

REMOVAL OF NITRATES FROM WATER IN THE PRESENCE OF COMPETITORS ANIONS USING PUROLITE RESINS

BY

ANCA CEICĂ*, LAURA BULGARIU**, LILIANA LAZĂR*,
IGOR CREȚESCU** and ION BALASANIAN*

Abstract. In this paper was studied the influence of competitors anions, chloride and sulphate ions in the nitrates retention process from aqueous solutions, using three anion exchange resin: two non-selective anion exchange resin Purolite A-100 and Purolite A-847 and a selective resin, specially designed for the removal of nitrates from waters, Purolite A-520E. The resins performance was determined for aqueous solutions containing 50 mg NO_3^-/L and 100 mg NO_3^-/L nitrate concentrations, using batch mode study. To determine the influence of sulphate ions on the nitrate retention, the sulphate concentrations were increased gradually, in the range of 20 – 90 mg $\text{SO}_4^{2-}/\text{L}$, and for the influence of chloride ions their concentration was 19 – 115 mg Cl^-/L . For all three resins studied was observed the negative influence of individual competing anions by decreasing of retentions capacity of nitrates. For each resin was determined the concentrations of competitors ions which does not affect the retention capacity of nitrates.

Key words: anionic resins, competing anions (Cl^- , SO_4^{2-}), nitrates removal, ion exchange.

1. Introduction

Nitrate anions represent very important wastewater pollutants [1]. While nitrate is a common nitrogenous compound due to natural processes of the nitrogen cycle, anthropogenic sources have greatly increased the nitrate concentration, particularly in groundwater [2]. The most anthropogenic sources are wastewater from industrial processes, septic tanks, application of nitrogen-rich fertilizers to agricultural areas and turf grass, and agricultural processes [1]. The nitrates are extremely soluble in water and can move easily through soil into the drinking water supply by rainfall and irrigation, thus increasing their concentration in drinking water

sources [3], [4]. Excess of nitrate contributes to eutrophication of surface waters due to excess of nutrients [1]. Possible health consequences of nitrate ingestion are related to methemoglobinemia the blue-baby syndrome in infants under 6 months of age [2], [3].

The European Union and World Health Organization (WHO) have both set the standard for nitrate in potable water at 11.3 mg nitrogen per litre (50 mg NO_3^-/L), while the Environmental Protection Agency (EPA) has recommends a maximum limit of 45 mg NO_3^-/L in drinking water [1], [2], [4]. In the Romania, according to the 458/2002 law, a maximum limit of 50 mg NO_3^-/L NO_3^- and 0.5 mg NO_2^-/L in drinking water were permitted.

Nitrate is a stable and highly soluble ion with low potential for co-precipitation or adsorption. These properties make it difficult to remove using conventional water treatment technologies such as lime softening and filtration. Several methods for nitrate removal from drinking water resources have been applied [3], [5]. These methods available for the removal of nitrate are: reverses osmosis, ion exchange, biological denitrification, electro-dialysis and catalytic reduction [6]. The ion exchange process seems to be the most suitable for water suppliers contaminated by nitrate [5], [6]. Ion exchange is a process in which the target ion replaces another less well adsorbed ion on a resin. Nitrate ions can be removed from the water with a strong-base anion resin. This process is a promising technology for nitrate removal because of its simplicity, easy process control, effectiveness, selectivity, recovery and relatively low cost, an omitting possibility of final water treatment. Disadvantages of the process include decreased ion-exchange capability during the process, increased water corrosion and ion concentration change in the water [5],..., [7].

In the removal of nitrates from drinking water resources, using ion-exchange, the competing anions (especially sulphate and chloride ions) have an important role, which, depending on their concentration can influence the retention of nitrates [8], [9]. Some researchers have studied the retention of nitrates of drinking water, in the competitors anions presence (Cl^- and SO_4^{2-}), by anionic resins: Purolite A-520E [10], Amberlite IRN-78 [11], Purolite A-850 [12], Purolite A-300 [13], Amberjet 4200 [14], FIBAN A-1 [8].

The aim of this research is to setting performance the retention of nitrates onto two non-selective Purolite resins (Purolite A-100, Purolite A-847) and nitrate-selective Purolite resins (Purolite A-520E), in the presence of competing anions: chloride and sulphate ions. Specific objectives of this research were to: (a) investigate the influence of individual competing anions on ion exchange resin nitrate removal performance; (b) compare the performance of nitrate-selective and non-selective resins under a variety of conditions.

