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Experimental determination of nanofiltration models: application to nitrate removal

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ABSTRACT

Nowadays, nitrate contamination of groundwater is a growing problem both in developed and developing countries. Such contamination comes mainly from the intensive use of nitrogen fertilizers in agriculture. Other possible sources of water contamination by nitrates are improper discharges of industrial effluents, the inadequate management of leachate from landfill of municipal solid waste and effluents from intensive farming. The maximum permissible concentration of nitrates in the water for public consumption in the EU countries is 50 mg/L, although the World Health Organization established a value of 25 mg/L to be considered a quality water. This problem of nitrate contamination, widespread in countries of the European Union, is very important in Spain, where there are some regions with a high presence of aquifers with nitrate concentrations significantly higher than the established limits. Among the possible treatments for nitrate removal from contaminated waters, nanofiltration (NF) is a technically proven process that eliminates nitrates without causing a significant imbalance in the rest of dissolved salts. In this work, the removal of nitrates from water using a commercial NF membrane is studied. Synthetic feeds with levels of nitrate concentration higher than the ones usually found in contaminated aquifers have been considered. On the other hand, the experimental results have been fitted to the solution-diffusion model, having found mathematical expressions that can be used to predict membrane performance for feed concentrations higher than 100 mg/L. The model considers the concentration on the surface of the membrane, estimated by the film theory model. It has been proved that concentration polarization cannot be neglected for this application. The tested membrane allows nitrate decontamination of water (concentration below the legal limit) in one pass for values of feed concentration up to 100 mg/L, with a transmembrane pressure of 5 bar.

Keywords: Nanofiltration; Nitrate removal; Experimental results; Solution-diffusion model

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1. Introduction

Nanofiltration (NF) has increased its potential applications in last years, thanks to some advantages against reverse osmosis such as higher flux, which means lower energy consumption, and lower rejection of monovalent ions but high rejection of divalent ions, thus allowing fractionation of solutes, which is one of the reasons of the commercial growth of this process [1]. As a result, NF is widely applied at present in drinking water treatment, industrial effluent treatment and water processing for some industries such as food and beverage ones. In the area of drinking water production, the major application of NF is softening, but it is also usually applied for the removal of NOM, micropollutants, viruses and bacteria, nitrates or arsenic, or for partial desalination [2]. This paper is focused on the application of NF in drinking water production, specifically in the removal of particular pollutants as nitrates.

1.1. Nitrate contamination and removal

Due to its high water solubility, nitrate is one of the most widespread groundwater contaminant in the world. The increase of nitrate levels in groundwater can be linked to human activities, especially the intensive use of fertilizers in agriculture. This problem of nitrate contamination is quite significant in aquifers in the Mediterranean area of Spain, where the extensive use of fertilizers results in high levels of nitrates in groundwater. In 2010, between 20 and 30% of the aquifers in this area showed nitrate concentration higher than 50 mg/L, according to data of the Ministry of Agriculture, Food and Environment of Spain.

High nitrate concentrations stimulate algal growth thus promoting eutrophication [3]. But nitrate also produces negative health effects in human body when water concentration is excessive, due to nitrate reduction to nitrites which can cause methaemoglobinaemia in infants and certain types of cancers associated to nitrosamine formation. For this reason, the maximum permissible concentration of nitrates in the water for public consumption is normally limited by legislation. In the EU countries, nitrate concentration in water is limited to 50 mg/L, although the quality criterion established by the World Health Organization (WHO) is 25 mg/L.

Conventional processes used for nitrate removal in drinking water are ion exchange, biological denitrification and electrodialysis, but they are rather complex to perform. NF has been proved as an alternative for nitrate removal since rejection of this ion can be enough to achieve suitable quality for drinking water [4]. Besides, NF would allow at the same time reducing water hardness, as well as pesticides removal [5]. Simultaneous removal of nitrate and pesticides is very interesting since nitrate concentrations are often high in regions where pesticide contamination is also a problem [6]. Other NF advantages in comparison with other processes are: ease of operation, no chemicals are required, reliability, modular and it is easy to scale up [7].

In this paper, NF has been tested for the removal of nitrates with the aim to determine the level of feed nitrate concentration below which the membrane could produce permeate with nitrate concentration below 50 mg/L, with the lowest energy consumption. Thus, the membrane selected should have high permeate flux and the operation pressure should be as low as possible.

