

Parametric Studies on the Performance of Cation Exchange for the Ammonium Removal

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Abstract—Ion exchange performance to remove ammonium in drinking water was studied experimentally in batch and continuous operation systems under the various conditions. Data were collected using commercially available strong-acid cation-exchange resins of Na^+ and H^+ types. The performance was evaluated using equilibrium concentrations for the batch system or the effluent concentration histories for the continuous column system as a function of time or the solution volume passed through the experimental column until resins were exhausted. With high temperature or low initial feed concentration, ammonium removal characteristics of the batch system increase. At the solution concentrations of 0.5, 1.0, and 2.0 mg/L of NH_4^+ -N and the temperatures of 15, 25, and 35 °C, the selectivity coefficients of resin were determined between 1.38 and 1.43 for Na^+ type resin, and 3.22 and 3.47 for H^+ type resin. The selectivity coefficient was correlated as a function of temperature using Kraus-Raridon equation. The breakthrough curves obtained from the continuous column operation give some results; i) with small column diameter or large column height, ii) with low initial feed concentration, iii) with low volumetric flow rate, or iv) with high solution temperature, the ammonium removal for the typical macroporous type resin increase. The results of this study could be scaled up and used as a design tool for the water-purification systems of the drinking water treatment processes.

Key words: Ion Exchange, Denitrification, Ammonium Removal, Selectivity Coefficient

INTRODUCTION

Ammonium concentration in drinking water is increasing all over the world due to natural source or waste from animal including men and from various industries. Ammonium in water itself is not toxic to men, but toxic to some fishes as a function of pH, fish species and size, and ammonium concentration. Ammonium can cause oxygen depletion in water as it is oxidized to nitrite and then to nitrate which is toxic especially to pregnant women and infants. Ammonium is also an algal nutrient source for the growth-limiting factor to the causes of eutrophication. This causes a fatal damage to the sea food industries [Lee, 1999]. The Korean standard of 0.5 mg NH_4^+ -N/L or less in drinking water was established for the human health.

The solution of the ammonium contamination problem requires that appropriate water treatment and purification technologies be developed. Methods of removing ammonium from water and wastewater can be; either biological processes which include nitrogen release from microbial nitrification-denitrification, or chemical-physical processes which include ammonia stripping, ion exchange, and breakpoint chlorination. The processes of electrodialysis, reverse osmosis, distillation, and algae harvesting look less potential for general application at this time.

Ion exchange is a physicochemical process in which ions in the water are exchanged for chemically equivalent number of ions associated with the exchange material. Ion exchange is economical and convenient and can provide a suitable solution for small or medium sized water treatment plants containing comparably low ammonium level [Kim et al., 1999]. In the process, raw water contacts with ion exchange material, and ammonium ions in water are exchanged for similarly charged hydrogen or sodium ions.

Selective ion exchange is a very reliable means for the removal of ammonium and other ions. Clinoptilolite was found to be an effective zeolite to remove ammonium selectively from wastewater [Beler-Baykal, 1994]. However, clinoptilolite is suitable only for the wastewater treatment system which usually employs a combination of biological nitrification and physicochemical process. Ion exchange capacity of clinoptilolite is much less than that of synthetic ion exchange resin, and not applicable for the final polishing step where a high degree of purity water is required. Especially, drinking water needs a highly selective strong-acid cation exchange resin to remove ammonium ions in water.

This paper presents the ammonium removal performance of cation exchange resin under the various conditions. The results of this study include the effects of the important system parameters such as resin type, resin weight, initial ammonium concentration, volumetric flow rate, column diameter and depth, and temperature using batch and continuous experimental systems, and can be a part of the denitrification process development by the mixed-bed ion exchange.

