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Removal of ammonium salt from aqueous solution of glycolic acid with macroporous resin

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ABSTRACT

Effects of main properties of eight resins on the removal of $(NH_4)_2SO_4$ from the aqueous solution of glycolic acid (GA) were studied via the dynamic adsorption separation method in the fixed-bed column. The results indicated that the breakthrough capacity (Q) and the saturation capacity (Q_e) for eight resins gradually decreased with enhancing temperature and flow rate, and the breakthrough volume and the saturation volume had a similar variation trend. Moreover, higher polarity of resin or larger specific surface area of resin could enhance separation capability, and smaller pore size of resin was advantageous to the removal of $(NH_4)_2SO_4$. The change degrees of Q and Q_e of GA for different polarities of resins were greater than that of Q and Q_e of $(NH_4)_2SO_4$ under different temperatures, whereas Q and Q_e of smaller specific surface area of resin and larger pore size of resin showed a contrary trend. In addition, HPD950 resin with larger specific surface area and lower polarity and suitable pore size was suitable choice for removal of $(NH_4)_2SO_4$ from the aqueous solution of GA.

Keywords: Ammonium salt; Removal; Glycolic acid; Macroporous resin

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1. Introduction

Glycolic acid (GA), one of simple carboxylic acid, is usually used as the medical materials and the cleaner [1,2]. Currently, GA sold on the market is mainly obtained by the chemical synthesis method due to easy operation and good product quality [3,4]. Especially, the hydrolysis of glycolonitrile with the dilute mineral acid (such as sulfuric acid) is widely applied in the production of GA due to higher yield [5]. However, the ammonium salt (e.g., $(NH_4)_2SO_4$) will be produced when the dilute mineral acid is used to hydrolyze the glycolonitrile [6], and the hydrolysate of glycolonitrile containing ammonium salt is unfavorable for the crystallization of GA.

In order to meet the standard of aqueous solution of GA, it is very necessary to undertake the experimental research on the removal of ammonium salt. Some methods, e.g., rectification [1], electrodialysis [7] and extraction [8], have been used to treat the ammonium salt in the aqueous solution of GA, however, some intrinsic drawbacks affect its wide application, e.g., higher energy consumption, membrane fouling and residual extractant [9,10]. In addition, the surface imprinting technique is of some help to the purification of aqueous solution of organics [11–14]. Resin adsorption, higher efficiency and lower cost, may be a more suitable choice for the separation of substance from the aqueous solution. To date, resin adsorption has been successfully applied in separation purifications of substances, e.g., heavy metal [15], radioactive substance [16] and organics [17].

Macroporous resin has specific affinity force to the organics [18,19] and weaker adsorption force to the inorganics. The inorganics should firstly break through the resin bed due to the difference of adsorption force when the sample solution is continuously loaded. On the other hand, the removal of inorganic salt from aqueous solution of organics via resin adsorption method hardly needs the acid–alkali regeneration

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[20,21]. In addition, other substances, e.g., ion exchanger (e.g., ion-exchange resin and ion-exchange fiber) [22] and natural materials (e.g., modified lignocelluloses and modified zeolite) [23], have a certain removal capability of salt. However, ion exchanger is usually not suitable for high salt solution and need the acid–alkali regeneration. The removal efficiency of salt with the natural materials is relatively low and cannot use for many times, and the regeneration of natural materials will be more trouble and uneconomical. To the best of our knowledge, the removal of ammonium salt from the aqueous solution of GA via adsorption separation method of resin has not been addressed.

In order to develop an economically viable removal system of ammonium salt, the continuous fix-bed separation of ammonium salt from the aqueous solution of GA using macroporous resin was studied. Effects of polarity, specific surface area and pore size on the fixed-bed separation process were systemically analyzed in order to explore the interaction of the removal capability with the main properties of resin. Here, $(NH_4)_2SO_4$ was chosen as model inorganic salt due to the hydrolysis of glycolonitrile with dilute sulfuric acid.

2. Materials and methods

2.1. Materials

All reagents were of analytical grade without further purification and purchased from Sinopharm Chemical Reagent Co., Ltd. (China). Resins and ion-exchange fiber were provided by Wuxi Applied Clean Separation Technology Institute (China), and the main properties of macroporous resin were presented in Table 1. The deionized water was used to prepare the aqueous solution of GA.

2.2. Procedure

The relative polarity of resin was determined with the HPLC (High performance liquid chromatography) method according to the relative retention time relationship between the tested resin and the reference resin (the polarities of DA-201 and D101 were 100 and 0, respectively) [24].

Prior to use, resins were sequentially dipped in four times volume of 2% NaOH and 95% ethanol for 24 h, and then resins were repeatedly washed with the deionized water and dried in the vacuum oven at 50°C to the constant weight.

Table 1
Main physicochemical properties of resins

Resin	Brunauer-Emmett-Teller (BET) surface area (m^2/g)	Average pore size (Å)	Relative polarity
HPD80	345	94	4.9
HPD100B	567	106	1.0
HPD300	874	53	7.2
HPD450	567	96	50.0
HPD722	494	105	20.7
HPD950	1159	98	10.6
X-5	573	298	8.5
D4020	558	103	12.7

All experiments were conducted in the fixed-bed column (inner diameter 2 cm, length 25 cm). 4 g resin was packed in the fixed-bed column. The temperature of fixed-bed column was maintained with the circulation heating system. The aqueous solution ($(NH_4)_2SO_4$ 15 mg/mL, GA 10 mg/mL) was passed through the fixed-bed column at the given temperature (T, °C) and the flow rate (mL/min). At intervals, samples were taken and then determined with UV spectrophotometry and HPLC, respectively. The breakthrough volume (V_1, mL) and the saturation volume (V_2, mL) were sequentially obtained according to the determinations of samples.