1.2. Nanofiltration models for nitrate removal

In a NF process, solutes are rejected by the combination of three mechanisms: size effect (as in ultrafiltration), ion interaction with the charged membrane material and solution diffusion of solutes through the membrane material (as in reverse osmosis).

There are several models currently available to describe mass transfer through a NF membrane. Phenomenological equations, such as Kedem and Spiegler model, assume the membrane as a black box, so they do not offer description of the ion transport. There are some applications in which this model successfully predicts membrane performance [8]. However, this model has to be used with care since it does not consider electrostatic potential of the membrane and it cannot explain solute rejections at low concentrations, as some experimental results show [9]. Wang et al. used this model with success to estimate the separation of three different nitrate salts by NF [10].

Other approaches to explain NF mechanisms include ion transport through pores by means of electrokinetic space-charge modelling, such as Tsuru, Nakao and Kimura models, and the Donnan-Steric Partitioning Pore Model (DSPM) proposed by Bowen et al. [8]. A modification of this last model can be found in the work of Santafé-Moros et al. who developed a DSPM model including dielectric exclusion, which allows describing NF performance for some ternary ionic systems including nitrates [11]. Although these models have described NF performance in certain applications, their usefulness is limited by the complexity of numerical calculations.

Another alternative approach is the solution-diffusion model. Although this model has been predominantly used for reverse osmosis, it has also been applied for modelling NF with successful results [12,13]. Solution-diffusion model has been mainly used for predicting transport through NF membranes of the following species: organic solvents [14], metals such as copper [12] and ionic salts such as NaCl and $CaSO_4$ [15]. However, no reference has been found about the application of this model to study nitrate removal by NF.

Besides being less complex than other models, solution-diffusion model has resulted in suitable predictions when convective transport through NF membrane is very weak [15], as it is the case of the commercial membrane used in this work. For all these reasons, we have chosen the solution-diffusion model to fit experimental results of nitrate removal by NF.

1.3. Solution-diffusion model

The expressions used for permeate flux (J_v) and solute flux (J_S) through the membrane have been:

$$J_V = A \cdot (\Delta P - \Delta\pi) \tag{1}$$

$$J_S = B \cdot \Delta C \tag{2}$$

where A is the permeability parameter of the solvent and B is the permeability parameter of the solute; ΔP is the transmembrane pressure; and $\Delta\pi$ and ΔC are the difference of osmotic pressure and solute concentration across the membrane, respectively. Osmotic pressure has been calculated with Van't Hoff equation. To calculate the osmotic pressure and the nitrate concentration in the feed side of the membrane, we have estimated the concentration of the solute at the membrane surface on the feed side (C_m). According to film theory model, this concentration can be estimated by the following expression:

$$C_m = C_f + (C_f - C_P) \cdot \exp\,(J_V/k) \tag{3}$$

where C_f and C_P are the concentration of solute in feed and permeate, respectively; J_v is the volumetric flux, and k is the mass transfer coefficient, which can be determined by empirical expressions such as:

$$Sh = a\ Re^b\ Sc^c \tag{4}$$

where Sh, Re and Sc are the Sherwood, Reynolds and Schmidt numbers, respectively; and a, b and c the empirical constants.

The dimensionless numbers Re and Sc can be calculated by the following equations:

$$Re = \frac{\rho \cdot v \cdot d_h}{\mu} \tag{5}$$

$$Sc = \frac{\mu}{\rho \cdot D} \tag{6}$$

where ρ and μ are the density and the dynamic viscosity of the solution, respectively; v is the linear velocity, d_h the hydraulic diameter and D the diffusivity of the solute.

With these dimensionless numbers and the proper correlation (Eq. (4)), the Sherwood number can be obtained by Eq. (7) and then the value of the mass transfer coefficient, k:

$$Sh = \frac{k}{d_h \cdot D} \tag{7}$$

Once the k coefficient is determined, C_m can be estimated by Eq. (3) and it would allow calculating the real rejection of the membrane (R) by the following expression:

$$R = \frac{C_m - C_P}{C_m} \tag{8}$$

and compare values with observed rejection ones (R_o), calculated by the conventional expression:

$$R_o = \frac{C_f - C_P}{C_f} \tag{9}$$

The comparison of these two rejection parameters would allow to check if the polarization layer must be considered in the model, as some authors suggest [16].