2. Experimental

Experimental investigation of the influence of competing anions onto nitrate removal from aqueous solution on Purolite resins were performed using batch-mode study and synthetic solutions of sodium nitrate. These resins were chosen based on their characteristics from various commercial available resins used for nitrates removal from water (Purolite Int. Ltd.). The physical properties and specifications of the exchanger resins reported by the suppliers are given in Table 1 [15].

Table 1
Physico-Chemical Properties of Resins Purolite

Physico-chemical properties	Purolite A-100	Purolite A-847	Purolite A-520E
Polymer Matrix Structure	Macroporous, styrene-divinylbenzene	Gel acrylic, divinylbenzene	Macroporous, styrene-divinylbenzene
Functional groups	Tertiary amine	Tertiary amine	Quaternary ammonium
Ionic form, as shipped	Free base	Free base	Cl ⁻
Physical form and appearance	Hard spherical opaque beads	Translucent white spherical beads	Opaque cream Spherical beads
Total exchange capacity - wet, volumetric - dry, weight	1.3 mval/mL 3.9 mval/g	1.6 mval/mL 5.5 mval/g	min. 0.9 meq/mL min. 2.8 meq/g
Temperature limit	max. 100°C	40°C	max. 100°C
pH Range, stability pH Range, operating	0 – 14 0 – 9	0 – 14 0 – 9	0 – 14 4.5 – 8.5

Stock solution of 1254 mg NO₃⁻/L of sodium nitrate was prepared by salt NaNO₃ p.a. dissolving in twice-distilled water. Working solutions was obtained by dilution from the stock solution with twice-distilled water. All other reagents were of analytical reagents degree, and were used without further purifications. A batch-mode study was performed with intermittent stirring, using glass bottles placed on a vibrating platform (100 rpm), for 24 h. Working solution volume in each experiment was 50 mL.

Two anions, sulphate and chloride, were chosen as competitors anions. To determine the individual influence of chloride and sulphate ions on the nitrate retention, the concentrations were increased gradually, in the range: 19 – 115 mg Cl⁻/L and 20 – 90 mg SO₄²⁻/L, respectively. The resins performance was determined for nitrate removal from aqueous solutions containing 50.16 mg

NO_3^- /L and 100.32 mg NO_3^- /L nitrate concentrations. The concentration of nitrate remained in solution after completion of ion exchange was determined by spectrometric method with an UV-VIS Digital Spectrophotometer, model S104D/WPA, (1x1 cm glass cell, $\lambda = 412$ nm, chromotropic acid, against a blank solution), using a prepared calibration graph [20].

In a previous studies [16],..., [19] it has been shown that the ion exchange process of NO_3^- on Purolite A-100, Purolite A-847 and Purolite A-520E resins depends by several experimental parameters (initial solution pH, initial nitrate concentration, resin dosage, temperature). On the basis of experimental results, optimum conditions for each resin are listed in Table 2.

Table 2
Experimental Parameters for the Nitrates Removal on Purolite Resins

Experimental parameters	Anionic resin type Purolite A-100, A-847, A-520E
Initial solution pH	6.5 – 6.8
Resin dose	6 g/L
Temperature	Ambient temperature

The efficiency of nitrate ion removal (degree of retention) expressed as the percentage of removed nitrates (R , %), and the amount of nitrate retained per unit mass (q_e mg NO_3^- /g-dry resin) by the Purolite resins at equilibrium (equilibrium retention capacity), have been calculated by using eqs. (1) and (2):

$$(1) \quad R = \frac{C_{\text{NO}_3^-}^o - C_{\text{NO}_3^-}^e}{C_{\text{NO}_3^-}^o} \cdot 100$$

$$(2) \quad q_e = \frac{(C_{\text{NO}_3^-}^o - C_{\text{NO}_3^-}^e) \cdot V}{m}$$

where: $C_{\text{NO}_3^-}^o$ and $C_{\text{NO}_3^-}^e$ are the initial and equilibrium concentrations of nitrate ions, [mg NO_3^- /L]; m – the mass of dry anion exchanger resin, [g]; V – the volume of the nitrates initial solution used, [L].

