Adsorption of 5-sodiosulfoisophthalic acids from aqueous solution onto poly(2-vinylpyridine) resin

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ABSTRACT

In the present study, the performance, behavior and mechanism of synthetic poly(2-vinylpyridine) resin (WH-225) adsorbing 5-sodiosulfoisophthalic acids (SIPA) from the aqueous solution were investigated, and two commercial adsorbents, namely, hypercrosslinked adsorbent NDA-100 and macroporous adsorbent XAD-4 were employed as reference. Compared to NDA-100 and XAD-4, WH-225 has the highest capacity for adsorbing SIPA from the aqueous solution, which is verified by the related adsorption experiments. The investigation indicated that electrostatic interaction is an important factor in affecting the adsorption behavior of WH-225. The Freundlich isotherm equation was successfully applied to describe the adsorption isotherms. The negative values of the adsorptive enthalpy changes indicate an exothermic process for WH-225, and the absolute values ($<43 \, \text{kJ mol}^{-1}$) further manifest a physical adsorption process. The column adsorption and desorption tests further proved WH-225 is a promising adsorbent for field applications to remove and recover aromatic acids (e.g. SIPA) from aqueous solution.

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1. Introduction

5-Sodiosulfoisophthalic acid dimethylester (SIPM) is an important chemical intermediate of aromatic sulfonic acids, which is increasingly used in the dye industry and as optical brighteners [1,2]. The effluent from manufacturing SIPM often has the characteristics of high concentration of 5-sodiosulfoisophthalic acids (SIPA, $15,000$–$25,000 \, \text{mg L}^{-1}$) and strong acidity ([H+], 1.00–$1.30 \, \text{mol L}^{-1}$). Currently, it is not efficient to treat such wastewater by biological, chemical and physical methods owing to the high acidity of the wastewater, the strong chemical stability and hydrophilicity of SIPA [3–5].

In the past decades, the use of polymeric adsorbents (namely, synthetic resins) for removal of organic pollutants is very important not only in the environmental protection but also in the resource reuse, because the adsorbents can be regenerated and the adsorbates can be recovered without chemical change [6,7]. In comparison to the traditional adsorbents such as activated carbon, cellulose, clay, and silica gel, polymeric adsorbents have the advantages of their feasible adsorption-regeneration properties and perfect mechanical stability. Normally, the hydrophobic interaction is believed to be the dominant force for the separation of compounds from the aqueous solution on the prevalent

hypercrosslinked or macroporous resins. However, when the compound is highly water-soluble, the adsorption capacity of hypercrosslinked or macroporous polymers would conspicuously decrease [8–12].

To overcome the drawback of the traditional polymeric adsorbents (hypercrosslinked and macroporous polymers), a kind of hydrophilic adsorbent, poly(2-vinylpyridine) resin (WH-225), was synthesized by suspension polymerization in our laboratory [13], and two commercial hypercrosslinked resin NDA-100 and macroporous resin Amberlite XAD-4 were used as reference to study the performance, behavior and mechanism of WH-225 adsorbing SIPA by focusing on the following work: adsorption equilibrium of SIPA from the aqueous solution onto the three adsorbents, the influence of acid concentration and temperature on the adsorption, and column adsorption and desorption characteristics of SIPA on WH-225.

2. Experimental

2.1. Materials

Commercial divinylbenzene (grade 63% of DVB, containing a mixture of DVB and ethylvinylbenzene, obtained from Dongda Chemical Co., Shandong Province, China) was dried after washing with a 5% NaOH aqueous solution. 2-Vinylpyridine (2-VP, supplied by Dongfang Chemical Co., Shandong Province, China) was dried over CaH$_2$ and distilled under reduced pressure prior to use. 2, 2′-Azobisisobutyronitrile (AIBN, donated by Shanghai First

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Table 1
Physicochemical properties of the three resins[a].

Resin	WH-225	NDA-100	XAD-4
Structure			
Matrix	Polyvinylpyridine macroporous	Polystyrene hypercrosslinked	Polystyrene macroporous
BET surface area ($m^2\,g^{-1}$)	69.6	954.3	922.3[b]
Micropore area ($m^2\,g^{-1}$)	6.5	614.1	3.2
Average pore diameter (nm)	9.4	2.4	5.6
Nitrogen content (%)	7.61	–	–

[a] The data are cited from Ref. [13].
[b] Value measured in our study.

