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# Study on the adsorption of cadmium(II) from aqueous solution by D152 resin

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#### ABSTRACT

The feasibility of using D152 resin as an adsorbent for cadmium(II) was examined. The adsorption capacity of D152 resin for cadmium(II) ions was studied as a function of solution pH, temperature and contact time. The optimal pH for the adsorption of cadmium(II) ions was found to be 5.95 in the HAc–NaAc system. The maximum uptake capacity of cadmium(II) was estimated to 378 mg/g D152 resin at 298 K, at an initial pH value of 5.95. The results obtained from equilibrium adsorption studies were conformed to the Langmuir and Freundlich and the correlation coefficients have been evaluated. The apparent adsorption rate constant was  $k_{298K} = 4.01 \times 10^{-5} \, \text{s}^{-1}$ , and the apparent activation energy was 2.78 kJ/mol. Thermodynamic parameters,  $\Delta S$  was 108 J/(mol K), heat of adsorption ( $\Delta H$ ) value of 12.8 kJ/mol indicated the endothermic nature of the adsorption process, and a decrease of Gibbs free energy ( $\Delta G$ ) with increasing temperature also indicated the spontaneous nature of the process, respectively. The elution tests were carried out using various mixed reagents. The maximum elution percent of cadmium(II) ions were obtained when the reagent 0.5 mol/L HCl was used.

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#### 1. Introduction

As we all know, heavy metals are often detected in industrial wastewaters, which originate from metal plating, mining activities, smelting, battery manufacture, tanneries, petroleum refining, paint manufacture, pesticides, pigment manufacture, printing and photographic industries, etc. [1,2]. Presence of metal ions is of special concern as they can accumulate in different components of the environment, represent a serious threat to human populations, the fauna, etc. Cd(II) is one of the most toxic metals. The cadmium has been well recognized for its negative effect on the environment where it is non-degradable and accumulates readily in living systems. Adverse health effects due to cadmium are well documented and it has been reported to cause renal disturbances, lung insufficiency, bone lesions, cancer and hypertension in humans [3].

A number of technologies have been developed over the years to remove cadmium ions from water such as chemical precipitation, chemical reduction, ion exchange, membrane separation, adsorption and reverse osmosis [4–9]. Compared with the other method, ion exchange has increasingly received more attention in environmental treatment applications throughout the world in recent years because the method is simple, relatively low-cost and effective in removing heavy metal ions [10–12]. These adsorbents

were prepared from synthetic polymers. An organic ion exchange resin is composed of high-molecular-weight polyelectrolytes that can exchange their mobile ions for ions of similar charge from the surrounding medium.

D152 macroporous weak acid resin is a polymeric material containing a functional group (–COOH). It not only has proton that can exchange with cation, but also oxygen atom that can coordinate directly with metal ions. Its principal characteristics are great chemical and physical stability, high exchange capacity and good ability of regeneration so it can be very suitable to remove heavy metals from water and industrial wastewater. Moreover, it is cheaper comparing with imported resins. There has been reported that D152 resin along with its modified forms have been shown as effective adsorbents. Yang et al. [13] has reported the coupling oligo- $\beta$ -cyclodextrin on D152 beads media for separation of puerarin. Considering the resin performance and economic cost, D152 resin is feasible in industrial water treatment in China.

In this work, the removal of Cd(II) from aqueous solutions using batch and column adsorption methods was investigated by D152 resin. Some factors affecting adsorption, such as initial pH of solution, contact time and temperature were examined. Kinetics and isotherm adsorption experiments were carried out. Thermodynamic parameters of adsorption for Cd(II) were calculated. The experimental results may provide a path for the removal and recovery of Cd(II) from aqueous solutions in the environmental protection and hydrometallurgical systems.

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 Table 1

 General description and properties of resin.

Resins	D152
Functional group	-COOH
Structure	Macroporous
Capacity (mmol/g)	9.5
Average pore diameter (nm)	10.3
Surface area (m <sup>2</sup> /g)	4.8

#### 2. Materials and methods

#### 2.1. Apparatus

The cadmium was determined with Shimadzu UV-2550 ultraviolet–visible spectrophotometer. D152 resin dosage was measured by electronic balance of Sartorius BS 224S. Mettler toledo delta 320 pH meter was used for measuring pH. The sample was shaken in the DSHZ-300A and the THZ-C-1 temperature constant shaking machine. The water used in the present work was purified using Molresearch analysis-type ultra-pure water machine.