THEORY

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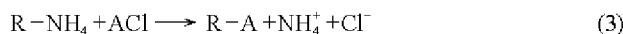
Ion exchange is a physicochemical process in which ions in water are exchanged for chemically equivalent number of ions associated with the exchange material. This ion-exchange material includes naturally occurring clays and synthetic resins. The resins are in the form of gel or macroporous granules. The resin structure consists of an interconnected network of hydrocarbons where soluble ionic functional groups are attached. In this process, raw water contacts with ion exchange resin, and ammonium ions in water are exchanged for similarly charged sodium or hydrogen ions. The process is continuous until the resin bed is exhausted, and then the resin is regenerated before continuing the process. The process is described as:



where A is Na^+ or H^+ , and the selectivity coefficient is defined as:

$$K_A^{NH_4^+} = \frac{[R - NH_4^+][A^+]}{[R - A][NH_4^+]} \quad (2)$$

and the regeneration process is:



Ion exchange is an exothermic process, and the temperature adversely affects on ion-exchange equilibrium and hence the selectivity coefficient. Selectivity is the preference of the particular ion exchanger for one ion over the other ion, and the selectivity coefficient can conveniently describe ion-exchange equilibria. The resin selectivity toward the exchanging ions is used to determine the sharpness of the exchange wave [Helfferich, 1962]. The selectivity coefficient is greater than one when ion in the solution phase is preferred by the resin phase. In this case, the equilibrium is said to be favorable.

Many methods of correlating the temperature dependency of the selectivity coefficient were proposed. Among these, Kraus-Raridon method provided convenient and practical method for determining the effect of temperature on equilibria [Divekar et al., 1987]. Using the Debye-Hückel theory of electrolytes, Kraus and Raridon [1959] developed a method for expressing the selectivity coefficients as a function of temperature. The expression for the selectivity coefficient is

$$\log K = \log K_t + C' \log (T/T_t) + C'' (1 - T/T_t) \quad (4)$$

where C' , C'' and K_t are constants and subscript t refers to a reference temperature. By fitting experimental data to Eq. (4), Kraus et al. [1960] determined the parameters C' , C'' and K_t by the least squares method. Divekar et al. [1987] verified the deviations between observed and calculated values of the selectivity coefficient within limits of experimental error. Divekar et al. [1987] also developed an equation for the cation- and anion-exchange selectivity coefficients by using experimental results especially reported on Dowex resins, and applied it to the mixed-bed ion exchange model for ultralow concentration. Their equation is easy to use but not practically proposed.

The selectivity of an ion-exchange resin is enhanced by increasing the degree of crosslinking and by decreasing the solution concentration. Ions with higher valence, smaller equivalent volume, and greater polarizability are preferred. The effect of the degree of crosslinking on the selectivity is illustrated by Myers and Boyd [1956].

Table 1. Characteristic values of cation exchange resins*

Parameter	Values	
	PK 228	Monosphere 650C
Resin type	Na^+ type	H^+ type
Appearance	Hard, Brown, Spherical beads	Hard, Black, Spherical beads
Bulk density (g/L)	780.0	800.9
Water retention capacity (%)	37-43	46-51
Capacity (meq/ml)	1.75	1.90
Diameter (mm)	0.40-0.55	0.65

*From the vendors.

The selectivity sequence for cations is: $Ag^+ > NH_4^+ > Na^+ > H^+$ [Zecchini, 1990]. This sequence shows that ammonium is preferred to sodium or hydrogen ions.

EXPERIMENTAL

The cation exchange process has been successfully used in removing ammonium from drinking water. In this study, both batch and continuous operation systems were used to show the ammonium removal characteristics of cation resins.

For the study, Na^+ -type cation exchange resin of PK228 provided by the Samyang Company, Limited, and H^+ -type of Dowex Monosphere 650C by the Dow Chemical Company, were used. The physicochemical properties of the resins are shown in Table 1. The resin was rinsed with high purity water and stored in plastic containers until they were used for the experimental runs. Ammonium chloride (NH_4Cl) of analytical reagent grade was the ammonium source in the experiments.

The batch system was used to investigate the effects of resin type, feed solution concentration, and temperature on the equilibrium data of ammonium. The system simply consists of a flask, a magnetic stirrer and bar, and a thermometer. Table 2 shows the experimental conditions of the system. Ion exchange resin and ammonium solution of the known weight and concentration, respectively, were added to the pure water in a 1 L flask container, and vigorously agitated. Water bath with a magnetic stirrer was used to maintain the constant temperature in the system. Water samples were collected periodically by hand using sample bottles. To avoid any leaching from the bottle itself, the samples were analyzed within at most 10 minutes using an ion chromatography (IC).

