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Studies on the Adsorption of 2-Naphthalenesulfonic Acid on Basic Resin from Effluents

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The adsorption reaction of 2-naphthalenesulfonic acid with the strong basic resin N201 was investigated experimentally with varying parameters (viz., the effects of pH, temperature, contact time, sodium sulfate, and fixed-bed column runs) and compared with the weakly basic resin D301. Results showed that the optimum initial pH for 2-naphthalenesulfonic acid adsorption by N201 was 2.4, and the equilibration time required was 2.5 h. The three single-component isotherms were analyzed with the Langmuir, Freundlich, and Redlich−Peterson equations. The Redlich−Peterson equation gave the lowest errors by use of the sum of the squares of the errors. The thermodynamic parameters (ΔG, ΔH, and ΔS) were also determined, and the adsorption process is more favored at low temperatures. The kinetics followed a pseudo-second-order rate equation very well. However, a dramatic decrease in adsorption capacity was observed for both resins upon addition of Na_2SO_4 into the 2-naphthalenesulfonic acid solution. The adsorption and stripping curves of 2-naphthalenesulfonic acid onto two resins was explored in fixed-bed column experiments. It was found that the dynamic adsorption capacity of N201 was higher than that of D301.

1. Introduction

2-Naphthalenesulfonic acid (2-NSA, CAS registry no. 120-18-3) is an aromatic sulfonate and an important structural unit for a variety of synthetic dyes. Wastewater originating from 2-NSA manufacture contains a large amount of 2-NSA, sulfuric acid, and so on.[1] In view of the toxic influence of these chemicals on humans and the environment, it is necessary to remove them before wastewater is discharged into water bodies. There are many techniques, like reverse osmosis, chemical oxidation, adsorption by solvent extraction, biochemical treatment, membrane filtration, ion-exchange adsorption, etc.,[2−5] for removal of 2-NSA from wastewater. Numerous sorbent materials like activated carbon and traditional polymeric adsorbents have been exploited for the treatment of aromatic sulfonates contaminated wastewater and industrial effluents. The removal of aromatic sulfonates by use of ion-exchange resins by adsorption and exchange was found to be an efficient and economic process, as it can generally remove all types of pollutants from wastewater.[6−11] So, a series of base anionic exchangers has been tested for removing aromatic anions from aqueous solution when inorganic anions coexist in industrial wastewater.[12−18] Some commercial base exchangers exhibit satisfactory performance in adsorption kinetics, selectivity, and feasible regeneration, which would facilitate them in field application. In the present study, the strong base resin N201 was proposed to remove aromatic sulfonates with good selectivity, large capacity, and low operational cost.

In extension of our previous work, weakly basic resins have been used systematically for 2-NSA adsorption from aqueous solution. Thus the adsorption of 2-NSA on N201 and D301 resins with varying solution pH, temperature, and contact time and the effect of sodium sulfate on 2-NSA uptake and fixed-bed column runs were explored in the present work. The most appropriate isotherm and the thermodynamics of the adsorption reaction were also studied.

Table 1. Physical Properties of Resins

	N201	D301
polymer matrix	styrene−DVB	styrene−DVB
styrene−divinylbenzene (DVB)	gel	macroporous
form as supplied	free base	free base
bead size, mm	0.35−0.4	0.35−0.55
moisture content, %	56	45
capacity, mmol·(cm^3 of dry resin)$^{-1}$	≥4.0	6.8

2. Materials and Methods

2.1. Materials. The basic anion exchanger N201 and weakly basic anion exchanger D301 were provided by Nankai Resin Co. Ltd. (Tianjin, China). The properties of the two resins are shown in Table 1. 2-NSA was purchased from Sigma−Aldrich (98.0 %). The other chemicals used in the study are of analytical grade and were provided by Shanghai Reagent Station (Shanghai, China).