#### 2.2. Resin, reagents and solutions

D152 resin was supplied by Nankai University and the properties were shown in Table 1. Standard solutions of metal ions were prepared from cadmium nitrate (AR). HAc–NaAc with pH 3.27–6.50 and HCl–Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> with pH 9.00 buffer solutions were prepared from the NaAc, HAc and Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> solutions. The colour reagent of 0.1% PAR-ethanol solution was obtained by dissolving 0.1000 g PAR ( $C_{11}H_9N_3O_2$ ) powder into 100 mL 95% ethanol solution. All other chemicals were of analytical grade and purified water was used throughout.

#### 2.3. Adsorption experiments

Experiments were run in a wide range of pH, temperature, contact time as well as adsorption isotherms. The operation for the removal of heavy metals is usually carried out in batch vessels and glass columns.

Batch experiments were performed under kinetic and equilibrium conditions. A desired amount of treated D152 resin was weighed and added into a conical flask, in which a desired volume of buffer solution with pH 5.95 was added. After 24 h, a required amount of standard solution of cadmium(II) was put. The flask was shaken in a shaker at constant temperature. The upper layer of clear solution was taken for analysis until adsorption equilibrium came. The procedure of kinetic tests was identical to that of the equilibrium tests. The aqueous samples were taken at preset time intervals and the concentrations of Cd(II) were similarly measured.

The dynamic adsorption was taken in a glass column. Continuous flow adsorption studies were conducted in a vertical glass column of 0.45 cm inner diameter and 23.5 cm height filled with Cd(II) ion solution. At the bottom of the column, a stainless sieve was attached followed by a layer of cotton wool. The particles were dropped in from the top of the column. Time taken by the particles to travel a distance of 7.4 cm in vertical direction was noted. The Cd(II) solutions at the outlet of the column were collected at regular time intervals and the concentration was measured using a ultraviolet–visible spectrophotometer at 526 nm. All the experiments were carried out at room temperature.

#### 2.4. Analytical method

A solution containing lower than 75  $\mu$ g of Cd(II) was accurately added into a 25 mL colorimetric tube, and then 1 mL colour reagent of 0.1% PAR-ethanol solution and 10 mL pH 9.00 HCl–Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> buffer solution were added. After the addition of purified water to the mark of colorimetric tube, the absorbency was determined in a 1 cm colormetric vessel at wavelength of 526 nm and compared with the blank test.

The adsorption capacity (Q), distribution coefficient (D) were calculated with the following formulas [14–16]:

$$Q = \frac{C_0 - C_e}{W} V \tag{1}$$

$$D = \frac{C_0 - C_e}{WC_e} V \tag{2}$$

where  $C_0$  is initial concentration of metal ion in solution (mg/mL),  $C_e$  is equilibrium concentration of metal ion in solution (mg/mL), V is total volume of solution (mL), W is D152 resin weight (g).

#### 3. Result and discussion

#### 3.1. Effect of pH on the adsorption for Cd(II)

The pH in solution has been identified as the most important variable governing metal adsorption on sorbents. The effect of pH on the adsorption behavior of D152 resin for Cd(II) was shown in Fig. 1 that the uptake of Cd(II) ion as a function of hydrogen ion concentration was in the pH range 3.27–6.50 for an initial metal concentration of 10.0 mg/30.0 mL at 298 K, 100 rpm.

The initial pH of the solution was significantly affected the adsorption capacity of adsorbent, adsorption capacity was the highest when pH was 5.95 with HAc–NaAc and decreased by either raising or lowering pH under the experimental condition. At lower values, the metal ion uptake was inhibited in this acidic medium and this can be attributed to the presence of H<sup>+</sup> ions competing with the Cd(II) ions for the adsorption sites. Contrarily, the metal ion was prone to deposit at higher values [17]. Therefore, all the following experiments were performed at pH 5.95 in the HAc–NaAc system.

#### 3.2. Adsorption isotherms

Adsorption isotherms describe how adsorbates interact with adsorbents. Therefore, the correlation of equilibrium data by either theoretical or empirical equations is essential to the practical design and operation of adsorption systems [18–20].

#### 3.2.1. Langmuir isotherm

For Langmuir isotherm model, four parts of 16.0 mg D152 resin and the initial cadmium ion concentration of



Fig. 1. Effect of pH on the distribution coefficient.



