



Selective nitrate removal from groundwater using a hybrid nanofiltration–reverse osmosis filtration scheme



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HIGHLIGHTS

- A hybrid NF/RO filtration scheme for nitrate removal is proposed and tested.
- Production of low salinity brines allowed to be discharged to sewerage systems.
- The new scheme can be applied in single or double NF stage modes prior to RO step.
- Appropriate NF membranes for the process should reject Cl^- better than NO_3^- .
- The results show the process to be both technically feasible and energy efficient.

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ABSTRACT

A novel and potentially cost effective filtration scheme for removal of nitrate from groundwater, characterized by production of low salinity waste brine that can be easily discharged to sewerage systems and high product-water recovery, is proposed. The inherent preference of particular NF membranes for rejecting chloride and sodium over nitrate ions is utilized in a preliminary NF stage to remove Na^+ , Cl^- , Ca^{2+} and Mg^{2+} to a side stream. In a second stage, RO is applied to remove NO_3^- and the RO permeate is mixed with the side stream of the NF stage to create product water low in nitrate, yet with a balanced composition consisting all the required species and minerals. The number of NF stages depends mainly on the rejection efficiency of the NF membrane. Based on Israeli regulations for both drinking water and required composition of brines discharged to the sewage, a treatment scheme composed of a single and double NF stages followed by RO is shown to reach water recoveries of 91.6% and 94.3%, respectively. Each NF stage raises the energy cost by approximately 0.5 cent/ m^3 product water. However, this cost is easily paid back by the inherent additional advantages of the combined scheme, i.e., less water treated by the RO, significant increase in total recovery ratio, no need in re-mineralization of the product water and minimization of calcium carbonate precipitation potential on the RO membrane. The principles for process design are described, making the specific treatment scheme proposed here easily adjustable to other regulatory requirements and other water characteristics. A provisional patent has been filed.

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1. Introduction

Increase in nitrate concentrations is observed in ground waters around the globe, mostly resulting from intensive application of fertilizers. In Israel, nitrate concentrations exceeding the 70 mg/L ($\sim 16 \text{ mg NO}_3^- \text{-N/L}$) standard is the main reason for closure of wells in the coastal aquifer, with an annual water loss of ~ 24 million m^3 . Application of advanced treatment technologies is required to reduce nitrate concentrations from 90–120 to below 60 mg/L in a cost effective and efficient fashion [1].

Advanced physical–chemical treatment techniques such as reverse osmosis (RO), ion exchange and electrodialysis are known to be effective for removing nitrates. However, all three methods produce waste concentrates (brines) containing high concentrations of nitrate, as well as other ions. In many inland places, local regulations regarding discharge of brines to the sewage system limit the application of physical–chemical technologies. In Israel, the threshold sodium and chloride concentrations for disposal to the sewage are 230 and 430 mg/L, respectively [2]. Treatment facilities installed for NO_3^- removal from drinking water in Israel are mainly based on separation by electrodialysis and RO, due to the relative simplicity and proven reliability of these methods [3]. On top of brine production, another drawback of membrane

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technologies is the potential for chemical fouling of the membrane. To minimize chemical fouling, operation with low water recovery ratios or the use of chemicals (antiscalants) is practiced [4]. Biological denitrification, the alternative treatment method, does not produce waste brine but requires an intensive post-treatment step to remove potential water contamination by organic matter and bacteria. Moreover, health concerns and public acceptance constraints limit the application of biological treatment of drinking water [5–7].

Nanofiltration (NF) is a promising technology which has been reported suitable for groundwater treatment. It is defined as a process with characteristics between RO and ultrafiltration and comprises a variety of membrane types with different retention efficiencies for either mono- and multivalent ions [8]. The main advantage of NF over RO is operation under lower pressures and higher recoveries [9]. The use of NF technology was extensively reported for water treatment processes as a sole treatment stage [8–14] or in combination with RO [15–17]. However, with respect to NO_3^- removal all of these processes, in their current development stage, are still limited by the aforementioned drawback associated with production of concentrated brines and the accompanied disposal issue.