2. Materials and methods

2.1. Nanofiltration membrane

There are some works on nitrate removal by NF membranes in which different commercial membranes have been proved to be very suitable for this application [4,5,7]. Among the tested membranes, NF270 manufactured by Dow Chemical has been used by different authors for removing several contaminants, showing some features that make this membrane a very efficient alternative in some applications. NF270 has higher resistance against fouling, exhibiting high flux stability [17]. This membrane has shown lower rejection values than other membranes for both monovalent (NO_3^-) and bivalent ions (Ca^{2+}, Mg^{2+}) [18], so it can be suitable for removing nitrate when concentration in water is not much higher than the standard limit (50 mg/L). Since it keeps STD relatively high, it requires lower remineralization of permeate, as it was suggested by Van der Bruggen et al. [7]. Furthermore, NF270 shows especially higher permeability in comparison to other NF membranes [4,18,19], which promises a decrease in energy consumption. As this work is intended to investigate decontamination possibilities of a membrane with the lowest energy consumption, NF270 was selected due to the higher permeated flux produced.

2.2. Pilot plant

Fig. 1 shows a schematic diagram of the pilot plant used in the experiments. Feed water was pumped from the feed tank (1) through a pressure pump (3) to the membrane module (6). The pilot plant was equipped with a 2540 spiral-wound NF270 NF membrane. It is a composite polyamide membrane with an active area of 2.6 m^2. Both permeate and concentrate were recycled back to the feed tank, working in a total recirculation mode. Experiments were performed at a temperature ranging from 22 to 25°C, using a cooler (2) to keep temperature under 25°C. Feed pressure was fixed by the regulation valve (8).

2.3. Experimental procedure

Experimental tests with osmotized feed water (conductivity around 15 μS/cm) were performed in a range of pressure from 5 to 12.5 bar, and with a constant feed flow of 650 L/h. Each test lasted about 2 h, measuring permeate flow (Q_p) every 15 min until steady state was reached. Permeate flow was determined by measuring the volume of permeate collected in a given time interval. The volumetric flux J_v was then calculated by the expression:

$$J_v = \frac{Q_p}{A_{memb}} \tag{10}$$

where A_{memb} represents the active area of the membrane.

Experimental tests with nitrate solutions were also performed. Feed solutions were prepared using sodium nitrate salt ($NaNO_3$) from Panreac, with concentration ranging from 35 to 250 mg/L. Transmembrane pressure was fixed at a value of 5 bar, and feed flow was again set at 650 L/h. Each test lasted between 3 and 4 h. Permeate flow and conductivity of each stream were measured every 15 min until steady state was reached. The volumetric flux J_v was determined by Eq. (10) after measuring the permeate flow as in the previous test. Conductivity was measured in permeate, feed and concentrate streams to calculate the respective nitrate concentrations by a calibration curve. With values of feed (C_f) and permeate (C_p) concentration, observed rejection values were calculated by Eq. (9).

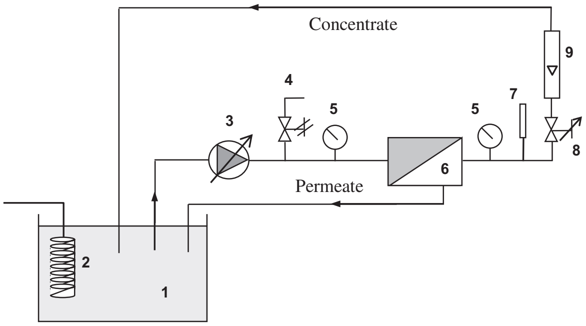

Fig. 1. Schematic diagram of the NF pilot plant.
Notes: (1) Feed tank, (2) Cooler, (3) pressure pump, (4) safety valve, (5) manometer, (6) membrane module, (7) thermometer, (8) regulation valve and (9) rotameter.

3. Results and discussion

3.1. Nitrate removal

As it has been explained in the previous section, experiments lasted until steady state was reached, both in permeate flux and permeate nitrate concentration values. Steady values for both parameters are shown in Table 1 for each experimental test.