Fig. 2. Langmuir isotherm curve.

 $[Cd^{2+}]_0 = 6.0 \text{ mg}/30.0 \text{ mL}, 8.0 \text{ mg}/30.0 \text{ mL}, 10.0 \text{ mg}/30.0 \text{ mL}, 12.0 \text{ mg}/30.0 \text{ mL}, 14.0 \text{ mg}/30.0 \text{ mL}$  were shaken until equilibrium at pH 5.95, 100 rpm.

Langmuir proposed a theory to describe the adsorption of gas molecules onto metal surfaces. The Langmuir adsorption isotherm has found successful applications in many other real adsorption processes of monolayer adsorption. Langmuir's model of adsorption depends on the assumption that intermolecular forces decrease rapidly with distance and consequently predicts the existence of monolayer coverage of the adsorbate at the outer surface of the adsorbent. The isotherm equation further assumes that adsorption takes place at specific homogeneous sites within the adsorbent. It is then assumed that once a Cd(II) molecule occupies a site, no further adsorption can take place at that site. Moreover, the Langmuire equation is based on the assumption of a structurally homogeneous adsorbent where all adsorption sites are identical and energetically equivalent. Theoretically, the sorbent has a finite capacity for the sorbate. Therefore, a saturation value is reached beyond which no further adsorption can take place.

The experimental data were fitted to the Langmuir equation:

$$\frac{C_e}{Q_e} = \frac{1}{Qb} + \frac{C_e}{Q} \tag{3}$$

where  $C_e$  (mg/mL) is equilibrium concentration of metal ion,  $Q_e$  (mg/g) is the adsorption capacity in equilibrium state, and *b* is the Langmuir constant which reflects quantitatively the affinity between the D152 resin and Cd(II) ions. It is equally used to analyse batch equilibrium data by plotting  $C_e/Q_e$  versus  $C_e$ , which yields a linear plot if the data conform to the Langmuir isotherm. According to the experiment data, plotting of  $C_e/Q_e$  versus  $C_e$  give a straight line and the correlation coefficient  $R^2$  are higher (Fig. 2 and Table 2). This is indicative of applicability of the proposed model for the process undertaken. In other words, the Langmuir-type adsorption isotherm was suitable for equilibrium studies.

#### 3.2.2. Freundlich isotherm

Freundlich isotherms were conducted as follows: 8.0, 12.0, 16.0, 20, 24.0 mg of D152 resin were weighed and the each initial cadmium ion concentration was 10.0 mg/30.0 mL at pH 5.95 and 288–308 K, 100 rpm.

Table 2
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Linearity relation of  $C_e/Q_e$  and  $C_e$ .

T (K)	Linearity relation of $C_e/Q_e$ and $C_e$	R <sup>2</sup>
288	y = 0.0013x + 0.0001	0.9983
298	y = 0.0022x + 9E - 05	0.9988
308	y = 0.0032x + 2E - 05	0.9992



Fig. 3. Freundlich isotherm curve.

The Freundlich adsorption equation has the general form:

$$Q_{\rm e} = aC_{\rm e}^{1/b}$$
 i.e.  $lg Q_{\rm e} = \frac{1}{b} lg C_{\rm e} + lg a$  (4)

where *b* and *a* are the isotherm parameters to be determined. The Freundlich adsorption isotherm represents the relationship between the corresponding adsorption capacity  $Q_e$  (mg/g) and the concentration of the metal in solution at equilibrium  $C_e$  (mg/mL). The plot of lg  $Q_e$  versus lg  $C_e$  for various initial concentrations was found to be linear in Fig. 3, indicating the applicability of the classical adsorption isotherm to this adsorbate–adsorbent system. The fit of the data to Freundlich isotherm (Table 3) indicates adsorption process is not restricted to one specific class of sites and assumes surface heterogeneity [21]. It has been stated by that magnitude of the exponent 1/*b* gives an indication of the favor-ability and capacity of the adsorption conditions. In most cases, the exponent between 1 < *b* < 10 shows beneficial adsorption [22].

