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Nitrate Removal from Groundwater by Strongly Basic Anion Exchange Resin, Duolite A161 (Type 1): Feasibility Study and Optimization

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Abstract

In this study, a powerfully basic anion exchange resin (type 1): Duolite A161 was used in batch mode sorption studies in order to evaluate its efficiency in removing nitrate ions. In the first part of this work, the optimization of the parameters influencing the efficiency of the resin, namely: The dose of the resin (0.5 to 8 g), the stirring speed (10 to 120 rpm), the contact time (5 to 30 min), the initial nitrate concentration (30 to 180 ppm) and the pH (4 to 11), was carried out. The second part was devoted to studying the competitiveness of anions and cations, commonly present in groundwater, namely: sulfates, bicarbonates, chlorides, fluorides, sodium, potassium, calcium and magnesium; under the predetermined optimum conditions so as to examine its effect on the retention of nitrate ions by the resin. The results confirmed the effectiveness of the strong base anion exchange resin, such that more than 99% elimination of nitrate ions was obtained at: 2 g of resin, 100 mg / L of nitrates, with stirring of 30rpm and during 18 min. On the other hand, the presence of anions: sulfates, chlorides, bicarbonates and fluorides resulted in an average reduction of 13%, 32%, 23% and 8% respectively. While the presence of cations: sodium, potassium, calcium and magnesium, had no effect on the effectiveness of the resin. Hence, the strong bases resin (type 1): Duolite A161 is one of the most attractive commercial exchangers, in terms of efficiency in removing nitrate ions from contaminated groundwater.

Keywords: Anion exchange resin, Duolite A161, Groundwater, Removing nitrate

1 Introduction

The pollution of groundwater by nitrates ions has become one of the most worrying global phenomenon for its harmful effects on human health and aquatic life [1]. In the absence of pollution, nitrates are naturally present in water in moderate concentrations which generally do not exceed a few milligrams per liter in groundwater. While, lately, their substance has carry on to increase in several regions of the world : Asia [2], Africa, the Middle East, the European Union, the United States, Australia, Canada, New Zealand [3] and Morocco [4]. This is principally attributed to agricultural activities based on the intensive use of synthetic fertilizers and manures, participating in the increase in the pollution of the water table by nitrates [5,7]. Decomposition of plant and animal matter, as well as deficient septic systems and uncontrolled landfills of urban wastewater, can also be sources of nitrates in water [7,8]. The risk of contamination becomes greater if the water table is shallow and if the soil covering the water table is vulnerable [4]. Similar to other countries in the world, Morocco is one of the affected countries; several studies have highlighted the contamination of groundwater by nitrates in several regions of the kingdom, with values that greatly exceed the standards for drinking water, up to 400 mg/l. According to these studies, it appears that the highest nitrate contents are generally found in regions where agricultural activities are more intense or in regions where the wells are close to uncontrolled landfills and the industrial zone and those where water resources are shallow [4,9]. From an ecological point of view, the presence of excess nitrates in the water is one of the causes, along with phosphates, of the proliferation of algae, which reduce the oxygen content, and therefore contributes to the appearance of the phenomenon of eutrophication or even dystrophication of aquatic environments [10]. From a healthy point of view, the reduction of nitrates ions to nitrites leads to the formation of methemoglobin (cyanosis disease) especially in infants (blue babies' syndrome) which, following anoxia cellular, may progress to death. Its presence can also contributes to the synthesis of nitrosamines which may, in the long term, cause cancer of the stomach or intestines in adults [1,7,11]. Faced with these potential dangers, the World Health Organization has adopted a threshold value of 50ppm of nitrates (NO_3^-) equivalent to 11.3 mg / l of nitrogen (N), for water intended for human consumption, after which, nitrates become toxic [12]. Currently, different processes exist to remove nitrates from

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contaminated water, namely: biological denitrification processes: heterotrophic or autotrophic [13], membrane processes: reverse osmosis, nanofiltration [14] and electrodialysis[15], in addition to the reduction processes reaches: catalytic, photo-catalytic or electrochemical [7]. Despite the effectiveness of these techniques, their application remains limited in view of their disadvantages, economically and / or technically [7,8], added to the risk of bacterial contamination. Screw biologically denitrified water requiring a germicidal post-treatment. In contrast, the ion exchange operation is among the most acceptable methods for small suppliers of water polluted with nitrates [7]. It offers attractive prospects in terms of simplicity, efficiency, selectivity, and cost-effectiveness in addition to the process's insensitivity to the pH of contaminated water and the effective long life of the resins [3,7,16].

