Desalination and Water Treatment
www.deswater.com

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doi: 10/5004/dwt.2010.1343

23 (2010) 3–12
November

Effect of pressure and pH over the removal of disinfection by-products using nanofiltration membranes in discontinuous systems

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Received 29 April 2009; Accepted 13 May 2010

ABSTRACT

This work studies the reduction by three nanofiltration membranes (NF90, NF270 and Desal-HL) of byproducts of subjecting natural water to a chlorination disinfection method. The studied disinfection byproducts were haloacetic acids (HAAs) and trihalomethanes (THMs). The experiments were carried out using natural water from the Amadorio swamp, evaluating the effects of pressure (between 100 and 400 kPa) and pH (2.7–9.0). The NF270 membrane generated a greater permeate flow (*J*). An increase in pressure causes an increase in permeate flow without causing large variations in the ratio J/J_o. An increase in the pressure slightly increases the fouling of membranes. For all studied membranes and pressures, the percentage reduction in HAA$_5$ and THMs formation was higher than 82%. An increase in pressure leads to greater generation of disinfection byproducts for NF90 and Desal-HL membranes. All membranes studied at natural water pH (8.2) had lower flow losses. More disinfection byproducts were generated at pH 2.7.

Keywords: Disinfection by-products; Haloacetic acids; Nanofiltration; Trihalomethanes

1. Introduction

Water is an essential resource for life and for the development of cities. In southeastern Spain, particularly in the Mediterranean area, lack of water is obvious and is widely studied [1,2]. This lack of water threatens both the supply of clean water to the population and the maintenance of proper farming areas in the region. Therefore, intensive efforts are being made to optimise the water resources of the area by reusing wastewater for irrigation and by improving the utilisation and quality of natural waters—both surface and groundwater—that are used to supply the population.

Natural waters contain varying concentrations of many organic compounds. The organic matter in natural water (natural organic matter, NOM) consists of a variety of complex organic compounds. In the process of purification of water by chlorination disinfection (the method used in 90% of the water purification in the community of Valencia), organic matter can react with chlorine to form different chlorination by products (DBPs) such as trihalomethanes (THMs), haloacetic acids (HAAs), haloacetonitriles (HANs), and chlorinated phenols that impair water quality and may have adverse health effects, such as the prevalence of development of certain types of cancers, including bladder, colon, and pancreas. [3–5].

At present, there are different regulations that govern the presence of disinfection byproducts in water fit for human consumption. In general, most regulations govern a maximum allowable concentration of THMs, but fewer countries or states regulate the presence of other products, such as HAAs. Current Spanish legislation only governs THMs, setting the threshold value as the sum of

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chloroform (CFM), bromodichloromethane (BDCM), dibromochloromethane (CDBM), and bromoform (BFM) totalling less than 100 mg/l [6]. In contrast, the United States of America, in addition to setting a maximum concentration of THMs to less than 80 mg/l, also sets a maximum allowable concentration of five haloacetic acids (HAA_5), or the sum of monochloroacetic (ClAc), dichloroacetic (Cl_2Ac), trichloroacetic (Cl_3Ac), monobromoacetic (BrAc), and dibromoacetic (Br_2Ac) acids, to less than 60 μg/l [7].

The guidelines of the World Health Organization (WHO) establish a maximum reference concentration of 300 μg/l for CFM, 60 μg/l for BDCM, 100 μg/l for CDBM, 100 μg/l for BFM, 50 μg/l for Cl_2AA, 20 μg/l for ClAA, 200 μg/l for Cl_3AA, 20 μg/l for dichloroacetonitrile, and 70 μg/l for dibromoacetonitrile [8].

For years, different techniques have been applied (coagulation–flocculation, biological degradation, ion exchange, activated carbon adsorption, ozonation, oxidation treatment, and advanced oxidation treatment) [9] to reduce the concentration of organic matter in natural waters and the formed disinfection by-products.

Since 1748, when J.A. Nollet discovered selectively permeable membranes, operations with membranes and their applications have evolved by leaps and bounds, including their possible application in the reduction of organic matter in natural waters and thus a method for reducing disinfection by-products.

The application of membrane techniques has the advantage that it can be performed continuously, at room temperature, with no phase changes, and generally does not require the addition of chemicals.

Extensive studies of DOC removal using ultrafiltration (UF) membranes have been made. Some studies have found reductions in DOC of between 70% and 85%, depending on the organic material and membrane used [10]. Other authors obtained removal efficiencies between 30% and 60% [11,12]. Although it is possible, in some cases, to obtain higher COD removal efficiencies, it has been found, in other cases, that the formation of products is not reduced using UF membranes applied to natural waters [13,14]. The organic substances found in natural waters have low molecular weights, which are only partially removed by UF; thus, it is necessary to apply membranes with smaller pore sizes, such as nanofiltration membranes [12].

