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Pilot plant comparison study of two commercial nanofiltration membranes in a drinking water treatment plant

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ABSTRACT

A wide range of commercial membranes were tested and compared at laboratory scale in order to select the most appropriate for improving the final water quality of a real drinking water treatment plant (DWTP). Most of the membranes tested showed a reduction of trihalomethanes formation potential (THMFP) higher than 90%. In this work, several NF membranes were tested at laboratory scale in order to evaluate the most suitable NF membrane to reduce THMFP. NF270 (Dow Chemical) and ESNA1LF2 (Hydranautics) were finally selected based on their permeability and inorganic salt rejection. These two membranes were tested in parallel in a pilot-scale plant. The effectiveness in THMPF removal was evident for all membranes tested. The comparison of both membranes was carried out simultaneously in a pilot plant installed in the DWTP of Manresa. In the spiral wound configuration, both membranes also showed effective separation of trihalomethanes (THM) precursors, reducing THMFP in treated water at values of approximately 90%, depending on the season.

Keywords: Pilot plant; Nanofiltration; Spiral-wound modules; NF270; ESNA1LF2; Drinking water; Trihalomethane formation potential

1. Introduction:

Increasing water pollution and frequent droughts occurring in cyclic periods endanger drinking water quality in some regions, as the Mediterranean. Water scarcity and an increasing population worsen the global water situation, requiring the implementation and development of new technologies into conventional processes for drinking water production. Conventional treatments for drinking water are usually based on four main steps: predisinfection, coagulation, filtration and final disinfection. Variations of these processes are frequent due to the high variability of the water source quality and the different requirements of each country in the final drinking water quality.

Disinfection is essential to control biological parameters in order to not endanger human health [1]. This key step could result in disinfection by-product (DBP) formation. The presence of DBP in drinking water is variable and their regulation is different in each country. Trihalomethanes (THM) are the most well-known chlorinated DBP and they have been recognised as a potential carcinogen [2,3]. Under [2,3]. Under current Spanish and European Union legisla-

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tion, the maximum content of THM in drinking water is $100\,\mu g\,L^{-1}$, while the standard value legislated in the USA is $80\,\mu g\,L^{-1}$ [4,5]. The total concentration of THM (tTHM) is represented by the sum of trichloromethane (TCM), bromodichloromethane (BDCM), dibromochloromethane (DBCM), and tribromomethane (TBM). The guidelines of the World Health Organization (WHO) and USA legislation also regulate other DBP as haloacetic acids (HAA) and haloacetonitriles (HAN) at levels lower than $100\,\mu g\,L^{-1}$ [6].

Natural waters present considerable amounts of natural organic matter (NOM), which reacts with chlorine or other oxidant agents employed as disinfectants. In these conditions the DBP formation is unavoidable and depends on the reaction conditions: type and concentration of disinfectant, NOM content and composition, temperature and pH [7,8]. Fortunately, drinking water process has been improved in several aspects, but the THMFP represents a challenge in many plants. Some solutions have been implemented, for example chlorine can be substituted for other oxidant agents, mainly potassium permanganate, dioxide chlorine, chloroamines and ozone [9]. This is a good alternative during the predisinfection, but it shows some inconvenience for the final disinfection, requiring the chlorine addition in this final step to assure stability of the microbiological quality [7]. Among other methods, removing NOM in the raw water leads to a decrease in the DBP content of the final water. In this way, nanofiltration (NF) has been gradually implemented in drinking water treatment as a result of its effectiveness in removing NOM and contaminants such as pharmaceuticals, heavy metals or pesticides [10–13].

Rejection mechanisms of NF mainly include size exclusion and electrostatic repulsion which allow compounds and ions removal from water [14–17]. NOM, as a precursor of DBP, deteriorates water quality and in this way, NF can increase the final water quality [18–21]. Furthermore, inorganic ions are partially removed depending on their concentration, electric charge, and molecular weight. From this point of view, groundwater and some brackish waters have been treated by NF technology in order to reduce hardness or remove heavy metals or other inorganic contaminants, such as sulphates or nitrates [22–24].

The main problems of NF technology are the high energy requirements in comparison to conventional processes and the fouling tendency of the membranes. Laboratory and pilot plant studies have investigated flux decline and availability to remove undesirable compounds in NF processes for different types of water composition and operational parameters [25–27].