2.3. Analysis

$(NH_4)_2SO_4$ was determined with the methanal–acetylacetone spectrophotometry method [25,26]. Hexamethylenetetramine was produced via the transformation of NH_4^+ with the excess methanal, acetylacetone reacted with the rest of methanal to produce 3,5-diacetyl-1,4-dihydropyridine which was determined with UV spectrophotometry (UV-1800, Shimadzu, Japan) at 414 nm. The subtraction method was used to calculate the content of $(NH_4)_2SO_4$ in aqueous solution of GA.

GA was determined by HPLC (LC-10AT, C18 column, Shimadzu, Japan) with UV detector at 225 nm. The mobile phases consisted of an 85/15 ratio of water to methanol (V/V) with a flow rate of 1 mL/min [27].

The equations of breakthrough capacity (Q, mg/g dry resin) and the saturation capacity (Q_e, mg/g dry resin) are presented as:

$$Q = \frac{C_0 \times V_1}{m \times (1 - w)} \tag{1}$$

$$Q_e = \frac{C_0 \times V_2 - \sum C_j \times V_j}{m \times (1 - w)} \tag{2}$$

where C_0 (mg/mL) is the initial concentration of component, C_j (mg/mL) is the component concentration at t min in effluent; V_1 (mL) and V_2 (mL) are the breakthrough volume and the saturation volume, respectively; V_j (mL) is the volume of effluent at t min; m is the weight of wet resin (g); and w is the water content of wet resin (%).

3. Results and discussion

3.1. Polarity of resin

3.1.1. Effect of temperature

The microcosm essence of resin polarity arises from difference in the chemical composition and the surface structural form, which influences the adsorption separation capability of resin. Thus, the effects of relative polarities of resins on the removal of $(NH_4)_2SO_4$ and the purification of aqueous solution of GA were systemically studied under different temperatures.

As shown in Fig. 1, Q and Q_e gradually decreased with increasing temperature of fixed-bed column of resin, and the increase of relative polarity of resin (Table 1) was prone to the enhancement of adsorption separation capability. Indicating that the increase of relative polarity of resin was obviously beneficial to removal of $(NH_4)_2SO_4$ from aqueous solution of GA due to the interaction force between resin and GA (Fig. 2), whereas the increase of temperature of fixed-bed column of resin was unbeneficial to the adsorption separation process. Thus, the removal of $(NH_4)_2SO_4$ should be performed at the lower temperature of fixed-bed column of resin due to greater differences of Q and Q_e. In addition, the adsorption capability of resin for GA was stronger than that for $(NH_4)_2SO_4$, indicating that the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA via the adsorption separation method of resin was feasible and effective in the industrial production.

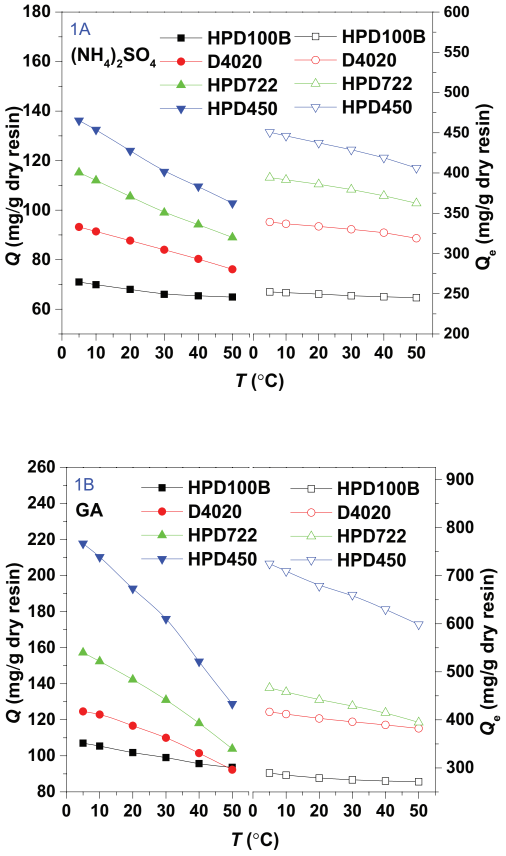


Fig. 1. (A) Q and (B) Q_e of resins (relative polarities of resins, HPD100B – 1, D4020 – 12.7, HPD722 – 20.7, HPD450 – 50).

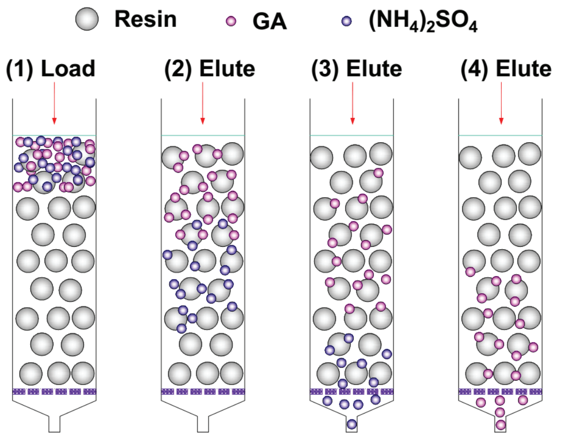


Fig. 2. Schematic diagram of separation of $(NH_4)_2SO_4$ from aqueous solution of GA.