To calculate the competitors ion concentration ($i = \text{Cl}^-$ or SO_4^{2-}) that not influence the equilibrium retention capacity of nitrates on studied resins were used linear regression equations [8]. The general form of the equation is:

$$(3) \quad q_e^* = -a \cdot C_i + b$$

where: q_e^* – is the equilibrium retention capacity of nitrate in absence of chloride ions; a – the slope; b – the intercept; C_i – the concentrations of chloride or sulphate ions.

3. Results and Discussions

Ion exchange is a reversible chemical process in which ions from an insoluble permanent solid medium (the ion exchanger-usually a resin) are exchanged for ions in a solution or fluid mixture surrounding the insoluble medium [5]. The direction of the exchange depends on the selective attraction of the ion exchange resin for the specific ions present and the concentration of the ions in the solution (for example Cl^- and SO_4^{2-}). Thanks to differences of affinity for different ions, common ion exchange resins can be used to remove selectively ions from water.

3.1. Effect of Chloride

The influence of the initial concentration of the chloride ions ($C_{Cl^-}^0$) on equilibrium retention capacity of nitrate ions from aqueous solution on Purolite resins is depicted in Fig. 1. Also, degree of retention of nitrates function of chloride ions concentration was calculated for comparative analysis (Table 3).

Table 3

Influence of Chloride Ions on Retention of NO_3^- Ions by Purolite Resins

$C_{Cl^-}^0$ [mg/L]	R, [%]					
	$C_{NO_3^-}^0 = 50.16$ mg/L			$C_{NO_3^-}^0 = 100.32$ mg/L		
	A-100	A-520E	A-847	A-100	A-520E	A-847
0	92.84	87.39	35.21	91.02	89.97	26.92
19.0378	90.51	85.97	31.14	88.19	88.75	25.66
38.0756	89.21	85.75	31.14	86.85	88.48	18.82
76.1512	88.19	85.64	21.83	84.46	85.86	21.83
114.226	86.83	84.98	17.45	83.44	83.78	17.18

From Fig. 1 *a* can see that for relatively low nitrate initial concentrations (50.16 mg NO_3^- /L), capacity of retention at equilibrium of the nitrate by Purolite A-100 resin was sensible higher compared with selective resin, specially designed for nitrate removal, Purolite A-520E. This trend is maintained until to concentration of the chloride ion equal to 80 mg Cl^- /L. Also, the negative slope of the linear equation (eq. (3)) suggest that, with increasing

concentration of chloride ions, retention process is negatively influenced for all three resins studied.

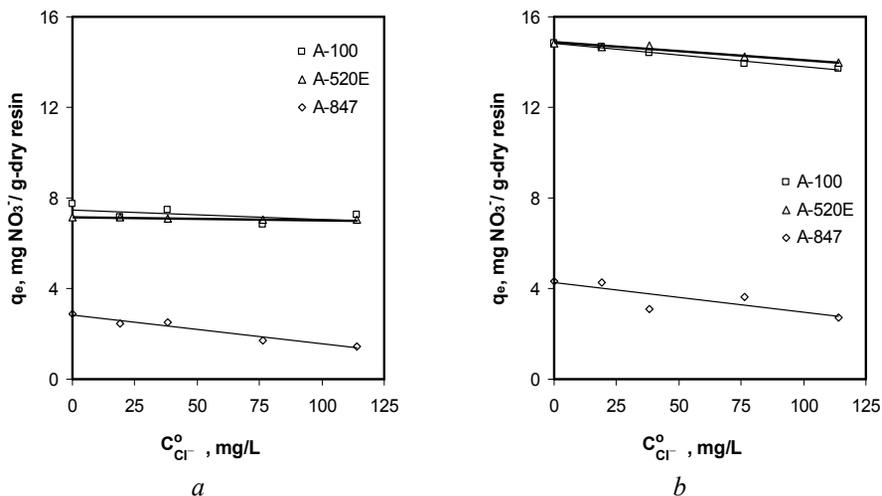


Fig. 1 – Influence of the initial concentration of Cl^- on the equilibrium retention capacity of nitrate ions from aqueous solution by the Purolite resins at 20°C and for

$$C_{NO_3^-}^0 = 50.16 \text{ mg } NO_3^- / L \text{ (a) and } C_{NO_3^-}^0 = 100.32 \text{ mg } NO_3^- / L \text{ (b).}$$