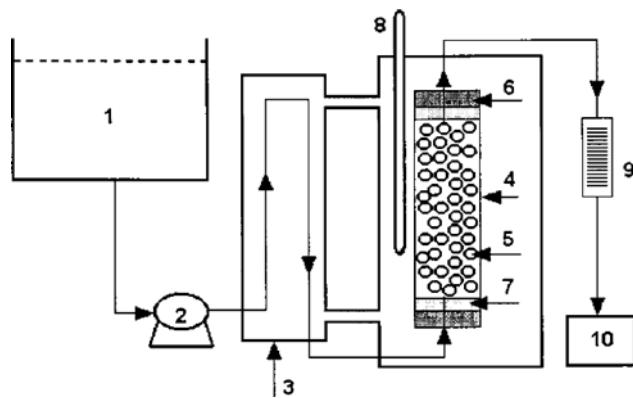
The continuous operation system was used to study the effects of resin type, temperature, volumetric flow rate, column diameter

Table 2. Experimental conditions of the batch reactor

Parameter	Values
Resin weight (g)	0.05
pH	5.0-7.0
Temperature (°C)	15, 25, 35
Feed concentration [NH_4^+ -N] (mg/L)	0.5, 1.0, 2.0
Agitation rate (rpm)	≥1000

Table 3. Experimental conditions of the column reactor

Parameter	Values
Feed concentration $[\text{NH}_4^+ \text{-N}]$ (mg/L)	0.5, 1.0, 2.0
Feed flow rate (L/hr)	3.6, 10.8
Temperature (°C)	20, 40, 50
Column diameter (cm)	0.5, 1.0
Resin depth (cm)	1.0-2.0
Resin weight (g)	0.5, 1.0

**Fig. 1. Schematic diagram of the continuous operation system.**

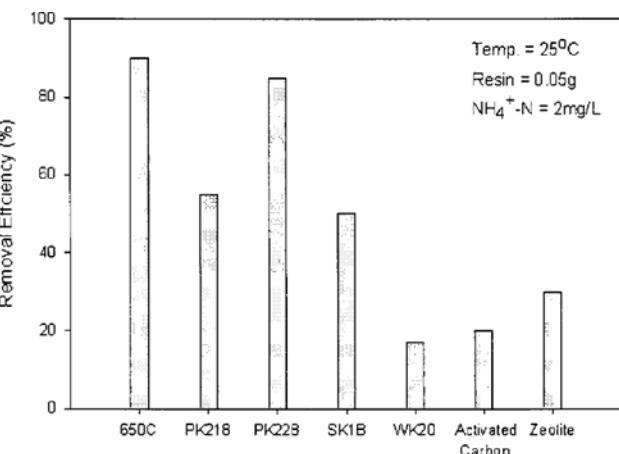
1. Feed storage	6. Silicone stopper
2. Tubing pump system	7. Glass wool
3. Water bath system	8. Thermometer
4. Glass column	9. Flow meter
5. Cation resin	10. Ion chromatography

and column depth on the breakthrough curves of ammonium. Table 3 shows the experimental conditions of the continuous system, and Fig. 1 shows the schematic diagram of the system. The experimental system is mainly consisted of a packed-bed column, its accessory for feeding, water bath, feed storage, and IC for measuring effluent concentrations. The ion-exchange column used for the experiment was made from Pyrex glass to see the resins loaded inside the column. Glass wool with stainless steel screens was used to support the resins and to ensure uniform flow distribution. Water bath was used to maintain the desired temperature inside the column. Feed solution was distributed from a 50 L Nalgene carboy to the column through a peristaltic pump. The pump drive was equipped with a speed controller for adjusting the flow rate to the design conditions. The feed solution was desired to simulate the water-purification system with the standard concentration of 0.5 mg $\text{NH}_4^+ \text{-N/L}$, and two and four times higher ammonium concentrations were also made to study the effect of feed solution concentration.

A Dionex IC (model DX-300) and Standard Methods [AWWA, 1992] were used to analyze the water samples for both batch and continuous operation systems. These two methods verified mutually the accuracy of the data.

