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Investigation of Selectivity and Kinetic Behavior of Strong-Base Ion Exchange Resin Purolite A 520E for Nitrate Removal from Aqueous Solution

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Abstract: The aim of this work is to present experimental results on the removal of nitrate by nitrate selective ion exchange resin, Purolite A 520E. The resin particle size, nitrate concentration, temperature, and stirring speed were investigated as experimental parameters and the optimum conditions for nitrate removal were determined. Nitrate removal by strong base anion exchange resin Purolite A 520E was carried out with the batch method in the presence of chloride and sulfate ions. The existence of a high concentration of competing ions in a solution resulted in a reduction of nitrate removal. Nitrate removal ratios decreased from 98% to 85% and 88%, respectively, in the presence of chloride and sulfate ions when the chloride and sulfate ratios were increased in solution. The process kinetics were predicted by using Homogenous Diffusion Models. It was seen that about 98% of nitrate in the aqueous solution could be removed using optimum conditions.

Keywords: Nitrate removal, ion exchange, strong-base ion exchange resin, Purolite A 520E, nitrate selective resin, selectivity, chloride, sulfate

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INTRODUCTION

Nitrate contamination of ground and surface water is an environmental problem in worldwide, especially in the south of Europe. The main causes of nitrate pollution are the excessive use of fertilizers in intensive agriculture, the irrigation with domestic wastewater and change in land-use patterns (1, 2). Possible health consequences of nitrate ingestion include methemoglobinemia, the blue-baby syndrome in infants under six months of age. In the stomach, nitrate is converted to nitrite. The nitrite in turn oxidizes hemoglobin to methemoglobin which does not carry oxygen to cell tissues (1). In addition, the reaction between nitrite and secondary or tertiary amine can result in the formation of *N*-nitroso compounds, some of which are known to be carcinogenic, teratogenic, and mutagenic. These include hypertension, increased infant mortality, central nervous system birth defects, diabetes, and changes of the immune system (1–4). As a result of the health consideration, the maximum contaminant limit for nitrate in drinking water has been set at 45 mg-NO_3^-/L by EPA. A similar guideline of 50 mg-NO_3^-/L has been set by the WHO, while the European Community (EC) standards allow a maximum admissible concentration of 50 mg-NO_3^-/L and a guide level of 25 mg-NO_3^-/L (5, 6).

Selection of the suitable technology depends on the efficiency to selectively remove nitrate and the cost associated in treating the wastes generated. There are physicochemical and biological processes available for the removal of nitrate. The processes include ion exchange (7–9), biological denitrification (6, 10), electrodialysis (11–13), and reverse osmosis (14). The challenge between different technologies depends on desired water quality, plant capacity, process automation, or access to manpower of suitable skills.

A biological denitrification method using degradation of microorganism offers the possibility of a very specific and selective reduction of nitrate to nitrogen. However, there are some limitations due to contamination of the drinking water with germs and metabolic substances. Because of this, an extensive reconditioning of the drinking water by filtration and germicidal treatment is necessary (15). RO and ED have better economics, larger automation possibilities, lower level in feed and process parameters control and no need for extensive post treatment are advantages (11, 14). However, the utility of these processes has been limited as they are relatively expensive and merely displace nitrate into concentrated waste brine that may pose a disposal problem (6). The advantage of a catalytic reduction process is the rapid removal of nitrate from water (16, 17). The disadvantage of this process is also its high capital. The ion exchange process seems to be the most suitable for small water suppliers contaminated by nitrate because of its simplicity, effectiveness, selectivity, recovery, and relatively low cost (7, 8). At very low feed solution salt concentrations, ion exchange is the most economical process. But its costs increase sharply with feed solution salinity. At about 500 mg/L,

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electrodialysis becomes the more economical process, while at around 5000 mg/L, reverse osmosis is the less costly process. At very high feed solution salt concentrations, in excess of 100,000 mg/L, multistage flash evaporation becomes the most economical process (Bungay et al. 1983). The disadvantage of the ion exchange is the necessity for the regeneration of chemicals.

Several research projects were developed on the denitrification of drinking water by anion exchange resins (18, 19); on macroporous resins, type IMAC HP-555 and type Dowex SBRP (20). Resins whose selectivity is better for nitrates than for sulfates (Duolite A 196, Amberlite IRA 996) were developed in 1985 (21) with the specific resin Purolite A 520E.