This physicochemical process consists of using anion exchange resins which make it possible to remove the anions present in the solution to be treated with the anions released by the resin until it is saturated. Once the resin is exhausted, its regeneration becomes necessary in order to bring it back to its original state and make the process profitable [10]. In the case of nitrates, strongly basic polystyrene anion exchange resins having a preferential affinity to nitrate, in particular, in the case of competing anions naturally present in water such as: sulphate, bicarbonate and chloride; are usually used. The nitrate-selective resins progressing in 1985 are those described by their prolonged alkyl chains where the number of carbon atoms around the ammoniacal nitrogen of the resin is considerable [1,17]. This is due to the longer carbon chain which makes the resin more hydrophobic, thus increasing its affinity for anions with reduce hydration energies such as nitrate [18].Among these resins, we can cite: Purolite A-520E [18], L20 [17], NDP-2 [19] and Indion NSSR [6].

In this study, the effectiveness of the anion exchange resin: Duolite A161, in removing nitrate ions, using batch sorption experiments, was investigated. This study will focus on two objectives: The first objective to define the optimum parameters giving better retention of nitrate ions: The dose of the resin, the rapidity of agitation, the connection time, the initial nitrate concentration and the pH of the medium. The second objective is to study the competitiveness of anions and cations commonly present in groundwater, in particular: sulphate, bicarbonate, chloride, fluoride, sodium, potassium, calcium and magnesium, under the predetermined optimum conditions in the purpose to assess its effect on the retention of the nitrate ions by the resin: Duolite A161.

2 Experiment

2.1 Feed solution

The denitrating solutions used in this study were artificial nitrate solutions. These model solutions were prepared by dissolving the Sodium Nitrate ($NaNO_3$) in distilled water. All other reagents used were analytical grade and used without purifications.

2.2 Materials

The Duolite A161 anion exchanger is a strongly basic resin (type 1). Its polystyrene matrix is prepared according to a special patented process which offers a flexible bridging with homogeneous structure obtained through careful cross linking. The porosity of Duolite A161 has been optimized to make it especially resistant to attrition and osmotic shocks. Performance and settlement of Duolite A161 have been attested through many years of application in several installations particularly in continuous ion exchange process and high speed condensate treatment [20]. The physical and chemical properties of the resin, aforesaid by the suppliers [20], are shown in Table 1.

Table 1: Physico-chemical properties of resin Duolite A161 [20]

PROPERTIES	
Matrix	Styrene divinyl benzene copolymer
Functional groups	$-N^+(CH_3)_3$ Min 95%
Physical form	Opaque Beads
Ionic form as supplied	Cl
Total exchange capacity	Min 1.15 eq / L (Cl^- form)
Moisture holding capacity	53-58% (Cl^-form)
Specific gravity	About 1.08 (Cl^- form)
Shipping weight	About 700 g / L (Cl^- form)
Operating pH range	0-14
Maximum operating temperature	60°C (OH^-), 100°C (Cl^-)

2.3 Methodology

Batch sorption experiments were executed in conformity with detailed experimental procedure described by [8,11,17]. In order to optimize the parameters influencing the efficiency of the resin studied: Duolite A161, batch sorption experiments (discontinuous) were carried out by introducing various quantities of resin into 250 ml of NaNO3 solution in 6 stirrers placed in parallel shows in figure 1 (Jar test disposal) at room temperature (25°C), measuring the pH of the solutions before shaking them. During these experiments, the factors influencing the efficiency of nitrate removal by the anion exchange resin were determined in the following order:

- The mass of the resin,
- The stirring speed,
- Contact time,
- The initial concentration of nitrates,
- The medium pH.