The use of NF in real water treatment plants has been increased in the last years. In 2001, one of the largest NF plant was built in the city of Boca Raton (Florida), to treat underground water for drinking water purposes [28,29]. In Paris, the Mery-sur-Oise drinking water treatment plant (DWTP) treats freshwater through NF process using NF200 membranes to remove pesticides and other contaminants. Pretreatment in this plant consists in predisinfection with ozone, sand and active carbon filtration previous to NF step. However, significant fouling was detected according to seasonal variations in the study of Her et al. [30]. A combination of physicochemical and hydrodinamic conditions affects fouling formation, increasing the flux decline and decreasing membrane performance. However the quality and flow of permeate water can be maintained if NF process is well designed and monitored. Pilot plant studies are useful to characterize fouling agents and find the best option to clean membranes.

This work consists in selecting two NF membranes from laboratory results in order to compare and evaluate the most suitable NF membrane to reduce THMFP during the pilot plant experimentation. The effectiveness in THMFP removal was noticeable for all membranes tested, consequently other parameters were evaluated in the laboratory experiments, as for example permeability and inorganic salt rejection, in order to select only two membranes. NF270 4040 (Dow Chemical) and ESNA1LF2 4040 (Hydranautics) modules were finally selected and its performance were evaluated in a pilot plant installed in the DWTP of Manresa, where $30{,}000\,m^3$/day of drinking water are produced from specific freshwater source located in this area.

2. Materials and methods

2.1. Feed water quality

Water from DWTP in Manresa (NE Spain) was used in this study. The hydric system that acts as a potable water source for the inhabitants of this city comes from Llobregat River. The deviation takes place 50 km after the Baells reservoir ($100\,Hm^3$), and before the mining salt intrusion located 10 km down through the river. Before entering the DWTP, where $30{,}000\,m^3$/day of drinking water is produced, water is stored in Llac de l'Agulla reservoir, a small reservoir ($0.3\,Hm^3$) that acts as water reserve for Manresa.

In a previous study, THMFP evolution along the hydric system of the Llobregat River and the ability of NF membranes to decrease it were shown [31] (Fig. 1).

Point 1 is near the source of the river, in the village of Pobla de Lillet. Point 2 is located 35 km

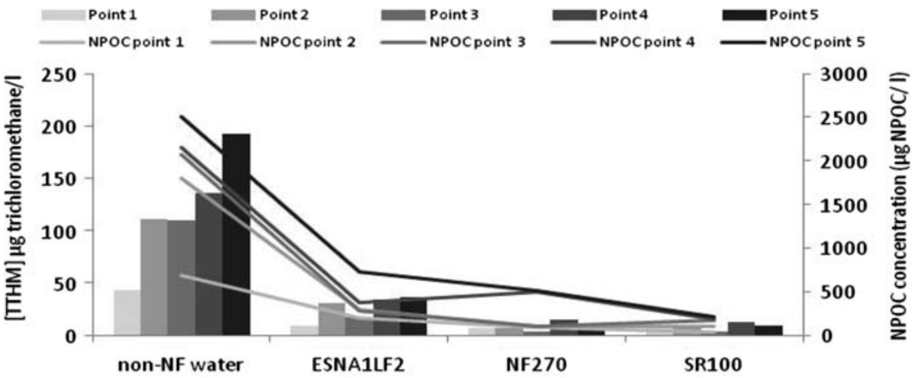

Fig. 1. THMPF and non-purgeable organic carbon (NPOC) evaluation through the hydric system selected in Llobregat River and their reduction by NF process at laboratory scale.

downstream, after La Baells reservoir. The water deviation takes place at point 3, in Balsareny, 50 km downstream of point 1. Point 4 is located 70 km downstream of the river source, before Llac de l'Agulla. Point 5 is located in the DWTP after prechlorination and pollyaluminium chloride sedimentation, followed by a sand filtration step. As can be seen, water reservoirs of La Baells and Llac de l'Agulla have a negative influence in final water quality of Llobregat's water, significantly increasing its THMPF.

NOM content in fresh water used during the experiments reacts with chlorine, as the disinfectant employed, to form THM. THMFP increased in waters that presented the highest contents of nonpurgeable organic carbon (NPOC), as it is shown in Fig. 1. The THMFP in point 5 is four times higher than THMFP in point 1, near the river source. These results indicate the important influence of water reservoirs in water quality from the point of view of THMFP. In any case, the ability of NF to reduce THM formation in disinfected waters in all points of the selected hydric system can be observed.

In this study, water used comes from point 5. Its average composition is shown in Table 1.

Inorganic water composition was considered practically constant during the experimental period. In order to avoid considerable fouling, an ultrafiltration (UF) step was implemented after sand filtration to treat feed water for NF spiral wound modules during the pilot study.

2.2. Selection of NF membranes at laboratory scale

Nine commercial NF membranes were selected to test its ability to remove THMPF precursors from water (Table 2). Three experimental campaigns carried out in the laboratory were performed in July 2009, November 2009, and March 2010. During each campaign, water was collected daily at point 5 in Manresa DWTP.