The change degrees of adsorption separation capabilities of resins with temperature of fixed-bed column increasing from 5°C to 50°C were further studied. As shown in Fig. 3, the change degrees of Q and Q_e gradually increased with the increase of relative polarities of resins for $(NH_4)_2SO_4$ and GA, meanwhile the change degrees of Q were higher than that of Q_e. Thus, the temperature of fixed-bed column of resin had a significant effect on the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA via the adsorption separation method of resin. In addition, the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA with the resin adsorption method should be an entropy increment process according to the experimental results. The GA molecule moved quickly in the fixed-bed column of resin with increasing the temperature, which could lead to lower removal efficiency of $(NH_4)_2SO_4$.

3.1.2. Effect of flow rate

The flow rate should be an important parameter to affect the retention time of substance in the fixed-bed column of resin. Thus, effect of flow rate on the breakthrough volume (V_1) and the saturation volume (V_2) under different flow rates (0.5–3 mL/min) were studied.

As shown in Fig. 4, V_1 and V_2 of four resins gradually decreased with the increase of flow rate, indicating that the breakthrough time was shorter and the breakthrough point shifted the left direction in the dynamic adsorption separation curves of resins (Fig. 5). The possible reasons of change trends of V_1 and V_2 were mainly induced by shortening the retention time and quicker movement of substance in the fixed-column bed of resin [28]. Moreover, V_1 and V_2 of $(NH_4)_2SO_4$ gradually increased with enhancing the relative polarity of resin, and the breakthrough point shifted the right direction in the dynamic adsorption curve of resin (Fig. 5),

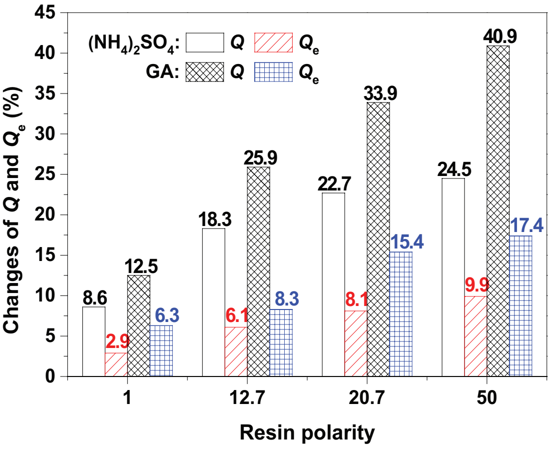


Fig. 3. Changes of Q and Q_e (relative polarities of resins, HPD100B – 1, D4020 – 12.7, HPD722 – 20.7, HPD450 – 50).

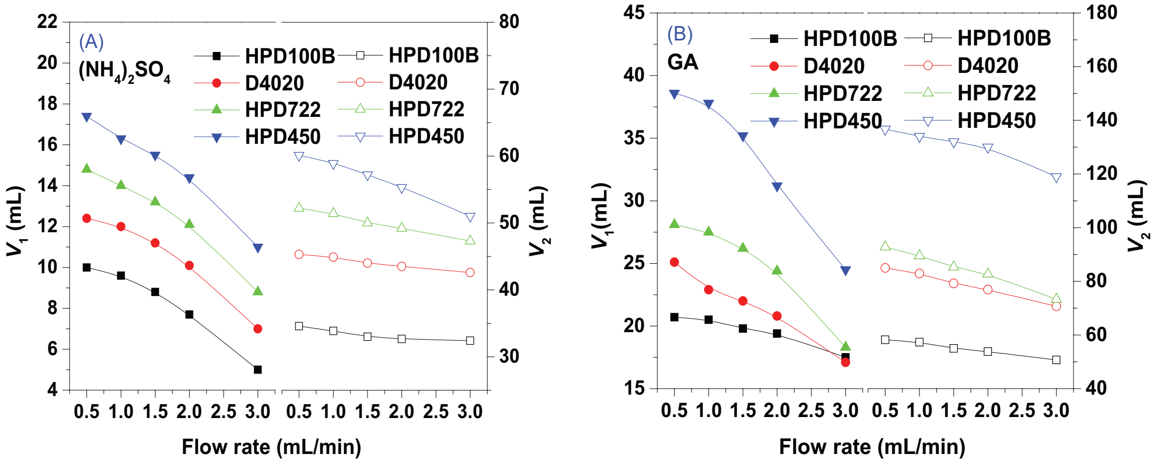


Fig. 4. (A) V_1 and (B) V_2 under different flow rates (relative polarities of resins, HPD100B – 1, D4020 – 12.7, HPD722 – 20.7, HPD450 – 50).