Concerning the study of the affinity of the resin towards the nitrate ions compared to the ions studied: sulfates, bicarbonates, chlorides, fluorides, sodium, potassium, calcium and magnesium, batch sorption experiments were realized, with 250 ml of solutions containing the nitrate ions and one of the studied ions, in dissolving nitrate salts: $NaNO_3$, KNO_3, $Ca(NO_3)_2$, $Mg(NO_3)_2$ and sodium salts: Na_2SO_4, $NHCO_3$, NaCl and NaF, under predetermined optimum conditions, at room temperature (25°C) and without pH adjustment.

Afterwards, the resin was separated from the sample by vacuum filtration. The remaining nitrate concentration was estimated utilizing the specific electrode method. The nitrate retention rate was determined utilizing the following equation (1) [21]:

$$\% R = \frac{C0 - Cr}{C0} \times 100) \qquad (1)$$

where R is the retention rate of nitrate content in the solution (%), C0 is the initial concentration of nitrate (mg/L), and Cr is the residual concentration of nitrate in (mg/l).

3 Results and Discussions

3.1 Determination of the optimal conditions for the elimination of nitrate ions by the resin: Duolite A161

3.1.1 Impact of the resin dose

The quantity of resin is one of the most important parameters, which can tell us about the capacity of an ion exchanger during the denitration process [8,18]. So as to find the optimal dose of the resin: Duolite A161, allowing the nitrate to be completely removed from the aqueous solution, varying amounts (0.5- 8 g) of Duolite A161 resin, were mixed with 250 ml of solution of nitrate (100 mg/l, pH 5.5). The solutions were stirred at 30 rpm during 10 min and at room temperature (25°C). The residual nitrate concentrations were measured after filtration of the solutions. The experimental results obtained are represented in Figure 2 which represents the nitrate elimination rates as a function of different amounts of Duolite A16 resin, keeping the other parameters constant. We observe from the figure that the efficiency of nitrate removal increases as the amount of resin increases.

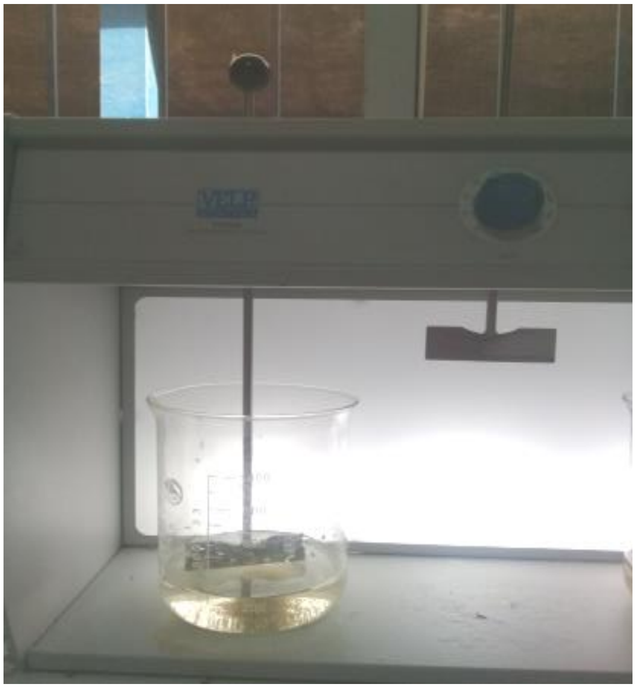

Figure 1: Test jar device

This can be explained by the way that increasing the dose of the resin more energetic sites to the retention of nitrate ions on the resin surface [8,11,14,18]. Based on the results obtained, the resin used proven high removal effectiveness even at low doses. For a quantity of resin ranging from 0.5 to 8g; the rate of removal of the nitrate ion increased from 50% to 92%. While beyond 2g of resin, the nitrate removal efficiency remained constant; even at higher resin doses where the differences between the values of nitrate removal rates were relatively negligible (89.73%; 90.91%; 92.75%; 92.24% et 92.68% at 2g; 3g; 4g; 6g et 8g of resin): (1-3%); which indicated the excess resin [14]. Therefore, it found that the optimum dose capable of removing nitrate quantitatively avoiding resin wastage, turned out to be 2g resin/250ml solution, with which, 89% nitrate removal was obtained [8].