RESULTS AND DISCUSSION

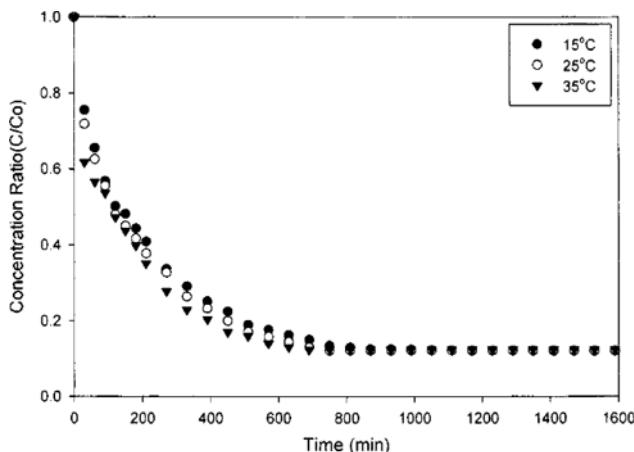
The experiments were performed using both batch and continuous operation systems under the various conditions. The results of water sample analysis were described as the ratio of the effluent

**Fig. 2. Comparison of ammonium removal efficiencies of activated carbon, natural zeolite, and cation resins.**

concentration (C) to the feed concentration (C_0) vs. run time or solution volume passed through the experimental column. The results from each experiment separately discussed the effects of the experimental variables such as resin type, temperature, feed concentration, flow rate and column diameter.

1. Batch Operation System

The results of the batch experiments were used to study the ion exchange reaction equilibrium between sodium or hydrogen in the resin phase and ammonium in the solution. Fig. 2 compares the performances of ion exchange resins with activated carbon, and zeolite, which are commercially available and known to be effective to remove ammonium in the solution. Especially, zeolite is naturally occurred clinoptilolite at Yongil area, Kyungpook, Korea and known to be selective for ammonium ion. In this figure, the ion exchange resins show better performance than activated carbon or zeolite, regardless of the resin type. The Samyang ion-exchange resin of PK228 and Dowex Monosphere resin of 650C show similar ammonium removal performance of more than 80%, while activated carbon and zeolite show less than 30%. This means that the ion exchange mechanism is more efficient than the adsorption

**Fig. 3. Effect of temperature on ammonium concentration profile in the batch system with $\text{NH}_4^+ \text{-N } 0.5 \text{ mg/L}$ and 0.05 g of PK228.**

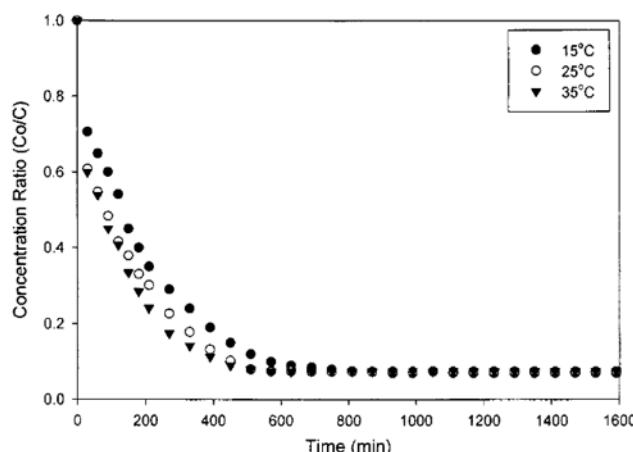


Fig. 4. Effect of temperature on ammonium concentration profile in the batch system with $\text{NH}_4^+ \text{-N } 0.5 \text{ mg/L}$ and 0.05 g of 650C.

mechanism of activated carbon or zeolite to remove ammonium. From this figure, it can be concluded that adsorption is not as a good method as ion exchange reaction to remove ammonium in the solution.