Negative influence of chloride ions was observed for solutions with higher nitrates content (100.32 mg NO_3^- /L and 114.22 mg Cl^- /L). At the same time can be seen from Fig. 1 *b* that the differences in equilibrium retention capacity are insignificant for Purolite A-100 resin (for 114.23 mg Cl^- /L, $q_e = 13.69$ mg NO_3^- /g-dry resins *versus* 13.99 mg NO_3^- /g-dry resins for Purolite A-520E). For Purolite A-847, the equilibrium retention capacity value is only 2.72 mg NO_3^- /g-dry resins. For the same experimental conditions, the values of the degree of retention are comparable for A-100 and A-520E resins (83.44% *versus* 83.78%), while for A-847 resin the degree of retention values is only 17.18 % (Table 3).

In order to determine selectivity of resins towards nitrates, in comparison with chloride as competitors ions, the selectivity coefficient ($\alpha_{NO_3^-/Cl^-}$) was used. The selectivity coefficient can be calculated as the ratio of concentration nitrate concentration to the interfering chloride ions concentration which gives a 5% of absorbance change, in a reference solution [17]. The results for both experiments (50.16 mg NO_3^- /L and 100.32 mg NO_3^- /L, respectively) are shown in Table 4.

Because in the case of the Purolite A-847 resin, both equilibrium retention capacity values and the degree of retention (Table 3) were lower compared with other exchange resins further, the analysis of selectivity coefficients of interest in the case of A-100 and A-520E resins. Can see that, to moderate concentrations of nitrate (50.16 mg NO₃⁻/L), the selectivity coefficients have comparable values for A-100 and A-520E resins.

Moreover, of Fig. 1 can see that the chloride ions concentrations not affect the retention of nitrates, in witch case were values is 70 mg Cl⁻/L *versus* 42 mg Cl⁻/L for nitrate-selective resin. To higher concentrations of nitrate (100.32 mg NO₃⁻/L and 114.226 mg Cl⁻/L) the degree of retention values for A-100 was similar with A-520E (83.44% *versus* 83.78%). For A-847 resin the degree of retention values is only 35.21%.

In terms of the influence of chloride ions, in the removal of nitrates from water, it can be concluded that, use of selective resin Purolite A-520E show no advantage in addition versus non-selective Purolite A-100 resin. The presence of chloride anion in the fed water might affect the capacity of the resins for low nitrate concentration.

Table 4

The Selectivity Coefficient in Retention of NO₃⁻ on Purolite Resins

Resin type	C _{NO₃⁻} ^o , [mg/L]	R ²	C _{Cl⁻} , [mg/L]	a _{NO₃⁻/Cl⁻}	ln a
Purolite A 100	50.16	0.324	69.75	0.719	-0.329
	100.32	0.983	4.12	24.379	3.193
Purolite A 520E	50.16	0.863	42.72	1.174	0.160
	100.32	0.947	1.13	88.772	4.48
Purolite A 847	50.16	0.955	6.29	7.958	2.074
	100.32	0.684	11.42	8.778	2.172

3.2. Effect of Sulphate

Dependence between the equilibrium retention capacity of nitrate ions from aqueous solution on Purolite resins and the initial concentration of the sulphate ions ($C_{\text{SO}_4^{2-}}^0$) is illustrated in Fig. 2.

From Fig. 2 *a* can see that for non selective A-100 resin (to moderate concentrations of nitrates of 50.16 mg NO₃⁻/L and sulphate ions concentrations of 90.07 mg SO₄²⁻/L), capacity of retention at equilibrium was higher compared with the other two resins studied, until to sulphate ions concentrations of about

90 mg SO_4^{2-} /L. The capacity of retention at equilibrium is 6.975 mg NO_3^- /g-dry resin for A 100 *versus* 6.894 mg NO_3^- /g-dry resin for A-520E. For A-847 capacity of retention at equilibrium value is only to 1.307 mg NO_3^- /g-dry resins.