As it can be seen in the table, flux decreased when feed concentration was higher. Anyway, flux decrease was not too high, the value for the highest tested concentration (245 mg/L) being 55.2 L/(m^2 h), which is around 13% lower than the value for the lowest tested

Table 1
Experimental results of nitrate removal by NF270 membrane

C_f (mg/L)	J_v (L/m^2 h)	C_p (mg/L)
35	62.9	8.7
60	60.2	22.5
100	59.0	50.4
190	60.3	106.2
245	55.2	135.7

concentration (35 mg/L) which is 63 L/(m^2 h), approximately. This decrease in flux values can be associated to the effect of osmotic pressure and to the effect of concentration polarization layer.

With regard to nitrate concentration in permeate, it can be seen from the table that, when feed concentration was lower than 60 mg/L, it was lower than the WHO quality standard (25 mg/L), and when feed concentration was of 100 mg/L, it was very close to the legal limit in EU (50 mg/L). According to these values, it can be stated that the tested membrane would be able to produce a permeate with a nitrate concentration below the legal limit of 50 mg/L when feed water shows a nitrate concentration up to 100 mg/L. Up to 60 mg/L, the permeate had a level of nitrate concentration even below the quality standard. For values of nitrate concentration in the feed water higher than 100 mg/L, more passes of NF to achieve the legal concentration would be necessary.

3.2. *Influence of membrane concentration*

For the estimation of the mass transfer coefficient, we used the correlation obtained by Schock and Miquel for commercial spacers [20]:

$$Sh = 0.065\ Re^{0.875}\ Sc^{0.25} \tag{11}$$

To calculate dimensionless numbers, we used values of density and dynamic viscosity for water at the average temperature of the experimental tests (25˚C), as feed solution can be considered as dilute since nitrate concentration was below 250 ppm. The value of diffusivity of nitrate in water for was taken from literature [21] for very low concentrations (0.005 M).

To calculate velocity, physical dimensions from the technical sheet of the NF270 membrane were used, and the hydraulic diameter was estimated as 2 h, *h* being the height of the channel of the spiral-wound module.

As a result, calculated values of Reynolds and Schmidt numbers were 256–631, respectively. These values resulted in a Sherwood number of 41.7, and finally the mass transfer coefficient was of 4.5×10^{-5} m/s, which is in agreement with values of k coefficients for spiral-wound modules [22]. This value of k was used to predict C_m values by Eq. (3), according to thin film theory.

Table 2 shows the results of C_m, and observed (R_o) and real rejection (R) values, calculated by Eqs. (8) and (9), respectively.

Table 2
Observed and real rejection values for nitrate removal with NF270 membrane

C_f (mg/L)	C_p (mg/L)	R_o (%)	C_m (mg/L)	R (%)
35	8.7	75.1	51.8	83.2
60	22.5	62.5	82.6	72.8
100	50.4	49.6	128.6	60.8
190	106.2	44.1	239.0	55.6
245	135.7	44.6	304.4	55.4

With regard to observed rejection values, it can be seen that it was around 75% for the lowest tested concentration (35 mg/L). For a nitrate concentration in the feed about 60 mg/L, retention index decreased to a value slightly higher than 60%; and for higher nitrate concentrations, removal percentages were around 44–50%. These results are in accordance with the type of nitrate salt used in the experiments. As a salt of type 1:1 ($NaNO_3$) was used, the retention decreases when the concentration increases. This is due to the decrease in the repulsion between the membrane and the nitrate ions because sodium cations neutralize the membrane charge [5,23].

As it can be seen in the table, values of C_m are significantly higher (>25% for all cases) than measured feed concentration. This means that there is a concentration polarization boundary layer that resists the permeating process. As a consequence, estimated values of real rejection are also higher than observed ones. Fig. 2 shows how values of rejection vary with feed concentration. The difference between observed