Table 1
Average composition of the feed water used

Parameter	Average value	Standard deviation	Parameter	Average value	Standard deviation
Conductivity (µS cm^{-1})	570	30	Ca^{2+} (mg L^{-1})	82	9
pH	7.8	0.2	Mg^{2+} (mg L^{-1})	12	2
SDI	1.2	0.9	Na^+ (mg L^{-1})	20	6
UVA (254 nm)	0.039	0.015	K^+ (mg L^{-1})	2.0	0.5
THMPF (µg L^{-1})	120	40	Sr (mg L^{-1})	1.05	0.05
NPOC (µg L^{-1})	2,560	540	Si (mg L^{-1})	0.91	0.93
AI (µg L^{-1})	51	13	HCO_3^- (mg L^{-1})	175	8
Ba (µg L^{-1})	43	10	SO_4^{2-} (mg L^{-1})	100	23
Fe_{total} (µg L^{-1})	7.5		CI^- (mg L^{-1})	35	7
B (µg L^{-1})	27	2	NO_3^- (mg L^{-1})	2.7	1.6
Mn (µg L^{-1})	0.14	0.17	F^- (mg L^{-1})	0.16	0.04

Table 2
Comercial NF membranes used in laboratory experiments

Manufacturer	Model	Manufacturer	Model
Dow Chemical	NF270	Hydranautics	ESNA1LF2
Dow Chemical	NF200	Koch	TFCS-SR3
Alfa Laval	NF99HF	Koch	TFCS-SR2
Osmonics	DK	Iberlact	PC D400
Osmonics	DL		

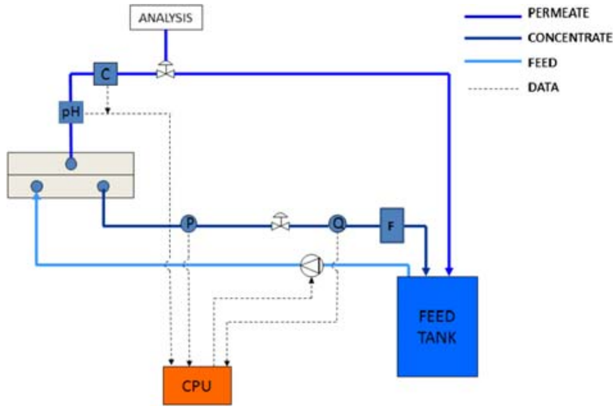

Fig. 2. Experimental system flow sheet at laboratory scale.

Fig. 2 shows the scheme of the lab-scale experimental system. The SEPA II module (GE Osmonics) was used for membrane testing. The NF experiments were performed recycling the concentrate and the permeate streams. In all experiments the temperature was maintained nearly to 25˚C with a thermal bath in order to obtain comparable results [32].

The operational conditions monitored and controlled in the laboratory-scale system were the operational pressure and the cross-flow velocity. The installation of a valve in the concentrate stream allowed controlling the feed pressure. Two pressure sensors, one in the feed and another in the concentrated streams measured the operational pressure values. A piston pump was used to feed the system and it was regulated to set the cross-flow at $1\,\mathrm{m\,s^{-1}}$, approximately.

Conductivity and pH of the permeate stream were measured online, using a conductivity cell (Crison 53 92) and a pH electrode (Crison 53 03). Five permeate samples, corresponding to filtered water at 3, 6, 9, 12, and 15 bars, were collected for each membrane in order to analyze major inorganic ions and NPOC concentrations. Measurements of UV light absorption were also performed for the previously indicated water samples. THMFP was only determined at 9 bars as the average value of the feed pressure.

2.3. NF pilot process

From the results obtained from laboratory two NF membranes were selected for the pilot plant study, NF270 and ESNA1LF2. The aim of this study was to compare simultaneously both membranes in terms of their performance and THMFP reduction ability in a period of six months.

Structural characteristics of NF membranes as electrical charge, hydrophobicity, molecular weight cut-off and roughness have been evaluated in previous studies [33], although a lack of information exists for ESNA1LF2. Table 3 shows some membrane characteristics such as pore size and membrane surface rugosity [34].

One of the rejection mechanisms of NF membranes is the steric exclusion, which is directly related with the membrane pore size. Membrane surface morphology is related to the membrane fouling potential. Vrijenhoek et al. demonstrated that more particles are deposited on rough membranes than on smooth membranes [35].