2.2. Analysis. 2-NSA in solution was analyzed on a high-performance liquid chromatograph (LC-10AT, Shimadzu Corp., Japan) and a UV−vis spectrophotometer (10A VP, Shimadzu Corp., Japan). Separation was performed on a C18 analytical column (150 mm × 4.6 mm i.d., particle size 5 μm). The mobile phase consisted of a solution of methanol−water (50/50) containing 0.5 % KH_2PO_4 as pH regulator at a flow of 0.8 cm^3·min^{-1}, and the detector wavelength was 275 nm. Under these conditions, the retention time for 2-NSA was 6.0 min. The sample solutions were filtered through 0.45 μm membrane filters, and the filtrates were analyzed for residual 2-NSA.[9]

2.3. Batch Adsorption Experiments. 2.3.1. pH Effect. The experiment for estimating the influence of initial solution pH on solute adsorption by the sorbent was carried out by mixing 0.100 g of resin particles with 0.1 dm^3 of 2-NSA solution (concentration 500 mg·dm^{-3}), adjusted at pH values ranging from 1.0 to 12.0, into separate glass flasks (0.25 dm^3). Sulfuric

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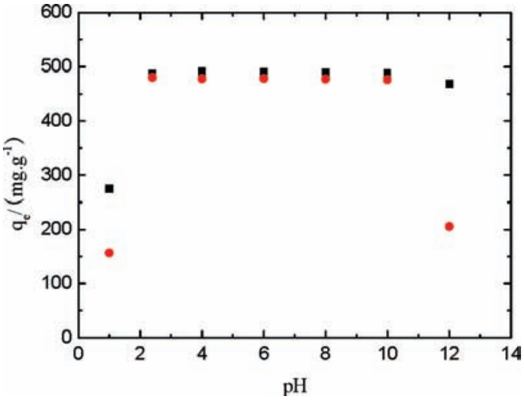

Figure 1. Effect of initial solution pH on 2-NSA adsorption by two resins (298 K): black squares, N201; red circles, D301.

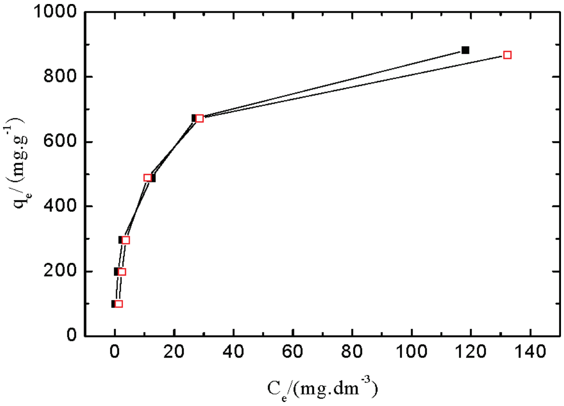

Figure 2. Adsorption isotherms of 2-NSA by N201 resin at different temperatures: black squares, 298 K; red squares, 323 K.

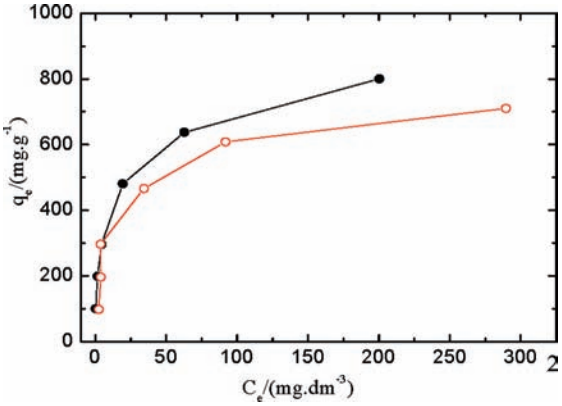

Figure 3. Adsorption isotherms of 2-NSA by D301 resin at different temperatures: black circles, 298 K; red circles, 323 K.

acid and sodium hydroxide were used to adjust the solution pH throughout the experiment when necessary.

2.3.2. Adsorption Isotherm. Batch adsorption runs were carried out in 0.25 dm^3 glass flasks. To start the experiment, 0.100 g of resin particles was introduced into a 0.1 dm^3 solution. The initial concentration of the solution was (100, 200, 300, 500, 700, and 900) mg·dm^{-3}. The flasks were then transferred to a shaker with a thermostat (SHA-1, Jintan Kexi Instrument Co. Ltd., China) and shaken at 180 rpm for 24 h at different temperatures to ensure the adsorption process reached equilibrium.