Negative influence of sulphate ions is observed at higher concentrations of nitrate (100.32 mg NO_3^- /L). From Fig. 2 *b* can see that, for non selective A-100 resin capacity of retention at equilibrium was higher *versus* A-520E, until to sulphate ions concentrations of about 65 mg SO_4^{2-} /L. In these conditions, it can be said that A-100 non selective resin shows good results compared with A-520E selective resin, for water with low nitrates and sulphate concentrations. The macroporous matrix and special ion exchange group functionality imparts ideal nitrate selectivity to A-520E resin making this resin particularly suitable for nitrate removal even when moderate to high concentrations of sulphate are present. Hence this resin gives superior performance in nitrate removal applications when compared with standard exchange resins.

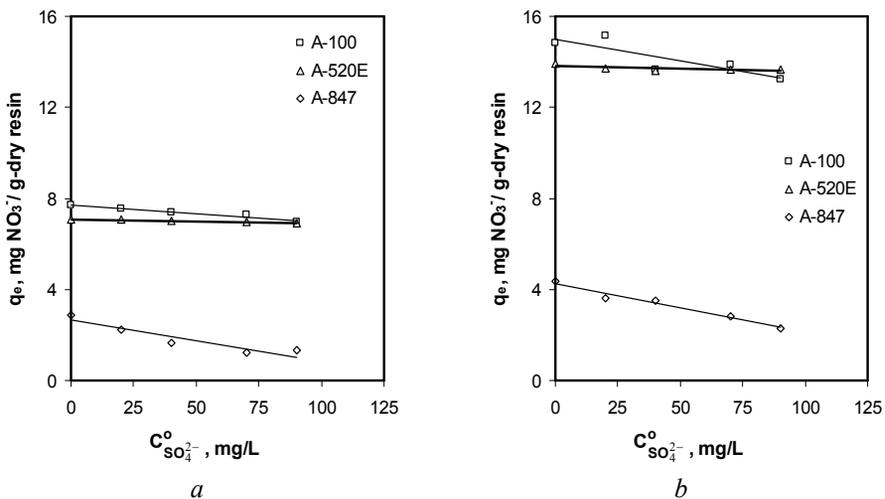


Fig. 2 – Influence of the initial concentration of SO_4^{2-} on the equilibrium retention capacity of nitrate ions from aqueous solution by the Purolite resins at 20°C and by

$C_{\text{NO}_3^-}^0 = 50.16$ mg NO_3^- /L (a) and $C_{\text{NO}_3^-}^0 = 100.32$ mg NO_3^- /L (b).

Also, the degree of retention of nitrates function of the sulphate ions concentration was calculated for comparative analysis (Table 5).

In the case of sulphates (90.07 mg SO_4^{2-} /L) it observed that, for both initial concentrations of nitrates studied (50.16 mg NO_3^- /L and 100.32 mg NO_3^- /L) the degree of retention is higher in case of A-100 non-selective resin,

86.95 % and 82.65 % versus 85.21% and 81.92 % for A-520E resin. For A-847 resin, the degree of retention value is only 16.22 % and 14.18 % respectively.

Table 5

Influence of Sulphate Ions in Retention of NO_3^- Ions on Purolite Resins

$C_{\text{SO}_4^{2-}}^0$ [mg/L]	R_s , [%]					
	$C_{\text{NO}_3^-}^0 = 50.16 \text{ mg/L}$			$C_{\text{NO}_3^-}^0 = 100.32 \text{ mg/L}$		
	A-100	A-520E	A-847	A-100	A-520E	A-847
0	92.84	86.29	35.22	91.02	84.11	26.90
20.02	90.90	85.20	27.82	91.26	84.98	22.39
40.03	89.32	85.20	20.19	87.06	85.21	21.57
70.05	87.85	84.66	14.58	85.82	81.92	17.25
90.07	86.95	85.21	16.22	82.65	81.92	14.18

Negative slope value (eq. (3)) shows the negative influence of sulphate ions in nitrates retention. Using linear regression equations were calculated selectivity coefficients (Table 6), for both initial concentrations of nitrates studied.