A schematic view of the NF pilot plant is presented in Fig. 3. The two-staged pilot plant used has three arrays of pressure vessels (PV) placed in a 2:1 configuration. The first stage has two rows of six NF elements each one with ESNA1LF2 4040 and NF270 4040 membranes in each separate row (stage 1.1 and stage 1.2). The second stage contained a combination of both membranes in a single row in order to increase global pilot plant recovery (stage 2). Due to the high silt density values (SDI) observed in the feed water an UF step was added to improve the quality of NF feed water. Metabisulphite and antiscalant (Genesys LF) addition was required to remove free chlorine and to avoid inorganic and colloidal scaling.

Table 3
Pore size and membrane surface rugosity for NF270 and ESNA1LF2

Membrane	rp (nm)	Rms (nm)
NF270	0.50	5.35
ESNA1LF2	0.49	49.07

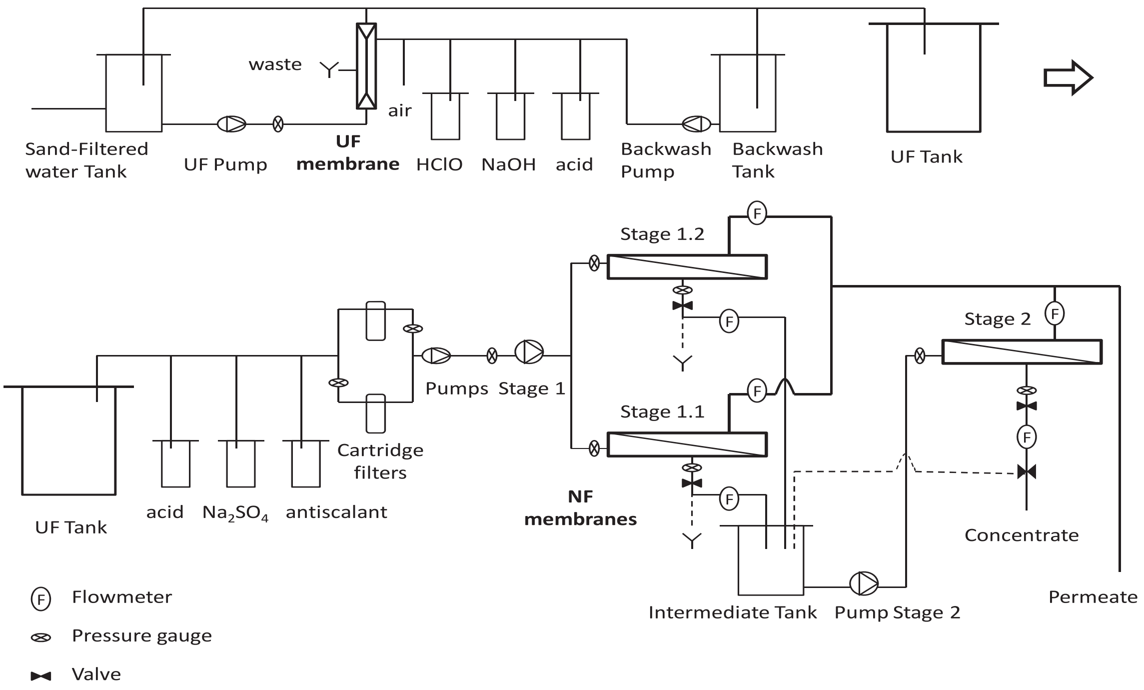

Fig. 3. Schematic representation of the pilot plant experimental set-up in a DWTP.

NPOC, total inorganic carbon (TIC), major inorganic anions (sulphates, nitrates, chloride) and cations (calcium, magnesium, sodium and potassium), conductivity, pH and UV absorbance were monitored weekly in the permeate water from stages 1.1, 1.2, and 2.

THMFP analysis was monitored monthly in feed and permeate samples of stages 1.1 and 1.2. Additionally, THMFP of blended samples using permeated water from both stages and water from point 5. 25%, 50%, and 75% of permeate water from each NF membrane were mixed with water from point 5 in order to study the influence of blending permeate water with non-filtered water.

The pilot process was run six months applying chemical cleanings when normalized permeate flow decline was higher than 10%. During the first four months the global recovery of the plant (including both stages) was 75%. In the last two months, the pilot plant was operated in a recirculation mode of approximately 10 l min^{-1} of concentrate flow to reach a global recovery of 90%. Transmembrane flux (TMF) was varied from 20 to 32 lmh in order to study its influence in permeability and the final water quality.

2.4. Analytical methods

In order to determine the rejection of the studied components in the feed water, several analytical methods have been used for the analysis of the feed water and permeate samples.

The rejection (%*R*) of components analyzed is defined as follows:

$$\%R = \frac{C_f - C_p}{C_f} \cdot 100$$

where C_f and C_p are the concentrations in the feed and in the permeate flows, respectively.