2-NSA uptake capacity at equilibrium, q_e (or at time t, q_t), of sorbent was calculated from the following mass balance:

$$q_e \text{ (or } q_t) = (C_0 - C_e)(V/W) \tag{1}$$

where C_0 and C_e are, respectively, the initial and residual concentrations (mg·dm^{-3}) of 2-NSA in solution and V and W are the test solution volume (dm^3) and mass (g) of sorbent, respectively, used for the test.

2.3.3. Adsorption Kinetics. A batch method was used to study the kinetics of 2-NSA adsorption by N201 and D301 at pH 2.4 and temperature 298 K. Here, 0.1 dm^3 of 2-NSA solution (concentration 500 mg·dm^{-3}) was taken with 0.100 g of resin into 20 0.25 dm^3 glass flasks, which were placed in a thermostated bath to attain the desired temperature. A measured volume of the reaction mixture, 0.005 dm^3, was sampled at definite time intervals until equilibrium was reached. The sample solutions were filtered through 0.45 μm membrane filters, and the filtrates were analyzed for residual 2-NSA.

2.4. Column Adsorption Experiment. Column experiments were carried out with a glass column (12 mm diameter and 230 mm length) equipped with a water bath to maintain a constant temperature. A BT-200F pump was used to ensure a constant flow rate. All column runs were performed under the hydrodynamic conditions: temperature was 298 K, 2-NSA concentration was 1.00 g·dm^{-3}, bed volume (BV) of dry resin was 0.01 dm^3, and liquid velocity was 3 BV·h^{-1}.

3. Results and Discussion

3.1. Effect of pH on Adsorption. The effect of solution pH on 2-NSA adsorption by N201 and D301 resins at 298 K is illustrated in Figure 1. A very high adsorption capacity was attained at a solution initial pH ranging from 2.4 to 10.0, and 2-NSA uptake even approaches 98.1 % (pH 2.4). But if pH < 2.4 or pH > 12.0, the 2-NSA adsorption capacity decreased dramatically. The maximum adsorption capacities of the solute onto both resins were observed over a large pH range, which resulted from the ion-exchange mechanism of the adsorption, and only the protonated amine groups took an effective role in adsorption of negatively charged aromatic sulfonate anions. A higher solution pH is unfavorable for protonation of the amine group and therefore resulted in a little lower adsorption capacity. As the solution pH values are less than the optimal one (such as pH 2.4 for 2-NSA adsorption), adsorption capacities decrease due to the added sulfate anions for pH adjustment. In fact, there may be electrostatic or Columbic interactions with the protonated amine groups and sulfate anions when loaded on both sorbents. Sulfate anions also result in competitive adsorption with aromatic sulfonates.[19]

3.2. Adsorption Isotherm. To assess the efficiency of N201 and D301 resins for the removal of 2-NSA, the equilibrium adsorption of the two resins was studied as a function of concentration on N201 and D301 resins at different temperatures, and the adsorption isotherms obtained are shown in Figures 2 and 3, respectively. A comparison of adsorption capacity at 298 and 323 K showed that the adsorption capacity increased with decreasing temperature, indicating that the process was apparently exothermic. Similar results were obtained by other workers.[7,20] The effect of temperature can be explained on the basis of changing 2-NSA solubility. Because the solubility of 2-NSA decreased with decreasing temperature, the lower solubility of adsorbate enhanced the adsorption reaction and the adsorption capacity increased with decreasing temperature.

To evaluate the nature of the adsorption reaction, the following most commonly used isotherm model equations were used for the data analysis: the Langmuir isotherm[21]

$$q_e = \frac{q_{max} b C_e}{1 + b C_e} \tag{2}$$

the Freundlich isotherm[22]

$$q_e = k C_e^{1/n} \tag{3}$$

and the Redlich−Peterson isotherm[23,24]

$$q_e = \frac{K_R C_e}{1 + b_R C_e^{m}} \tag{4}$$

where C_e ($mg \cdot dm^{-3}$) and q_e ($mg \cdot g^{-1}$) are, respectively, the equilibrium concentration and capacity. q_{max} ($mg \cdot g^{-1}$) and b ($dm^3 \cdot mg^{-1}$) are the Langmuir constants related to monolayer adsorption capacity and adsorption equilibrium constant, respectively. k ($mg \cdot g^{-1}$) and n $(dm^3 \cdot mg^{-1})^{-1/n}$ are the Freundlich constants related to adsorption capacity and intensity, respectively. K_R, b_R, and m are the Redlich−Peterson constants.