A novel filtration scheme is presented for removal of nitrate from groundwater, accompanied by the production of low concentrated waste brine and high recovery. The methodology is based on an inherent preference of particular NF membranes for removing chloride and sodium over nitrate ions [18]. This membrane selectivity is utilized for removing a relatively high fraction of the chloride and sodium ions to a side stream in a preliminary stage prior to a main stage in which nitrate is removed by RO. The side stream of the NF stage is then mixed with the product water after nitrate has been removed from it in the RO step. As a result, the salinity of the waste brine is relatively low and can be discharged to the sewage according to local regulations. Moreover, the nitrate-rich brine contains relatively low Na^+ and Cl^- concentrations and can thus be used for irrigation purposes. The various alternatives for process design such as the NF membrane type, the number of NF stages and the recovery ratio of each filtration stage depend mainly on the planned usage of the waste brine and on the local regulations for both brine and product water. These aspects, along with other considerations related to the proposed process are exemplified in this paper on a specific nitrate-contaminated groundwater and brine discharge according to local Israeli regulations. However, the principles for process design are described in a manner enabling the suggested treatment scheme to be easily adjusted to other discharge criteria and/or water characteristics.

2. Description of the proposed treatment scheme

The combined system consists of a NF step followed by RO filtration. The NF step can be applied in single- or double-stage modes, according to the removal efficiency of the NF membrane, as discussed in Section 4. The suggested single and double NF stage treatment schemes are illustrated schematically in Figs. 1 and 2, respectively. Fig. 1 also demonstrates concentrations of Cl^- and NO_3^- -N together with percentages of feed water (Q) and pressures in the different stages of the process. These numbers are based on specific feed water characteristics (Zur Moshe well), Israeli regulations and calculations elaborated later in this work.

The NF stage serves as a selective barrier for passage of nitrate to the permeate water over the passage of other monovalent ions (e.g. chloride and sodium). It also rejects Ca^{2+} , SO_4^{2-} and HCO_3^- ions to a large extent thereby reducing the precipitation and scaling potential of (mainly) calcium-based precipitants in the following

RO stage. Additionally, it reduces the amount of water needed to be treated by RO. In the RO step, nitrate and the remaining ions are rejected and removed as brine with relatively low salinity. The concentrated stream from the NF step is mixed with the product water emerging from the RO step (i.e. RO permeate), thus re-mineralization of the product water is unnecessary.

3. Materials and methods

3.1. Experimental setup

A bench scale nanofiltration system operating in cross-flow mode with a flat sheet membrane cell was used for all membrane tests. The total membrane surface area was 48 cm^2 . Water was recirculated from the feed tank over the membrane cell with an applied inlet pressure of 8 bars and cross flow rate of 10 L/min. Water temperature was maintained constant at 24 °C. Six different NF membranes were tested: DL and DK (GE Osmonics), NF90, NF245 and NF270 (Dow) and TS80 (Trisep). For each membrane test, permeate and retentate were collected for further analysis throughout the whole process. The final NF recovery ratio was 70%. RO filtration was not tested experimentally. All experiments were carried out with real groundwater brought from the Zur Moshe well located on the coastal aquifer of Israel. The main groundwater quality parameters are shown in Table 1.

3.2. Analyses

Nitrate and chloride were measured using ion chromatography (761 Compact IC, Metrohm). Calcium, magnesium and sodium were measured with ICP-OES spectrometer (iCAP 6000 series, Thermo Fisher Scientific). Conductivity and pH were measured with standard lab conductivity and pH meter (MeterLab). Alkalinity was measured according to Standard Methods (Method 2320). Estimation of precipitation potential of minerals was done using the PHREEQC software (USGS).

3.3. Mass balance for determining the adequate recovery ratio for each filtration stage

The selection of the recovery ratio to be applied in each filtration stage is imperative for successful process design. Maximization of product water recovery, minimization of waste brine, minimization of water volume to be treated in further steps (second NF stage, if applied, and RO), salinity of the waste brine, nitrate concentration in the product water and reduction of chemicals dosing – all are strongly affected by the recovery ratio of the different filtration stages. Operating the preliminary NF stage with a minimal recovery ratio can meet most of these goals. NF with a lower recovery ratio produces less saline NF permeate, thus also reducing the salinity of both the RO feed stream and the resulting waste brine. However, operating the NF with a lower recovery ratio produces a higher volume of NF retentate contaminated with nitrate and raises the nitrate concentration in the product water. The RO recovery ratio controls both the total process recovery ratio and the TDS concentration in the final brine. Therefore, optimal recovery ratios in both the NF (first stage NF in the double NF scheme) and RO steps should be pre-determined in order to meet regulations of both product water and waste brine to be discharged to the sewage. The effect of the recovery ratio of the second NF step on the various process considerations (relevant only to the double NF scheme) is less prominent and was thus fixed at 90% during the following preliminary process design calculations.