Ion Chromatography (Dionex ICS-2000) was used to analyze anions (SO_4^{2-}, Cl^-, NO_3^-, F^-) and cations (Ca^{2+}, Mg^{2+}, Na^+, K^+), and Total Carbon Analyzer (AnalytikJena Multi NC 3,100) was used to analyze TIC and NPOC. UV absorbance was measured using a spectrophotometer (Shimadzu UV_1603).

THMFP was determined applying the 5710B standard method and THM concentrations were analyzed by HS-GC/ECD (Agilent 7694E-Hewlett Packard 6890). Reference materials and spiked samples were analyzed together with samples in each analysis batch, and the recoveries were always between 90% and 110%.

3. Results and discussion

3.1. Selection of NF membranes at laboratory scale

The most important parameters characterizing the performance of the tested membranes were evaluated at laboratory scale in three experimental campaigns (March 2010, November 2009 and July 2010).

Fig. 4 shows conductivity rejection is in front of permeate fluxes obtained at 3, 6, 9, 12, and 15 bars.

Conductivity reduction of feed water takes into account the main dissolved inorganic ions and their behavior in front of membrane surface properties, basically electrostatic ones [36]. Results from Fig. 4 indicate the variability between membranes regarding the permate flux and the conductivity reduction, being TFC SR3, D400, 99HF, and NF200 the most impermeable in front of inorganic salts. TFC-SR2, NF270, and ESNA1LF2 showed the highest permeabilities with the feed water used. These tendencies were observed for all campaigns and it is important to note that inorganic composition in the feed water was quite constant for all sampling period.

Dissolved organic matter was measured by non-purgeable organic carbon (NPOC). UV absorbance and THMFP were determined as indicators of NOM composition and its content of THM precursors. Fig. 5 shows the NF membranes performance in terms of organic matter rejection for March 2010 and July 2009 laboratory campaigns, respectively.

With the exception of DK membrane, NPOC rejection was higher than 90% in all cases. The humic fraction of NOM is recognized as the major fraction that absorbs ultraviolet radiation (UVA) at 254 nm and the most hydrophobic fraction of NOM [37]. In this study, feed water presents low values of UVA, aromatic compounds being less representative than aliphatic compounds [38]. These UVA values could indicate the variation of organic composition in feed water depending on the experimental campaign which is probably related to temperature and microbiological activity.

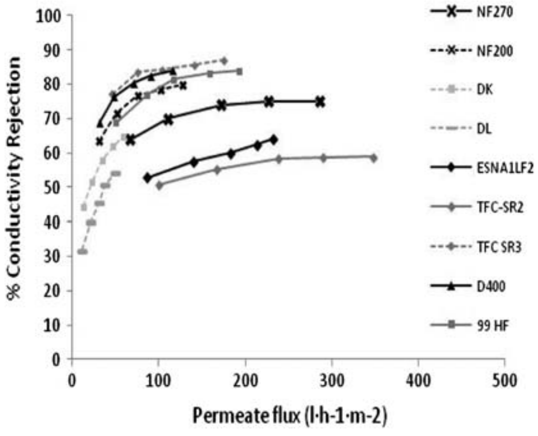

Fig. 4. Conductivity rejection of the nine commercial NF membranes during the laboratory experimentation in November 2009. Each point was obtained at a fixed pressure (3, 6, 9, 12 and 15 bars).

Fig. 6 shows THMFP evolution in the permeate samples. In the campaign of March 2010 higher THMFP were observed in the feed water indicating an increase in the THM precursors content during this seasonal period. Only TCM and BDCM were quantified during THMFP determinations, this is related to low bromide concentrations in feed and permeate water samples.

All membranes demonstrated effective THMFP reduction although seasonal variations in NOM content, presenting values higher than 90% in most cases (NF270, NF200, SR100, D400, 99HF). The average NPOC for feed water was 1.92 and 3.05 mg l^{-1} corresponding to November and March experimental periods, respectively.