3.1.2 Contact time effect

The touch time of an anion exchanger with a given anion plays a crucial role in realizing better reduction of nitrate ions. To decide the touch time necessary to reach sorption equilibrium, 9 parallel experiments were carried out by introducing 250 ml of solution (pH 5.5) containing100 mg/L of nitrates and 2 g of resin: Duolite A161 at 30 rpm, for different times (5-30 minutes). Residual nitrate concentrations in the recovered filtrates were calculated. The change in the concentration of nitrate ions during different contact times was studied under fixed operating conditions as mentioned in figure 3. From the results obtained, we can see that the nitrate concentration decreases with increasing touch time. Among the different times, the highest nitrate retention rate was obtained after 18 minutes when the percent elimination reached 90.6%. After the first 18 minutes, the removal yields remained constant; indicating that a contact time of 18 min was enough to reach sorption balance. The relatively low values of the touch time required to reach the balance state, indicate that the nitrate elimination process might be due to electrostatic interplay between the functional groups of the resin and the nitrate ions as a result of the ion exchange process [11]. Therefore, the optimum contact time ensures the equilibrium of sorption during denitration by the anion exchange resin: Duolite A161 was found to be 18 min.

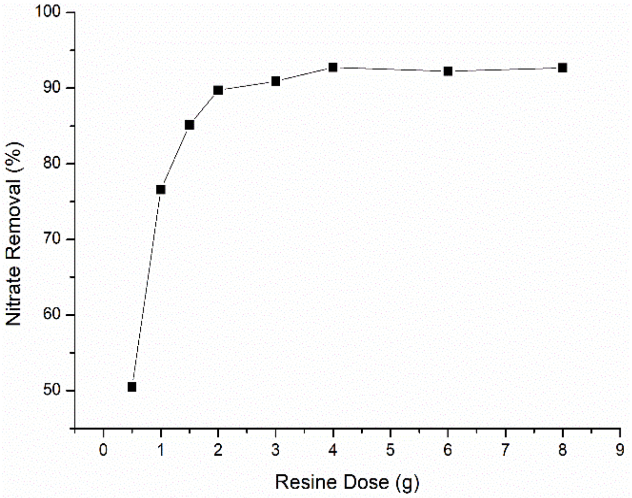

Figure 2: Impact of resin dose on nitrate elimination efficiency from aqueous solution

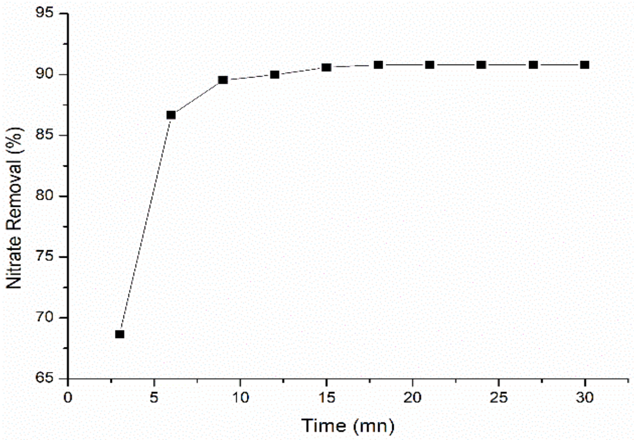

Figure 3: The impact of contact time on nitrate elimination efficiency from aqueous solution

3.1.3 Impact of stirring speed

The stirring speed in the ion exchange process is an interesting parameter to have a good reduction of nitrates, while consuming less energy. To determine the optimum speed, sorption studies in batch mode were carried out by putting a solution of 250 ml (pH 5.5) containing 100 mg/L of nitrates and 2g of resin under different stirring speeds ranging from 10 to 120 rpm for 18 minutes. Residual nitrate concentrations were measured after filtering the samples.

