

**Fig. 1.** Freundlich and Langmuir equation fitting curve for 717 resin.

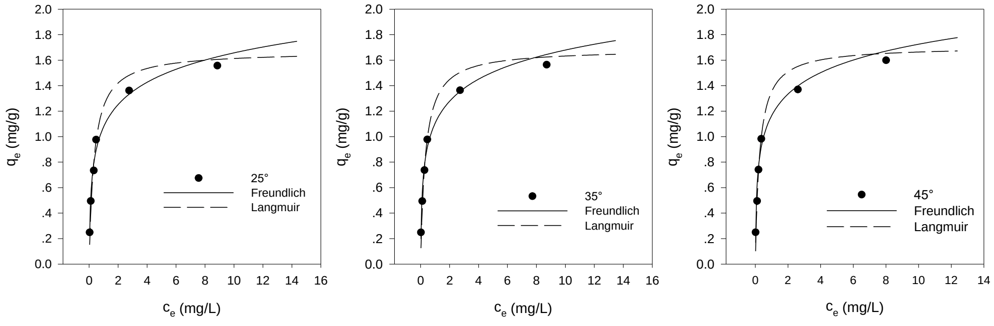

**Fig. 2.** Freundlich and Langmuir equation fitting curve for IRA 402 resin.

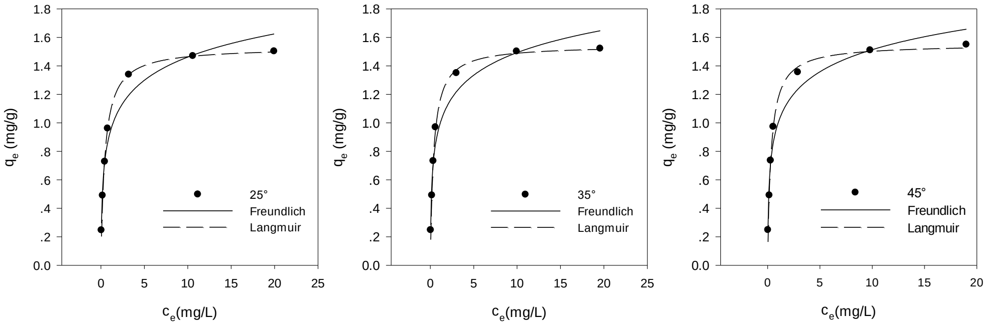

**Fig. 3.** Freundlich and Langmuir equation fitting curve for IRA 900 resin.

**Table 3.** Freundlich and Langmuir isotherm parameters for adsorption of $NO_3^-$ on three resins at different temperature.

| Resin type | Temperature/K | Parameters for Freundlich equation | | | Parameters for Langmuir equation | | | |
|---|---|---|---|---|---|---|---|---|
| | | $K_f$ | $n$ | $R^2$ | $b$ | $q_m$ | $k_l$ | $R^2$ |
| 717 | 298 | 0.84 | 3.37 | 0.89 | 6.10 | 1.27 | 0.22 | 0.98 |
| | 308 | 0.87 | 3.48 | 0.89 | 8.00 | 1.31 | 0.16 | 0.97 |
| | 318 | 0.91 | 3.70 | 0.89 | 9.71 | 1.33 | 0.14 | 0.99 |
| IRA 402 | 298 | 0.89 | 3.53 | 0.90 | 8.55 | 1.14 | 0.15 | 0.98 |
| | 308 | 0.91 | 3.62 | 0.91 | 11.49 | 1.21 | 0.10 | 0.96 |
| | 318 | 0.96 | 3.80 | 0.91 | 18.87 | 1.30 | 0.06 | 0.95 |
| IRA 900 | 298 | 0.78 | 3.32 | 0.88 | 4.22 | 1.24 | 0.32 | 0.99 |
| | 308 | 0.82 | 3.45 | 0.88 | 5.95 | 1.29 | 0.22 | 0.98 |
| | 318 | 0.85 | 3.55 | 0.88 | 7.69 | 1.37 | 0.16 | 0.97 |

**Table 4.** Thermodynamic equilibrium parameters for adsorption of $NO_3^-$ on three resins at different temperature.

| Resin type | $T$ (K) | $K_0$ | $\Delta G$ (kJ/mol) | $\Delta H$ (kJ/mol) | $\Delta S$ [J/(mol·K) ] |
|---|---|---|---|---|---|
| 717 | 298 | 4.65 | -11.51 | 16.35 | 93.51 |
| | 308 | 6.29 | -16.10 | | 105.37 |
| | 318 | 7.29 | -19.28 | | 112.05 |
| IRA 402 | 298 | 6.57 | -16.28 | 29.76 | 154.52 |
| | 308 | 9.53 | -24.40 | | 175.86 |
| | 318 | 16.58 | -43.85 | | 231.48 |
| IRA 900 | 298 | 3.09 | -7.65 | 22.86 | 102.40 |
| | 308 | 4.63 | -11.86 | | 112.73 |
| | 318 | 6.18 | -16.35 | | 123.32 |

$\Delta G$ of $NO_3^-$ adsorption process was negative at different temperature for three kinds of resin. It illustrate the adsorbate tend to adsorbing from solution to the surface of resin and the adsorption process can occur spontaneously. And with the increase of temperature, absolute value of ΔG increases, which further demonstrate that temperature rising accelerate the process of $NO_3^-$ adsorption on resins.