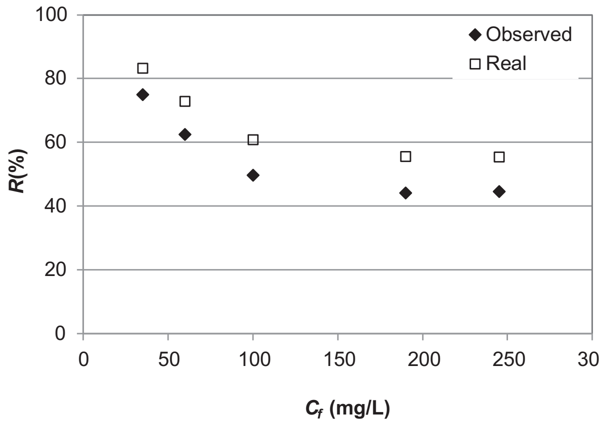

Fig. 2. Evolution of observed and real rejection values with feed concentration.

and real rejection values increases from 10 to 25%, approximately, as feed concentration becomes higher. This shows that concentration polarization increases with feed concentration and it cannot be neglected in the prediction of membrane performance. Therefore, the parameters of the solution-diffusion model have been determined by considering estimated values of C_m in the feed side of the membrane.

3.3. Solution-diffusion model

Fig. 3 shows the evolution of volumetric flux with transmembrane pressure (a) and the evolution of solute flux with concentration difference across the membrane (b). The linear regressions from these evolutions allow determining the parameters of the solution-diffusion model. According to Eq. (1), from the first representation (a), the permeability parameter of the solvent (A) has been determined with a mean-squared regression value of 0.994. This parameter has resulted in a value of 11.042 ($\mathrm{L\,m^{-2}\,h^{-1}\,bar^{-1}}$). From the other representation (*b*), and according to Eq. (2), the value of the permeability parameter for the solute (B) has resulted to be 42.52 ($\mathrm{L\,m^{-2}\,h^{-1}}$) with a mean-squared value of 0.896. This last regression coefficient has not been as good as expected due to the low values of J_S at low feed concentrations, which may be subjected to experimental error due to the low values of conductivity measured.

Therefore, it can be stated that the equation of the solution-diffusion model for volumetric flux fits better with experimental results than the equation of solute flux, which does not show linear performance for feed concentration below 100 ppm.

Finally, the solution-diffusion model has been validated by comparing experimental and model results of the volumetric flux and the permeate concentration. Results of these estimations are shown in Figs. 4 and 5.

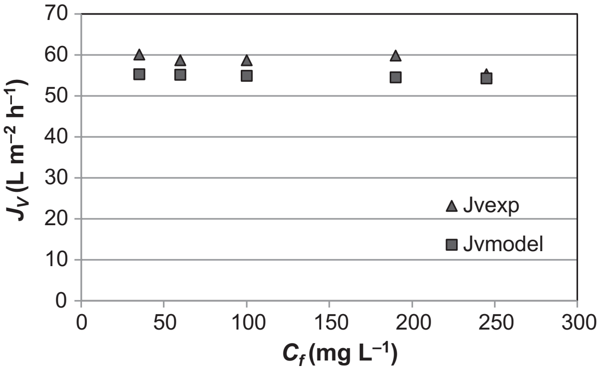

Fig. 4. Comparison of experimental and predicted values of volumetric flux for different feed concentrations.

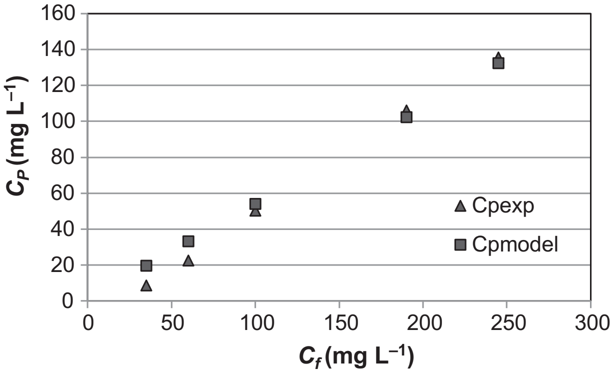

Fig. 5. Comparison of experimental and predicted values of permeate concentration for different feed concentrations.