Chemical Reagent Plant, Shanghai, China) was purified by recrystallization from methanol. NDA-100 was obtained from Jiangsu N&G Environmental Technology Co., Ltd. (Jiangsu Province, China). The commercial spherical resin Amberlite XAD-4 was purchased from Rohm and Haas Co. (Philadelphia, USA). Prior to use, these two commercial resins were rinsed with methanol in a glass column at 323 K and dried under vacuum at 333 K for 8 h. SIPA (purity: 98%) was kindly offered by Shandong Haihua Co., Ltd. (Shandong Province, China). The other chemicals, methanol, sulfuric acid, gelatin and n-heptane were purchased from Shanghai Chemical Reagent Plant (Shanghai, China) and used in the study without further purification. Aqueous solutions were prepared using distilled deionized water.

2.2. Adsorbent preparation

The poly(2-vinylpyridine) resin, WH-225, was prepared by suspension polymerization, in which 2-vinylpyridine was used as the monomer, divinylbenzene as the cross-linked reagent, n-heptane as the porogen, and 2, 2′-azobisisobutyronitrile (AIBN) as an initiator. The synthetic process had been described in detail in our previous paper [13]. The resulting WH-225 was obtained in spherical bead form. The polymer was thoroughly washed several times with hot water, extracted with methanol in a Soxhlet apparatus for 8 h, and then dried in vacuum at 333 K for 8 h. The beads were then sieved to get 30–60 mesh for the experiments.

N_2 adsorption isotherm of WH-225 at 77 K was measured using an ASAP 2010 (Micromeritics Instrument Co., USA). The BET surface area was determined from the N_2 adsorption isotherms by means of the BET equation. The elemental analysis of the resins was performed using a CHN-O-Rapid Elemental Analytical Instrument (Heraeus, Germany).

2.3. Adsorption assay

The equilibrium sorption experiments of SIPA with different concentrations of sulfuric acid, i.e., 0.25, 0.5, 1.0, and 1.5 mol L^{-1}, were carried out at 288 K. Firstly, 0.1000 g resin was introduced into a series of 150 mL conical flasks, respectively, and 100 mL aqueous solution of SIPA with known concentration was added into each flask. The initial concentrations (C_0) of the solutions were 200, 400, 600, 800, and 1000 mg L^{-1}. The flasks were then completely sealed and placed in a model G25 incubator shaker (New Brunswick Scientific) at a pre-set temperature with shaking speed of 120 rpm for 24 h to ensure the equilibrium (preliminary tests indicated that adsorption equilibrium was achieved within about 12 h).

Finally, the residual concentration of SIPA in the solutions (C_e) was analyzed by high performance liquid chromatography (HPLC) equipped with a Waters 600 controller, a Waters 600 pump (Waters Assoc., USA) and a Waters 2487 Dual Absorbance UV detector at 240 nm. The mobile phase consisted of 30% methanol and 70% water with flow rate at 1 mL min^{-1}. The amount of SIPA in the supernatant subtracted from the amount initially added provided the amount of SIPA adsorbed on the resins. The adsorption capacity q_e (mmol g^{-1}) was calculated using the following equation:

$$q_e = \frac{V(C_0 - C_e)}{MW}$$

(1)

where V is the volume of solution (L), W is the mass of resins (g) and M is the molecular weight of SIPA.

3. Results and discussion

3.1. Properties of resins

The copolymer WH-225 in the current study is a macroporous vinylpyridine-divinylbenzene matrix, of which the BET surface area is only 69.6 m^2 g^{-1}. Amberlite XAD-4 is a macroporous styrene-divinylbenzene matrix without any functional groups [14]. NDA-100 is consisted of hypercrosslinked macroporous polystyrene-divinylbenzene [15], the BET surface area of which is the biggest among these resins. The content of nitrogen is not existed in Amberlite XAD-4 and NDA-100. Table 1 lists the properties of the three resins.

