

Study on the Adsorption Properties of D113 Cation Exchange Resin for Zn^{2+}

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Abstract: *The adsorption properties of D113 cation exchange resins on Zn^{2+} were researched systematically using the traditional batch method. Under different initial Zn^{2+} concentrations, reaction times, and temperatures, the adsorption efficiency and capacity were measured respectively. In addition, the adsorption equilibrium of D113 resin for Zn^{2+} obeyed the Langmuir adsorption isotherm model well; the adsorption process agreed with pseudo second-order kinetics model, and was dominated by chemisorption; $\Delta H > 0$ and $\Delta G > 0$ at different temperatures indicated the adsorption was a spontaneous endothermic process in nature. Finally, SEM and FT-IR were properly used to characterize the resins.*

Keywords: *D113 cation exchange resin, Zn^{2+} , batch adsorption, isothermal adsorption model, kinetic model*

1. Introduction

Ion exchange resin is a kind of polymer mainly composed of the skeleton and functional groups, and it is generally applied for separation, adsorption, and purification [1-4]. In recent years, ion exchange resin has been widely used in the preparation of inorganic materials, including layered double hydroxides [5, 6], nano-ZnO [7], etc. [8, 9]

Weak acid ion exchange resin has a strong exchange capacity, good cyclicality, and high efficiency [10]. D113 resin is a kind of macroporous weak acid acrylic acid cation exchange one as an adsorbent to remove some harmful ingredients, such as ammonium-nitrogen [11], copper [12], etc.[13], and as a catalyst to synthesize three-dimensional activated graphene network [14], polyoxymethylene dimethyl ethers [15], etc. Moreover, D113 resin is usually applied in the field of materials preparation [16,17]. In this field, D113 resin acts as a donor of exchangeable ions and adjusts the morphology of inorganic materials by its unique structure. The amount of adsorption capacity depends on the amount of resin used in the water treatment industry; Furthermore, the morphology, yield and purity of the materials are greatly affected by the adsorption capacity of exchangeable ions on the resin. Therefore, enhancing the adsorption capacity is very necessary, and it is very important to study the adsorption conditions and the interrelation between exchangeable ions and resins.

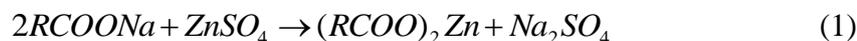
In this work, the adsorption of Zn^{2+} on D113 cation exchange resin was investigated systematically. Through the traditional batch method, the adsorption efficiency and capacity of the ion exchange resin for Zn^{2+} under different reaction conditions were measured in detail, and the optimal adsorption condition, the optimum isothermal adsorption model and kinetic model were explored systematically, which were helpful enthusiastically for adsorption performance study of ion exchange resin for some cations. In addition, this study also provided useful data support for the precursor's preparation of inorganic materials, such as nano-ZnO, nano-ZnS, micron-grade $Zn(OH)_2$, nano- $ZnFe_2O_4$, etc. by ion-exchange method. In the same time, this study also expanded advantageously the application of D113 resin in the water treatment industry, especially in the heavy metal removal progress [18].

2. Materials and methods

All of the reagents used were analytical grade and were obtained from the Chinese Xilong Chemical Co., Ltd. Deionized water was used in all of the experiments. The ion exchange resins were supplied from the Chemical Plant of NanKai University, Table 1 shows the properties of the resin selected.

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A certain amount of D113 resin after pretreatment and $ZnSO_4$ solution were efficiently mixed in a ground-glass stoppered flask, and then the flask was placed in a Digital display water bath temperature oscillator (SHA-C, Jiangnan instrument factory in Jintan city, China), to set the temperature and adjust the oscillation amplitude for a certain time, to measure the Zn^{2+} concentration before and after reaction by the following EDTA Titration. The chemical reaction equation is shown by equation (1):



The Zn^{2+} concentration was determined as follows: 1-2 drops of xylenol orange disodium salt as the indicator were added into a certain amount of Zn^{2+} solution, to add the hexamethylenetetramine solution into the Zn^{2+} solution until the solution presented stable amaranth, and to add more 5 mL. EDTA standard solution titrated the solution from amaranth to bright yellow, i.e. the endpoint. The exact concentration of Zn^{2+} at different conditions was calculated according to the dosage of EDTA standard solution. Moreover, to illustrate the adsorption of Zn^{2+} on resins, three same experiments, that is parallel, were carried out under each experimental condition, subsequently, the average value was obtained.