Eq. (1) [19] can be used to assess specific ion concentrations in permeate or retentate of NF for a given recovery ratio:

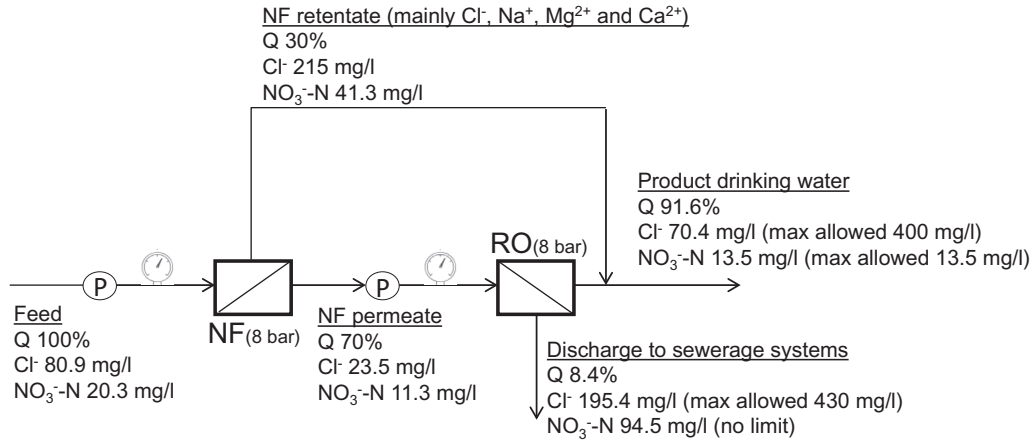


Fig. 1. Suggested treatment scheme with a single NF stage (numbers based on Zur Moshe well water and Israeli regulations).

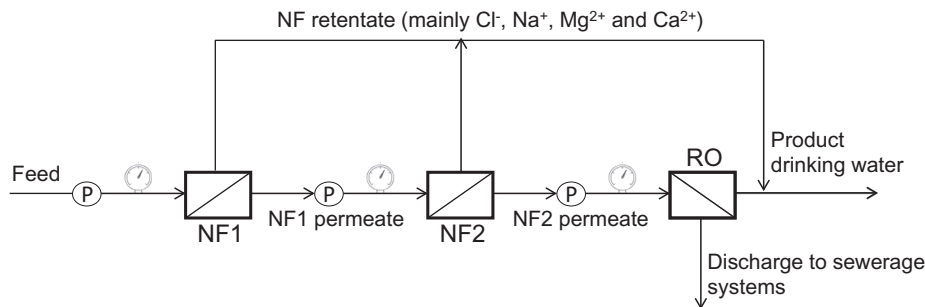


Fig. 2. Suggested treatment scheme with a double NF stage.

Table 1
Composition of source water.

Parameter	Source
pH	7.2
Conductivity [$\mu\text{S}/\text{cm}$]	774
Alkalinity [mg/L as CaCO_3]	166
NO_3^- -N [mg/L]	20.3
Cl^- [mg/L]	80.9
Na^+ [mg/L]	45.9
SO_4^{2-} [mg/L]	14.3
Ca^{2+} [mg/L]	83.7
Mg^{2+} [mg/L]	11.5

(R) was determined by measuring the initial rejection at $Y < 0.01$. In the RO step R was assumed to be 1 for all components. Determination of the final set of operation conditions was carried out based on the calculated results for the NF step. Finally, the recovery ratio of each filtration stage was determined according to the Israeli nitrate drinking water regulations (i.e. maximum of 13.5 mg NO_3^- -N/L) and the maximal chloride and sodium concentrations in brines discharged to the sewage (430 and 230 mg/L, respectively).

4. Results and discussion

The results presented in this section include the following: (a) Results from preliminary experiments aimed at selecting NF membranes with preference to chloride and sodium rejection over nitrate; (b) Validation of the mass balance equation based on experimental results using the selected NF membranes at different recovery ratios; (c) Application of the mass balance equation for determining the permeate and brine composition as a function of NF recovery ratio at different RO recovery ratios, and selection of the most suitable recovery ratios to meet the Israeli regulations.