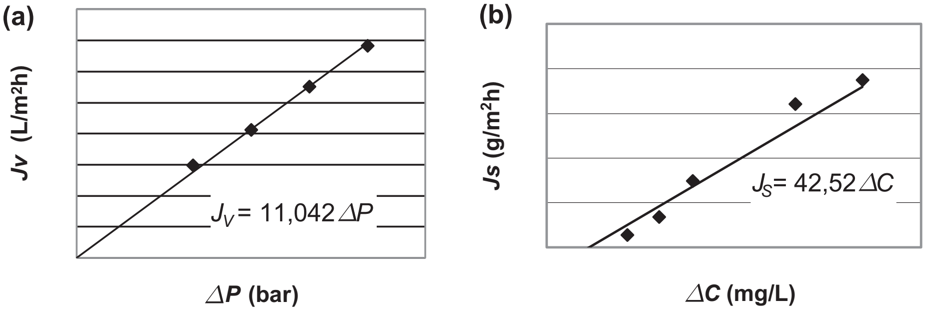

Fig. 3. Regression of solution-diffusion parameters: (a) permeability parameter of the solvent and (b) permeability parameter of the solute.

In relation to volumetric flux, it is shown in Fig. 4 that predicted values are below experimental ones for almost all feed concentrations. Anyway, differences in values are not too high (around 6–8% in most cases). So, it seems that the solution-diffusion model can explain quite satisfactorily the permeate flux through the NF270 membrane when removing nitrates for feed concentration below 250 ppm.

With regard to permeate concentration, Fig. 5 shows the similar trend of these values to increase with feed concentration. Predicted and experimental concentrations become closer as feed concentration increases above 100 mg/L. Differences for low feed concentrations (below 100 ppm) are quite significant, probably due to imprecision on the measurement of very low values of conductivity. Anyway, the determined solution-diffusion model could be used to predict permeate concentration for feed concentrations higher than 60 mg/L, since experimental values are quite close to theoretical ones (relative error below 8%).

4. Conclusions

In this work, NF270 membrane has been tested for nitrate removal. Taking into account it is a low rejection membrane for monovalent ions, but it produces higher permeate flux than other NF membranes, the following conclusions have been obtained:

(1) The tested membrane seems suitable for nitrate decontamination up to 100 mg/L of nitrate in the feed water. Further studies should be performed with real water as the interactions with other substances can modify nitrate removal by means of the membrane.

(2) Concentration polarization cannot be neglected in this process since values of estimated concentration at the membrane surface are significantly higher than nitrate concentrations measured in the feed tank.

(3) The parameters of the solution-diffusion model for the removal of nitrates between 35 and 245 mg/L of feed concentration have been obtained experimentally. The equations of the model with the parameters obtained in this work could be used to predict volumetric flux and nitrate concentrations in permeate for feed concentration values higher than 60 mg/L, with errors below 8%.

Nomenclature

A — permeability coefficient of the solvent ($\mathrm{L\ m^{-2}\ h^{-1}\ bar^{-1}}$)
A_{memb} — effective membrane area ($\mathrm{m^2}$)
B — permeability coefficient of the solute ($\mathrm{L\ m^{-2}\ h^{-1}}$)
C_{f} — feed concentration ($\mathrm{mg\ L^{-1}}$)
C_{m} — concentration of solute at the membrane surface ($\mathrm{mg\ L^{-1}}$)
C_{P} — permeate concentration ($\mathrm{mg\ L^{-1}}$)
D — diffusion coefficient ($\mathrm{m^2\ s^{-1}}$)
d_{h} — hydraulic diameter (m)
h — channel height (m)
J_{v} — volumetric permeate flux ($\mathrm{L\ m^{-2}\ h^{-1}}$)
J_{S} — solute flux ($\mathrm{g\ m^{-2}\ h^{-1}}$)
k — mass transfer coefficient (m/h)
Q_{P} — permeate flow ($\mathrm{L\ h^{-1}}$)
R — real rejection (%)
R_{o} — observed rejection (%)
Re — Reynolds number (dimensionless)
Sc — Schmidt number (dimensionless)
Sh — Sherwood number (dimensionless)
μ — dynamic viscosity ($\mathrm{kg\ m^{-1}\ s^{-1}}$)
ρ — density ($\mathrm{kg\ m^{-3}}$)
ΔC — difference of concentration between both sides of membrane ($\mathrm{mg\ L^{-1}}$)
ΔP — transmembrane pressure (bar)
$\Delta\pi$ — difference of osmotic pressure between both sides of membrane (bar)

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