This research evaluated the characteristics of three commercial nanofiltration membranes (NF90, NF270, and Desal-HL) in reducing the formation of two types of disinfection byproducts (trihalomethanes and haloacetic acids) in natural water pretreated with these membranes. The effect of pressure and pH on the potential formation of haloacetic acids (HAAFP) and trihalomethanes (THMFP) has also been investigated.

2. Experimental

2.1. Feed waters

For this study, we used natural water from the Amadorio swamp. This bog is located in the southern Spanish province of Alicante and is used to supply different municipalities such as Benidorm and Vilajoiosa.

The samples from the swamp were initially filtered using a Wattman GF/C fibreglass filter of 0.45 μm pore size to remove suspended organic matter.

The reservoir water was characterised by determining the concentration of dissolved organic carbon (DOC), the SUVA rate (UVA 254/DOC), pH, and conductivity. The apparent molecular weight (MW) distribution of the natural water was determined too by means of sequential filtration through membranes of decreasing molecular weight cutoff (MWCO) [12]. Source water was fractioned in a 64 mm diameter stirred cell (model 8200, Amicon, Beverly, MA) by means of a series of regenerated cellulose acetate UF membranes (Millipore YM30, YM10, YM3 and YM1) of 30,000, 10,000, 3000 and 1000 Da nominal MWCO, respectively, and a cellulose acetate UF membrane (Millipore YC05) of 500 Da nominal MWCO, while bearing in mind that parameters such as the pH, ionic strength, type of membrane, pressure and calibration might affect the MW distribution as determined using this method [15].

Table 1 shows the values of DOC, SUVA, pH, conductivity, and the percentage of distribution of molecular weight (MWCO) of dissolved organic carbon from the used natural water.

Table 1
Values of DOC, SUVA, conductivity, pH, and apparent molecular weight distribution (MWCO) in water from the Amadorio swamp

	Amadorio
DOC (mg/l)	1.6
SUVA (l/mg m)	1.8
pH	8.2
Conductivity (μS/cm)	745
% DOC	
MWCO 30,000	8
MWCO 10,000	5
MWCO 1000	25
MWCO 500	12
MWCO <500	22

2.2. Analytical methods

The concentrations of organic matter in the reject and the permeate were determined by measuring the dissolved organic carbon (DOC) using a Shimadzu

TOC 5000-A. The UVA was measured with a UV/Vis spectrophotometer (Shimadzu UV-1601) in samples previously adjusted to pH 7 by adding NaOH or HCl. Specific UVA (SUVA) was calculated as the ratio of UVA to DOC. The conductivity and pH were measured using a Mettler-Toledo compact conductivity pH-meter.

To determine HAAFP and THMFP, prior chlorination of the samples was performed using the chlorination method 5710B of the "Standard Methods for the Examinations of Water and Wastewater" with some modifications [16]. The applied Cl_2 dose was 20 mg/l, and the reaction was maintained at the conditions described in the standard method for 72 h. Subsequently, the reaction was stopped to avoid decomposition of some DBPs [17]. THMs and HAAs formed after chlorination were determined through the USEPA 551.2 [18] and USEPA 552.2 [16] methods, respectively.

2.3. Membranes

For this study, three different nanofiltration membranes (NF90, NF270 and Desal-HL) were used. All membranes had a circular section of 63.5 mm in diameter, with an effective area of 2780.5 mm^2.

Table 2 shows the general characteristics of the studied membranes.

2.4. Nanofiltration process

Nanofiltration experiments were carried out in a dead-end stirred-cell filtration system (Fig. 1). The system consists of a filtration cell (model 8200, Millipore corp.) with a volume of 200 ml and allows the use of membranes with a surface area of 2780.5 mm^2. The module is pressurised by nitrogen gas.

2.5. Filtration protocol

Before the initial membrane use, it was placed into an Amicon module and compacted for 8 h at a pressure of 400 kPa. Once compacted, permeate flow of the membrane was measured at the experimental pressure. This flow was taken for each membrane as the reference value of the clean membrane (J_o: m^3/s·m^2).

Once the membrane was fitted, the stirred cell was filled with distilled water at the desired pH and conductivity for the experiment, and the system pressurised to the desired pressure. The variation of permeate flow rate with time at constant pressure was measured until the flow stabilised. Subsequently, the stirred cell was

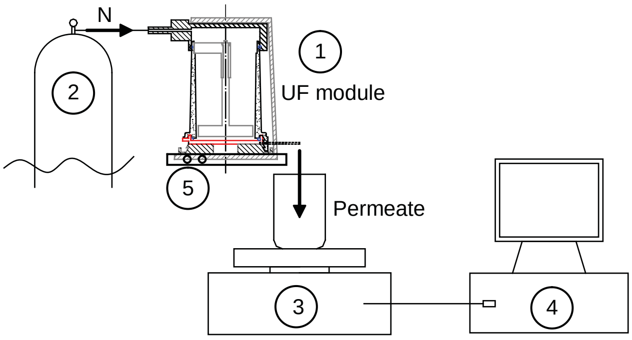

Fig. 1. Diagram of the experimental setup. 1. Nanofiltration module (Amicon 8200); 2. nitrogen-pressurised bottle; 3. analytical balance; 4. PC for data logging; 5. magnetic stirrer.