Table 6

The Selectivity Coefficient in Retention of NO_3^- on Purolite Resins

Resin type	$C_{\text{NO}_3^-}^0$, [mg/L]	R^2	$C_{\text{SO}_4^{2-}}$, [mg/L]	$a_{\text{NO}_3^-/\text{SO}_4^{2-}}$	$\ln a$
Purolite A 100	50.16	0.941	8.59	5.839	1.764
	100.32	0.733	5.92	17.181	2.844
Purolite A 520E	50.16	0.903	38.19	1.313	0.270
	100.32	0.507	65.36	1.532	0.425
Purolite A 847	50.16	0.879	15.67	3.201	1.163
	100.32	0.976	5.912	16.966	2.831

3.3. Performance of Nitrate-Selective and Non-Selective Resins

Non-selective strongly basic anion exchange resins (conventional resins) have a stronger affinity (or a better selectivity) for the nitrate or sulphate ion than for the chloride ion, the order of affinity being: $\text{SO}_4^{2-} > \text{NO}_3^- > \text{Cl}^-$.

Sulphate has two negative charges (the valence) that give it a greater affinity to standard non-selective strong-base anion resin (Purolite A-100) than nitrate with one negative charge. The reason for investigating effect of anions individually was to better understand the adsorption characteristics of the resins tested. This is important since nitrate can be “dumped” from these types of resin. Dumping is defined as the elevation of nitrates in the treated water over

the level of nitrates in the raw (influent) water. As the nitrate system reaches exhaustion, sulphates will displace nitrates, causing a significant increase in nitrate leakage corresponding to the nitrate and sulphate levels in the raw water.

The nitrate-selective resins (Purolite A-520E) have a different exchange group that makes it possible for nitrates to be removed from water without the potential for dumping. With these types of resins the nitrate is held preferentially over sulphate (Fig. 3). Nitrate-selective resins are formulated in such a way as to adsorb nitrate preferentially *versus* sulphate and are able to exchange nitrate for sulphate as well as other anions. The exchange capacity of Purolite A-520E resin was not affected to the same extent as that of Purolite A-100 resin ones, but the differences are not significant (Fig. 3).

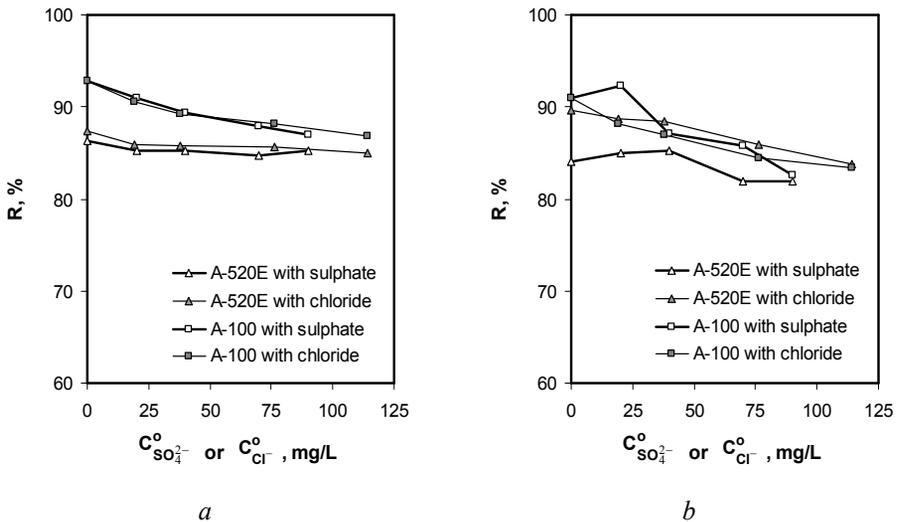


Fig. 3 – Individual influence of the initial concentration of SO_4^{2-} and Cl^- on the degree of nitrate retention from aqueous solution by the Purolite resins - chloride form at 20°C and by $C_{NO_3^0} = 50.16 \text{ mg NO}_3^-/\text{L}$ (a) and $C_{NO_3^0} = 100.32 \text{ mg NO}_3^-/\text{L}$ (b).

It can be seen that sulphate couldn't challenge Purolite A-520 more than chloride ions did, and therefore this resin's performance was the best amongst the resins tested when faced with a blend of competing anions. Overall, Purolite A-100 appears to be the alternative by Purolite A-520E for removing nitrate from highly concentrated waters. Also, it can be concluded that the non-selective resins Purolite A-847 may not be reliable for removing nitrate from real waters that contain background anions. The reason is that they are highly affected by the presence of competing anions, and might start releasing previously adsorbed nitrate if a careful regeneration practice is not maintained.