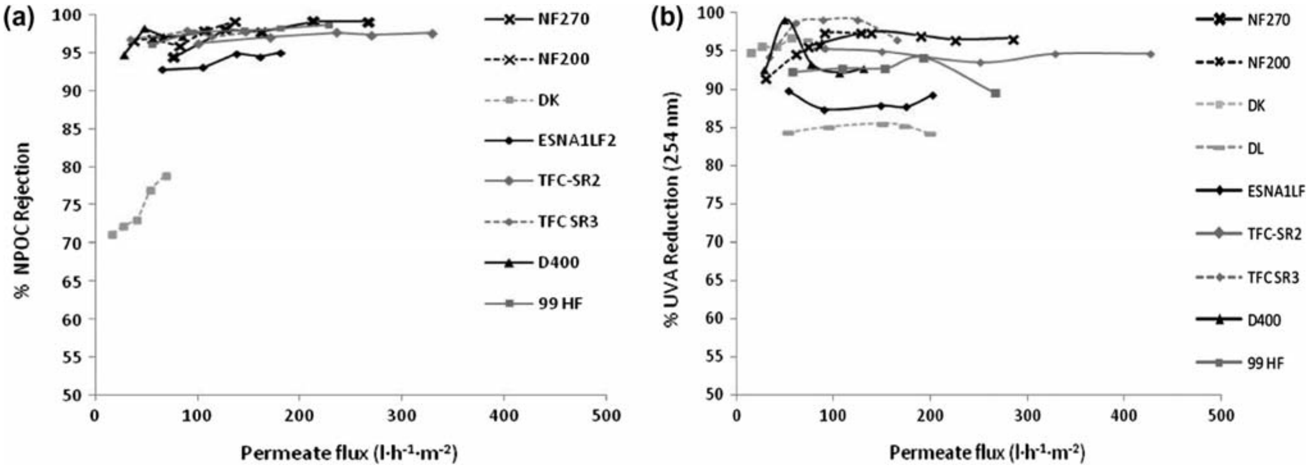

Fig. 5. Organic components rejection for NF membranes tested in the laboratory experimentation period. Each point was obtained at a fixed pressure (3, 6, 9, 12 and 15 bars). (a) NPOC rejection in March 2010. (b) UVA reduction in July 2009.

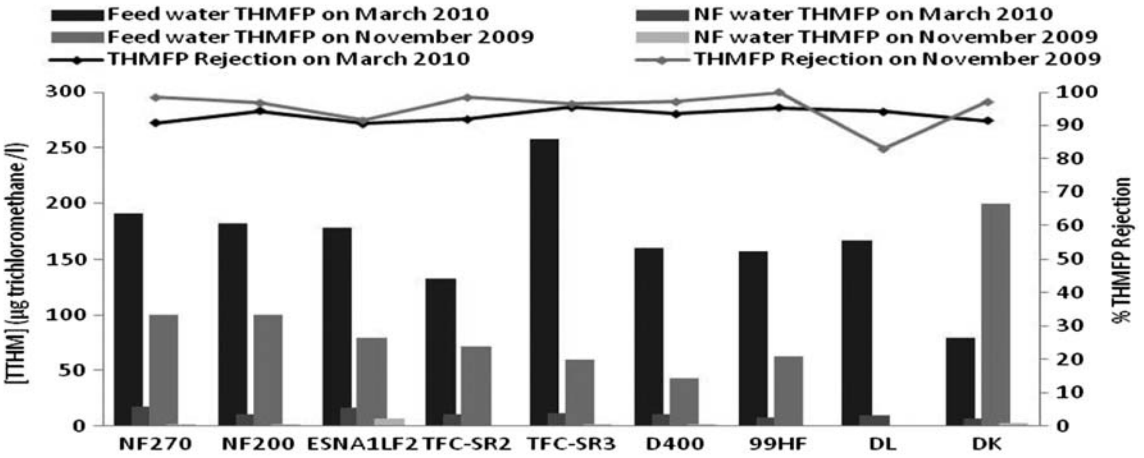

Fig. 6. THMFP reduction using 10 NF membranes in two experimental campaigns at laboratory scale.

The lower values of THMFP in permeates (<30 µg THM l^{-1}) could indicate that the membranes tested were not able to remove low molecular weight organic compounds [39,40]

Due to the high THMFP removal capacity of all the membranes tested, permeability was chosen as the main criteria for membrane selection. As TFC-SR2 was no longer commercially available, ESNA1LF2 and NF270 were selected for the pilot-scale study.

3.2. *Simultaneous evaluation of NF270 and ESNA1LF2 in a pilot plant*

Two NF membranes were selected from previous laboratory experiments, NF270 4040 and ESNA1LF2 4040, as a result of their permeability, similar THMFP reduction and different salt rejection. Both membranes were tested in parallel at the same operational recovery and TMF in a pilot-scale plant.

Fig. 7 shows the normalized permeate flow for both membranes during the whole study.