However, and with regard to their contamination by nitrates, the underground waters are often charged by sulfates. These latter, whose valence is higher than nitrates, have the property to be fixed preferentially by the resins, consequently reducing the cycle of production of the exchanger. For this reason, resins whose selectivity is better for nitrates than for sulfates (Duolite A 196, Amberlite IRA 996) were developed in 1985 (21) with the specific resin Purolite A 520E (7). Both the benzyltrimethyl ammonium type and the benzyldimethylethanol ammonium type resins have a significantly stronger affinity for nitrate over either chloride or bicarbonate but a lower affinity for nitrate as compared to sulfate at ionic concentrations typical of potable waters. This lower affinity for nitrate over sulfate leads to two difficulties. One is a limited useful capacity as the sulfate to nitrate ratio. The second difficulty is commonly referred to as “Nitrate Dumping.” The need to find resins selective for nitrate over sulfate was the subject of EPA funded studies including work done by Clifford and Weber 1977 (22). Work done by G. Guter 1984 demonstrated that the affinity of strong base anion exchangers for nitrate could be modified by increasing the size of the carbon side chains surrounding the nitrogen in the strong base site (23).

In this paper, nitrate removal by ion exchange employing strong base nitrate selective anion exchange resin, Purolite A 520E is presented. The aim of this study is to investigate the effect of various parameters such as initial nitrate concentration, resin particle size, temperature, and the effect of the presence of other ions on nitrate removal. The selectivity and kinetic behavior of Purolite A 520E resin was studied by the batch method.

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EXPERIMENTAL

Materials

Nitrate selective strong base anion exchange resin Purolite A 520E (Cl^- form) was kindly sent by Purolite Int. Ltd. This resin has a macroporous structure and was produced as nitrate selective resin. The chemical and physical properties of resin are given in Table 1. Resins are used as provided by the company.

Inorganic chemicals were supplied by Merck and Riedel-de Haën as analytical-grade reagents. Model nitrate solutions were prepared by dissolving $NaNO_3$ in deionized water. The solutions of sulfate and chloride were prepared through the dissolution of Na_2SO_4 and NaCl respectively, in deionized water.

Batch Mode Sorption

The effects of various parameters such as initial nitrate concentration, temperature, stirring speed, resin particle size, the ionic form of the resin, and the effect of the other ions were investigated.

The kinetic behavior of Purolite A 520E resin was investigated using 3.0 g of dry resin in order to get a measure of the relative performance of the resin. Nitrate concentration in the solution was monitored with time. The experimental parameters were 200, 250, 300 rpm for stirring speed, 10, 50, 100, 150 mg-NO_3^-/L for nitrate concentration, 288, 293, 298, 308 K for temperature, 0.500–0.710 and 0.355–0.500 mm for resin particle size range. Kinetic tests were carried out in a 1 L of three–necked round bottom glass flask immersed in a water bath at the desired temperature. A 1 L of nitrate solution of a given concentration was put into the flask. The system was stirred by a mechanical stirrer at the desired stirring speed. The nitrate concentration was monitored for 5, 10, 15, 20, 30, 45, 60, 90, 120, 150, and 1440 min periods by taking 3 mL of supernatant from the solution.

Analyses

The analyses of nitrate, chloride, and sulfate ions were carried out using a Shimadzu model ion chromatography equipment (Model LC 10 A*i*).

Table 1. Physico-chemical properties of resin Purolite A 520E

Skeleton	Polystyrene crosslinked with DVB of the macroporous type
Functional groupings	Triethylammonium
Physical aspect	Opaque balls, beige color
Granulometry	0.3–1.2 mm
Ionic form	$C1^-$
Total exchange capacity	0.9 meq/mL
Humidity	45–52%
Limit of temperature	100°C
Limits of pH	0–14
Real density	1.06

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RESULTS AND DISCUSSIONS

Effect of Stirring Speeds

The results given in Fig. 1 show that the resin reached equilibrium faster at a larger stirring speed. Film-diffusion depends on the degree of agitation (24). The film thickness on the particle surface is decreased with each agitation. Thus diffusion of nitrate into the resin increases with the agitation rate. The time for equilibrium attainment is not much affected by the stirring speed. The bigger difference is observed at that nitrate removal level which is smaller than 90%.

Effect of the Concentrations of Nitrate

The initial concentration of the nitrate in the solution affects the film-diffusion. Concentration gradients exist only in the films. This shows that the flux is proportional to the solution concentration and to the inter-diffusion coefficient in the film. The diffusion increases with concentration (24). Figure 2 shows that the value of C/C_o increased with increasing initial nitrate concentration in the solution. This is the case only for that nitrate removal which is larger than 90% ($C/C_o < 0.1$) at the nearly equilibrium state. The initial nitrate concentration affects the equilibrium state. However, there is no observable effect within the nitrate uptake range where C/C_o is larger than 0.2.