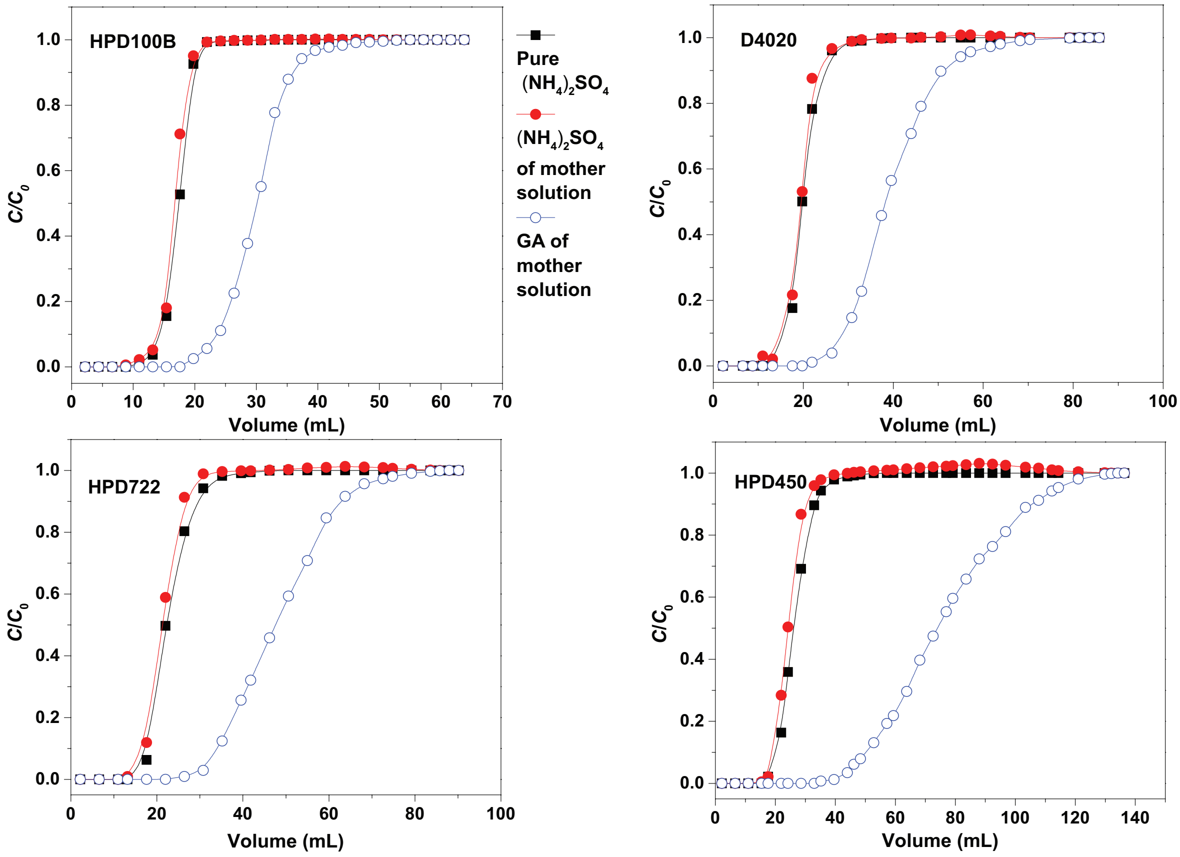


Fig. 5. Dynamic adsorption curves of $(NH_4)_2SO_4$ and GA (relative polarities of resins, HPD100B – 1, D4020 – 12.7, HPD722 – 20.7, HPD450 – 50).

which was beneficial to the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA.

V_1 and V_2 of $(NH_4)_2SO_4$ markedly decreased when the flow rate was beyond 1.5 mL/min, thus the flow rate of liquid should be below 1.5 mL/min with the dynamic adsorption separation method of resin to remove $(NH_4)_2SO_4$ from the aqueous solution of GA. Moreover, the results of dynamic adsorption curves of resins showed that the separation capabilities of $(NH_4)_2SO_4$ for four resins reduced to 2.7%, 4.4%, 6.4% and 8.3%, respectively (the area difference of breakthrough curves), indicating that higher polarity of resin was beneficial to the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA. Meanwhile, the part of protrude from the dynamic adsorption curve of resin occurred (C/C_0 > 1) and gradually increased with the increase of polarity of resin, indicating the $(NH_4)_2SO_4$ on the resin was gradually displaced by the GA molecule due to stronger interaction force between resin and GA molecule.

3.2. Specific surface area of resin

3.2.1. Effect of temperature

As shown in Fig. 6, the increase of specific surface area of resin was beneficial to the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA due to the increase of Q and Q_e of resin, whereas the increase of column temperature had an inhibition effect on the removal of $(NH_4)_2SO_4$. Larger specific surface area of resin had more available reaction active site and more open pore structure, which was beneficial to faster and easier internal diffusion of substance inside pore [20,29]. Smaller pore volume of resin with less available active sites would render it difficult for the removal of $(NH_4)_2SO_4$ from aqueous solution of GA.

The change degrees of adsorption separation capabilities of resins were further studied with the temperature of fixed-bed column of resin increasing from 5°C to 50°C. As shown in Fig. 7, Q and Q_e of resin were significantly influenced by changing the temperature of fixed-bed column of resin, and Q and Q_e of higher specific surface area of resin obviously changed, whereas Q and Q_e of lower specific surface area of resin slightly changed. Indicating that the removal of $(NH_4)_2SO_4$ from aqueous solution of GA with higher specific surface area of resin should be performed at low column temperature. The possible reason of separation capabilities of $(NH_4)_2SO_4$ and GA could arise from quicker movement of adsorbate at higher column temperature, which might lead to getting rid of the interaction force between resin and adsorbate as well as insufficient diffusion of substance inside the pore [30].

3.2.2. Effect of flow rate

As for the movement of adsorbate in the fixed-bed column of resin, it should be affected by the flow rate of liquid. As shown in Fig. 8, the increase of flow rate obviously resulted in the decreases of V_1 and V_2 for $(NH_4)_2SO_4$ and GA, indicating that higher flow rate of liquid was unbeneficial to the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA due to shorter retention time. Moreover, V_1 and V_2 for GA and $(NH_4)_2SO_4$ gradually increased with enhancing specific surface area of resin. The breakthrough point shifted the right direction in the dynamic adsorption curve of resin, and the breakthrough time was prolonged. This phenomenon indicated that higher specific surface area of resin was prone to strengthen the bound effect of GA on the resin.