Table 2
Characteristics of membranes used in the study

Membrane	NF90	NF270	Desal-HL-51
Manufacturer	Dow-Filmtec		GE Osmonics
Material[a]	Polyamide TF	Polypiperazine amide	Polyamide TF
MWCO[a]	200	200–300	150–300
W. Permeability (m^3/s m^2 kPa)[b]	2.4×10^{-8}	3.3×10^{-8}	2.8×10^{-8}
Contact angle (°)	54[c]	27[c]	52[d]
Charge			
(pH 3)	–14[f]	2.4[e]	3.5[e]
(pH 7)	–26[f]	–21.6[e]	–14.2[e]
(pH 11)	–30 (pH9)[f]	–26[e]	–17.4[e]

[a] Information provided by manufacturer.
[b] Estimated in this study.
[c] Ref. [19].
[d] Ref. [20].
[e] Ref. [21].
[f] Ref. [22].

emptiedand refilled with water from the swamp to study its properties and to pressurise the system.

The permeate flow (J: $m^3/s{\cdot}m^2$) was measured continuously using an analytical balance. Permeate samples were collected periodically for subsequent analytical determinations. At the end of the swamp water filtration experiment, the stirred cell was emptied and refilled with distilled water at the same pH, conductivity, and pressure as the initial experiment, and the flow variation over time was also determined. This latter experiment allowed instantaneous determination of irreversible fouling of the membrane.

Finally, another experiment was performed with distilled water (pure water) to clean the membrane. If the change in flow was greater than 5% of that of the clean membrane, the membrane was discarded, and a new one was used in the following experiment.

All experiments were carried out at room temperature 20 ± 2°C. In all experiments, a constant agitation speed of 200 rpm was maintained. The pressure study was performed between 100 and 400 kPa. The pH study was conducted by adjusting the pH between 2.7 and 9.0 and maintaining the pressure at 400 kPa.

3. Removal of organic matter from natural water

3.1. Effect of pressure

The study to determine the effect of pressure on the reduction of THM and HAA formation was performed at a pressure range between 100 and 400 kPa.

The results of the flow variation obtained for the three membranes at the three studied pressures are shown in Figs. 2–4. Each membrane is plotted as the permeate flow and reduced flow (J/J_0) versus nanofiltration time.

Table 3 shows the values of the percentage reduction of permeate flow rate after an initial washing with distilled water at the experimental pressure, which allows instantaneous determination of irreversible fouling of the membrane.

As it can be seen for the three studied membranes, an increase in pressure leads to higher permeate flow. This behaviour can be mathematically described by Darcy’s law.

The membrane that generates the greatest flow of permeate is NF270, followed by the Desal-HL membrane, with NF90 generating the lowest permeate flow. This is

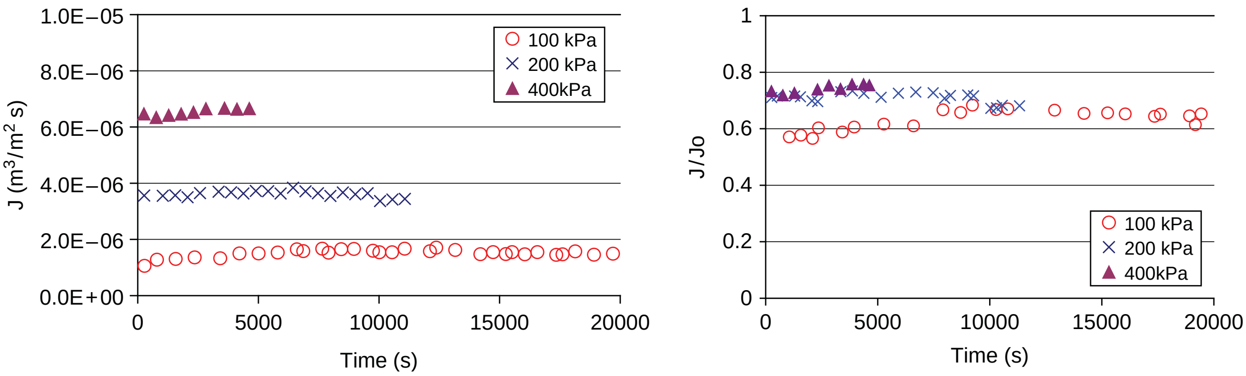

Fig. 2. NF90 membrane. Permeate flow (J) and reduced flow (J/J_0) versus time for the three pressures studied.