The isotherm data shown as symbols (Figures 4 and 5) have been analyzed with the aforesaid equations (eqs 2, 3, and 4) by nonlinear methods by use of the Origin software package, and the fits of curves are also shown in Figures 4 and 5. The related isotherm parameters evaluated from the nonlinear analyses of the data are given in Table 2. On the basis of the linear regression coefficient (R^2) values (Table 2), it could be said that the fits of the present equilibrium data were better with the Redlich−Peterson isotherm (eq 4) than the Langmuir (eq 2) and Freundlich (eq 3) isotherms. The Redlich−Peterson linear regression coefficient (R^2) obtained from the nonlinear method of analysis was more than 0.95. Meanwhile, from Table 2, it can be seen that N201 had a larger capacity than D301 and was a fairly good scavenging agent for 2-NSA at normal conditions compared to many other sorbents.

If liquid-phase adsorption follows the Redlich−Peterson model, the thermodynamic parameters of the adsorption process can be calculated as[9,25,26]

$$\Delta G = -RT \int_0^{Ce} q \frac{dc}{c} \tag{5}$$

$$\ln (1/C_e) = -\frac{\Delta H}{R}\frac{1}{T} + K_0 \tag{6}$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \tag{7}$$

where R is the universal gas constant, 8.314 $J \cdot (mol \cdot K)^{-1}$, T is the absolute temperature, and q is the calculated adsorption capacity calculated by the Redlich−Peterson equation. The enthalpy change is determined by plotting ln $(1/C_e)$ versus $1/T$. As presented in Table 3, ΔH for the present reaction onto the two resins is found to be negative, which proves the exothermic nature of the adsorption reaction. For N201, ΔS of the process is found to be positive, which indicates increasing randomness at the solid−liquid interface when the solute adsorption takes place on the solid. This was presumably due to the increase of released solvent molecules when solvated solute distributes on the solid phase from the aqueous solution and the number of molecules increasing at the solid−liquid interface. The negative ΔH and positive ΔS values implied that 2-NSA adsorption onto N201 was an enthalpy- and entropy-driven process simultaneously. But for adsorption of D301, ΔS of the process was negative, and upon reaction there is a decrease of randomness at the solid−liquid interface. The Gibbs energy change (ΔG) is found to be negative for both resins, and the value decreases with increasing temperature. This suggested the spontaneous nature of the adsorption reaction at the studied temperatures, and the reaction spontaneity decreased with increasing temperature, which was characteristic of an exothermic surface reaction.

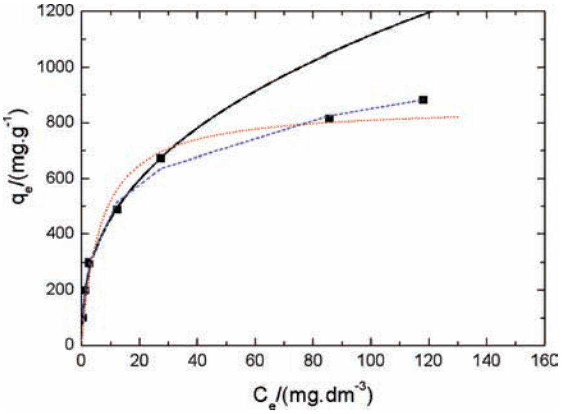

Figure 4. Equilibrium data for 2-NSA adsorption by N201 resin at 298 K and pH = 2.4. Symbols represent the experimental data, and lines show the model fit: solid black line, Freundlich isotherm; dashed red line, Langmuir isotherm; dotted blue line, Redlich−Peterson isotherm.