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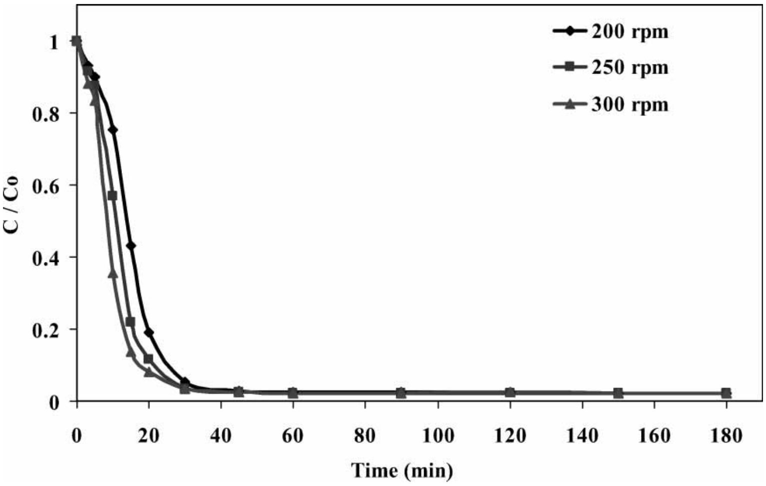

Figure 1. Effect of stirring speeds on removal of nitrate (Condition: 3.0 g of dry resin (0.500–0.710 mm); 100 mg-NO_3^-/L; stirring at 25°C).

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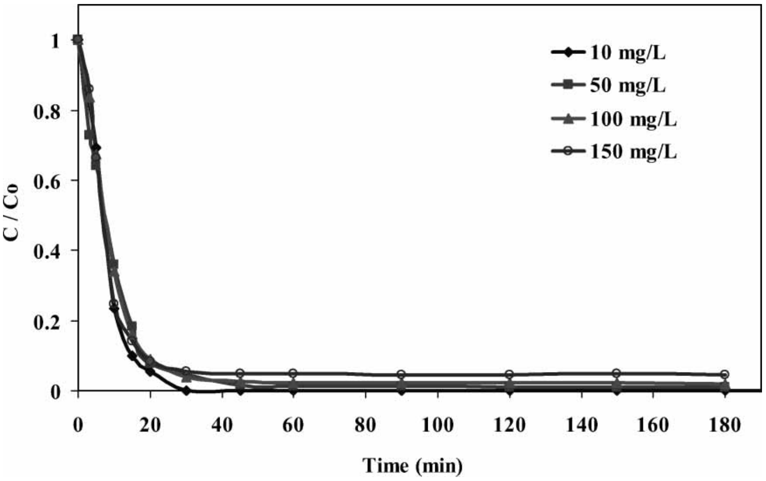

Figure 2. Effect of nitrate concentration on kinetic behavior of Purolite A 520E (Condition: 3.0 g of dry resin (0.500–0.710 mm)/L; stirring at 25°C, 250 rpm).

Effect of Resin Particle Size

The effect of the resin particle size was investigated with resins at particle size ranges of 0.500–0.710 and 0.355–0.500 mm. In the experiments, a nitrate concentration of 100 mg NO_3^-/L, temperature at 25°C and 3.0 g-dry resin were kept constant. The results are given in Fig. 3. The particle size influenced

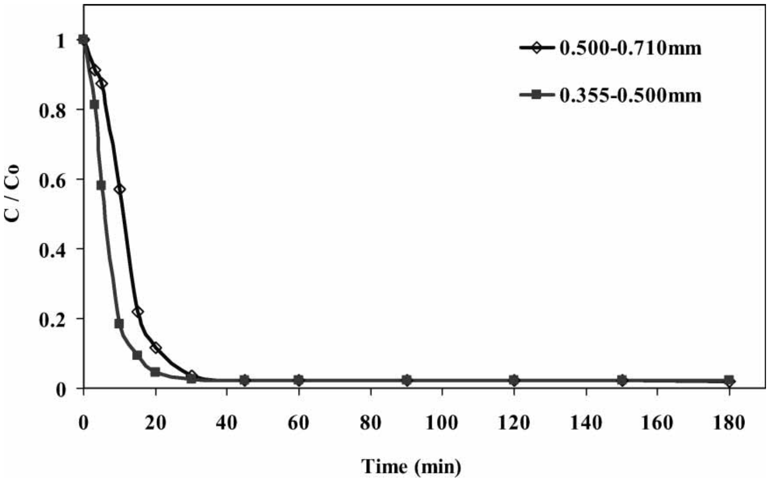

Figure 3. Effect of resin particle size on kinetic behavior of Purolite A 520E (Condition: 3.0 g of dry resin/L; 100 mg-NO_3^-/L; stirring at 25°C, 250 rpm).