#### 3.3. Determination of adsorption rate constant

The adsorption kinetics was determined according to the following procedure: 30 mL mixture solution of buffer solution and metal ion solution (10.0 mg/30.0 mL) was shaken with 16.0 mg of D152 resin at 288, 298 and 308 K. At predetermined intervals, aliquots of 0.2 mL solution were taken out for analysis and the concentration of metal ion was determined. After the remains kept constant and volume was corrected, a series of data were obtained (Fig. 4). The results obtained indicate that an increase of the temperature deals induce an increase in the capacity of cadmium adsorption. It means that the adsorption process is an endothermic process. Therefore, the adsorption reaction is a chemical adsorption. From the Fig. 4, the loading half time is  $t_{1/2} = 5$  h, the time of equilibrium t = 51 h.

According to Brykina method, the adsorption rate constant k can be calculated from:

$$-\ln(1-F) = kt \tag{5}$$

**Table 3** Linearity relation of  $\lg Q_e$  and  $\lg C_e$ .

T (K)	Linearity relation of $\lg Q_e$ and $\lg C_e$	$R^2$	b
288	y = 0.2779x + 2.6828	0.9983	3.60
298	y = 0.5467x + 2.9870	0.9919	1.83
308	y = 0.7165x + 3.1746	0.9923	1.40



Fig. 4. Adsorption curve of Cd(II).



Fig. 5. Determination of adsorption rate constant.

where  $F = Q_t/Q_\infty$ ,  $Q_t$  and  $Q_\infty$  are the adsorption amounts at certain time and at equilibrium time, respectively. The experimental results accord with the equation and a straight line was obtained by plotting  $-\ln(1-F)$  against t (Fig. 5). Therefore, the adsorption rate constant can be found from the slope of the straight line, which is  $k_{298K} = 4.01 \times 10^{-5} \text{ s}^{-1}$ . The correlation coefficient ( $R^2 = 0.9959$ ) was obtained via linear fitting. The other results were shown in Table 4. According to Boyd form the linear relationship of  $-\ln(1-F)\sim t$ , it can be deduced that the liquid film spreading was the predominating step of the adsorption process [23].

According to the Arrhenius equation

$$lg k = -\frac{E_a}{2.303RT} + lg A \tag{6}$$

where  $E_a$  is the Arrhenius activation energy for the adsorption process indicating the minimum energy that reactants must have for the reaction to proceed, A is the Arrhenius factor, R is the gas constant (8.314 J/(mol K)), k is the adsorption rate constant and T is the

# **Table 4** Adsorption rate constants (k) for the adsorption of Cd(II) with D152 resin and its correlation coefficient ( $R^2$ ).

Temperature (K)	288	298	308
$\frac{k(\times 10^{-5} \text{ s}^{-1})}{R^2}$	2.92	4.01	4.89
	0.9910	0.9959	0.9965

Thermodynamic parameters for Cd(II) on D152 resin.

$\Delta H(kJ/mol)$	$H(kJ/mol)$ $\Delta S(J/(Kmol))$		$\Delta G (\mathrm{kJ/mol})$	
		T=288 K	T=298 K	<i>T</i> =308 K
12.8	108	-18.3	-19.4	-20.5

solution temperature. The slope of straight line is made by plotting  $-\lg k$  versus 1/T, and calculated by linear fitting, yields the apparent activation energy of  $E_a = 2.78$  kJ/mol, which could be considered as a low energy barrier in this study. It can be deduced that the adsorption speed accelerated when the temperature rose within the scope of experimental temperature [24].

# 3.4. The influence of adsorption temperature and the thermodynamic parameters

Four parts of 16.0 mg resins were weighed. Under the experimental condition, distribution coefficient of the resin for Cd(II) over the range of temperature from 288 to 308 K was measured. There are three important thermodynamic parameters  $\Delta H$ ,  $\Delta S$  and  $\Delta G$ . They are the change in enthalpy, entropy and Gibb's free energy, respectively.

The slopes and intercept of lg*D* versus 1/T was used for the  $\Delta H$  and  $\Delta S$ . The linear equation is y = -0.6665x + 5.6233 and the correlation coefficient 0.9988.The slope of straight line is  $K_{\text{slope}} = -0.6665$ . According to

$$lg D = -\frac{\Delta H}{2.303RT} + \frac{\Delta S}{2.303R}$$
(7)

where  $\Delta H = -K_{slope} \times 2.303R = 0.6665 \times 2.303 \times 8.31 = 12.8 (kJ/mol)$ . The results are presented in Table 5 along with the Gibbs free energy calculated using equation given below:

$$\Delta G = \Delta H - T \Delta S \tag{8}$$

 $\Delta G$  values were negative, indicating that the adsorption process led to a decrease in Gibb's free energy and confirming the feasibility of the process and spontaneous nature of the adsorption under the experimental condition. Positive values of  $\Delta S$  refer to the increased randomness at the solid–solution interface. The positive values of  $\Delta H$  indicate that the adsorption process is an endothermic process. So the adsorption reaction is a chemical adsorption [25,26].