Table 1. The properties of ion exchange resin

Items	Properties
Name	D113
Polymer structure	Macroporous weak acid acrylic acid
Functional group	-COOH
Total exchange capacity	(a) ≥ 10.8 mmol/g (dry, H type) (b) ≥ 4.2 mmol/mL (wet, H type)
Physical form	Milky or yellowish opaque spherical beads
Effective size (mm)	0.315-1.25
Maximum service temperature ($^{\circ}C$)	100
pH range of application	5-14

A pH Meter (ST2100, Ohaus instrument Corporation in Changzhou, China) was used for measuring pH of solutions. Resins were dried before characterization using Electrothermal constant temperature drying oven (DHG-9030A, Shanghai Yiheng corporation, China) to remove the adsorbed water under $60^{\circ}C$. FT-IR spectra of the resins were obtained from a FT-IR spectrometer (Tensor II, Bruker, Germany). The morphology of resins was observed and analyzed by SEM (Zeiss Supra55VP, German).

Finally, based on the experimental data, the adsorption efficiency, the adsorption capacity and the partition coefficient under different reaction conditions were accurately calculated. The related calculation formulas were followed as (2), (3) and (4).

$$\text{Adsorption efficiency} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (2)$$

$$q_e = \frac{(c_0 - c_e) \times V}{W} \quad (3)$$

$$K_d = \frac{(C_0 - C_e)}{C_e} \times \frac{V}{W} \quad (4)$$

where q_e is the adsorption capacity of resins ($\text{mg}\cdot\text{g}^{-1}$), C_0 means the initial concentration of Zn^{2+} ($\text{mg}\cdot\text{L}^{-1}$), C_e is the equilibrium concentration of Zn^{2+} ($\text{mg}\cdot\text{L}^{-1}$), K_d is the partition coefficient ($\text{mL}\cdot\text{g}^{-1}$), V represents the solution volume (L), and W is the resin quality (g).

3. Results and discussions

3.1. Effect of initial concentration on adsorption

D113 resin of 2 g and ZnSO_4 solution with a different concentration completely mixed and reacted at 70-80 rpm and 303 K for 2 h, and the molar amount of Zn^{2+} remained unchanged for 13 mmol and was excessively compared with resin dosage. The effect of the different initial concentrations, including 0.2, 0.5, 0.8, 1.0, and 1.2 mol/L, on the adsorption of ion exchange resin for Zn^{2+} ion, was researched carefully.

Figure 1 is adsorption efficiency and capacity curves under different ZnSO_4 concentrations. It shows that the adsorption efficiency remained basically between 74 and 76 % with the increase of ZnSO_4 concentration, whereas q_e increased gradually. It was noteworthy that when the concentration was greater than 1.0 mol/L, the corresponding q_e was unchanged, and 405 mg/g was the maximum adsorption capacity, which was bigger than 184.5 mg/g of some novel organic chelating resin [19]. The total exchange capacity of D113 resin was more than 10.8 mmol/g (dry) in Table 1. According to equation (1) and the molar mass of Zn, the theoretical adsorption capacity of 2 g D113 resin for Zn^{2+} ion was more than 353.02 mg/g. Therefore by comparison the above result was very ideal and believable. In addition, during the reaction by multiple determinations pH value of the solution under different concentration was the same, maintaining between 5.63 and 5.85, that is, the adsorption was in a relatively small pH range, and the adsorption environment was beneficial to the stable release and exchange of ions.