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the time required to establish the equilibrium. A decrease in the particle size thus shortens the time required for equilibration. The particle size of the resin also affects internal diffusion. In case of small resin particle size range (0.355–0.500 mm), the resin Purolite A 520E reached equilibrium with 95% of removal in about 20 min., while the resin with bigger particle size required 30 min. to reach 95% of the removal.

Effect of Temperature

The effect of temperature on nitrate removal was studied at 288, 293, 298, and 308 K. The experiments were made by using 3.0 g dry resin immersed into 1 L of 100 mg-NO_3^-/L-$NaNO_3$ solution using a mechanical stirrer with 250 rpm. The results are shown in Fig. 4. Nitrate removal by Purolite A 520E resin increased with increasing the temperature. This result supports the fact that the reaction rate is controlled by the chemical reaction. The temperature generally affects the diffusion rate. Thus both mechanisms could be considered as the rate determining steps.

Effect of Chloride and Sulfate Ions on Nitrate Removal

The removal of nitrate by Purolite A 520E resin was studied in the presence of chloride and sulfate ions with batch method. The experiments were carried out with nitrate (50; 100; 150 mg-NO_3^-/L) paired only with either chloride or sulfate ions and mixtures containing both chloride and sulfate ions along

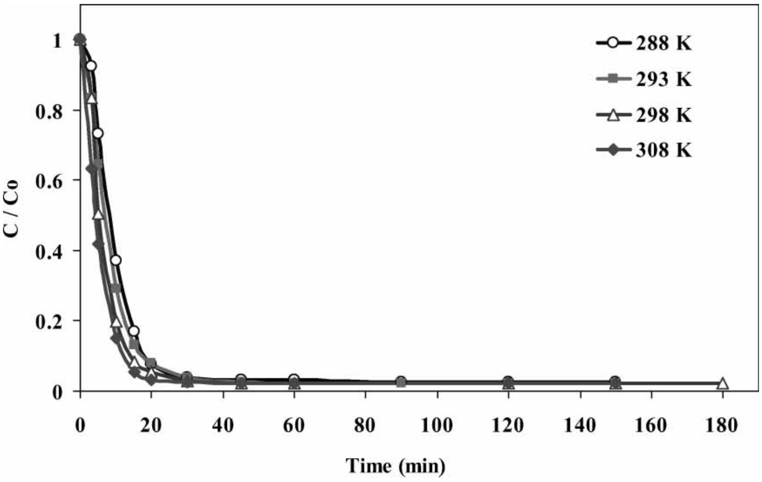

Figure 4. Effect of temperature on the removal of nitrate (Condition: 3.0 g of dry resin (0.500–0.710 mm)/L; 100 mg-NO_3^-/L; stirring at 250 rpm).

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Table 2. Equivalent concentration ratios of ionic species for nitrate solutions

Equivalent concentration ratio	Equivalent concentration values		
	For 50 mg-NO_3^-/L	For 100 mg-NO_3^-/L	For 150 mg-NO_3^-/L
NO_3^- : Cl^-	1:2	1:2	1:2
	1:5	1:5	1:5
	1:10	1:10	1:10
NO_3^- : SO_4^{2-}	1:1	1:1	1:1
	1:5	1:5	1:5
	1:10	1:10	1:10
NO_3^- : SO_4^{2-} : Cl^-	1:10:10	1:10:10	1:10:10

with nitrate ion. Concentrations of chloride and sulfate are one, five, or ten times of nitrate concentrations as equivalent concentration. Chloride concentration is also twice of nitrate concentrations as equivalent concentration. These concentration ratios are summarized in Table 2.