*Enthalpy change of adsorption process (ΔH).* Enthalpy change ($\Delta H$) in the process of $NO_3^-$ adsorption on resins was estimated by the following Van't Hoff equation (7) [24]. Using $\ln(1/c_e)$ and $1/T$, a linear relationship model was built, and the slope was obtained. After conversion, $\Delta H$ can be estimated.

$$\ln(1/c_e) = A + (-\Delta \mathrm{H}/RT) \qquad (7)$$

In order to calculate $\Delta H$, $NO_3^-$ adsorption experiments with a concentration of 5 mg/L were added for three kinds of resin at 30 °C (303 k) and 40 °C (313 k). $NO_3^-$ equilibrium concentration after adsorption was measured and drew with different temperature (Figure 4). $\Delta H$ for three kinds of resin was obtained by the slop in the figure.

As shown in table 4, the enthalpy change affected by the temperature was insignificant, therefore the enthalpy change at different temperature was identical. $\Delta H$ was all greater than zero for three kinds of resin. $K_0$ increased as the temperature rising, showed that the adsorption behaviors were endothermic process for the three kinds of resin. Temperature helps $NO_3^-$ adsorbing on resins.

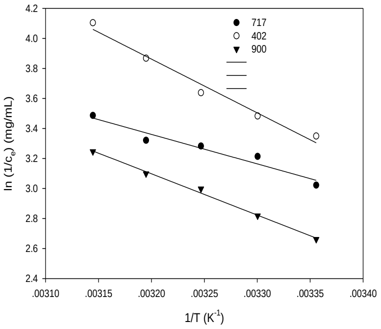

**Fig. 4.** The liner fitting curve between $\ln(1/c_e)$ and $1/T$ for three resins.

*Entropy change of adsorption process (ΔS).*

$$\Delta S = (\Delta H - \Delta G)/T \qquad (8)$$

The entropy change ($\Delta S$) in the process of $NO_3^-$ adsorption on resins was estimated by the following Gibbs-Helmholtz equation (8) [24].

The calculation results were shown in table 4, Δ S are greater than zero for three kinds of resin, which show that $NO_3^-$ adsorption on the resin is an entropy increase process. For the solid-liquid adsorption behavior, adsorbate molecules adsorbing to the resin surface by liquid phase will lose some freedom, which is a process of entropy decrease. Adsorbate adsorbs on the adsorbent (resin), and exchange with the ion exchange group of resin phase. The group and the hydration water molecules releasing into the liquid phase is the process of entropy increase. So the total entropy of adsorption processes are the sum of the both [24-25]. Due to the large volume of the exchange groups of three kinds of resin, especially for the 717 and IRA 402 (Table 1), therefore $\Delta S$ is positive in the adsorption process.

## CONCLUSION

This paper studied $NO_3^-$ adsorption thermodynamic properties on strongly basic anion exchange resin, 717, IRA 402 and IRA 900. Two kinds of adsorption isotherm at different temperature were measured and thermodynamic parameters, $\Delta G$, $\Delta H$ and $\Delta S$ in the adsorption process were estimated.

The adsorption effect of $NO_3^-$ for IRA 402 resin is the best, 717 is the second, and IRA 900 is the last, but there are an insignificant difference. Adsorption efficiency was outstanding for adsorption of 5 mg N/L $NO_3^-$ on three resins, which greater than 98% and indicate these three kinds of resin have excellent selectivity on adsorption of $NO_3^-$. Compared with the common 717 resin, IRA 402 and IRA 900 also can be better resins for $NO_3^-$ adsorption.

Adsorption efficiency was very high for adsorption of 5-20 mg N/L $NO_3^-$ on three resins, which greater than 96%, but slightly lower than adsorption efficiency of the dynamic ion exchange column. As the initial concentration of $KNO_3$ solution rising to 30 mg N/L, adsorption efficiency appeared decrease trend. It indicate ion exchange resin have certain saturation capacity for the exchange of ions. With the increase of temperature, so did the $NO_3^-$ equilibrium adsorption capacity and adsorption efficiency for resin. It demonstrate that temperature rising is helpful to the resin adsorption, but the influence of temperature is not significant.

Freundlich and Langmuir equation fitting correlation coefficient $R^2$ of $NO_3^-$ adsorption isotherm for three kinds of resin were greater than 0.85. And the Langmuir equation coefficient was greater, more than 0.95, which indicated that the Langmuir isotherm equation can well describe the adsorption process of $NO_3^-$.

$\Delta G$ of $NO_3^-$ adsorption process was negative at different temperature for three kinds of resin. Which illustrate the adsorption process can occur spontaneously. And with the increase of temperature, absolute value of $\Delta G$ increases, which further demonstrate that temperature rising may accelerate $NO_3^-$ adsorption process for resin.

$\Delta H$ and $\Delta S$ were all greater than zero in the process of $NO_3^-$ adsorption on three kinds of resin, showing that adsorption of $NO_3^-$ is endothermic and entropy increasing process. Therefore increasing temperature is conducive to the more complete and faster adsorption. Therefore, increasing temperature is conducive to the adsorption process more complete and faster.

The research conclusion for strongly basic anion exchange resin to remove nitrate from water have certain guiding and application significance. In the future, adsorption dynamics of $NO_3^-$ on strongly basic anion exchange resin and elution recycling can be further analyzed and discussed. Our goal is using cheap, available and good selective adsorption resin to dispose nitrogen wastewater, and reduce the damage and pollution to the environment.

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