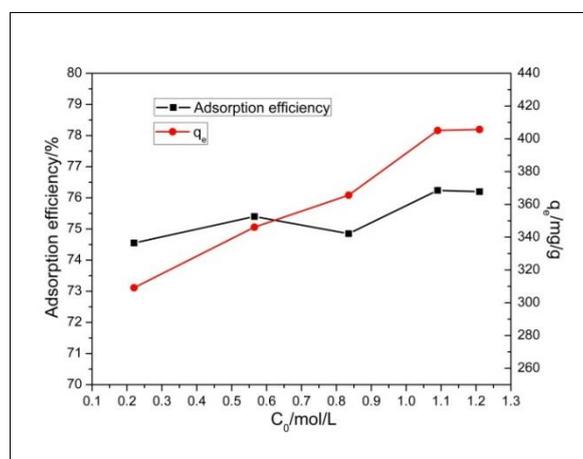


Figure 1. Effect of different initial concentration on Zn^{2+} adsorption

There were lots of larger channels in the internal of the resin beads, and there were a large number of exchangeable ions in the inner wall of the channel and on the bean surface [20]. At a certain time and temperature, the initial concentration of ZnSO_4 was greater, the contact between Zn^{2+} ions and the resins was more adequate, and more Zn^{2+} ions were adsorbed onto the resins, which induced the gradual enhancement of q_e . When these exchangeable positions were fully occupied, q_e naturally remained unchanged. In addition, because of the bigger initial concentration, its adsorption efficiency was consistent with the other results according to formula (2). Thus, the adsorption efficiency could be kept within a certain range.

Furthermore when the Zn^{2+} concentration was higher, the resistance between ion exchange and adsorption increased gradually. Especially when the adsorption capacity of the resin reached saturation,

the surface free energy also decreased, and the steric hindrance to Zn^{2+} adsorption also enhanced further, so that the adsorption ability of the resin for Zn^{2+} decreased. Therefore, the increase of the adsorption capacity slowed down until equilibrium.

Therefore, it was very distinct that the adsorption efficiency wasn't almost relative with the initial concentration of $ZnSO_4$, but the greater the initial concentration was, the greater the adsorption capacity of resin for Zn^{2+} was.

Langmuir and Freundlich isotherm adsorption equation are two common adsorption isothermal models for the study of the adsorption process [21,22]. Using these adsorption isotherm models, the adsorption mechanism of ion exchange resin on Zn^{2+} is studied. If it is in agreement with Langmuir isothermal adsorption equation, then the adsorption process belongs to monolayer homogeneous adsorption. The equation is shown by formula (5):

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \quad (5)$$

where K_L is the related parameter of adsorption energy; q_m is the maximum adsorption capacity of resins ($mg \cdot L^{-1}$).

If it is in agreement with Freundlich isothermal adsorption equation, then it could show that the adsorption process of ion exchange resin on Zn^{2+} belongs to inhomogeneous adsorption. The equation is presented by formula (6):

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (6)$$

where K_F is the parameter related to adsorption capacity; n is the parameter related to adsorption intensity.

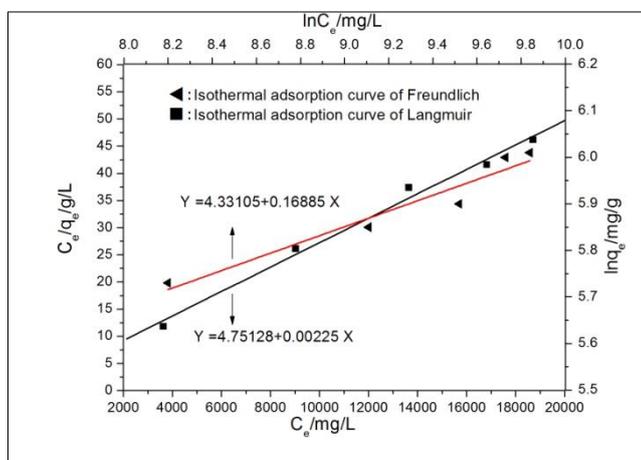


Figure 2. Isothermal adsorption curves of Langmuir and Freundlich for adsorption of Zn^{2+} by resin

Figure 2 illustrates Langmuir and Freundlich fitting curves, and the corresponding fitting equations were obtained, and the related parameters were further calculated by the slope and the intercept and listed in Table 2. The results exhibited that the linear correlation coefficients about Langmuir and Freundlich isotherm adsorption curves of D113 resin on Zn^{2+} were $R^2=0.99006$ and $R^2=0.94309$ respectively, which could explain that the adsorption process of D113 resins on Zn^{2+} accorded with Langmuir adsorption isotherm model and belonged to the monolayer adsorption. From the parameters of Langmuir isothermal adsorption equation, the maximum adsorption capacity of 1 g D113 resin for Zn^{2+} was $444.44 \text{ mg} \cdot \text{g}^{-1}$.