Figs. 3 and 4 show the effect of temperature on the equilibrium curve of ammonium with PK228 and 650C, respectively. Closer inspection of the figures indicates that as the temperature increases, the concentration profile of ammonium has sharper slope and reaches the equilibrium state faster. This trend well agrees with the model simulation of Divekar et al. [1987], who evaluated the temperature effect on the performance of the mixed-bed ion exchange at concentrations approaching the dissociation of water. With the increased temperature, the selectivity coefficient and solution viscosity decrease, while the ionization constant of water and ionic diffusion coefficient increase. They believed that the selectivity coefficient and viscosity change to a much smaller extent than the other parameters and that this change is overridden by the other two factors to improve the performance of the mixed-bed ion exchange. Figs. 3 and 4 also show that the equilibrium concentration increases as the temperature increases. This means that the opera-

tion capacity of the resin is not likely changed significantly by temperature. Therefore, it can be said that temperature affects the ion exchange rate more than the operation capacity of resin.

Fig. 5 contains the same data as Figs. 3 and 4 at 25°C and shows the difference between the performance of PK228 and 650C. The 650C resin, which is H^+ type, shows the sharper slope of the curve than the PK228 resin, which is Na^+ type. The slopes of the curves imply that the exchange rate of ammonium with hydrogen is higher than that with sodium, which means the selectivity of the 650C resin for ammonium over hydrogen is larger than that of PK228 for ammonium over sodium. The equilibrium concentration of 650C is lower than that of PK228 and this is because 650C has the bigger total capacity than PK228.

The selectivity coefficients of ammonium over sodium or hydrogen were obtained using the equilibrium data and given in Table 4. These data were applied to the equation of Kraus-Raridon [1959] based on Debye-Hückel theory. Using the selectivity coefficient at 15, 25, and 35°C , the equation for PK228 was correlated as;

$$\log K_{\text{Na}^+}^{\text{NH}_4^+} = 1.4102 - 0.1196 \log \left(\frac{T}{25} \right) + 0.0322 \left(1 - \frac{25}{T} \right) \text{ at } 0.5 \text{ mg/L} \quad (5)$$

and for 650C;

$$\log K_{\text{H}^+}^{\text{NH}_4^+} = 3.3923 - 0.3515 \log \left(\frac{T}{25} \right) \text{ at } 0.5 \text{ mg/L} + 0.1029 \left(1 - \frac{25}{T} \right) \text{ at } 0.5 \text{ mg/L} \quad (6)$$

Fig. 6 shows how the variation of initial ammonium concentra-

Table 4. Selectivity coefficients of cation exchange resins with $\text{NH}_4^+ \text{-N } 0.5 \text{ mg/L}$

Temperature ($^\circ\text{C}$)	Selectivity coefficient (K)	
	PK 228 ($\text{Na}^+ \text{-NH}_4^+$)	Monosphere 650C ($\text{H}^+ \text{-NH}_4^+$)
15	1.43	3.47
25	1.41	3.39
35	1.38	3.22

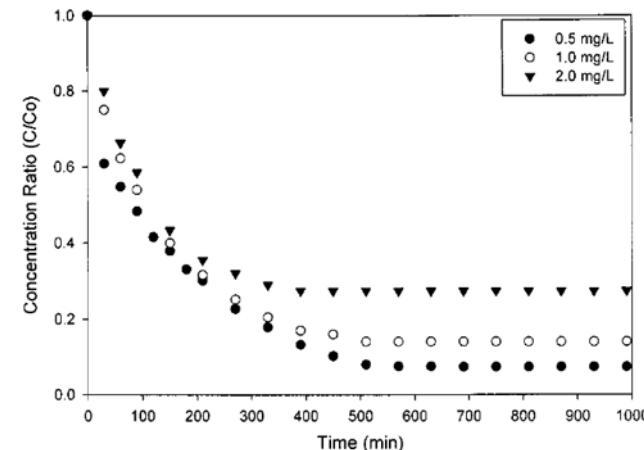


Fig. 5. Effect of resin type on ammonium concentration profile in the batch system with $\text{NH}_4^+ \text{-N } 0.5 \text{ mg/L}$ at 25°C .

Fig. 6. Effect of initial feed concentration on ammonium concentration profile in the batch system at 25°C (650C).