Figures 5–7 show variation of C/C_o values versus time for nitrate removal in the presence of chloride and sulfate ions. Three concentration values of nitrate have similar results. The excess of chloride in solution hindered the interaction between nitrate ions and ions on the resin. The

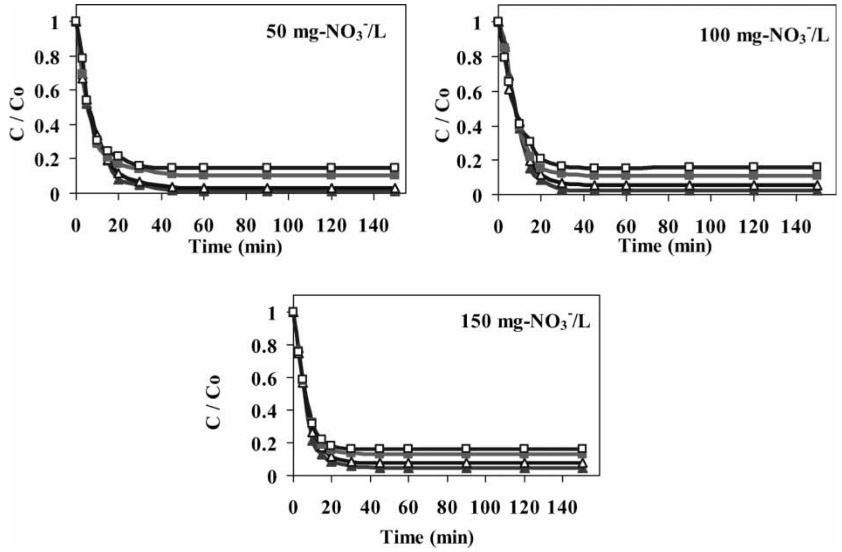

Figure 5. Effect of chloride at various concentration ratios on nitrate removal (Condition: 3.0 g of dry resin (0.500–0.710 mm)/L; stirring at 25°C, 250 rpm) (NO_3^- : Cl^-) = ▲ (1:1), △ (1:2), ■ (1:5), □ (1:10).

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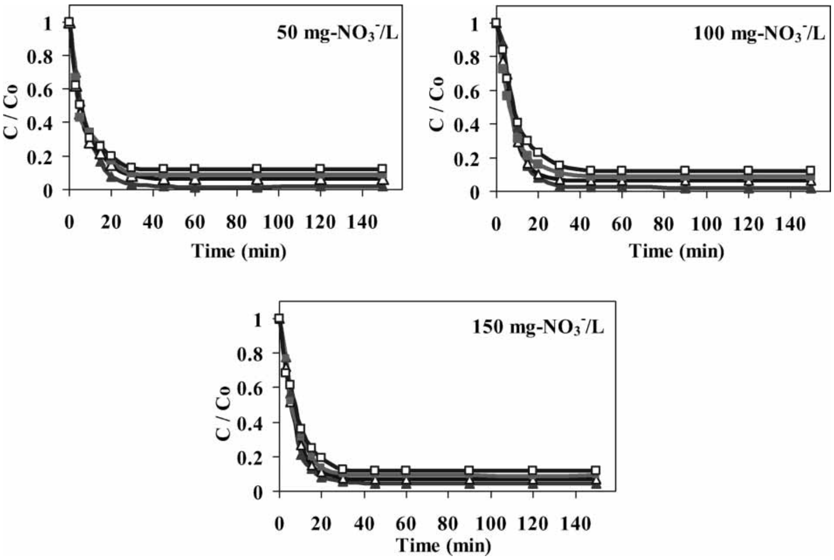

Figure 6. Effect of sulfate at various concentration ratios on nitrate removal (Condition: 3.0 g of dry resin (0.500–0.710 mm)/L; stirring at 25°C, 250 rpm) (NO_3^- : Cl^-) = ▲ (1:1), △ (1:2), ■ (1:5), □ (1:10).