4.1. Preliminary NF membrane tests

Six NF membranes were tested in order to assess their selectivity for nitrate and chloride rejection. The results are shown in Fig. 3.

Three of the membranes showed better rejection for chloride. The combination of high chloride rejection with a reasonable selectivity ratio was observed specifically for TS80. Lower rejections of both ions, yet with prominent selectivity for chloride removal, were attained by NF270. Accordingly, it was decided to further

$$\frac{C_P}{C_F} = \frac{1 - (1 - Y)^{1-R}}{Y} \quad (1)$$

where C_P and C_F are the concentrations of a specific ion in permeate and feed, respectively, R is the local rejection and Y is the corresponding recovery ratio. Eq. (1) was suggested for rough estimations since the osmotic feed pressure does not increase significantly with the increase in recovery in NF processes (for solutions dominated by monovalent ions, namely Na^+ and Cl^-). Therefore, a constant flux can be roughly assumed for all recoveries and the rejection in the computed segment (i.e. the local rejection R) remains also approximately constant throughout the process. Having said this, validation of the suitability of Eq. (1) for design purposes is needed.

Based on the above considerations (along with technical constraints), a maximal (relatively low) recovery ratio of 70% was selected for the first NF stage. Experimental results (measured concentrations of specific ions in the permeate water at different recoveries) were used for validating Eq. (1). The local rejection

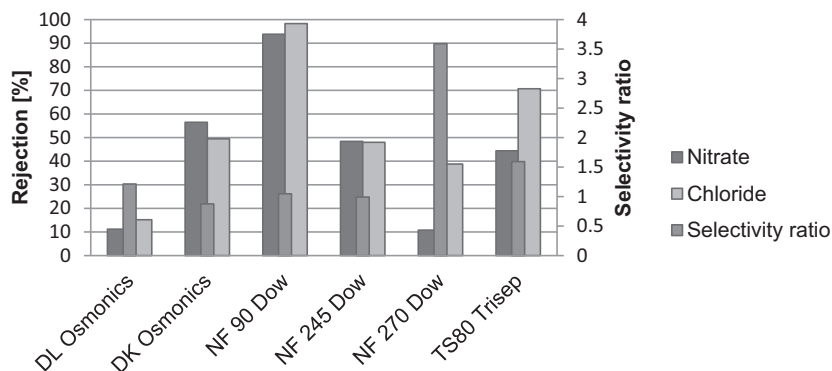


Fig. 3. Final rejections (70% recovery) of nitrate and chloride for different NF membranes and the corresponding chloride rejection: nitrate rejection selectivity ratio.

evaluate the suitability of TS80 and NF270 for a filtration scheme consisting of single and double NF stages, respectively. However, reevaluation of removal capacity of NF membranes should be considered for different composition of source water.

4.2. Validation of the mass balance equation

The initial ($Y \approx 0.01$), middle ($Y \approx 0.35$) and final ($Y = 0.7$) permeate concentrations of nitrate, chloride, sodium and calcium for the single and double NF experiments were measured and compared to the calculated concentrations based on Eq. (1). In general, the higher the recovery ratio was, the higher were the deviations between measured and calculated values. Negligible deviations were obtained at $Y = 0.01$ and $Y = 0.35$ (data not shown). Figs. 4 and 5(A) and (B) present the measured and the calculated ion concentrations at $Y = 0.7$ for the single and double NF experiments, respectively.

The maximal deviation between measured and calculated ion concentrations in the final permeate was lower than 10%. These minor deviations corroborate the use of the Eq. (1) for predicting the ion concentrations throughout the recovery ratio range.

4.3. Determination of the recovery ratio at each stage using Eq. (1)

Concentrations of relevant ions (NO_3^- , Cl^- , Na^+) at different stages of the process as a function of the recovery ratio were assessed using Eq. (1) for both the single and the double NF filtration scheme. RO tests were not performed in this work, so that the assumption of full rejection ($R = 1$) for all components in RO stage must be carefully taken into account in future calculations. The calculated NO_3^- -N concentrations are shown for the product water, while those of Cl^- and Na^+ are shown for the waste brine.