3.2. Adsorption isotherms

The adsorption isotherms can supply information on the adsorption strength and capacity. Fig. 1 compares the equilibrium adsorption isotherms of SIPA adsorbed onto WH-225, NDA-100 and XAD-4 from aqueous solutions with different acid concentrations at 288 K.

It is well known that the surface area of adsorbent plays an important role in adsorption process. However, the order of adsorption capacities for SIPA is not in agreement with their specific surface areas. The specific surface area of WH-225 is only about one-thirteenth of that of NDA-100 or XAD-4 (Table 1), but the adsorption capacity on WH-225 is much higher than that on NDA-100 or XAD-4. For example, in the case of acid concentration 0.25 mol L^{-1} and the same residual concentration (C_e) of 1.0 mmol L^{-1}, adsorption capacity of WH-225 for SIPA is 291% more than that on NDA-100 and about 21 times higher than that on XAD-4. When C_e is 3.0 mmol L^{-1}, the adsorption capacity of WH-225 is 440% higher than that on NDA-100 and about 11 times higher than that on XAD-
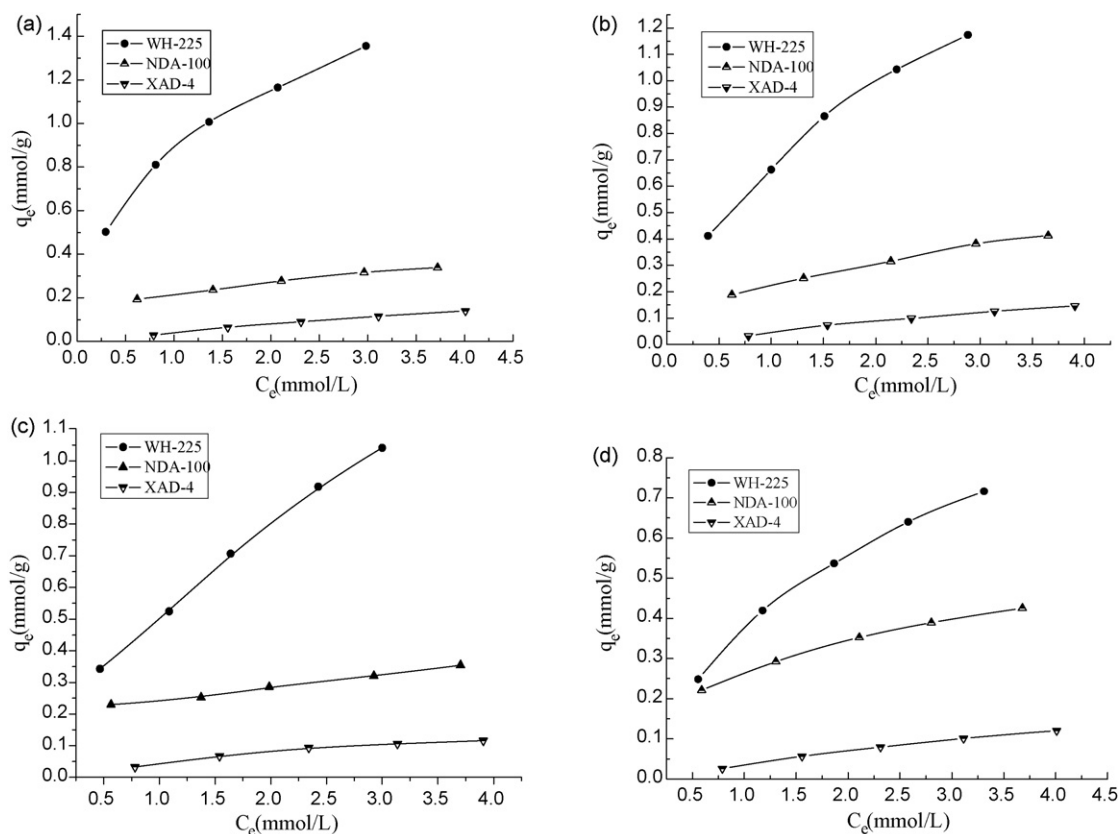
Fig. 1. Equilibrium adsorption isotherms at 288 K for SIPA on three resins for different acid concentrations: (a) 0.25 mol L^{-1}; (b) 0.50 mol L^{-1}; (c) 1.00 mol L^{-1} (the data are cited from Ref. [13]); and (d) 1.50 mol L^{-1}.