#### 3.5. Saturated capacity method

The maximum uptake capacity of Cd(II) was determined according to the following procedure: 30 mL mixture solution of buffer solution and metal ion solution (10.0 mg/30.0 mL) was shaken with 16.0 mg of D152 resin at 288–308 K, 100 rpm. The maximum adsorption capacity was calculated by using the above-mentioned method until adsorption equilibrium [14]. At 288, 298, 308 K, the maximum adsorption capacity of D152 resin for Cd(II) was 325, 378, 409 mg/g, respectively.

#### 3.6. Dynamic adsorption curve

The dynamic leakage curves on D152 resin were obtained based on the volume of effluent liquid and the Cd(II) concentration herein. The adsorption effect of macroporous resin was determined by surface adsorption and surface electrical property, hydrogen bonding interactions, etc.

The dynamic experiments were carried out in glass columns wet-packed. Sample solution flowed through the glass column at constant flow rate and the Cd(II) contents in the effluent liquid were monitored by ultraviolet–visible spectroscopy analysis. A solution having a known concentration of 0.200 mg/mL of Cd(II)

#### Table 6

The elution test of Cd(II).

Composition of HCl-NaCl (mol/L)	Adsorption capacity [mgCd(II)/15.0 mg]	Elution capacity (mg)	Elution percentage (%)
0.5 mol/L HCl–0.5 mol/L NaCl	0.375	0.367	97.8
0.5 mol/L HCl-1.0 mol/L NaCl	0.377	0.373	99.1
0.25 mol/L HCl-0.50 mol/L NaCl	0.375	0.362	95.7
0.75 mol/L HCl-0.25 mol/L NaCl	0.378	0.378	100
0.5 mol/L HCl	0.375	0.375	100



Fig. 6. Dynamic adsorption curve.

was continuously fed into the column. When the concentration of Cd(II) emerging from the bottom of the column was equal to that entering the top of the resin bed, the experiment was terminated, then a plot of  $C_e/C_0$  versus the volume of effluent gave a typical breakthrough curve in Fig. 6. The dynamic saturated adsorption capacity is 349 mg/g resin. It is less than the maximum uptake capacity (378 mg/g) because the contact time of the ion concentration with resins was transitory in the dynamic adsorption process.

#### 3.7. Elution

16.0 mg resin was added into a mixed solution composed of pH 5.95 buffer solution and desired amount of standard solution of Cd(II). After equilibrium reached, the concentration of Cd(II) in the aqueous phase was determined, and then the adsorption capacity of the resin for Cd(II) was obtained.

Then, the resin separated from aqueous phase was washed three times with pH 5.95 buffer solution. The resin adsorbed Cd(II) was shaken with 30.0 mL eluant. After equilibrium arrived, the concentration of Cd(II) in aqueous phase was determined and then the percentage of elution was obtained. The results listed in Table 6 show that the percentage of elution is different when the composition of HCl–NaCl is changed. It shows that the percentages of elution can reach 100%. So Cd(II) adsorbed on D152 macroporous weak acid resin can be recovered.

Considering the environmental pollution and economic cost, the experimental results show that the 0.5 mol/L HCl is the best.

#### 4. Conclusions

In this study, we can conclude that D152 resin can be used for removal of Cd(II) very effectively. It was observed that Cd(II) adsorption is highly dependent on pH. In addition, D152 resin dosage and initial Cd(II) concentration are effective on Cd(II) adsorption. The adsorption behavior could be modeled using the Langmuir isotherm and Freundlich isotherm. Maximum uptake capacity of cadmium(II) was estimated to 378 mg/g D152 resin by batch method at 298 K. The apparent adsorption rate constant is  $k_{298K} = 4.01 \times 10^{-5} \text{ s}^{-1}$ . The apparent activation energy is 2.78 kJ/mol. The adsorption parameters of thermodynamic are  $\Delta H = 12.8 \text{ kJ/mol}, \Delta G_{298K} = -19.4 \text{ kJ/mol}, \Delta S = 108 \text{ J/(mol K)}.$  In summary, D152 resin has many advantages to remove Cd(II) in the solution.

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