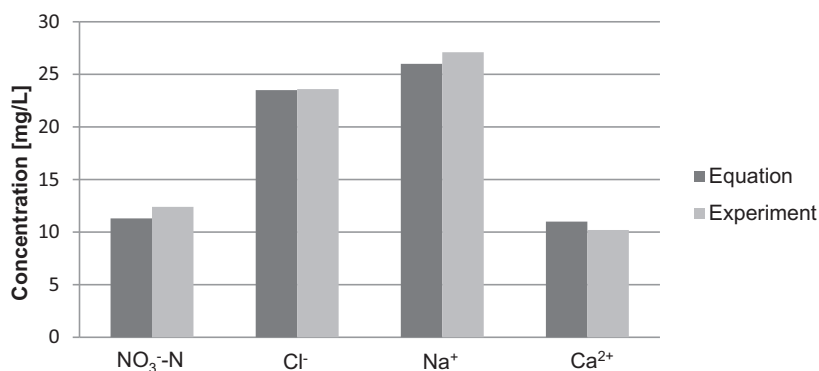


Fig. 4. Comparison between measured and calculated (Eq. (1)) ion concentrations of for the single NF using TS80.

4.3.1. Single NF scheme with TS80

Fig. 6 presents nitrate concentration in the product water as a function of the NF and RO recovery ratios.

At higher NF recoveries, higher volumes of nitrate-rich water are treated in the RO step, resulting in lower nitrate concentration in the product water. The higher the RO recovery, the higher is the dilution of the product water by the nitrate-free RO permeate. As shown in Fig. 6, the minimal recovery that can be applied for NF in order to meet the drinking water standards of 13.5 mg NO_3^- -N/L, together with reasonable and high RO recovery, is 69% (correlates with 90% recovery of RO).

Figs. 7 and 8 present chloride and sodium concentrations in the brine discharged to sewage, as a function of the recovery ratio in the NF step at different RO recovery ratios.

As expected, operation of NF1 at higher recovery ratios produces RO brine with higher salt concentrations. At higher recovery ratios, the NF permeate is more concentrated, thus raising also the TDS concentration in the feed stream to the RO step and its corresponding brine. The RO recovery ratio affects directly the brine's salinity by a dilution factor. As shown in Fig. 8, the limitation of 230 mg Na^+ /L for discharge to the sewer limits RO recovery to a maximum of 88%. Higher RO recoveries do not meet this standard under the relevant NF recoveries above 69%. Chloride concentrations are lower than the standard (430 mg Cl^- /L) and therefore do not limit the value of RO recovery. However, operation at 88% RO recovery requires a minimal NF recovery of 70% in order to meet product water nitrate concentrations meeting the Israeli standard (Fig. 6). Therefore, recoveries of 70% and 88% for NF and RO, respectively, are adequate for the Israeli regulations, resulting in maximal overall water recovery, minimum water volume to be treated in the RO step and minimum chemical dosing to eliminate scaling. The total recovery ratio obtained with the above recovery ratios is 91.6% (Fig. 9).