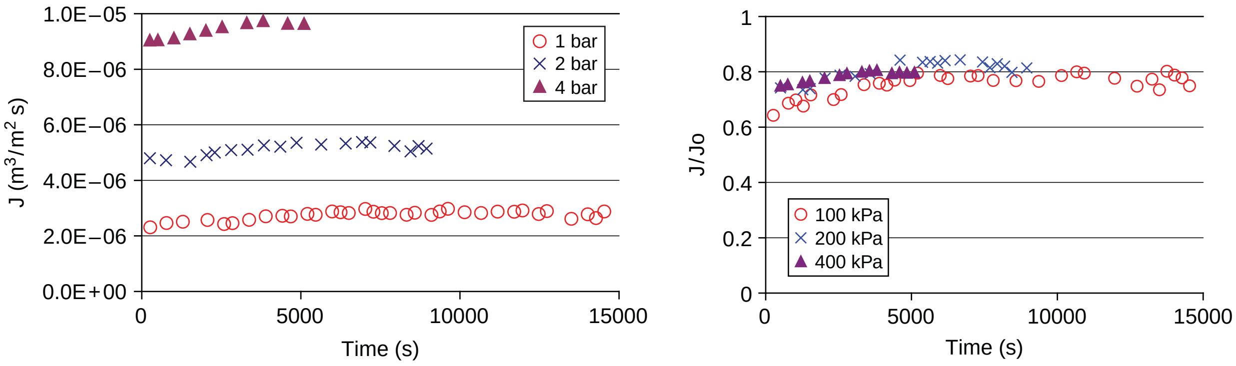

Fig. 3. NF270 membrane. Permeate flow (J) and reduced flow (J/J_0) versus time for the three pressures studied.

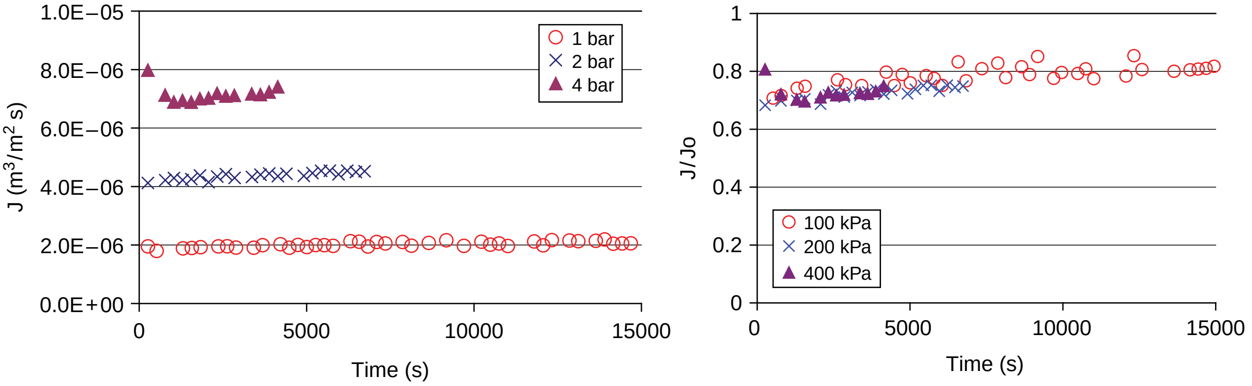

Fig. 4. Desal-HL membrane. Permeate flow (J) and reduced flow (J/J_0) versus time for the three pressures studied.

Table 3
Irreversible fouling of the membrane after post-experiment cleaning with distilled water

Pressure (kPa)	Irreversible fouling (%)		
	NF90	NF270	Desal-HL
100	0.1	0.1	0.1
200	3.5	1.3	2.5
400	10.7	5.1	12.2

partly due to the lower MWCO of NF90 (Table 2) and its more hydrophobic character (it has a contact angle with water of 54 °C).

Noting the values of loss of flow through the membrane (J/J_0), we observe that the NF90 membrane has a higher volumetric loss than do the other two membranes. At a pressure of 100 kPa, the NF90 membrane shows a value of J/J_0 equal to 66% at the end of the experiment, while the NF270 and Desal-HL membranes reach 77% and 80%, respectively. For all membranes, an increase in pressure produces a higher permeate flow, but there are no large variations in the loss of flow through the membranes with respect to their initial conditions.