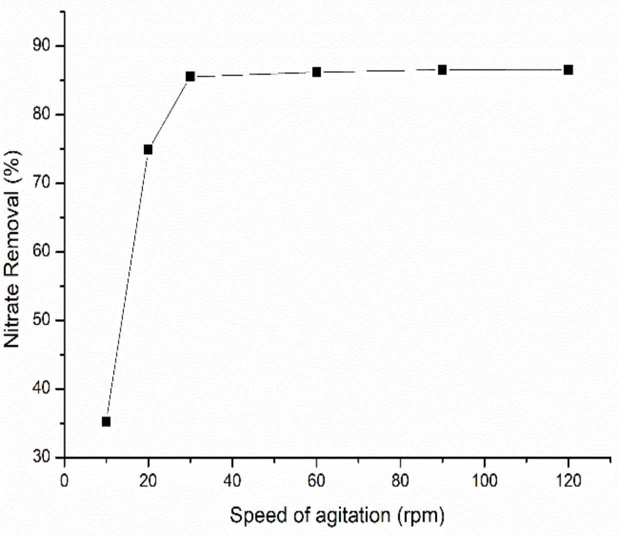

Figure 4: Impact of speed of agitation on nitrate elimination efficiency from aqueous solution

The influence of the stirring rate on the nitrate concentration is shown in Figure 4. During the 18 min of stirring, it is observed that the nitrate ion content decreases from 10 to 30 rpm where the nitrate retention rate increases from 35% to 85%, and begins to stabilize from 60 up to 120 rpm, for which the nitrate removal percentages were almost unchangeable (85.5% - 86%). Therefore, to avoid the energy consumption of the agitation system, it was revealed that 30 rpm is the optimal agitation speed for our study, under which, 85% nitrate removal was obtained.

3.1.4 Impact of the initial concentration of nitrate ions

The impact of the initial nitrate ions concentrations on the elimination efficiency of Duolite A161 resin was tested by varying the initial nitrate ions concentration from 30 to 180 mg/L: the initial concentration of maximum nitrate ions utilized for this study was restricted to about 180 ppm because their concentration in polluted drinking water is between 100 and 200 ppm [6]. Solutions were prepared by diluting NaNO3stock solution. 250 ml of each solution (pH 5.5) were mixed with 2 g of resin and stirred at 30 rpm for 18 min. The residual nitrate concentrations were measured after filtering the samples. From the experimental results shown in Figure 5, it is noticed that the heighten the initial nitrate concentration from 30 to 180 mg / L; led to a diminishing in nitrate retention rates from 93% to 81%, these results are comparable to those obtained by [8,11]. This can be connected to the restricted number of sorption sites obtainable on the surface of the Duolite A161 resin relative to the number of nitrates in the aqueous solution to be denitrated [21]. Indeed, Duolite A161 resin showed relatively high elimination efficiency despite the grow in the initial concentration of nitrate ions contained in the solution to be treated. Therefore, at a dose of 2 g resin solution / 250 ml, the availability of active sites on the resin surface relative to the number of nitrate ions was not a limiting factor [6].

3.1.5 Impact of pH

The initial pH of the aqueous fluid to be denitrated is one of the most significant variables to study for its influence on the adsorption performance of resins [17]. To assess the effect of pH on resin removal efficiency: Duolite A161, six solutions (180 ppm of nitrates / 2g resin / 250 ml) of different pH (4, 5, 7, 8) were stirred at 30 rpm. The initial pH values of the model fluid were changed by adding HCl (0.01 M) or NaOH (0.01 M). The final nitrate concentrations of the filtered samples were measured.

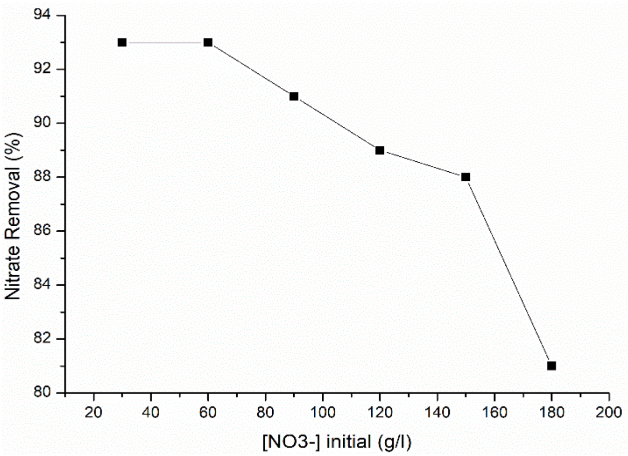

Figure 5: Impact of initial nitrate ions concentrations on nitrate elimination efficiency from aqueous solution