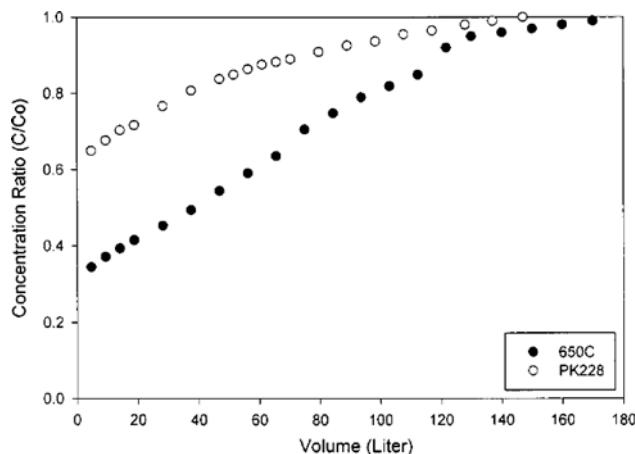


Fig. 7. Ammonium breakthrough curves with PK228 and 650C at 30 °C (NH_4^+ -N 0.5 mg/L).

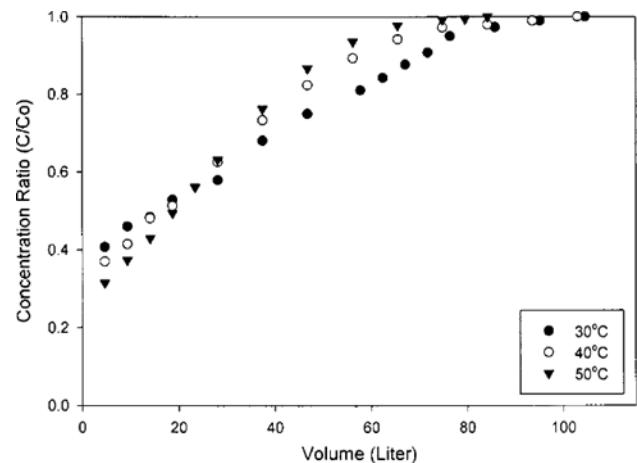


Fig. 8. Effect of temperature on ammonium breakthrough curves with NH_4^+ -N 1.0 mg/L and 0.5 g of 650C.

tion affects the ion-exchange performance. The figure shows the higher equilibrium concentration and the worse performance as the initial concentration increases. Based on the figure, it is possible to suggest that the low initial concentration needs longer reaction time to reach equilibrium.

2. Continuous Operation System

The results of the continuous column experiments are given in Figs. 7 to 12, from which one can see that the different levels of initial leakage of ammonium appear in the effluent of the exchange process. The initial leakages might be due to the insufficient contact time between the solution and the resins, which result from the high flow rate and the relatively short bed depth. These conditions were selected for the reasonable duration of the experimental runs.

The performances of two cation exchange resins, 650C and PK228, for the ammonium removal were compared in Fig. 7. It is observed in this figure that 650C shows the lower level of the initial leakage and much longer time to reach the equilibrium state than PK228. This can be explained by the differences in the selectivity coefficients and the capacities of those resins. The selectivity coefficient directly relates to the ability of the resin to remove an ion over another. Considering the results obtained through the batch experiments (see Fig. 5), it could be expected that 650C showed much better performance for the ammonium removal in the continuous system than PK228, resulting in the lower initial leakage. The capacity of the resin is defined as the net number of ionic sites utilized in a given weight of the resin in a given process. As shown in Table 1, the capacity of PK228 is lower than that of 650C, and as a result, PK228 is exhausted and reaches the equilibrium state faster than 650C. It is obvious from the figure that the selectivity coefficient and the capacity of resin influence the shape of the breakthrough curve significantly.

Fig. 8 shows the effect of temperature on the effluent concentration profile of ammonium. With the higher temperature, the breakthrough curve shows the lower initial leakage and the steeper slope. This results in two distinct zones in the curve. In one zone, the higher temperature gives better removal performance; and in the other, the reverse effect is noticed. Thus, it can be concluded that temperature enhances the exchange rate below the resin capacity and causes faster exhaustion to the equilibrium state.