Noting the values in Table 3, which show the instant irreversible fouling, it was observed that an increase in pressure for all membranes generates greater irreversible fouling. At pressures of 200 and 400 kPa, the NF270 membrane has a lower irreversible fouling than do the other two membranes. This is because the NF90 and Desal-HL membranes have an increased hydrophobic character, as evidenced by their larger contact angle with water (Table 2), which allows organic matter to adsorb onto the surface, creating slightly higher fouling than that produced in the NF270 membrane, which has a less hydrophobic character.

Given the potential formation of THMs and HAAs, Fig. 5 shows the percentage reduction of THMFP and HAAFP at the three studied pressures for the three studied membranes. For the NF90 and Desal-HL membranes, increased pressure produces lower THMFP and HAAFP elimination, while the NF270 membrane shows a slight increase in removal of disinfection byproducts. These trends can be more clearly seen in Fig. 6, which shows for the studied membranes the rates of reduction of the five haloacetic acids covered by American law (HAA_5) and of the sum of the four THMs.

The NF90 and Desal-HL membranes have a more hydrophobic character than does the NF270 and are more likely to adsorb molecules onto their surface, which may explain the greater fouling of those membranes. Additionally, increased pressure in these membranes causes poorer performance in terms of organic matter removal and disinfection byproduct formation due to increased with pressure the thickness of the gel layer and the phenomenon of concentration polarisation on the surface [23]. Similar results were found by other authors [24].

The NF270 membrane has a less hydrophobic character (contact angle with water of 27 °C) and, with an increase of pressure, produces slightly higher removal efficiencies of disinfection byproducts. At high pressures, the permeability of the solvent (water) rapidly increases compared with that of the solute; thus, more solvent molecules can pass through the membrane. This may reduce the passage of solute through the membrane, generating a slight increase in retention.

Other authors found the same trend when the concentration of organic matter was low [22]. At low pressure, because the membrane has a low hydrophobic character, there is little accumulation of organic matter on the surface. An increase in pressure favours the approach of organic matter to the surface, forming an additional layer that prevents the passage of larger molecules, improving removal efficiencies. Other authors have obtained similar results when working with low concentrations of organic matter [24].