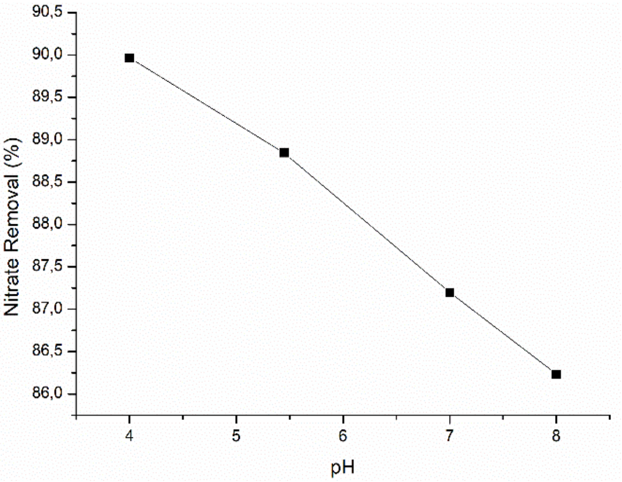

Figure 6: Impact of pH on efficiency elimination of nitrate from aqueous solution

The Figure 6 shows the adjustment in the remaining concentration of nitrate ions as an element of the initial pH of the fluid to be corrected. From the figure, we can notice that the percent nitrate removal by the resin was significantly higher (89.97%-88.84%) for acidic solutions (pH<7), compared to basic or neutral solutions (pH ≥ 7), where the retention rates were between 87.19% and 86.23%. The relatively small decrease in retention rates can be attributed to competition between nitrate anions (NO_3^-) and hydroxide ions (OH^-) present in solutions with a pH less than or equal to 7 at the binding sites of the anionic resin A161 [22]. Since the nitrate removal percentages were almost unchanged at different pH ranges, the resin exchange process: Duolite A161 was discovered to be independent of the initial pH of the aqueous solution. Based on these results, Table 2 summarizes the optimum conditions which allowed better retention of nitrate ions by the resin: Duolite A161 obtained in this study.

Table 2: Optimal conditions

Parameters	**Values**
Mass of resin	2g
Contact time	18min
Stirring speed	30rpm
NO_3 concentration	120 mg / l
pH	pH < 8

3.2 Competitiveness of transfer of anions and cations through the resin: Duolite A161

The behavior of resins towards a given anion, in the presence of another competitive anion (co-ions), is an important factor to study during the ion exchange process [23]. In addition, the cations naturally present in water, as counter-ions, can negatively influence the removal efficiency of anionic resins [24]; hence the interest in evaluate their effect on the ion exchange process. In this study, eight ions were selected to examine their effect on the removal efficiency of Duolite A161 anion exchange resin; anions known as competitive ions to nitrate ions, namely: sulfate (SO_4^{2-}), bicarbonate (HCO_3^-), chloride (Cl^-) and fluoride (F^-) as well as cations commonly found in ground water: sodium (Na^+), potassium (K^+), magnesium (Mg^{2+}) and calcium (Ca^{2+}).

3.2.1 Impact of cations on efficiency of resin

To study the impact of cations on the ion exchange process, four synthetic nitrate fluids of 100 mg/L were ready by dissolving in 250 ml of distilled water: sodium nitrate ($NaNO_3$), potassium nitrate (KNO_3), magnesium nitrate ($Mg(NO_3)_2$) and calcium nitrate ($Ca(NO_3)_2$) separately. The prepared fluids were agitated for 18 min after adding 2 g of resin to each of the solutions. From Figure 7, it can be seen that bivalent cations had the greatest impact. But in general, the cations do not have a significant effect in terms of the percentage of nitrate ion removal, (reduction from 2 to 6%) where the retention rates obtained were: 99%, 98%, 93.6% and 93.31% in the presence of Na^+, k^+, Mg^{2+} and Ca^{2+} respectively. The outcomes obtained prove that the presence of the cations: Na^+, k^+, Mg^{2+} and Ca^{2+}, has a relatively negligible effect on the process of elimination of nitrate ions.