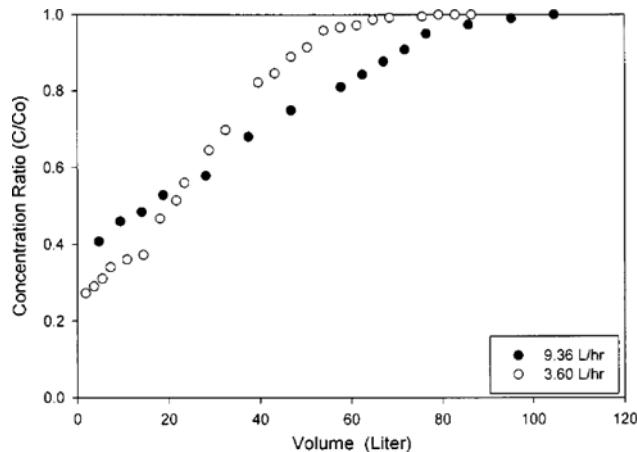


Fig. 9. Effect of volumetric flow rate on ammonium breakthrough curves with NH_4^+ -N 1.0 mg/L at 30 °C (650C).

The effect of the volumetric flow rate on the breakthrough curve of ammonium is shown in Fig. 9. The figure indicates that the initial leakage increases and the slope becomes broader as the flow rate increases. This might be because the contact time between the solution and the resin decreases with the increased flow rate. An interesting point observed in the figure is that the slope of breakthrough curves is steep compared with the results of Yoon et al. [1995], who studied the effect of cation to anion resin ratio on the mixed-bed ion exchange performance at ultralow NaCl solution concentration. They used the Ambersep 200H of Rohm and Haas Company, especially manufactured for the mixed-bed ion exchange. The resin selectivity coefficient of 200H was 2.0-2.5 for Na^+ - H^+ exchange, smaller than the resin used in this experiment. The higher selectivity coefficient make the steeper slope of the breakthrough curve. The flow rate appears not to affect the operation capacity of the resin significantly.

Figs. 10 and 11 show the effect of feed concentration on the ammonium breakthrough curve with the different weight of the resin. As expected, the curves in both figures indicate the steeper slope and the faster breakthrough when the concentration increases. While the initial leakage also increases with the feed concentration when

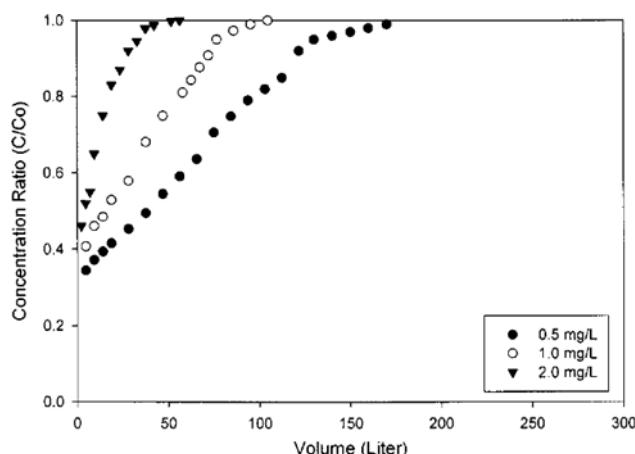


Fig. 10. Effect of feed concentration on ammonium breakthrough curves with 0.5 g of 650C at 30 °C.

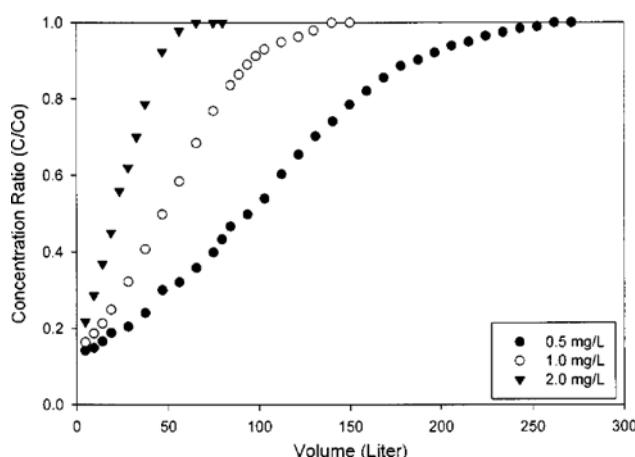


Fig. 11. Effect of feed concentration on ammonium breakthrough curves with 1.0 g of 650C at 30 °C.

the weight of the resin is 0.5 g in Fig. 10, it is almost constant regardless of the concentration with 1.0 g of the resin in Fig. 11. These weights correspond to 1 cm and 2 cm of bed depth of the continuous column, respectively. The ratio of the treated solution volume, which is the left-hand side area of the breakthrough curves, agrees with the ratio of the resin weights. In Fig. 11, the treated solution volumes are 94.5, 48.4, and 23.2 L for feed concentrations of 0.5, 1.0, and 2.0 mg NH_4^+ -N/L, respectively. Therefore, it can be said that the operation capacity of the resin is not likely to be changed by the variation of the initial feed concentration.