Three chemical cleanings using NaOH and sodium dodecylsulfate (SDS) were realized in order to recover the permeate flow (25/5/2011, 12/7/2011 and 01/8/2011). At the final of the pilot plant experimentation, two modules were sacrificed in order to analyze the cause of permeability decrease. No evidence of fouling deposition was detected. Another reason to explain TMF decline could be the membrane compaction [41]. Not enough filtered water by UF could be supplied in

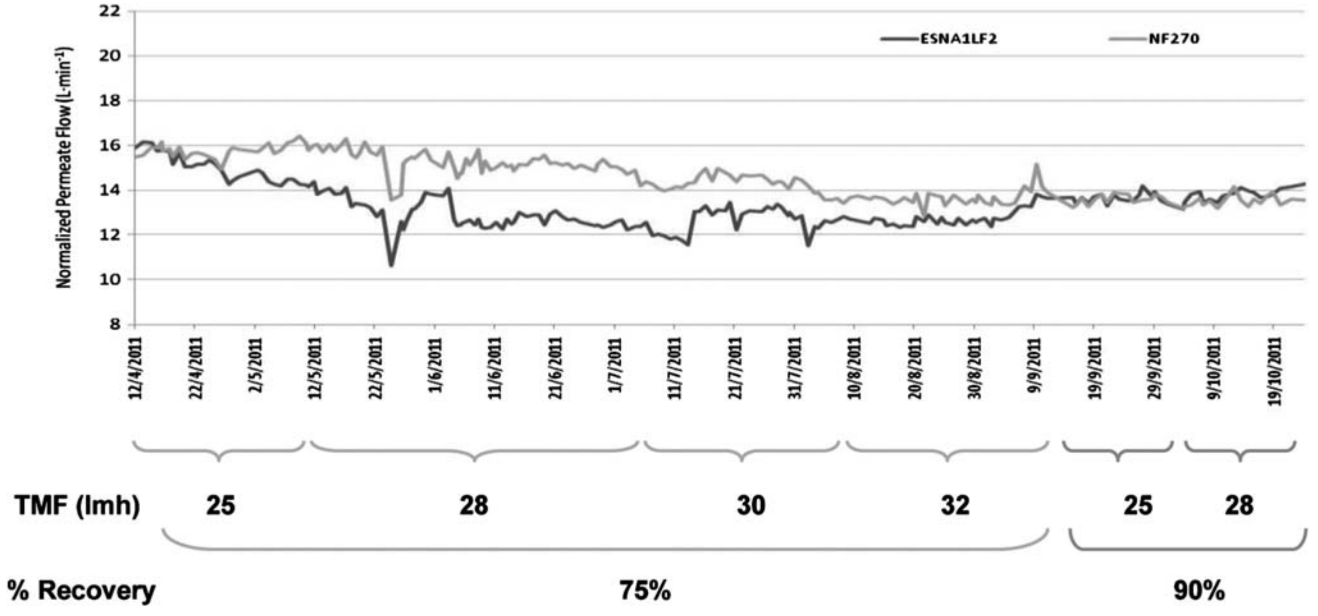

Fig. 7. Normalized permeate flow of ESNA1LF2 and NF270 membranes.

Table 4
Average component rejection during NF process at pilot plant experimentation

Parameter	NF270		ESNA1LF2	
	Average rejection (%)	Standard deviation (%)	Average rejection (%)	Standard deviation (%)
Ca^{2+}	63	4	90	2
Mg^{2+}	71	4	89	2
Na^{+}	32	4	56	9
K^{+}	36	6	63	7
HCO_3^{-}	43	4	74	5
SO_4^{2-}	98	1	99	0.4
Cl^{-}	7	4	71	5
Conductivity	57	4	83	5
NPOC	96	2	97	2
UVA 254	99	2	99	2

order to continuously feed the NF membranes which resulted in intermittent operation of the pilot plant which could lead to membrane compaction.

Table 4 shows the average rejection values of inorganic feed water components.

Lower salt rejection for NF270 has some beneficial implications for DWTPs where the goal is to decrease THMFP. Salt rejection is related to the membrane permeability. Higher salt rejection results in higher osmotic pressure differences in both membrane sides which imply higher feed pressures and energy requirements. NF270 required a feed pressure between 3 and 4 bars while ESNA1LF require a feed pressure between 4 and 5 bars. Furthermore, lower corrosive potential of permeate water will result in lower salt rejection which will decrease the post-treatment costs.

Fig. 8 represents the NF270 and the ESNA1LF2 ability to decrease THMFP in feed water.

THMFP reduction was nearly proportional to the percentage of permeate water in both membranes tested. A minimum of 50% of permeate water ensures a THMFP lower than 100 μg THM l^{-1} in all the experimental period. Even in the warm period between June and September, when the highest THMFP in the feed water was observed and membranes run at the highest TMF and recovery, both membranes demonstrated their ability to significantly reduce the THMFP.