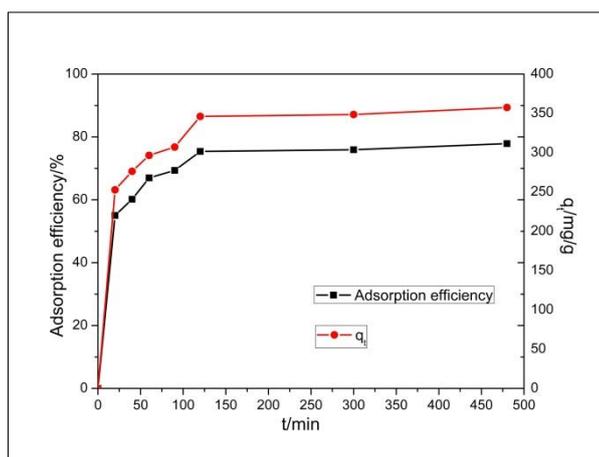
Table 2. Parameters of Langmuir and Freundlich isothermal for adsorption of Zn²⁺ by resin

Langmuir isothermal adsorption equation			Freundlich isothermal adsorption equation		
q _m (mg·g ⁻¹)	K _L (mg·L ⁻¹)	R ²	K _F (mg ¹⁻ⁿ ·g ⁻¹ ·L ⁿ)	1/n	R ²
444.44	0.0004736	0.99006	76.02	0.16885	0.94309

3.2. Effect of reaction time on adsorption

Effect of different reaction times (20, 40, 60, 90, 120, 300 and 480 min) on adsorption of 2 g D113 resin for Zn²⁺ (Figure 3) was investigated at 70-80 rpm and 303 K with the initial concentration of 0.5 mol/L and the Zn²⁺ molar amount of 13 mmol. It was seen that within 20 min the adsorption efficiency had reached 55 %, and then the adsorption efficiency and adsorption capacity increased gradually with the extension of time, eventually tended to be stable, basically reached equilibrium during 120-480 min, the adsorption capacity reached approximately 350 mg/g.

The removal rate of Zn²⁺ in solution, i.e. the adsorption efficiency of the resin for Zn²⁺, and the adsorption capacity change of resin with reaction time directly reflected the adsorption speed of resin for Zn²⁺, which was a reflection of the microscopic adsorption process of resin beads for Zn²⁺. In the initial stage of adsorption, the adsorption for Zn²⁺ was very fast because of the electronegativity of resin beads and a large number of exchangeable ions in the channels, which made the curve steep. Then, with the increase of time, the surface free energy of resin beads gradually decreased, and the exchangeable sites in the channels decreased accordingly, so that the adsorption speed slowed down, the adsorption efficiency and adsorption capacity increased slowly until they reached equilibrium. In summary, it could be determined that 120 min was the optimum adsorption time.

**Figure 3.** Effect of different reaction times on Zn²⁺ adsorption

To further study the influence of reaction time on the adsorption of Zn²⁺ by D113 resin, pseudo first order and pseudo two order adsorption kinetic models are adopted to research the adsorption speed [23, 24]. Two fitting equations are given in formulas (7) and (8):

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (7)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (8)$$

where q_t is the adsorption capacity of resins at various time t (mg·g⁻¹); k₁ is the coefficient of pseudo first-order kinetics model (min⁻¹); and k₂ is the coefficient of pseudo second-order kinetics model (g · mg⁻¹ · min⁻¹).

According to the above equations, the adsorption kinetics data are fitted, the corresponding graphs are shown in Figure 4. Based on the intercepts and slopes of the fitting curves, q_e , k_1 , k_2 , R^2 are calculated and listed in Table 3.