The dynamic adsorption curves of resins indicated that the removal capacity of $(NH_4)_2SO_4$ from the aqueous solution of GA for HPD80 resin, D4020 resin and HPD950 resin reduced to 2.7%, 4.4% and 4.7%, respectively (Figs. 5 and 9). Thus, higher specific surface area of resin was beneficial to the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA due to the decrease of $(NH_4)_2SO_4$ on the surface of resin. In addition, $(NH_4)_2SO_4$ on the resin was gradually displaced by GA molecule during the dynamic separation process (protrude part of dynamic adsorption curve, $C/C_0 > 1$) due to stronger interaction force between resin and GA.

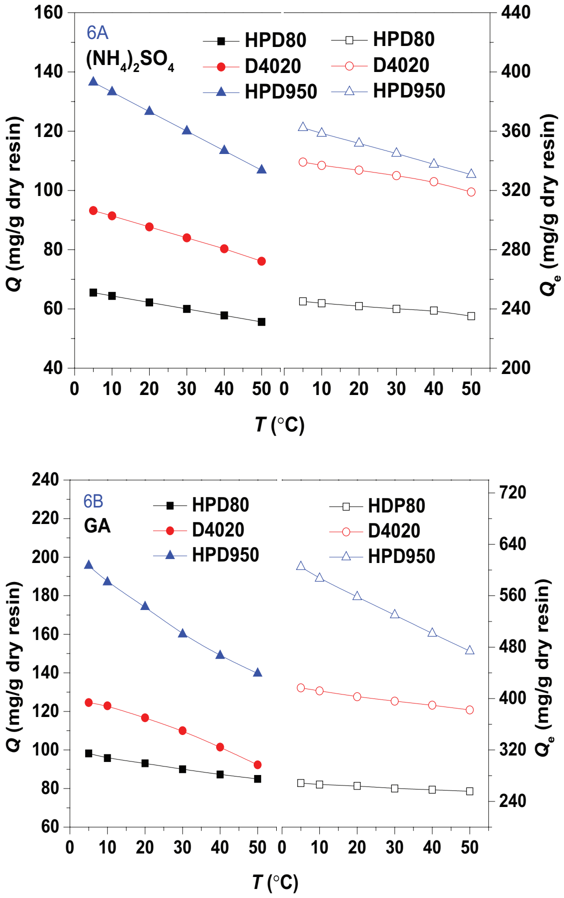


Fig. 6. (A) Q and (B) Q_e of resins (specific surface areas of resins, HPD80 – 345 m²/g, D4020 – 558 m²/g, HPD950 – 1,159 m²/g).

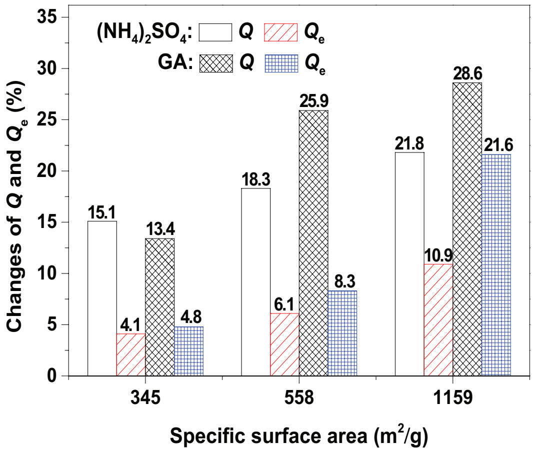


Fig. 7. Changes of Q and Q_e (specific surface areas of resins, HPD80 – 345 m²/g, D4020 – 558 m²/g, HPD950 – 1,159 m²/g).

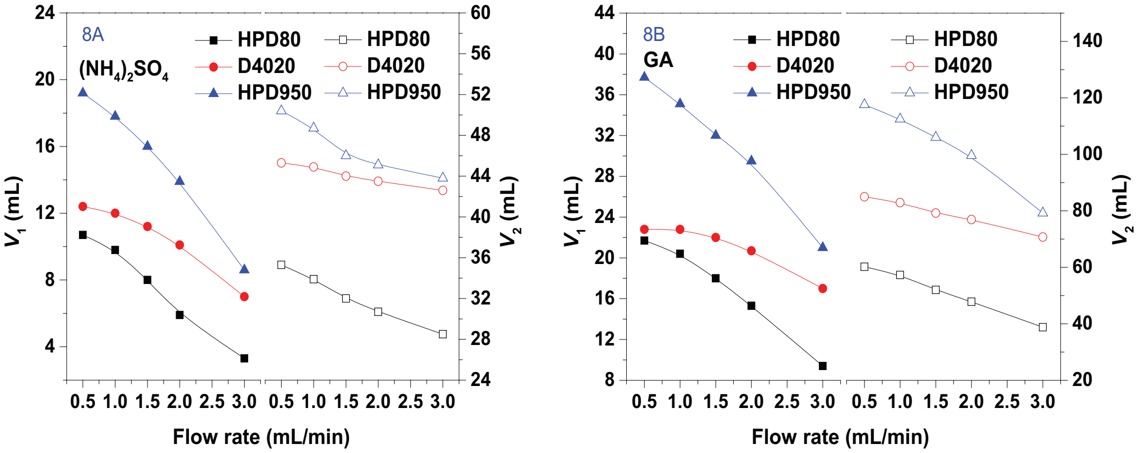


Fig. 8. (A) V_1 and (B) V_2 of resins under different flow rates (specific surface areas of resins, HPD80 – 345 m^2/g, D4020 558 – m^2/g, HPD950 – 1,159 m^2/g).