4 (Table 2). The highest adsorption capacity of WH-225 towards SIPA is thus contributed by some other factors rather than the difference in the surface areas. It is known that adsorbents NDA-100 and XAD-4 have polydivinylbenzene network matrix, in this case, the adsorption of SIPA was based on the presence of weak van der Waals force between the polymer network and SIPA, and the π–π interactions due to the aromatic rings presented in the DVB and SIPA [9,16,17]. Different from NDA-100 and XAD-4, WH-225 contains the nitrogen in the pyridine ring, and the increase of the adsorption capacity was mainly due to the presence of such interactions as electrostatic interaction between the protonated nitrogen and the SIPA anion (Fig. 2a), besides the interaction of van der Waals force and π–π interaction between the polymer network and SIPA.

Contrary to XAD-4, NDA-100 has predominant contribution in micropore area (Table 1), which favors a solute–solute interaction for SIPA according to capillary condensation theory [18]. Therefore, the adsorption capacity of NDA-100 is much higher than that of XAD-4.

To further explain adsorption isotherms, many theories with different complexity have been proposed. The Freundlich model has been widely used to depict the adsorption isotherms [19]. The Freundlich equation in linear form is given by:

$$\ln q_e = \ln K_F + n \ln C_e \qquad (2)$$

where K_F and n are characteristic constants; generally, K_F is an index of the adsorption capacity, and the exponent n denotes the heterogeneity of the surface and the intensity of adsorption.

The regression equations for the adsorption isotherms are listed in Table 3. All the correlation factors (r) are larger than 0.99, suggesting that all the equations are reliable. The values of K_F indicate
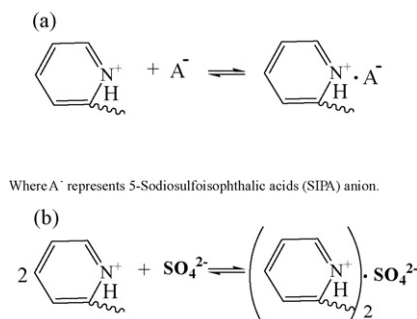
Table 2
Equilibrium adsorption capacity q_e (mmol g^{-1}) for SIPA on WH-225, NAD-100 and XAD-4 with four acid concentrations at 288 K.

Acid concentration (mol L^{-1})	Adsorbent	$C_e = 1.0$ (mmol L^{-1})	$C_e = 3.0$ (mmol L^{-1})
0.25	WH-225	0.8637	1.3801
	NAD-100	0.2205	0.3132
	XAD-4	0.0386	0.1121
0.5	WH-225	0.6771	1.2189
	NAD-100	0.2289	0.3755
	XAD-4	0.0441	0.1217
1.0	WH-225	0.5346	1.0318
	NAD-100	0.2340	0.3275
	XAD-4	0.0415	0.0991
1.5	WH-225	0.3638	0.6988
	NAD-100	0.2673	0.3973
	XAD-4	0.0342	0.0973

Where A$^-$ represents 5-Sodiosulfoisophthalic acids (SIPA) anion.

Fig. 2. (a) Electrostatic interaction between the protonated N and adsorbate SIPA anion. (b) Adsorption between the protonated N and free SO$_4^{2-}$.

Table 3
Regression equations of $\ln q_e$ vs. $\ln C_e$ for Freundlich isotherms at 288 K.