It could be seen from Table 3, the linear correlation coefficients of pseudo first order and pseudo two order adsorption kinetic model were 0.71924 and 0.98949 respectively, and the simulation calculated value, $q_e=413.22 \text{ mg}\cdot\text{g}^{-1}$ obtained by fitting pseudo two order adsorption kinetic model was closer to the equilibrium adsorption experimental value, $q_e=419.25 \text{ mg}\cdot\text{g}^{-1}$. It showed that the adsorption of D113 resin for Zn^{2+} conformed to the pseudo second order adsorption kinetic model, and the adsorption process was dominated by chemisorption.

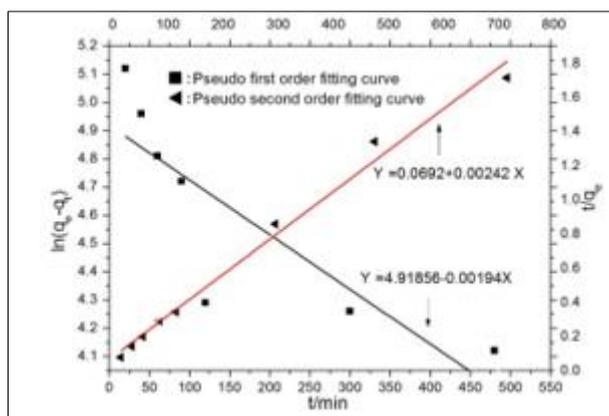


Figure 4. Pseudo first order and second order fitting curves for Zn^{2+} adsorption by resin

Table 3. Pseudo first and pseudo second order kinetic parameters of Zn^{2+} adsorbed on D113 resin

q_e (exp) $\text{mg}\cdot\text{g}^{-1}$	Pseudo first order kinetics model			Pseudo second order kinetics model		
	k_1 min^{-1}	q_e (cal) $\text{mg}\cdot\text{g}^{-1}$	R^2	k_2 , $\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1}$	q_e (cal) $\text{mg}\cdot\text{g}^{-1}$	R^2
419.25	0.00194	136.81	0.71924	8.46×10^{-5}	413.22	0.98949

3.3. Effect of reaction temperature on adsorption

The adsorption of 2 g D113 resin for Zn^{2+} at various temperatures (293, 303, 313 and 323 K) is illustrated in Figure 5, and the initial concentration of ZnSO_4 was 0.5 mol/L for Zn^{2+} molar amount of 13 mmol. When the temperature increased from 293 K to 303 K, the adsorption efficiency raised rapidly; but when the temperature continued to rise until 313 K, the adsorption efficiency increased slightly; when the temperature increased to 323 K, the adsorption efficiency reached more than 90%. Therefore, the temperature had a significant impact on the adsorption of the resin.

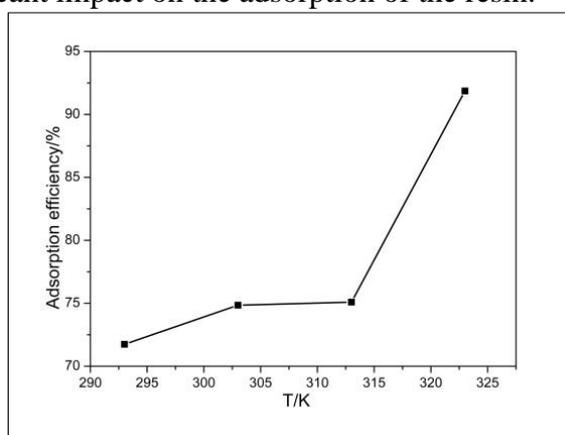


Figure 5. Effect of different reaction temperatures on Zn^{2+} adsorption

This was because the resin was heated, they had a certain degree of expansion [25], the internal channels also enlarged; while the increase of temperature driven Zn^{2+} in the solution to move more quickly [26], which made more Zn^{2+} enter into the channels and had the fast replacement reaction with the exchangeable ions, and the effective adsorption and the ion exchange reaction were greatly improved. Therefore, the enhancement of temperature could further raise the adsorption efficiency on Zn^{2+} .

Through the change of Gibbs free energy ΔG , enthalpy change ΔH and entropy change ΔS , the influence of temperature on the adsorption process of D113 resin was further considered. The values of these thermodynamic functions could be calculated by the followed formula (9):

$$\ln K_d = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (9)$$

where $R=8.314 \text{ J}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$, is the gas molar constant; T means the absolute temperature (K).