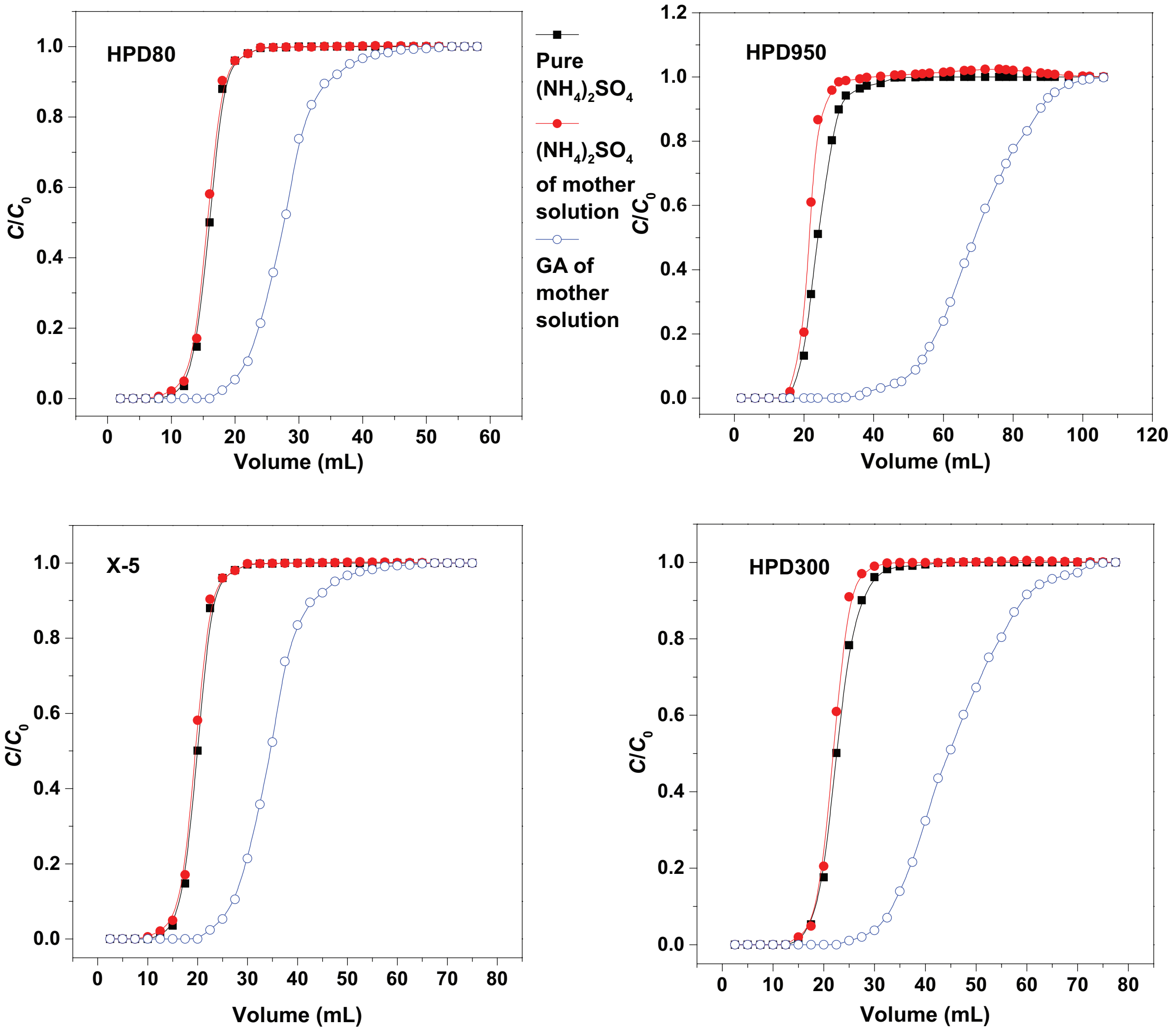


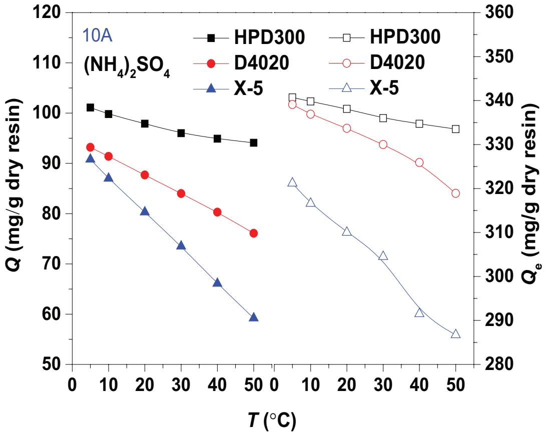
Fig. 9. Dynamic adsorption curves of $(NH_4)_2SO_4$ and GA (specific surface areas of resins, HPD80 – 345 m^2/g, D4020 – 558 m^2/g, HPD950 – 1,159 m^2/g).

3.3. Effect of pore size

3.3.1. Effect of temperature

As shown in Fig. 10, *Q* and Q_e of $(NH_4)_2SO_4$ gradually decreased with enhancing the temperature of fixed-bed column of resin, thus the increase of column temperature was unbeneficial to the removal of $(NH_4)_2SO_4$ from aqueous solution of GA. *Q* and Q_e of $(NH_4)_2SO_4$ gradually decreased with the increase of pore size of resin (HPD300 > D4020 > X-5), meanwhile *Q* and Q_e of GA had similar trend to that of $(NH_4)_2SO_4$. Moreover, *Q* and Q_e of $(NH_4)_2SO_4$ were higher than that of GA, which was more advantageous to the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA due to difference of adsorption capability, and the differences of *Q* and Q_e of resins were mainly from the physicochemical property of resin and the concentration of substance inside pore of resin [22]. Larger pore size of resin should be better for the diffusion of GA and the adsorption rate of GA, which could enhance the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA. In addition, the length of GA molecule was about 5–15 Å and far below the pore size of resin [31], thus the ammonium salt in aqueous solution of GA was efficiently removed with higher polarity and higher specific surface of resin.