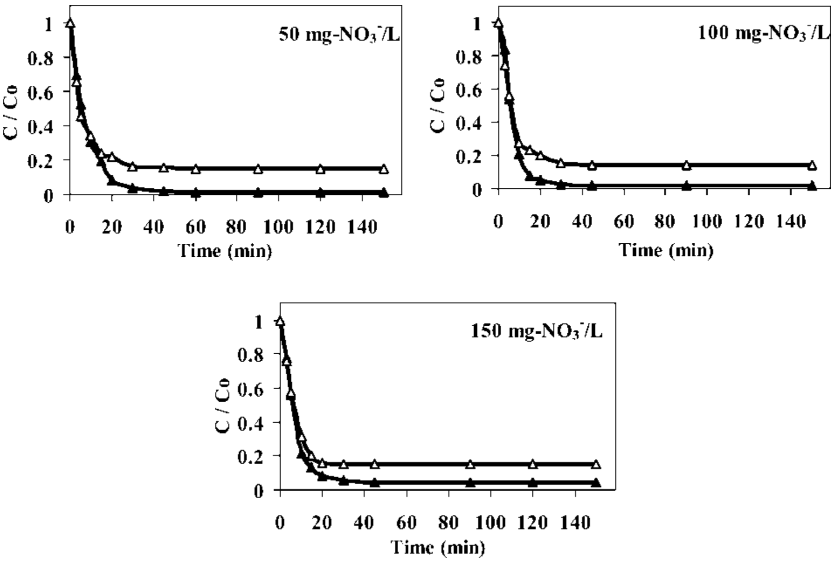

Figure 7. Effect of sulfate and chloride at various concentration ratios on nitrate removal (Condition: 3.0 g of dry resin (0.500–0.710 mm)/L; stirring at 25°C, 250 rpm) ▲ NO_3^- alone (NO_3^- : SO_4^{2-} Cl^-) = △ (1:10:10).

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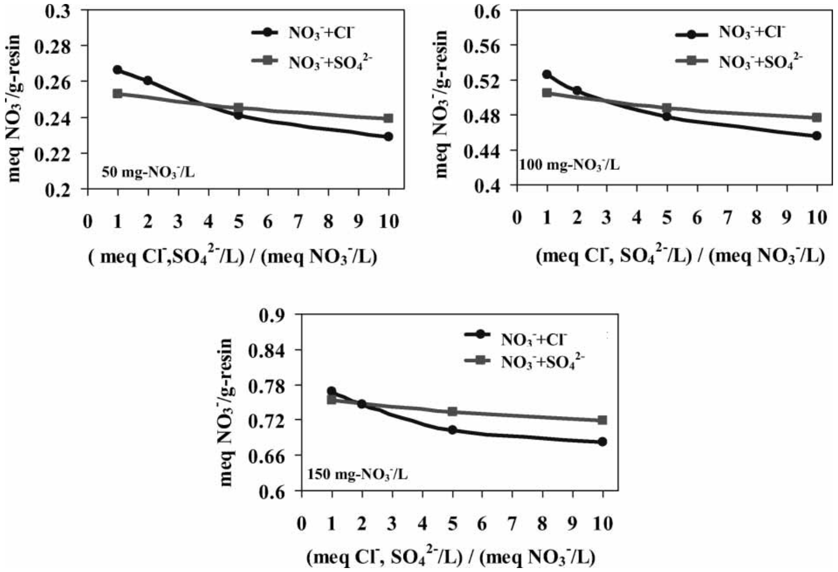

Figure 8. Resin capacity on nitrate removal in the presence of chloride and sulfate (Condition: 3.0 g of dry resin (0.500–0.710 mm)/L; stirring at 25°C, 250 rpm).

sorption of nitrate by the resin was affected by the existence of sulfate ions. The presence of both chloride and sulfate ions together resulted in a remarkable influence on the uptake of nitrate.

Figures 8 and 9 indicate the resin capacity for the equilibrium state versus concentration of chloride and sulfate ions. As seen in these figures, the amount of nitrate uptake per unit weight of resin decreased in higher concentration ratios of chloride and sulfate ions. There is a clear difference between the variation of resin capacity in the presence of sulfate and chloride ions (Fig. 8). Chloride ions caused a significant shift in the uptake of nitrate. The chloride ions compete with nitrate ions since the resin tried to have a chloride form again in the presence of excess chloride ion in the solution. The decrease in nitrate uptake of the resin was more remarkable in the presence of chloride than that in the presence of sulfate ions. While chloride ions on the resin phase are removed by the sulfate ions in the solution due to the electrostatic interaction, nitrate ions will reach onto the resin and take position of chloride ions due to the high selectivity of resin to nitrate ions.

As seen in Fig. 9, the resin capacity for nitrate removal decreased by about 13.9%, 12.4%, and 10.4% for the solutions containing 50, 100, and 150 mg-NO$_3^-$/L, respectively, in the presence of both chloride and sulfate ions (NO$_3^-$:SO$_4^{2-}$:Cl$^-$ = 1:10:10). It can be observed that the presence of sulfate and chloride ions significantly influenced the uptake of nitrate by Purolite A 520E.