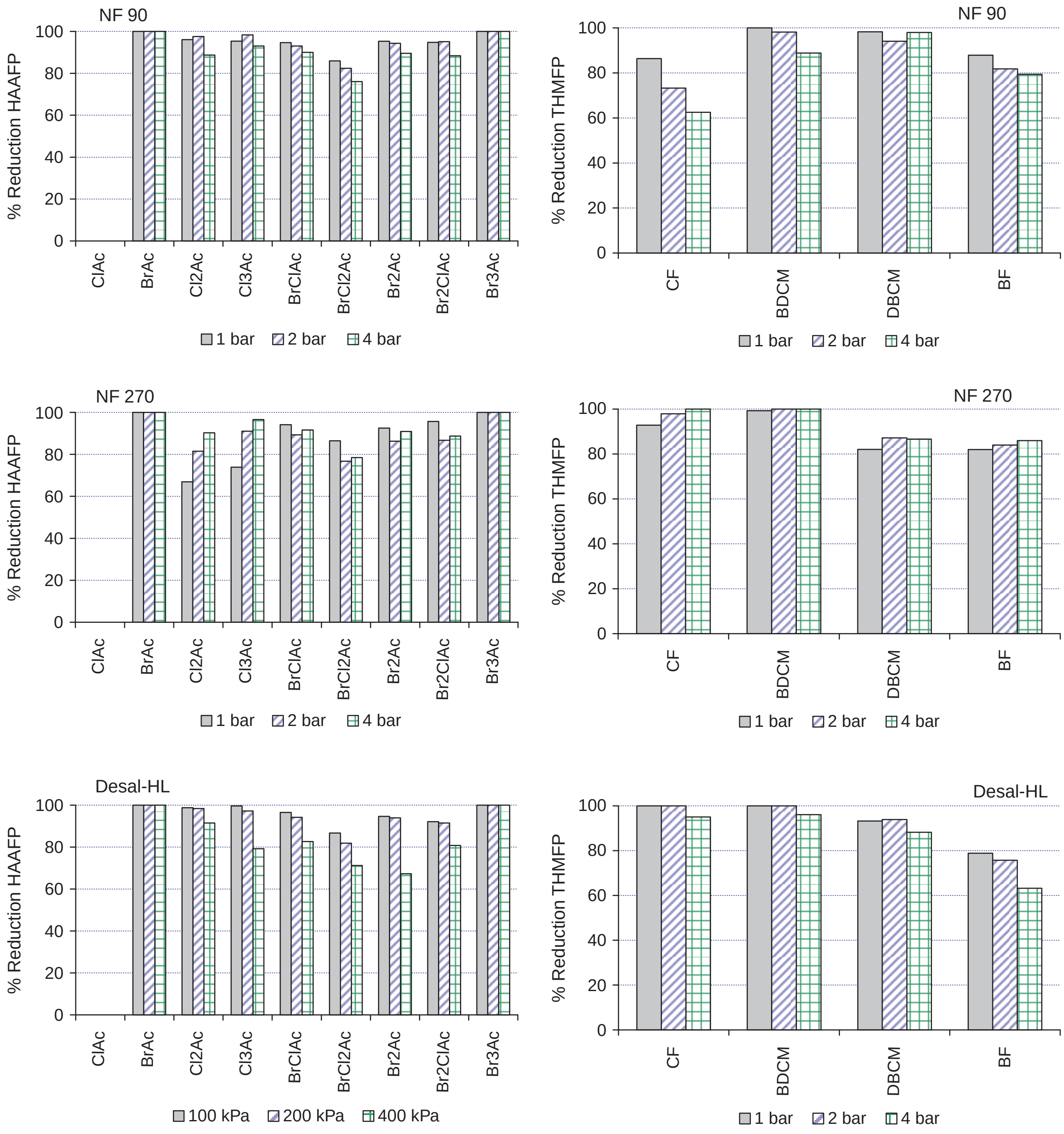

Fig. 5. Percentage reduction of THMFP and HAAFP for NF90, NF270, and Desal-HL membranes.

3.2. *Effect of pH*

Experiments were performed to determine the effect of pH on nanofiltration of natural waters for a pH range between 2.7 and 9.0. This range is wider than the one usually found in natural waters (7.0–8.5). The range of the study was extended to analyse possible effects if membranes were applied to other water types with variable pH. The pH was adjusted by adding HCl or NaOH. All experiments were performed with Amadorio swamp water, with an initial concentration of NOM around 1.6 mg/l. All experiments were conducted at a pressure of 400 kPa and constant agitation at 200 rpm.

Fig. 7 shows the reduction of flow (J/J_0) with time at different pH for all membranes.

It can be seen that at acidic or very acidic pH (6.0, 4.0 and 2.7), the value of J/J_0 is less than the value of J/J_0 with natural water (pH 8.2) for all membranes. This is

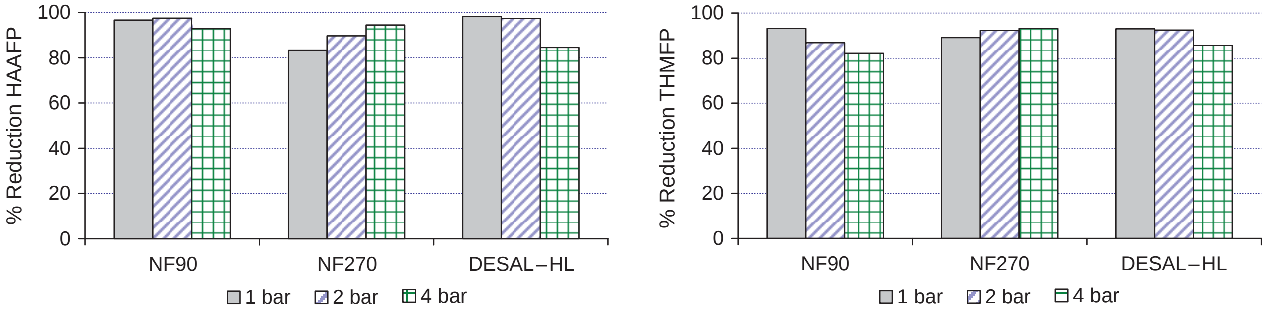

Fig. 6. Percentage reduction of (a) HAA_5FP ($ClAc + BrAc + Cl_2Ac + Cl_3Ac + Br_2Ac$) and (b) THMFP (CF + BDCM + DBCM + BF) for NF90, NF270, and Desal-HL membranes.