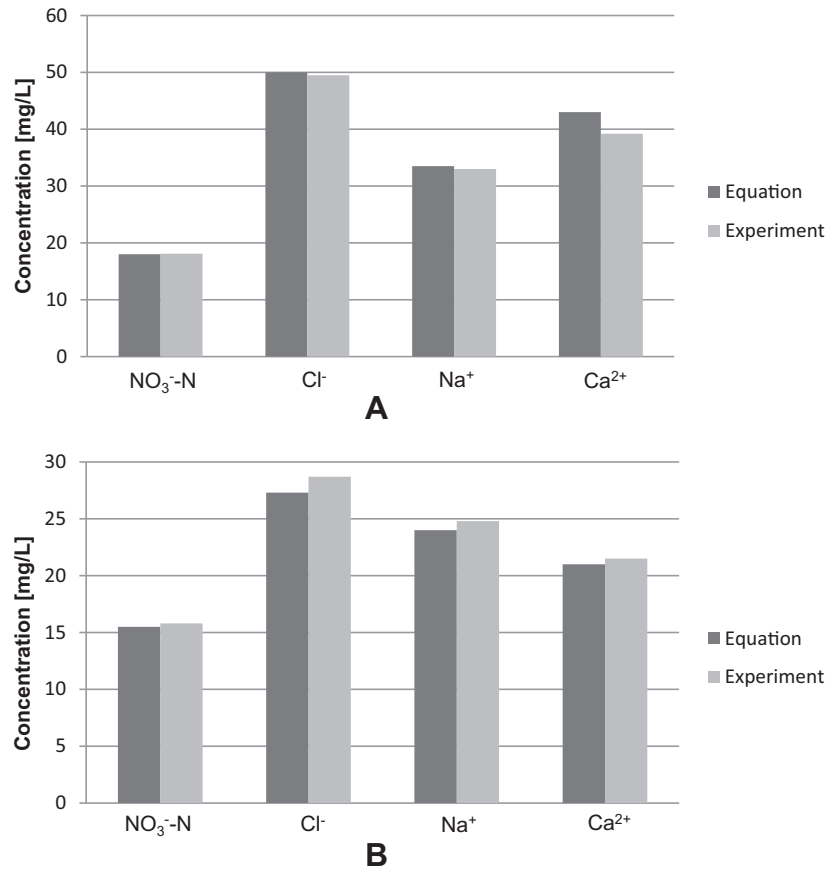


Fig. 5. Comparison between measured and calculated (Eq. (1)) ions concentrations for first stage NF filtration (A) and second stage NF filtration (B) using NF270.

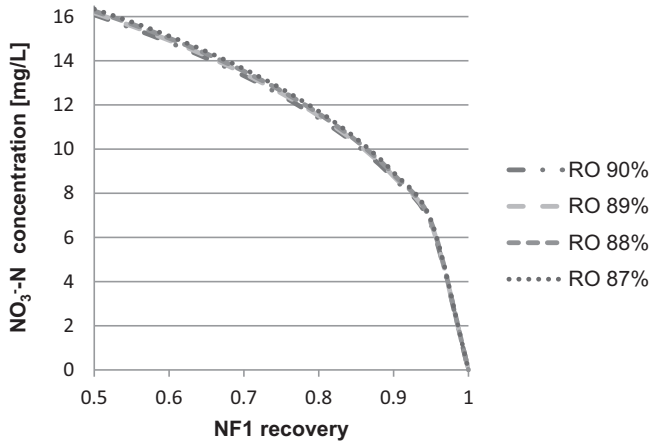


Fig. 6. Nitrate concentration in product water as a function of NF recovery ratio at different RO recovery ratios.

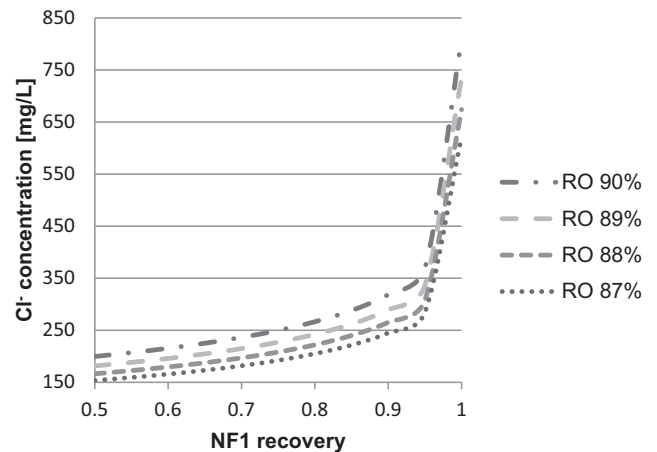


Fig. 7. Chloride concentrations in the brine discharged to the sewage as a function of NF recovery ratio at different RO recovery ratios.

4.3.2. Double NF scheme with NF270

A similar procedure for determining recovery ratios was applied for the double NF scheme using NF270. In general, the trends were similar to those observed in the single NF experiment. However, the lower rejection efficiencies of the NF270 membrane led to less concentrated NF1 retentate and more concentrated NF1 permeate at a given recovery. Therefore, the concentration of nitrate in the product water decreased, the level of both chloride and sodium in the waste brine increased and the recovery of NF1 had, thus, to be reduced.

The adequate recoveries for the Israeli regulations were 53%, 90% and 88% for NF1, NF2 and RO, respectively. Calculated nitrate-N concentration in product and chloride and sodium concentrations in waste brine with the above recoveries were 13.5, 257 and 216 mg/L, respectively. The total recovery was 94.3%.