The change degrees of separation capabilities of resins with the change of pore size (from 5°C to 50°C) were further investigated. As shown in Fig. 11, *Q* and Q_e of smaller pore size of resin for $(NH_4)_2SO_4$ slightly changed, whereas *Q* and Q_e of larger pore size of resin significantly changed. Moreover,



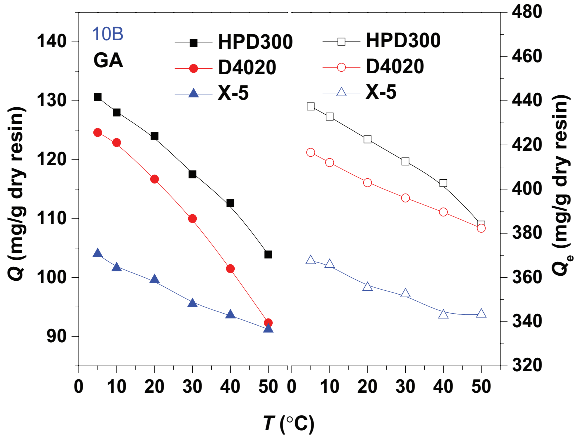


Fig. 10. (A) *Q* and (B) Q_e of resin (HPD300 – 53 Å, D4020 – 103 Å, X-5 – 298 Å).

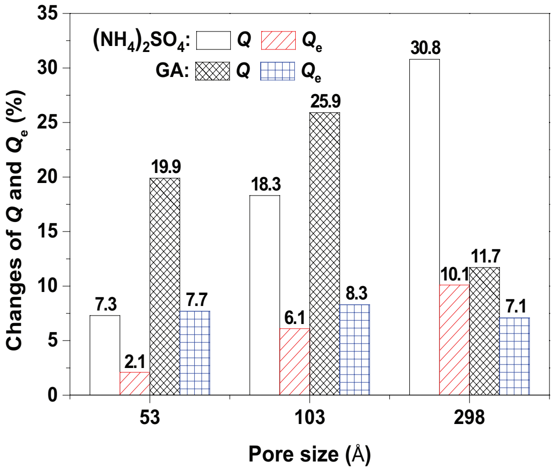
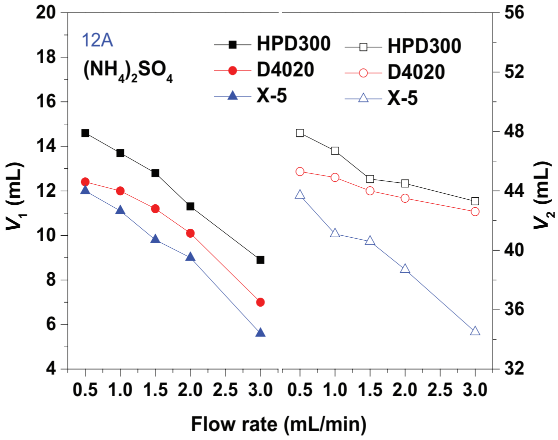


Fig. 11. Changes of *Q* and Q_e (HPD300 – 53 Å, D4020 – 103 Å, X-5 – 298 Å).



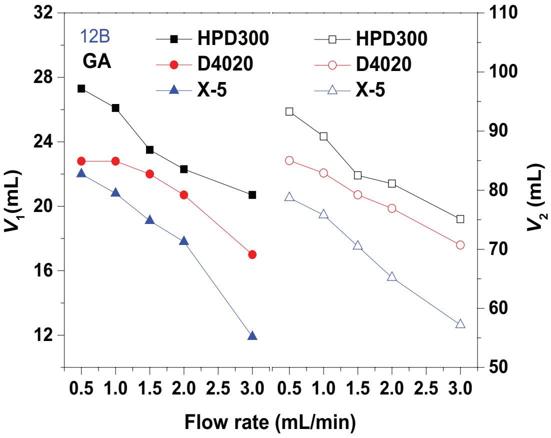


Fig. 12. (A) V_1 and (B) V_2 under different flow rates (HPD300 – 53 Å, D4020 – 103 Å, X-5 – 298 Å).

the removal capability of $(NH_4)_2SO_4$ reduced to 2.3%, 4.2% and 4.4% with increasing the pore size of resin, respectively (Figs. 5 and 9). Indicating that smaller pore of resin was more advantageous to the removal of $(NH_4)_2SO_4$ due to stable separation capability of $(NH_4)_2SO_4$.