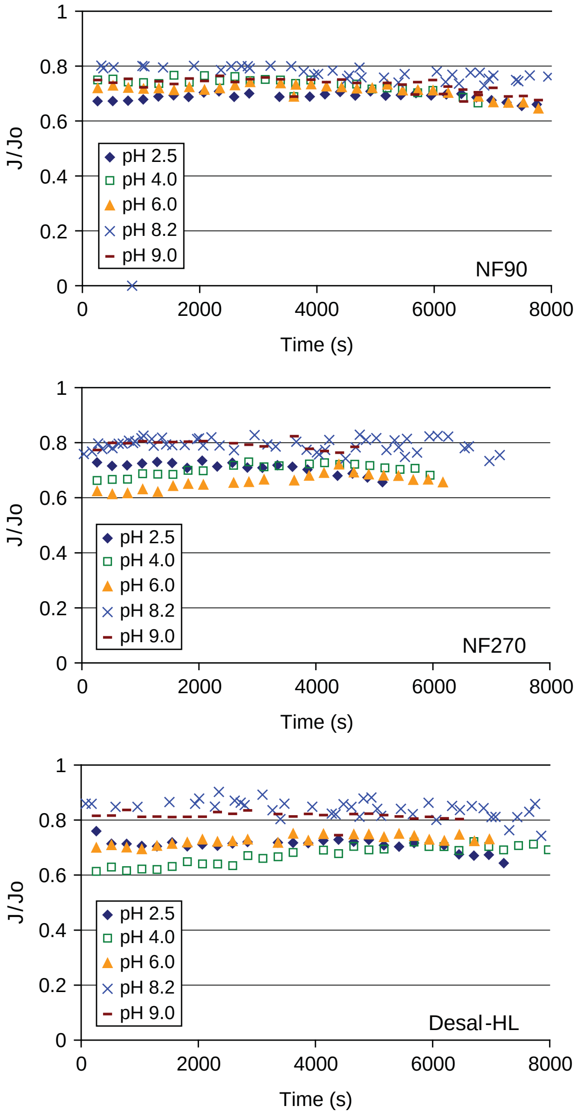

Fig. 7. Permeate flow reduction with time for the NF90, NF270, and Desal-HL membranes at different pH.

because samples prepared with HCl acidification have increased conductivity, resulting in a reduced flow rate [25], in addition to acidic pH causing the membranes to have less negative charge, which favours the adsorption of organic matter on the membrane surface. The values of J/J_0 for water at natural pH and water at pH 9.0 are practically equal and greater than those obtained in experiments performed at lower pH.

Fig. 8 shows the percentage reductions for THMFP and HAAFP for the three permeated membranes at the different studied pH levels, and Fig. 9 represents the percentage reduction in HAA_5FP and in the sum of the four THMs.

In general, for the three studied membranes, the percentage of HAA and THM formation is almost constant at pH values between 4 and 9.2. For all membranes, the byproduct removal efficiencies at pH 2.7 are slightly lower. The smaller reduction of THMFP and HAAFP at low (acidic) pH can be attributed, in part, to the membranes having less negative charge than at neutral or basic pH (Table 2). This decreases the repulsion of organic matter from the membrane surface; thus, organic matter can more easily reach the surface [26]. Moreover, at low pH, humic acid can form hydrogen bonds, thereby reducing its molecular size and negative charge. This causes the nanofiltration membrane to have better adsorption to its surface and better diffusion through it [22]. Other authors have obtained similar results [27].

4. Conclusions

Removal of THM and HAA decreases with increased pressure for NF90 and Desal-HL membranes. Using the NF270 membrane, a pressure increase improves the efficiency of removal of the studied byproducts, which is slightly higher for low concentrations of organic matter.