4.4. Calcium carbonate precipitation and assessment of chemical addition

Calcium carbonate precipitation can occur at the different stages of the process. Magnesium and sulfate concentrations were

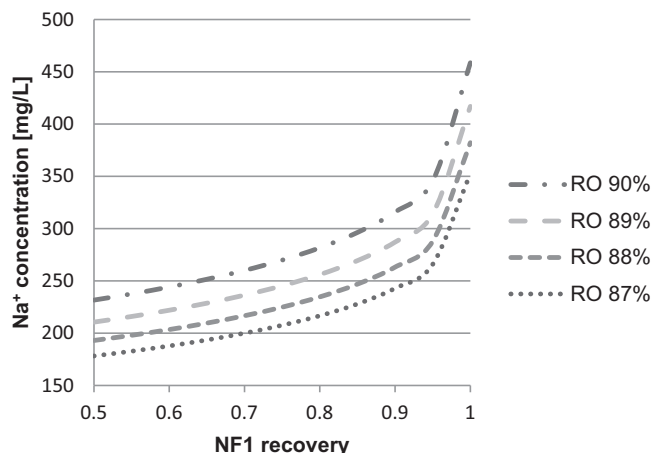


Fig. 8. Sodium concentration in brine discharged to the sewage, as a function of NF recovery at different RO recoveries.

too low for inducing precipitation (Table 1). Fig. 10 presents the calcium concentration in the NF and RO retentates as a function of the NF recovery for the single NF scheme. Fig. 11 presents calcium concentration in the retentate of both NF and RO stages as a function of the recovery of the NF1 for the double NF scheme. RO recovery was set at 88% as found to be an optimum in the previous sections for both filtration schemes.

In general, the calcium concentration in the retentate of NF1 depends positively on the recovery of NF1 due to a dilution factor. The calcium concentration in the retentate of NF2 and RO depends also positively on NF1's recovery due to the formation of higher concentrated permeates at the higher NF recoveries. Each permeate serves as the feed stream for the next filtration stage, thus also affecting positively the corresponding retentate of the specific stage. At the relevant NF1 recovery range determined in the previous section (i.e. 50–70%), the highest precipitation potential was calculated in RO retentate for the double NF scheme with the corresponding assessed parameters of pH 8.14, alkalinity 250 mg/L (as calcium carbonate) and calcium 213 mg/L. For this case, the CaCO₃ precipitation potential calculated was 56 mg/L. The scaling potential can be minimized by the addition of a small amount of antiscalant. Due to the difficulty of the addition of antiscalants to drinking water, a similar effect can be achieved by lowering source water pH from 7.2 to 6.7 by the addition of 0.5 mM HCl or 0.25 mM H₂SO₄. The addition of chlorides or sulfates in the process is negligible. Similar acid concentration is required in the single NF scheme.

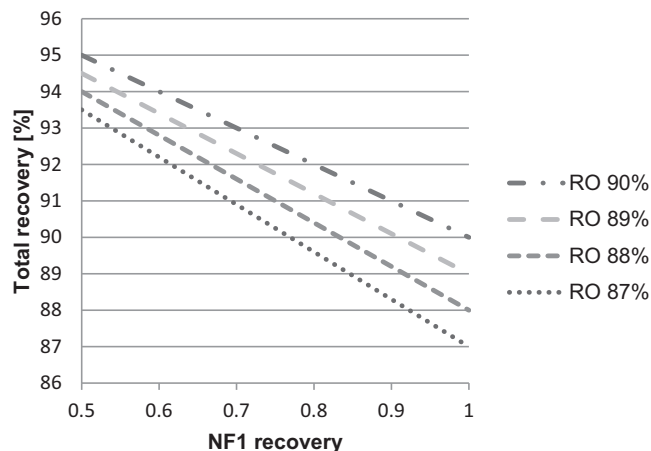


Fig. 9. Total recovery as a function of NF recovery for different RO recoveries.

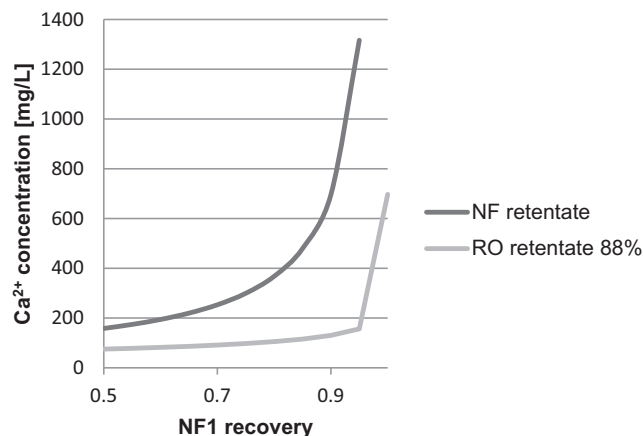


Fig. 10. Calcium concentration in the retentates of the NF and the RO (at 88% recovery) stages as a function of NF recovery, for the single NF scheme.