Acid concentration (mol L^{-1})	Adsorbent	Freundlich equation	K_F	n	r^2
0.25	WH-225	$\ln q_e = 0.4266 \ln c_e - 0.1465$	0.8685	0.4266	0.9972
	NAD-100	$\ln q_e = 0.3196 \ln c_e - 1.512$	0.2190	0.3196	0.9910
	XAD-4	$\ln q_e = 0.9712 \ln c_e - 3.2554$	0.04219	0.9712	0.9900
0.5	WH-225	$\ln q_e = 0.535 \ln c_e - 0.3899$	0.6798	0.5350	0.9970
	NAD-100	$\ln q_e = 0.4505 \ln c_e - 1.4743$	0.2268	0.4505	0.9947
	XAD-4	$\ln q_e = 0.9247 \ln c_e - 3.122$	0.04825	0.9247	0.9901
1.0	WH-225	$\ln q_e = 0.5984 \ln c_e - 0.6261$	0.5306	0.5984	0.9991
	NAD-100	$\ln q_e = 0.306 \ln c_e - 1.4525$	0.2327	0.3060	0.9949
	XAD-4	$\ln q_e = 0.7915 \ln c_e - 3.1811$	0.04387	0.7915	0.9907
1.5	WH-225	$\ln q_e = 0.5942 \ln c_e - 1.0112$	0.3717	0.5942	0.9943
	NAD-100	$\ln q_e = 0.3607 \ln c_e - 1.3193$	0.2676	0.3607	0.9992
	XAD-4	$\ln q_e = 0.9502 \ln c_e - 3.3742$	0.03749	0.9502	0.9920

that the adsorption capacities of SIPA on the three resins are in the following order: WH-225 > NDA-100 > XAD-4. The exponents (n) are less than 1 in all cases, indicating favorable adsorptions.

3.3. Effect of acid concentration on adsorption

In order to find out the effect of acid concentration on adsorption, the adsorption of SIPA onto WH-225 from the aqueous solution with different acid concentrations was run at 288 K, in which the initial concentrations of SIPA solutions were 1000 mg L^{-1}. Fig. 3 illustrates the effect of the acid concentration on adsorption capacity of SIPA on WH-225. It was found that the adsorption capacity of SIPA on WH-225 increases with the increasing acid concentration of SIPA solutions when acid concentration is less than 0.20 mol L^{-1}. As mentioned above, SIPA is likely adsorbed by WH-225 through electrostatic interaction between SIPA anion and positively charged N on the pyridine (described in Fig. 2a). It is known that the positively charged functional groups on the pyridine are a result of nitrogen protonated by hydrogen ion of the acid. Therefore, the more protonated nitrogen in the solution as the acid concentration of SIPA solutions increased, the more electrostatic interaction could be formed to enhance the adsorption capacity.

When acid concentration is higher than 0.20 mol L^{-1}, the equilibrium adsorption capacities of WH-225 decrease with the increasing acid concentration of the solutions (as shown in Fig. 3). Such result may be attributed to the lessened electrostatic interaction between SIPA anion and positively charged N on the pyridine as well as competitive adsorption between free SO$_4^{2-}$ and SIPA anion on WH-225 (seen in Fig. 2b). From Fig. 3, it is believed that the disso-

ciation of SIPA molecules in the solution would be restrained when the acid concentration increased and the amount of negatively charged SIPA would decrease. Therefore, the amount of adsorbed SIPA on WH-225 would decrease through electrostatic interaction between negatively charged –SO$_3^-$ and positively charged N on the pyridine. On the other hand, the sulfuric acid could also occupy the effective adsorption sites by its electrostatic interaction, resulting in the competitive adsorption between free SO$_4^{2-}$ and SIPA anion on WH-225 (seen in Fig. 2b).

3.4. Effect of temperature on adsorption and thermodynamic analysis

Usually, temperature is an important factor that directly affects the adsorption capacity. To study the effect of temperature on the adsorption performance and behavior of WH-225, three different temperatures (288, 303 and 318 K) were employed in the static adsorption experiments. The equilibrium adsorption isotherms of SIPA on WH-225 were showed in Fig. 4. The acid background 1.00 mol L^{-1} was selected to study the effect of temperature on the adsorption due to the practical wastewater containing 1.00 mol L^{-1} acid. It can be seen that lower temperature is favorable for adsorption of SIPA onto WH-225, indicating an exothermic nature of the adsorption. The isosteric enthalpies of adsorption were calculated with a derivative Van't Hoff equation [20]:

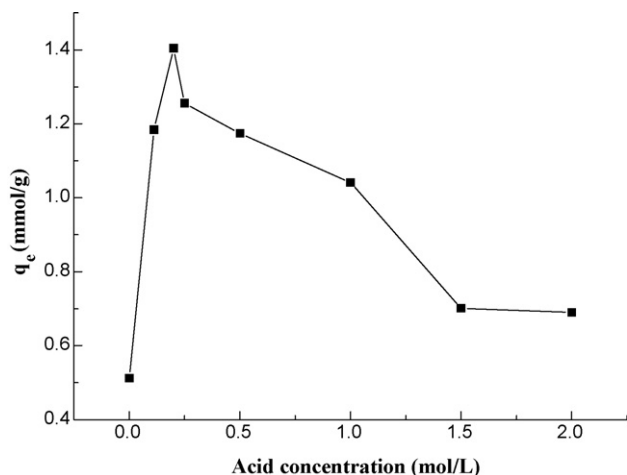$$\log\left(\frac{1}{C_e}\right) = \log(K_0) + \frac{-\Delta H}{2.303RT} \qquad (3)$$

Fig. 3. The effect of the acid concentration on adsorption capacity of SIPA on WH-225 resin with initial concentrations of SIPA 1000 ppm at 288 K.
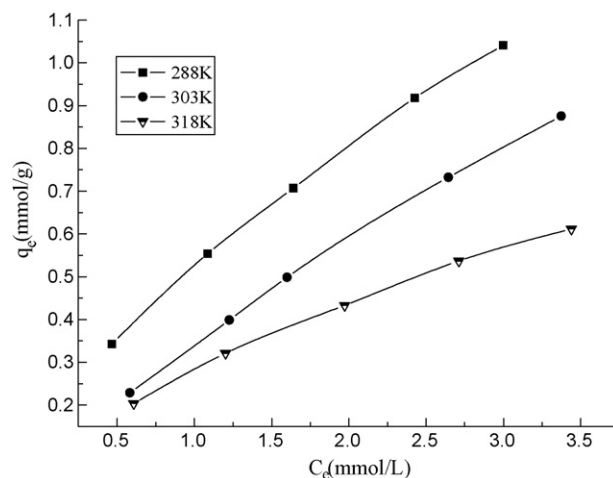

Fig. 4. Equilibrium adsorption isotherms at 288, 303, and 318 K for SIPA on WH-225 for 1.00 mol/L acid concentrations.

Table 4
Adsorption thermodynamics parameters of WH-225 towards SIPA.

q_e (mmol g^{-1})	ΔH (kJ mol^{-1})	ΔG (kJ mol^{-1})			ΔS (J mol^{-1} K^{-1})		
		288 K	303 K	318 K	288 K	303 K	318 K
0.4	−26.4	−3.9	−3.3	−4.2	−78.1	−76.2	−70.9
0.6	−25.3	−3.9	−3.3	−4.2	−74.3	−72.6	−67.4

where ΔH is the isosteric enthalpy of adsorption, R is the gas constant, C_e is the equilibrium concentration of solute in moles per liter at the absolute temperature T, and K_0 is a constant. At different temperatures (288, 303, and 318 K), C_e was obtained from the well-fitted isotherms. ΔH was calculated from the slope of line plotted vs. $1/T$.

Other thermodynamics parameters, such as the apparent energy (ΔG) and the entropy (ΔS), can be calculated by Eqs. (4) and (5), respectively [20]:

$$\Delta G = -RT \int_0^X \left(\frac{q}{X}\right) \, dX \tag{4}$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \tag{5}$$

where X represents the molar fraction of the adsorbate in solution, q is then replaced by the adsorption isotherm equation. The use of the Freundlich isotherm would lead to a value for the apparent free energy independent of q [21]:

$$\Delta G = -nRT \tag{6}$$

The calculated isosteric enthalpies of adsorption are summarized in Table 4. The negative values of the enthalpies indicate an exothermic adsorption process for WH-225 adsorbing SIPA, while the range of their magnitudes (<43 kJ mol^{-1}) further show a physical adsorption process. The negative values of ΔG, then, indicate the spontaneous nature of the adsorption processes. Finally, the negative values of the adsorption entropy are in agreement with restricted mobilities of the adsorbed SIPA molecules as compared with those in solution. The absolute entropy values decrease slightly with the increase of temperature. The molecular interaction makes the system more ordered and therefore decreased the entropy.