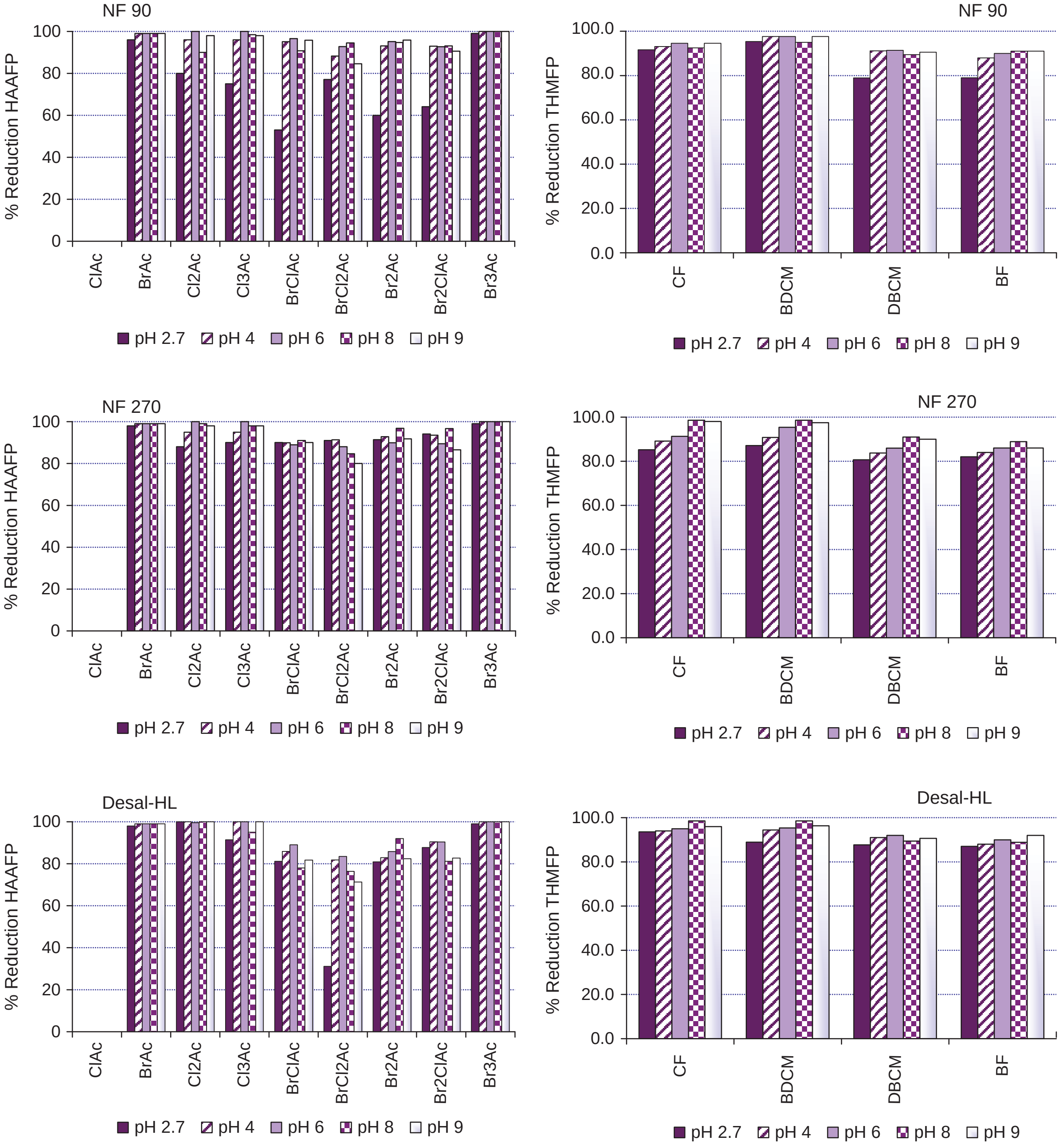

Fig. 8. Percentage reduction of HAAFP and THMFP for NF90, NF270, and Desal-HL membranes.

In all cases, the reduction of the total potential HAA_5 and THM formation is greater than 82%. At pressures of 400 kPa, the NF270 membrane generates a greater reduction as compared to other membranes of the formation of THM and HAA.

At low pH (2.5–6.0), lower values of J/J_0 are obtained than in experiments performed at pH 8.2 and 9.0. For the three membranes, using natural water yielded the lowest flow reduction as compared with other pHs.

The analysis sought to find the effect of pH on the potential HAA and THM formation for the three studied membranes. The results show that at very low pH (2.7), there is a lower percentage of reduction of disinfection by products. At all other pH values, the byproduct reduction is similar.

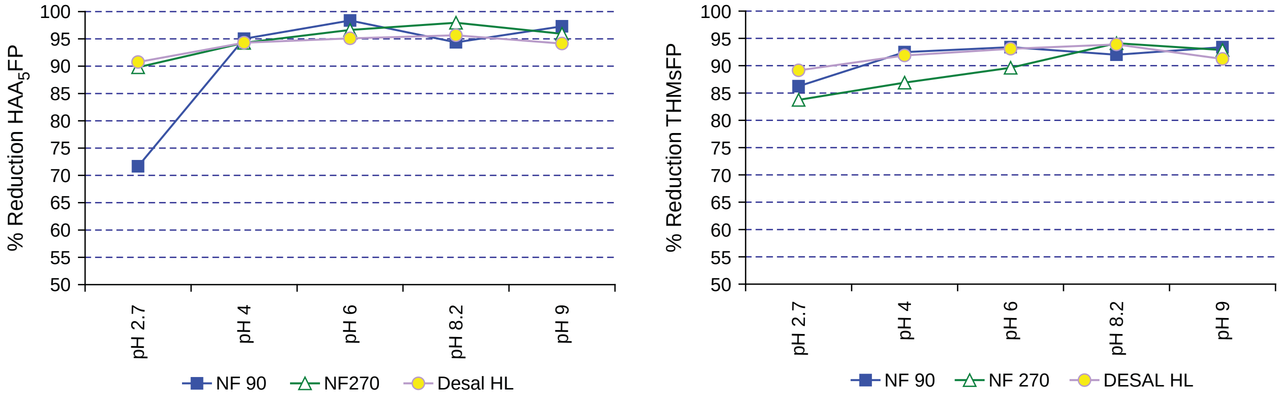

Fig. 9. Percentage reduction of (a) HAA_5FP (ClAc + BrAc + Cl_2Ac + Cl_3Ac + Br_2Ac) and (b) THMFP (CF + BDCM + DBCM + BF) for NF90, NF270, and Desal-HL membranes at different pH values.

Acknowledgment

This study was partially financed by the project “Water resources generation by means of membrane processes” (NF MBR/NF) CTQ2007-66780.

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