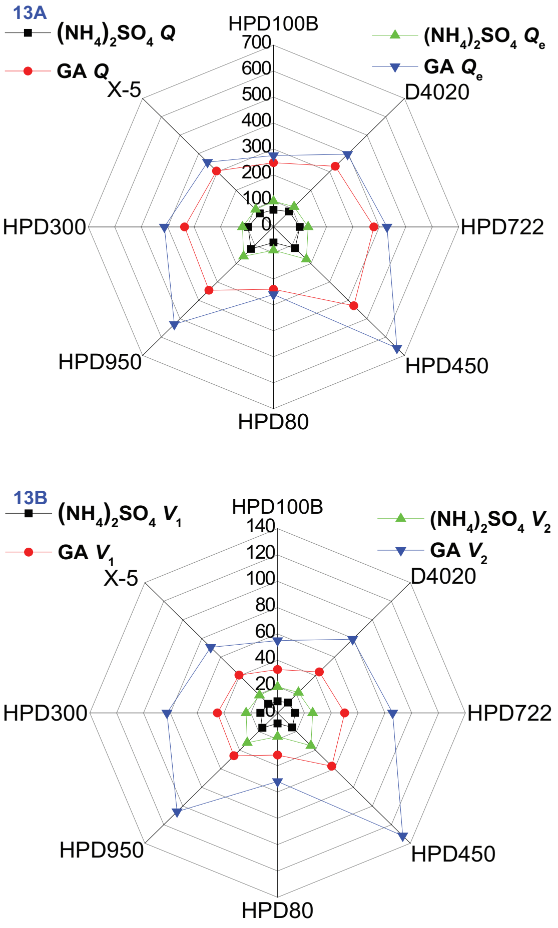


Fig. 13. Spider plots of: (A) Q and Q_e and (B) V_1 and V_2 for resins.

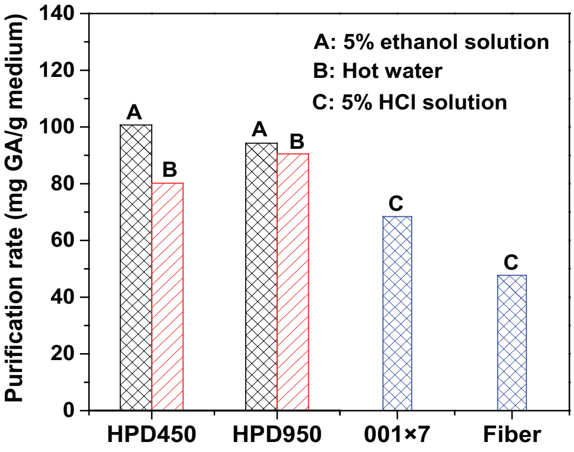


Fig. 14. Comparison of removal capacity of different media.

3.3.2. Effect of flow rate

As for effect of flow rate, the results (Fig. 12) indicated that V_1 and V_2 gradually decreased with enhancing the flow rate of liquid, and V_1 and V_2 of larger pore size of resin significantly decreased. This phenomenon could arise from the decrease of specific surface area for larger pore size of resin. Thus, suitable pore size of resin was very important to remove the ammonium salt from the aqueous solution of GA.

3.4. Comprehensive analysis

Main purpose of spider analysis was to set up the brief pictorial diagram of resin property on the removal of ammonium salt from the aqueous solution of GA. As shown in Fig. 13(A), Q and Q_e of HPD450 and HPD950 were higher for the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA, whereas Q and Q_e of the other resins were lower. HPD450 (higher relative polarity) was prone to the interaction between reactive site of resin and GA due to stronger absorption force. As for HPD950 (larger specific surface area) could provide more adsorption reactive site per unit volume of resin, which was advantageous to the removal of $(NH_4)_2SO_4$ from aqueous solution of GA. Thus, it could be concluded that the removal of $(NH_4)_2SO_4$ from aqueous solution of GA via adsorption separation method of resin should consider higher polarity of resin firstly, specific surface area secondly and pore size thirdly.

As shown in Fig. 13(B), values of V_1 and V_2 for HPD450 were higher than that of the other resins, and the difference values of V_1 and V_2 for HPD450 were also greater. Thus, higher polarity of resin could enhance the separation capability and was beneficial to the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA. However, the removal of $(NH_4)_2SO_4$ with higher polarity of resin probably raised some questions, e.g., increase of energy consumption, choosing higher polarity of eluent. Thus, HPD950 (higher specific surface area, lower polarity) was suitable choice for the removal of $(NH_4)_2SO_4$ from aqueous solution of GA.

In order to evaluate the removal efficiency of ammonium salt, the representative resins (HPD450 and HPD950) and two other ion exchangers (strong-acid cation exchange resin 001×7, strong-acid cation exchange fiber) were further studied. The eluents were water (50°C) and 5% ethanol solution (25°C). The regenerant of two ion exchangers was 5% HCl solution (25°C). As shown in Fig. 14, the removal efficiency of ammonium salt via adsorption separation of macroporous resin was significantly higher than that of the other medium, and the eluant containing ethanol was beneficial to the removal of ammonium salt with macroporous resin. The possible reason of above phenomenon arises mainly from different desalination mechanisms, and the removal of salt with ion exchanger is the exchange of functional groups, whereas macroporous resin is the physical adsorption.

4. Conclusion

The breakthrough capacity (Q) and the saturation capacity (Q_e) gradually decreased with enhancing the temperature of fixed-bed column of resin as well the flow rate of liquid. Higher polarity of resin or larger specific surface area of resin could raise the separation capability of resin and the effluent volume (V_1 and V_2), and values of Q and Q_e for higher polar resin and larger specific surface area of resin greatly changed with the temperature change of column bed. Moreover, Q and Q_e and the effluent volumes (V_1 and V_2) of $(NH_4)_2SO_4$ decreased with the increase of pore size of resin, and the enhancement of resin pore size was not always better for the removal of $(NH_4)_2SO_4$. HPD950 with higher specific surface area and lower polarity was suitable choice for the removal of $(NH_4)_2SO_4$ from the aqueous solution of GA and beneficial to the elution with common eluent.

Symbols

C_0 — Initial concentration of component, mg/mL
C_j — Component concentration in effluent, mg/mL
V_1 — Breakthrough volume, mL
V_2 — Saturation volume, mL
V_j — Volume of effluent, mL
m — Weight of wet resin, g
w — Water content of wet resin, %

Acknowledgments

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