The initial leakage depends on the bed depth for the favorable equilibrium. The limiting slope of the breakthrough curve is reached in a shorter height of bed for a favorable equilibrium, while an unfavorable equilibrium gives a more gradual and broad breakthrough curve [Vermeulen and Hiester, 1959]. As a function of bed height, cation breakthrough curves in Figs. 10 and 11 show different levels of initial leakage. This is because of the relatively short bed of the system and thus, insufficient contact between the solution and the resin for the relatively favorable equilibrium. The results in this experiment are coincided with the results of Yoon et al. [1999], who studied the temperature effects on the mixed-bed ion exchange per-

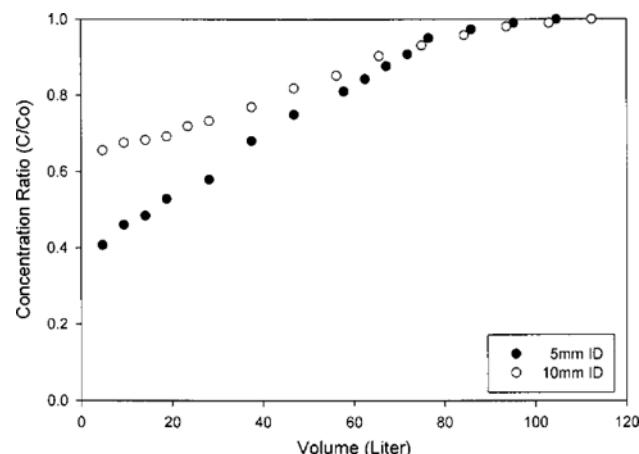


Fig. 12. Effect of column diameter on ammonium breakthrough curves with NH_4^+ -N 1.0 mg/L at 30 °C (650C).

formance at ultralow NaCl solution concentration. They used the Dowex Monosphere 650C with the selectivity coefficient of 1.13 manufactured by Dow Chemical Company, and showed the broader cation breakthrough curve than anion breakthrough curve and different levels of breakthrough as a function of cation to anion resin ratio.

Column diameter with a constant flow rate influences the performance of continuous column, especially at the early stage of the process. Fig. 12 shows the effect of column diameter on the ammonium breakthrough curve. Serious increase of initial leakage for 10 mm inside diameter might be due to the short bed. The ratio of 10 to 5 mm inside diameter corresponds to the bed height ratio of 4 to 1. The effect of bed height was described previously in Figs. 10 and 11.

CONCLUSIONS

The effects of the system parameters, such as resin type, feed concentration, temperature, flow rate, and column diameter, on the performance of an ion exchange unit for the ammonium removal were experimentally evaluated. Through both batch and continuous column experiments, the following conclusions are drawn:

1. The selectivity coefficient and the total capacity of resin directly affect the ability of the resin to remove ammonium. The H^+ type cation resin shows the better results for the ammonium removal than the Na^+ type resin. This is because the selectivity of the former resin for ammonium over hydrogen is bigger than that of the latter for ammonium over sodium. The equilibrium concentration of ammonium decreases as the resin capacity increases.
2. Temperature strongly affects positively the performance of ion exchange for the ammonium removal below the resin capacity. As temperature increases, the exchange rate increases and the equilibrium is more favorable.
3. As the initial feed concentration increases, the equilibrium concentration decreases and the breakthrough time is shorter. However, the total amount of ammonium removed is not changed.
4. With the increased volumetric flow rate and column diameter, the contact time between the solution and the resin decreases, re-

sulting in the increased initial leakage and the steeper slope of the curve. The operation capacity of the resin is not appeared to be affected significantly by these parameters.

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