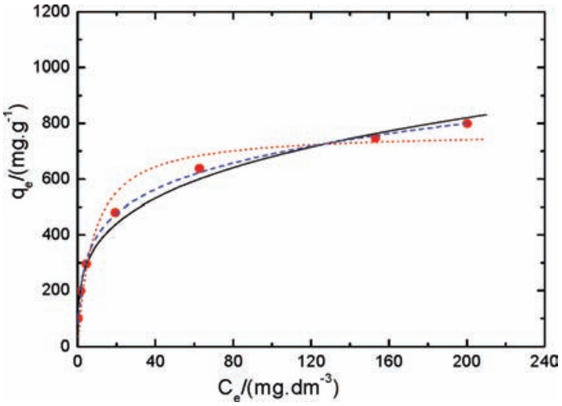

Figure 5. Equilibrium data for 2-NSA adsorption by D301 resin at 298 K and pH = 2.4. Symbols represent the experimental data, and lines show the model fit: solid black line, Freundlich isotherm; dashed red line, Langmuir isotherm; dotted blue line, Redlich−Peterson isotherm.

3.3. Kinetic Analysis. Figure 6 shows the kinetic data of 2-NSA adsorption by N201 and D301 obtained at pH 2.4 and temperature 298 K. It is found that 80 % of total 2-NSA uptake takes place in 1 h, and the time taken to reach equilibrium was 2.5 h for the N201 resin. It also can be seen from Figure 6 that the adsorption rate was low in the late adsorption stage, and consequently, the adsorption process spent the most time in the late stage. The data (Figure 6) have been analyzed by the nonlinear least-squares fit method using the appropriate coordinate of each equation by pseudo-first-order[27] (eq 8) and pseudo-second-order[28] (eq 9) kinetic equations, and the fits are shown with the experimental data in Figures 7 and 8.

$$\log (q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \tag{8}$$

Table 2. Values of Isotherm Equation Parameters Estimated by the Nonlinear Analysis of Equilibrium 2-NSA Adsorption Data on Resins

		Langmuir			Freundlich			Redlich−Peterson			
	T	$q_{\max}$	b		k			k_R	b_R		
resin type	K	$mg \cdot g^{-1}$	$dm^3 \cdot mg^{-1}$	R^2	$mg \cdot g^{-1}$	n	R^2	$dm^3 \cdot g^{-1}$	$(dm^3 \cdot mg^{-1})^m$	m	R^2
N201	298	873.211	151.590	0.9550	219.508	3.320	0.9709	363.965	1.020	0.804	0.9953
N201	323	969.492	95.400	0.9898	203.366	3.354	0.9404	113.612	0.158	0.949	0.9973
D301	298	759.558	132.510	0.9454	195.342	3.659	0.9829	406.262	1.486	0.794	0.9978
D301	323	681.130	108.210	0.9301	159.992	3.650	0.9143	110.939	0.311	0.874	0.9546

Table 3. Values of Thermodynamic Parameters Evaluated for 2-NSA Sorption

	T	ΔG	ΔS	ΔH
resin type	K	$kJ \cdot mol^{-1}$	$J \cdot (mol \cdot K)^{-1}$	$kJ \cdot mol^{-1}$
N201	298	−3.014	5.684	−1.321
N201	323	−2.791	4.551	
D301	298	−2.932	−8.221	−5.382
D301	323	−2.624	−8.541	

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^{\ 2}} + \frac{t}{q_e} \tag{9}$$

where q_e and q_t are the adsorption capacities ($mg \cdot g^{-1}$) at equilibrium and at time t (h), respectively; and k_1 (h^{-1}) and k_2 ($g \cdot mg^{-1} \cdot h^{-1}$) are the rate constants, respectively, related to eqs 8 and 9. The kinetic parameters estimated from the nonlinear plots for the kinetic equations used are shown in Table 4. The R^2 (regression coefficient) values estimated for the aforesaid solute concentrations from the pseudo-second-order equation were higher than those from the pseudo-first-order equation. Thus, on the basis of coefficient of determination (R^2) values, it could be generalized that the pseudo-second-order equation was more suitable than the pseudo-first-order equation to describe the adsorption of 2-NSA on both resins.