The variations of $\ln K_d$ vs $1/T \times 10^{-3}$ were demonstrated in Figure 6, ΔH and ΔS were computed by the intercept and the slope respectively, and the ΔG at different temperatures were calculated according to formula (10). The values of the above thermodynamic functions were listed in Table 4.

$$\Delta G = \Delta H - T\Delta S \quad (10)$$

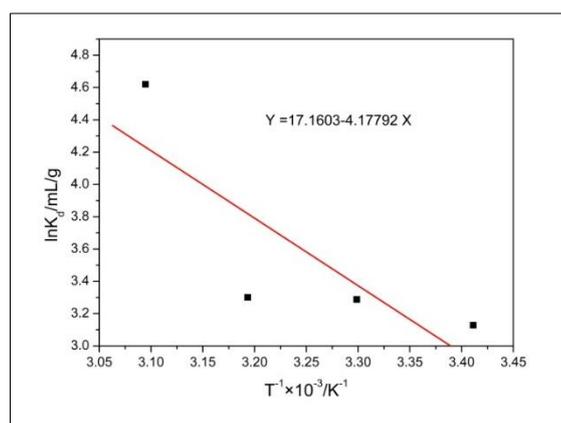


Figure 6. The variations of $\ln K_d$ vs $1/T \times 10^{-3}$ for Zn^{2+} adsorption by resin

Table 4. Thermodynamic parameters for adsorption of Zn^{2+} on ion exchange resins

Adsorbent	ΔH $\text{J}\cdot\text{mol}^{-1}$	ΔS $\text{J}\cdot\text{mol}^{-1}$	$\Delta G \text{ J}\cdot\text{mol}^{-1}$			
			293 K	303 K	313 K	323 K
D113 resin	34734.56	142.67	-7089.15	-8515.85	-9942.55	-11369.3

In Table 4, $\Delta H > 0$ suggested the adsorption of D113 resin on Zn^{2+} was endothermic; $\Delta S > 0$ exhibited the entropy was increased during the adsorption process; at different temperatures (293, 303, 313 and 323 K) $\Delta G < 0$ indicated that this process was spontaneous.

3.4. Characterization of resins

The morphology and surface structure of D113 resins before and after Zn^{2+} adsorption are shown in Figure 7. Compared with the smooth surface of resin before Zn^{2+} adsorption, the surface of resin after Zn^{2+} adsorption changed thicker and coarser with so many uneven ravines, just as a wrinkled ball. In addition, the surface of the cation exchange resin was negatively charged, and Zn^{2+} ions were easily adsorbed on the surface of the resin. So it was very distinct that the resin adsorbed a lot of Zn^{2+} ions.