To compare the capacity decrease for Purolite A 520E, the strong base anion exchange resin, Amberlite IRA 904, having the trimethylammonium

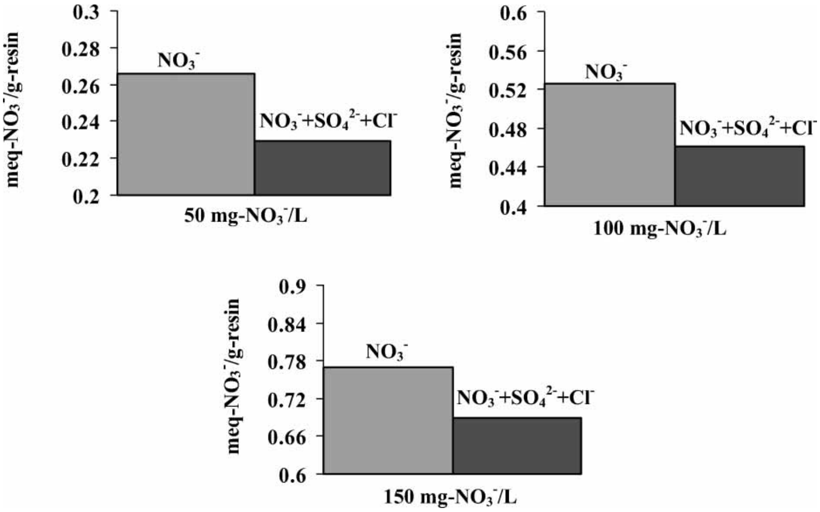

Figure 9. Resin capacity on nitrate removal by Purolite A 520E resin in the presence of both chloride and sulfate (Condition: 3.0 g of dry resin (0.500–0.710 mm)/L; stirring at 25°C, 250 rpm).

group was used for removing the nitrate from a solution containing only 100 mg-NO_3^-/L and from a solution containing 100 mg-NO_3^-/L along with both chloride and sulfate ions at the same conditions (Fig. 10). In a solution containing only 100 mg-NO_3^-/L, 98% of the nitrate was removed by Purolite A 520E resin while 96% of the nitrate by the Amberlite IRA 904 resin. The decreasing capacity of the Amberlite IRA 904 resin is larger (23%) than that of the Purolite A 520E resin in the presence of both chloride and sulfate ions (NO_3^-:SO_4^{2-}:Cl^- = 1:10:10). Purolite A 520E exhibited a higher percent nitrate removal (86%) than Amberlite IRA 904 resin (74%) in the presence of both chloride and sulfate ions. Because Purolite A 520E resin is functionalized with triethylamine exchange sites (25, 26). It was reported that this resin represents a class of materials with increasing selectivity for nitrate over sulfate as the carbon side chain length increases. Selectivity for nitrate increases from the trimethyl to tributhyl by two log units (25). As the chain length increases, the volume capacity of the resins decreases. The groups larger than butyl are not recommended for nitrate removal. (27).

Kinetic Study

Kinetic tests were performed by using 3.0 g of dry Purolite A 520E resin (0.500–0.710 mm and 0.355–0.500 mm) immersed into a 1 L of $NaNO_3$

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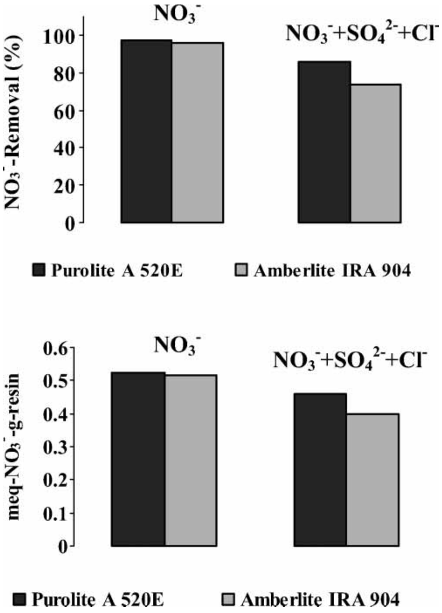

Figure 10. Resin capacity on nitrate removal by Purolite A 520E and Amberlite IRA 904 resin in the presence of both chloride and sulfate (Condition: 3.0 g of dry resin (0.500–0.710 mm)/L; stirring at 25°C, 250 rpm).

solution (100 mg-NO_3^-/L). The nitrate concentration was monitored for 5, 10, 15, 20, 30, 45, 60, 90, 120, 150, and 1440 min periods. Fig. 11 illustrates the relative concentration decrease of nitrate versus time. The removal of nitrate increased with time and reached the equilibrium in 45 min with 97.6% of nitrate removal.

The ion exchange between the counter ion in the solution and the exchangeable ion on the resin is well described by a heterogeneous process. The models for process dynamics cover both the diffusional steps and the exchange reaction on the active sites (28). Kinetic models developed for spherical particles in different cases of the rate-determining steps are given in Table 3. X is the fractional attainment of equilibrium or extent of resin conversion. X value was calculated according to equilibrium time after 24 h.