3.5. Column adsorption and desorption of SIPA on WH-225

Since WH-225 is to be developed as a polymeric adsorbent for removal and recovery of organic pollutants from wastewater, the column adsorption and desorption studies are thus practically very important. An aqueous 20,000 mg L^{-1} solution of SIPA with 1.00 mol L^{-1} acid was passed through a column containing WH-225. In the current study, breakthrough capacities and total capacities of adsorbents for the adsorption of SIPA were determined by the continuous flow column methods when the effluent concentration from the column reached 5% and nearly 100% of the initial concentration, respectively. The adsorption curve of SIPA on WH-225 was plotted in Fig. 5a. As the effluent volume was about 14 BV, where BV represented the total volume of resin bed, SIPA from the column began to leak. The breakthrough capacities and total capacities of the adsorbent are 1.0 and 1.2 mmol mL^{-1}, respectively. After finishing the column adsorption, 2 wt% sodium hydroxide was used to regenerate WH-225 and recover SIPA from the resin. Desorption efficiency was observed to attain 100% by treating with 4.25 bed volume of the alkaline solution at room temperature (seen in Fig. 5b).
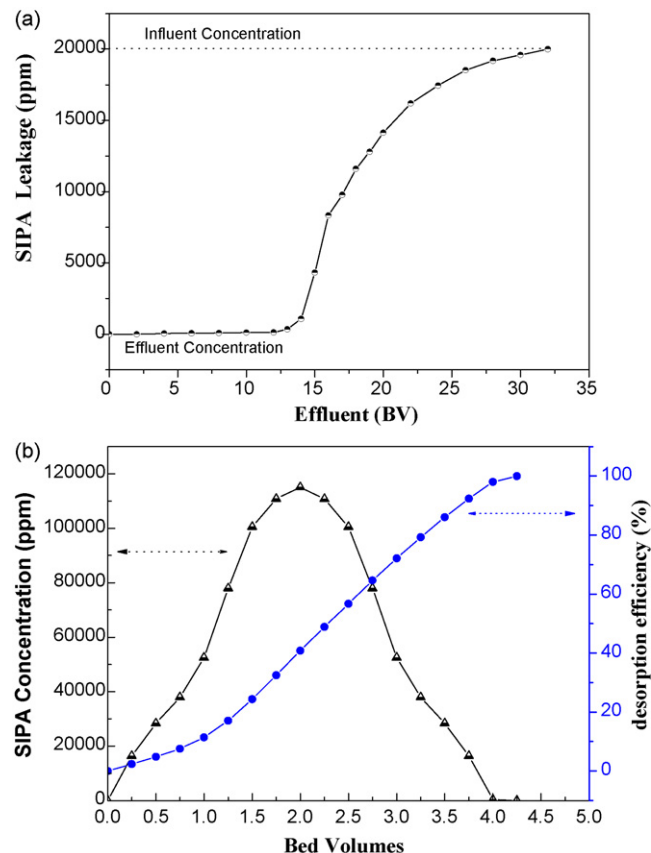

Fig. 5. (a) Column adsorption for SIPA on WH-225 at a rate of 2 BV h^{-1} with 1 mol L^{-1} acid concentration. (b) Column dynamic desorption and desorption efficiency curve of SIPA on WH-225 resin using 2 wt% sodium hydroxide as the eluant by continuous flow column method at a rate of 1 BV h^{-1}.

4. Conclusions

Poly(2-vinylpyridine) resin, WH-225, was proved to be the most efficient adsorbent for separating SIPA from aqueous solutions in comparison with NDA-100 and XAD-4 at the same static sorption conditions, which mostly attributes to its electrostatic interaction. All the adsorption data of SIPA on the three resins fit the Freundlich model very well.

Thermodynamic analysis showed that negative values of the adsorptive enthalpy changes indicate an exothermic process for WH-225 adsorbing SIPA, and the magnitudes (<43 kJ mol^{-1}) further manifest the adsorption process is physical.

In the column assay, the breakthrough capacities and total capacities of WH-225 towards SIPA are 1.0 and 1.2 mmol mL^{-1}, respectively. Besides, SIPA can be easily desorbed from WH-225 with 2 wt% sodium hydroxide and 100% desorption efficiency was achieved as expected, which further indicates WH-225 is a quite effective sorbent for removal and recovery of aromatic acids from aqueous solution, such as SIPA.

Acknowledgements

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