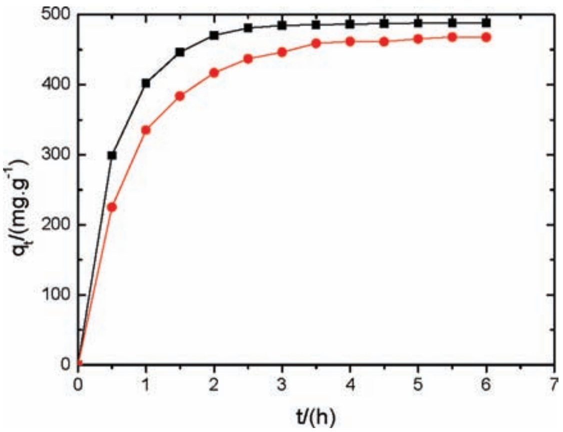

Figure 6. Adsorption kinetics of 2-NSA on two resins at 298 K and pH = 2.4: black squares, N201 resin; red circles, D301 resin.

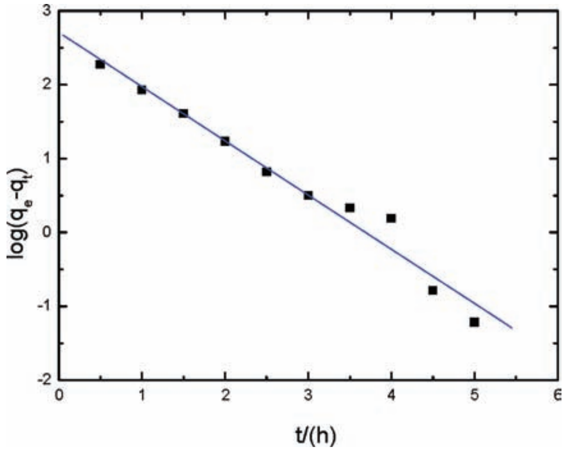

Figure 7. Experimental data for pseudo-first-order equation fit on N201 resin at 298 K and pH = 2.4: symbols, experimental data; line, fit to equation.

3.4. Effect of Na_2SO_4 on 2-NSA Uptake. The concentration of Na_2SO_4 in industrial 2-NSA wastewaters can be at a relatively high level. The effect of Na_2SO_4 on 2-NSA uptake by N201 and D301 resins was determined, and the results are depicted in Figure 9. 2-NSA adsorption decreased by the addition of 1 % Na_2SO_4, and a further increase of Na_2SO_4 in solution resulted in a gradual increase of 2-NSA uptake. Similar results are also reported elsewhere.[1,7,19] Adsorption capacities of 2-NSA decrease when sulfate anions are present due to the competition for available sites between adsorbable components, and electrostatic interaction plays a positive role in 2-NSA adsorption. On the basis of the "salting-out" effect,[29,30] the adsorption capacity of 2-NSA may be increased by further removal of Na_2SO_4.

3.5. Column Adsorption Experiments. The fixed-bed column runs of N201 and D301 resins to test their reliability for 2-NSA removal from wastewater were studied. From Figure 10, we can see that N201 exhibited more satisfactory column performance than D301 for 2-NSA removal. 2-NSA can be efficiently removed by N201 within about 308 BV per run before a significant breakthrough occurred, and the 2-NSA concentration in the effluent solution within 308 BV was about 0.35 $mg \cdot L^{-1}$,

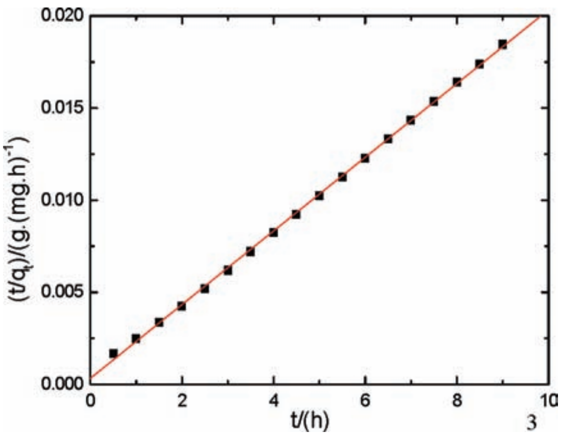

Figure 8. Experimental data for pseudo-second-order equation fit on N201 resin at 298 K and pH = 2.4: symbols, experimental data; line, fit to equation.