$X = q_t/q_e$ where q_e and q_t (both in mg/g resin) are the amount of nitrate adsorbed per unit mass of the resin at equilibrium and time t, respectively.

These models were applied to fit the kinetic data of the resin Purolite A 520E (0.500–0.710 mm; 0.355–0.500 mm). Since the resin Purolite A 520E reached equilibrium after about 45 min, the data obtained at contact times longer than 45 min were neglected in this application of kinetic models. Slopes and linear correlation coefficients for five models of kinetic for Purolite A 520E are given in Table 4.

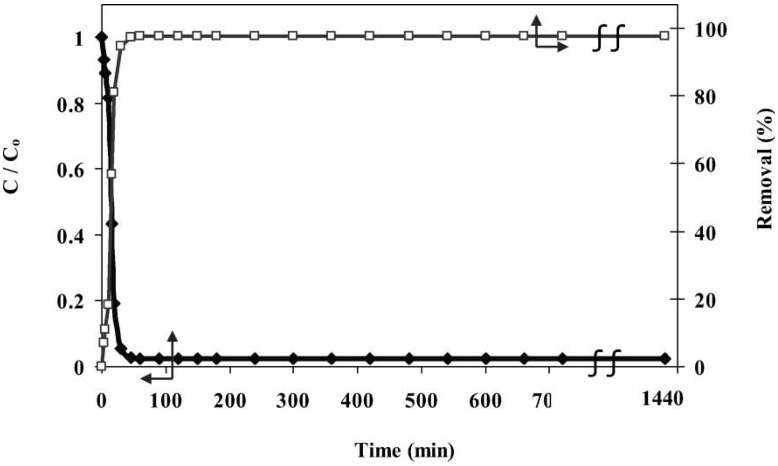

Figure 11. Removal of nitrate versus time (Condition: 3.0 g of dry Purolite A 520E resin (0.500–0.710 mm)/L;100 mg-NO_3^- /L; stirring at 25°C, 250 rpm).

Linear correlation coefficients help to decide the rate determining step. According to the infinite solution volume models (ISV), it can be said that kinetic behavior of resin Purolite A 520E is film diffusion controlled. As seen in Table 4, the maximum of the correlation coefficients for unreacted core models (UCM) shows that the reaction is chemical reaction controlled. These results are the same for two different resin particle size ranges.

CONCLUSIONS

In this study, the removal of nitrate from aqueous solutions was investigated by using nitrate selective anion exchange resin, Purolite A 520E. The removal

Table 3. Diffusional and reaction models

Method	Equation	Rate controlling step
ISV	$-\ln(1 - X) = kt$, where $k = Dr\pi^2/r_o^2$	Film diffusion
ISV	$-\ln(1 - X^2) = k_{li}t$, where $k = 3DC/r_o\delta Cr$	Particle diffusion
UCM	$X = (3C_{Ao}K_{MA}/ar_oC_{So})t$	Liquid film
UCM	$3\text{-}3(1 - X)^{2/3} - 2X = (6D_{CR}C_{Ao}/\ ar_o^2 C_{So})t$	Reacted layer
UCM	$1 - (1 - X)^{1/3} = (k_sC_{Ao}/\ r_o)t$	Chemical reaction

X, the fractional attainment of equilibrium or extent of resin conversion; k, rate constant (L/s); t, time (s); Dr, diffusion coefficient in solid phase (m^2/s); r_o, average particle radius (mm).

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Table 4. Evaluation of kinetic models for Purolite A 520E (0.500–0.710 mm)

	0.500–0.710 mm		0.355–0.500 mm	
Method	Slope	Linear correlation, r^2	Slope	Linear correlation, r^2
$-\ln(1 - X)$	0.1721	0.9998	0.1794	0.9978
$-\ln(1 - X^2)$	0.1502	0.9897	0.1370	0.9663
X	0.0455	0.8143	0.0585	0.8246
$3\text{-}3(1 - X)^{2/3} - 2X$	0.0346	0.9405	0.0380	0.9654
$1 - (1 - X)^{1/3}$	0.0376	0.9495	0.0401	0.9916

of nitrate from the aqueous solution depends on stirring speed, initial nitrate concentration, resin particle size, and temperature. In the presence of chloride and sulfate ions, uptake of nitrate by the resin decreased. The amount of decrease for Purolite A 520E resin having triethylamonnium functional groups was smaller than the Amberlite IRA 904 resin. According to infinite solution volume models (ISV), the adsorption rate is controlled by the film diffusion process while the maximum of the correlation coefficients for unreacted core models (UCM) shows that the reaction rate is chemical reaction controlled.

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