3. Conclusions

In this study, efficiency of nitrate removal from water onto Purolite resins was investigated, in the presence of other background anions. The ion exchange resins used in this experiment are based on styrene-divinylbenzene copolymer attached to a tertiary amine functional group (non-selective resins/Purolite A-100 and Purolite A-847) or a quaternary ammonium functional group (nitrate-selective resin/Purolite A-520E).

The ions of sulphate (SO_4^{2-}), and chloride (Cl^-), were chosen as competing anions, and the effects were investigated individually using two reference concentrations of nitrates (50.16 mg NO_3^-/L and 100.32 mg NO_3^-/L). The exchange capacity for all tested resins for nitrate removal was negatively impacted in the presence of competing anions, especially sulphate. The study presented shows that, to moderate concentrations of nitrates and chloride (50.16 mg NO_3^-/L nitrates concentrations and 114.22 mg Cl^-/L chloride concentrations), Purolite A-100 non-selective resin shows better results ($q_e = 7.23$ mg $\text{NO}_3^-/\text{g-dry resin}$; $R = 86.83\%$) compared with Purolite A-520E selective resin ($q_e = 7.02$ mg $\text{NO}_3^-/\text{g-dry resin}$, $R = 84.98\%$), in the retention of nitrates from water. For Purolite A-847 capacity of retentions is only 1.42 mg $\text{NO}_3^-/\text{g-dry resin}$ and degree of retentions of 17.45%.

For water with higher concentrations of nitrate (100.32 mg NO_3^-/L) and moderate concentrations of chloride (114.22 mg Cl^-/L), the value of equilibrium capacity of retention was higher for Purolite A-520E selective resin. But, differences were insignificant for Purolite A-100 resin, $q_e = 13.68$ mg $\text{NO}_3^-/\text{g-dry resin}$ versus 13.99 mg $\text{NO}_3^-/\text{g-dry resin}$ for Purolite A-520E resin. In the case of sulphate ions, observed that, although this does not significantly influence in the retention of nitrates onto Purolite A-520E resin, the capacity of retention at equilibrium value was higher for Purolite A-100, for moderate nitrates concentrations, 50.16 mg NO_3^-/L . At higher concentrations of nitrates, 100.32 mg NO_3^-/L , the capacity of retention at equilibrium value was higher for Purolite A-100 until to sulphate ions concentrations of about 65 mg $\text{SO}_4^{2-}/\text{L}$.

Thus, using non nitrate-selective resins for removing nitrate from a wastewater containing other background anions is not suggested at full-scale unless a very robust regeneration practice is achievable. Therefore, if feed water contains elevated concentrations of sulphate or total dissolved solids, resins become exhausted sooner and regeneration would be needed at more frequent intervals. Purolite A-520E nitrate-selective resin is the most promising of the

resins tested for removing nitrate from the wastewater, when faced with a blend of competing anions.

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“Gheorghe Asachi” Technical University of Iași,

*Department of Chemical Engineering

e-mail: ancaceica@ch.tuiasi.ro

**Department of Environmental Engineering
and Management

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ELIMINAREA NITRAȚILOR DIN APĂ ÎN PREZENȚA ANIONILOR COMPETITORI UTILIZÂND RĂȘINI PUROLITE

(Rezumat)

În această lucrare a fost studiată influența ionilor competitori, clorură respectiv sulfat, în procesul de reținere a nitraților din soluții apoase utilizând trei rășini schimbătoare de ioni: două rășini non-selective Purolite A-100 și Purolite A-847 și o rășină selectivă, special destinată pentru reținerea nitraților din ape, Purolite A-520E. Performanțele rășinilor au fost studiate în regim static utilizând două concentrații inițiale de nitrați (50.16 mg NO₃⁻/L respectiv 100.32 mg NO₃⁻/L). Pentru determinarea influenței ionului sulfat, concentrația ionului sulfat a fost crescută treptat în domeniul 20 – 90 mg SO₄²⁻/L, iar pentru influența ionului clorură concentrația acestuia a fost crescută în domeniul 19 – 114 mg Cl⁻/L. Pentru toate cele trei rășini studiate a fost observată influența negativă a ionilor competitori prin scăderea capacității de reținere a nitraților odată cu creșterea concentrației acestora. S-a determinat pentru fiecare rășină în parte, valoarea concentrației maximum admisă a ionilor competitori care nu influențează procesul de reținere a nitraților din ape.