Table 4. Values of Kinetic Equation Parameters Estimated for Adsorption of 2-NSA (pH = 2.4, T = 298 K)

	pseudo-first-order			pseudo-second-order		
	k^{-1}	q_e		k_2	q_e	
resin type	h^{-1}	$mg \cdot g^{-1}$	R^2	$g \cdot (mg \cdot h)^{-1}$	$mg \cdot g^{-1}$	R^2
N201	1.690	511.754	0.9718	0.0120	500.000	0.9997
D301	0.540	212.761	0.9309	0.00380	515.462	0.9993

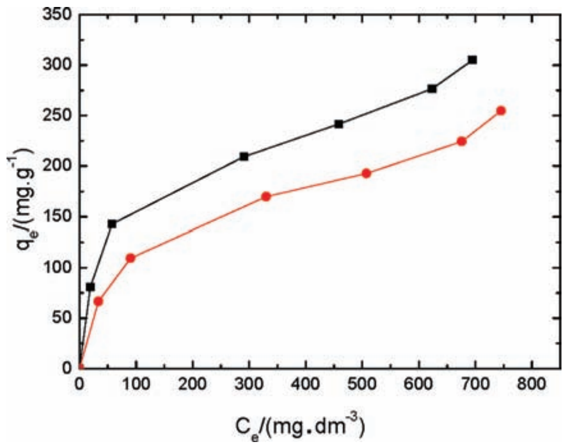

Figure 9. Effect of added Na_2SO_4 on 2-NSA uptake onto N201 and D301 resins at 298 K (0.100 g of resin was introduced into 0.1 dm^3 of solution containing an initial 2-NSA amount of 1000 $mg\cdot dm^{-3}$ and 1 % Na_2SO_4). Black squares, N201; red circles, D301.

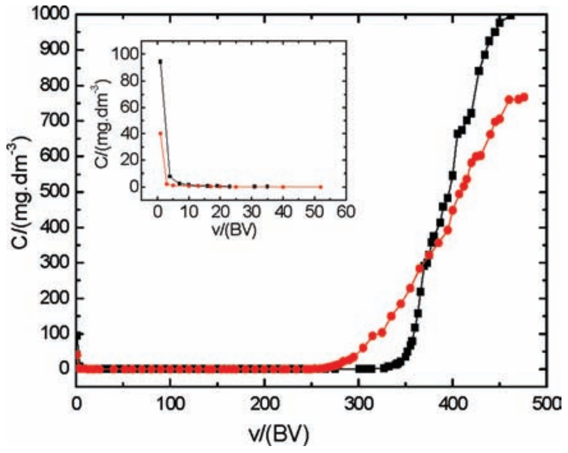

Figure 10. Column adsorption of 2-NSA onto two resins at 298 K: black squares, N201; red circles, D301.

on average. In other words, the removal efficiency of 2-NSA by N201 is as high as 99.96 %. Furthermore, the entire regeneration of both exhausted resins could be readily achieved by using (1 to 2) M NaOH solution, as has been reported previously.[6]

4. Conclusions

- N201 showed better adsorption capacity than D301 over a wide range of initial solution pH.

- The adsorption pattern of 2-NSA by both sorbents fit well to the Redlich−Peterson model. For N201 at 298 K, the reaction was exothermic ($\Delta H = -1.321\ kJ\cdot mol^{-1}$), which took place with increasing entropy ($\Delta S = +5.684\ J\cdot mol^{-1}\cdot K^{-1}$) and a negative Gibbs energy change ($\Delta G = -3.014\ kJ\cdot mol^{-1}$) indicating the spontaneous nature of the adsorption reaction and the reaction spontaneity decreased with increasing temperature.

- The adsorption reaction took 2.5 h to attain equilibrium for N201. The pseudo-second-order equation was more suitable than the pseudo-first-order equation to describe the kinetic data of 2-NSA adsorption onto both resins.

- A dramatic decrease in the adsorption capacity was observed for both resins upon addition of Na_2SO_4 into the 2-NSA solution.

- The adsorption and stripping curves of 2-NSA onto N201 and D301 in the fixed-bed column experiment indicated the dynamic adsorption capacity of N201 was higher than that of D301.

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