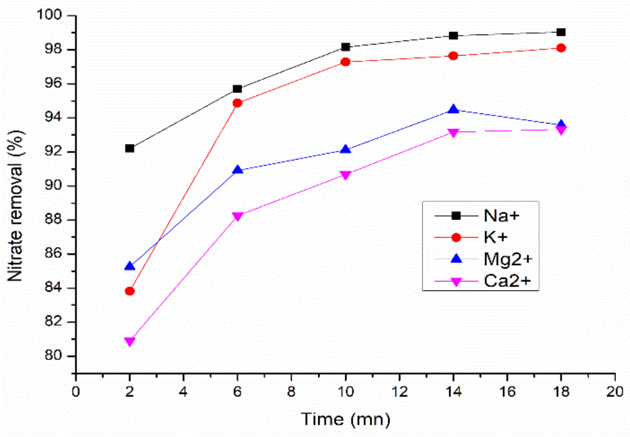

Figure 7: Impact of cations on efficiency elimination of nitrate from aqueous solution

3.2.2 Impact of anions on efficiency of resin

Concerning the impact of anions, binary solutions of 250 ml, containing a fixed concentration of nitrate (100 mg/L) and one of the anions, were prepared, varying their initial concentrations from 50 to 200 mg/L, with a quantity of 2g of Duolite A161 resin. All solutions were prepared without pH adjustment and agitated at a rapidity of 30 rpm, at chamber temperature for 18 min. Residual nitrate concentrations were measured after filtration of the samples using the specific electrode method.

In the case of anions Figures 8, 9, 10 and 11, the results indicate that each competing anion caused, as its concentration increased, a significant reduction of: (6 - 11%), (11 - 15%), (14 - 29%) and (18 - 42%) in the presence of: F^-, SO_4^{2-}, HCO_3^- and Cl^- respectively. Fluoride ions had the lower effect on the elimination of nitrate compared to SO_4^{2-}, HCO_3^- and Cl^-. The strong decrease in retention rates, indicates that Duolite A161 resin removes other anions, including: SO_4^{2-}, HCO_3^- and Cl^-, this may be related to the hydrophobicity of the anionic resin and the hydration energies of the anions present in the aqueous solution to be denitrated, according to which the resin preferentially binds one of the anions present leading to competition between the NO_3^-, SO_4^{2-}, HCO_3^- and Cl^- ions on the exchange sites of the resin [17,19]. The results obtained prove that the presence of the anions: SO_4^-, HCO_3^-, Cl^- and F^-, in the contaminated water, reduce significantly the efficiency of the resin: Duolite A161.

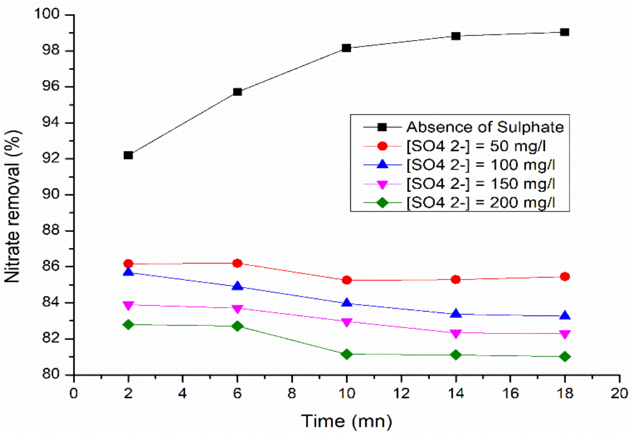

Figure 8: Impact of sulfate ions on efficiency eliinition of nitrate from aqueous solution

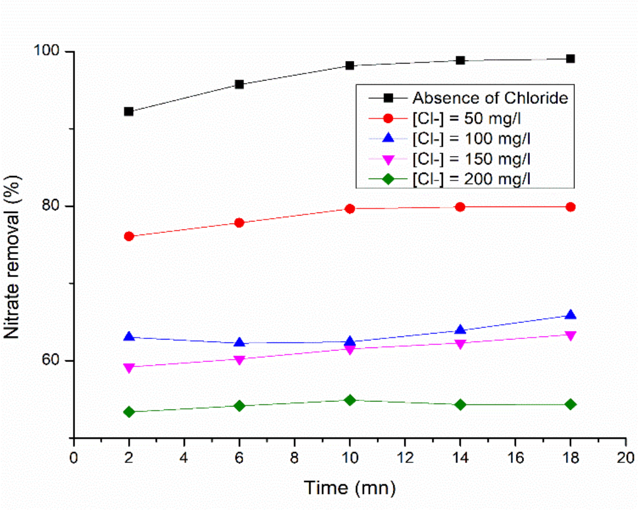

Figure 9: Impact of chloride ions on efficiency elimination of nitrate from aqueous solution