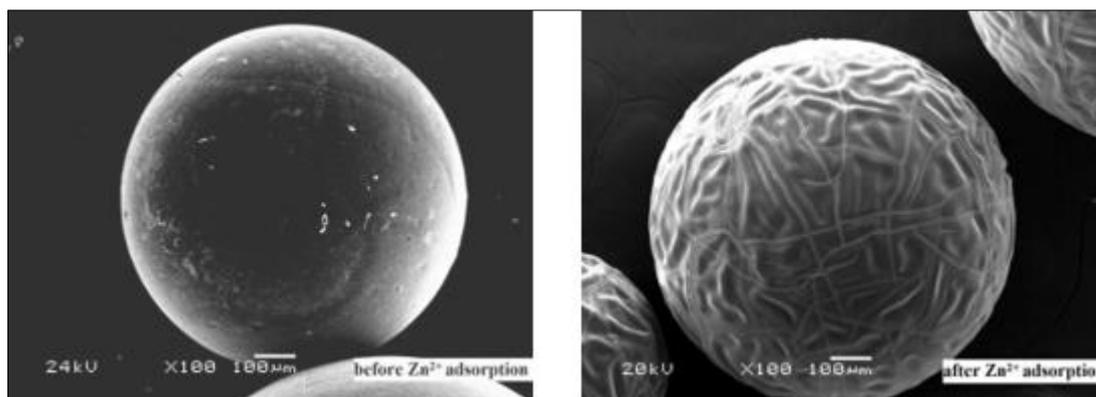


Figure 7. SEM images of D113 resin before and after Zn^{2+} adsorption

Moreover, FT-IR analysis is used to determine the resins adsorption mechanism for Zn^{2+} . Figure 8 shows FT-IR spectra of D113 resin before and after Zn^{2+} adsorption. The broad peak at around 2880 cm^{-1} corresponded to the C-H stretching vibration on acrylic skeleton. The absorption peaks at 1732 and 1397 cm^{-1} showed the presence of C=O and C-OH respectively in (a). After Zn^{2+} adsorption the above peaks all changed and transferred, as shown in (b). The absorption band at 1732 cm^{-1} disappeared for C=O, moreover the new peak formed at 1552 cm^{-1} for C=O, and the characteristic band of C-OH was indicated at 1409 cm^{-1} . During the vibrations of C=O and C-OH on resin matrix, because of the reaction of metal cations and these functional groups, the electronic distribution on the functional groups changed, the electron cloud density was averaged, and the stretching force constant declined, which could lead to the position moving of infrared absorption peaks [27]. Therefore, above analytical results explained the oxygen atoms and Zn^{2+} were connected by coordination bonds and H of C-OH was truly exchanged and displaced by Zn^{2+} . The results further confirmed the adsorption process was dominated by chemisorptions.

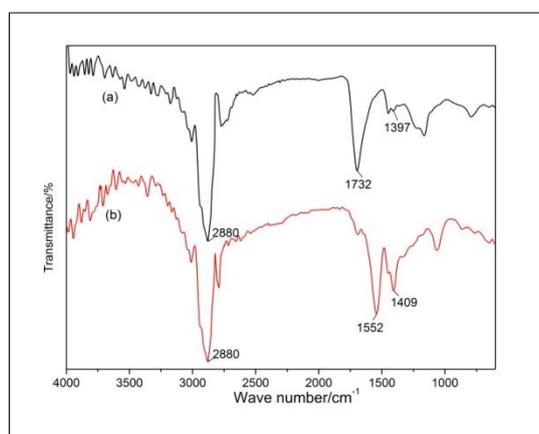


Figure 8. FT-IR spectra of D113 resin before and after Zn^{2+} adsorption (a) before Zn^{2+} adsorption, and (b) after Zn^{2+} adsorption

4. Conclusions

In this study, the traditional batch method was adopted to research the adsorption properties of D113 cation exchange resin for Zn^{2+} . To change initial Zn^{2+} concentration from 0.2 mol/L to 1.2 mol/L , reaction time from 20 min to 480 min, and reaction temperature from 293 K to 323 K , the curves of the adsorption efficiency and capacity vs reactive conditions were made respectively. The calculated results indicated that the adsorption efficiency of D113 resin on Zn^{2+} was not affected intensively by the initial Zn^{2+} concentration, and the adsorption capacity enhanced with the increasing of initial concentration

until equilibrium; the increase of temperature was energetically helpful to the adsorption of Zn^{2+} by D113 resin, and 120 min was the optimum adsorption time. In addition, the adsorption equilibrium of D113 resin for Zn^{2+} accorded with Langmuir adsorption isotherm model, the maximum adsorption capacity was $405 \text{ mg}\cdot\text{g}^{-1}$, which was a bigger adsorption capacity for Zn^{2+} compared with some consulting references; the adsorption process agreed with pseudo second-order kinetics model, and the linear correlation coefficients were 0.98949, and the adsorption was dominated by chemisorption; by calculating thermodynamics functions, $\Delta H > 0$, $\Delta S > 0$ and $\Delta G < 0$ at different temperatures indicated the adsorption process was spontaneous and endothermic. Moreover, SEM images and FT-IR spectra of resins further confirmed the reaction process was chemical adsorption.

Through the adsorption properties study of ion exchange resin for Zn^{2+} , the active data and the theory resources could support for the water treatment industry and the preparation of the inorganic material about the ion exchange resins as the precursor, which could broaden the preparation of inorganic materials, and expand its further application in the aspect of the preparation of the inorganic material by the structural characteristics of ion exchange resin.

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