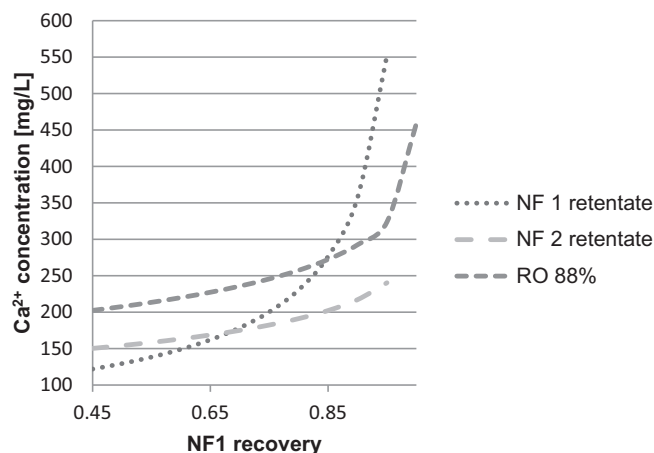


Fig. 11. Calcium concentration in the retentates of NF1, NF2 and RO (at 88% recovery) stages as a function of NF1 recovery, for the double NF scheme.

Table 2

Estimations of energy consumption and energy cost for the 3 treatment alternatives based on membrane filtration.

Treatment scheme	RO	Single NF + RO	Double NF + RO
Stage	RO	NF RO	NF1 NF2 RO
Stage recoveries [%]	80	70 88	53 90 88
Total recovery [%]	80	91.6	94.3
Pressure [bar]	10	8 8	8 8 8
Energy consumption [kWh/m ³ product]	0.46	0.55	0.63
Energy cost [cent/m ³ product]	2.76	3.3	3.78

4.5. Comparison of energy requirements in different filtration schemes

Table 2 presents rough estimations of energy consumption and energy cost for the 3 treatment alternatives: RO, single NF followed by RO and double NF followed by RO. Recoveries and pressures for the combined NF–RO schemes are based on the calculations above. In the case of RO as a sole treatment, recovery and pressure were chosen so that the process will meet the local Israeli standards for drinking water and wastewater.

Each NF stage increases the energy cost by approximately 0.5 cent/m³ product water. However, the addition of single and double NF stage increases significantly the total recovery and minimizes waste brine volume by a factor higher than 2 and 3, respectively, on top of much lower brine salinity.

5. Conclusions

A novel and potentially cost effective filtration scheme for removal of nitrate from groundwater, characterized by production of low salinity waste brine that can be easily discharged to sewerage systems and high product-water recovery, was investigated. The system consists of a NF step followed by RO filtration. Several nanofiltration membranes have shown their effectiveness for preferable removal of chloride and sodium over nitrate. This selectivity is utilized here to separate nitrate from other ions in a preliminary groundwater filtration stage. The nitrate-rich NF permeate is then filtrated by RO in a following stage to produce a salt-free permeate together with a waste brine with low salinity. The monovalent ions and the essential minerals removed in the NF step are mixed again with the RO permeate, making remineralization of product water unnecessary and minimizing scaling potential on the membranes. Two schemes for treating nitrate-contaminated groundwater with brine discharge to sewer are suggested in this work. High total recoveries of 91.6% and 94.3% can be achieved for the single and double NF scheme, respectively. CaCO_3 precipitation potential is low for both cases and can be removed completely by reasonable acid dosing.

The suggested scheme is based on a “fit to purpose” concept that links the quality of the treated water, the applied technology and the alternative destinations for waste brine such as direct disposal to the sewage, denitrification followed by disposal to the sewage or irrigation. The current work describes a specific treatment scheme for nitrate-contaminated groundwater with direct brine discharge to the sewage based on local regulations in Israel. However, the principles for the process design are set, so that the suggested treatment scheme can be easily adjusted to other demands.

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