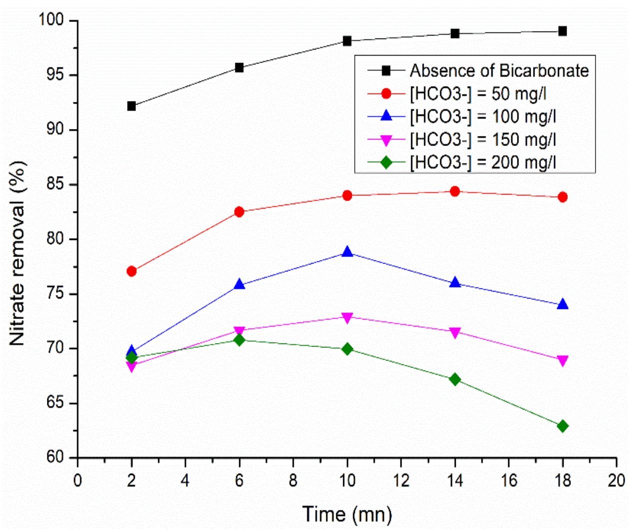

Figure 10: Impact of bicarbonate ions on efficiency elimination of nitrate from aqueous solution

4 Conclusion

In this study, the elimination of nitrates from watery solution was performed utilizing the strongly basic anion exchanger (type 1): Duolite A161. From the results obtained, it can be concluded, on the one hand, that the resin showed a high removal efficiency of nitrate ions was obtained, at small lots of resin (2g), using 100 mg/l as initial concentration of nitrates, with stirring at 30 rpm and for 18 min. The time desired to reach sorption equilibrium was very rapid. The operation of nitrate elimination by the resin was independent at the initial pH of the water solution to be treated. On the other hand, it was revealed that the existence of the anions reduced the resin effectiveness, especially in the case of: chlorides, bicarbonates, sulfates, and fluorides respectively. However, the presence of the cations studied: sodium, potassium, calcium and magnesium, had no effect on the competence of the resin. Therefore, the anion exchanger: Duolite A161 is one of the promising candidates for the elimination of nitrate ions from contamination groundwater. As research perspectives, a study of the regeneration of the resin: Duolite A161 will be carried out, to examine the cost-effectiveness of the process, for the purpose to verify the performance of the resin: Duolite A161, after its use in the elimination of nitrate ions from water contaminated, and assess its reusability after regeneration.

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Ethical issue

The authors declare that this manuscript is original from our laboratory (Advanced Materials and Process Engineering) and respects the publication standards with regard to: Double publication, references and results illustrated in this manuscript.

Competing interests

The authors appear their names in this scientific work, certify that they have no conflict of interest and that there is no funding for this work.

Author's contribution

All the authors contributed of the production of this manuscript including data collection, writing and statistical analysis.

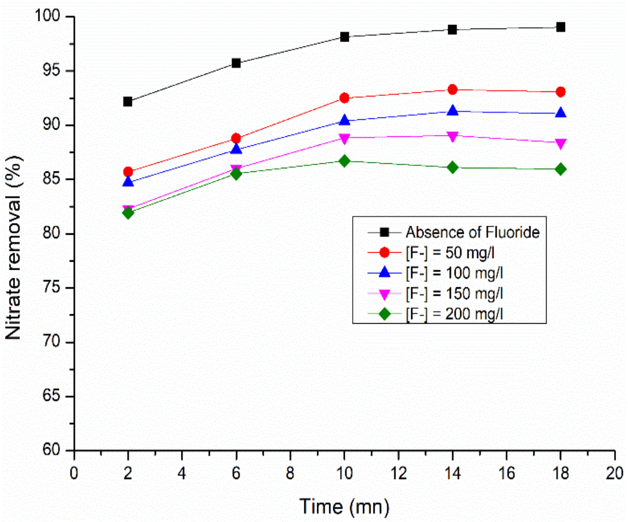

Figure 11: Impact of fluoride ions on efficiency elimination of nitrate from aqueous solution

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