Variability in temperature, NOM composition, and biological activity could affect THMFP. Specific ultraviolate absorbance (SUVA) (UVA/NPOC ratio) could act as an indicator of the THMFP surface waters [30,42,43]. Fig. 9 shows the relationship between THMFP of the feed and filtered water and its SUVA values.

This results show that SUVA is closely related to the THMFP of the studied water. Consequently, THMFP of the permeate samples is also linked to the

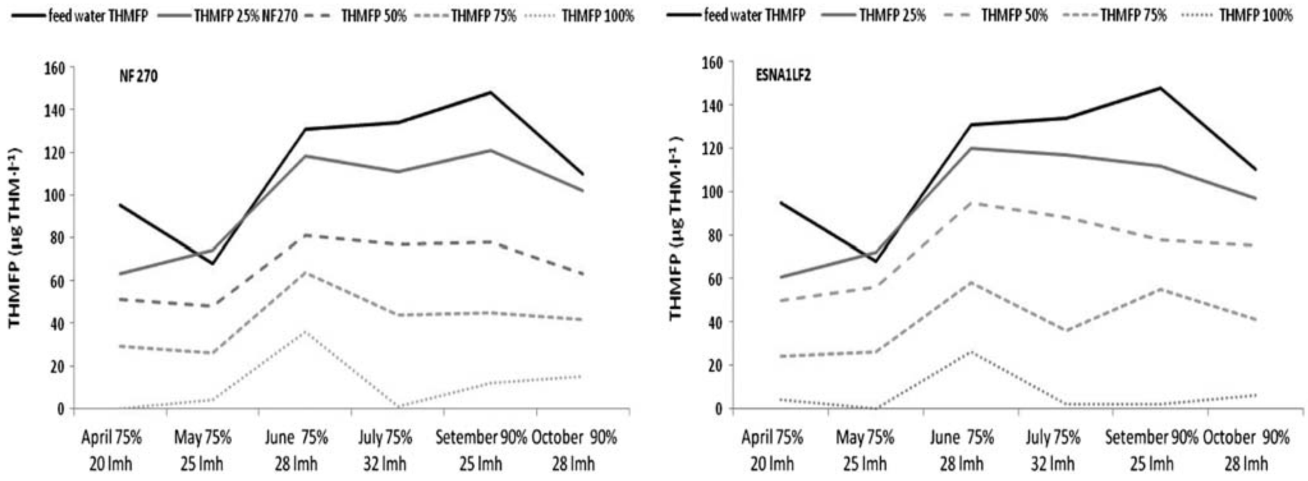

Fig. 8. THMFP reduction in blended water samples of NF permeate and feed water. Blending ratio indicates the percentage of filtered water to the feed water.

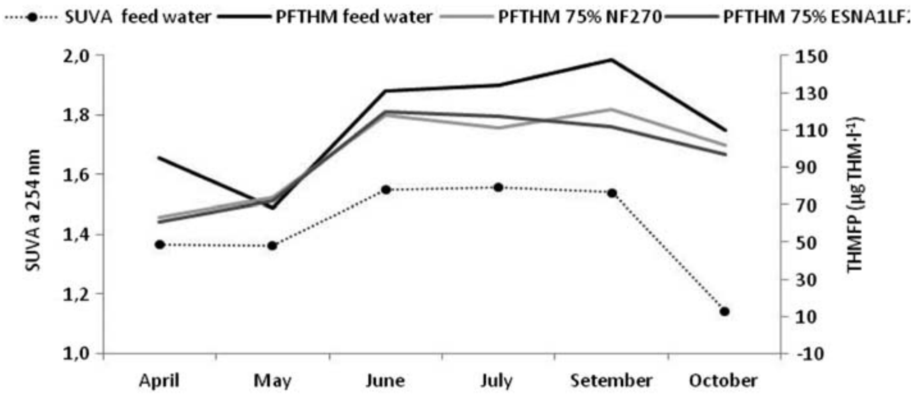

Fig. 9. Relation between SUVA ratio and THMFP in feed and permate water samples.

SUVA values of the feed water. Results obtained show that THMFP of the filtered water is mainly related to the NOM concentration and composition of the feed water and not to the TMF, recovery or feed water temperature at which membranes are operated. This means that low molecular weight fraction of the NOM is permeable to both membranes studied, resulting in a residual THMFP in the permeate water [39,40].

4. Conclusions

Nine NF commercial membranes were compared at laboratory scale showing different performances regarding salt removal and permeability but similar ability to remove THM precursor material from surface water at Manresa DWTP.

NF270 and ESNA1LF2 were selected for testing in equal operational conditions at pilot scale. The main difference between both membranes was salt rejection. ESNA1LF2 showed the highest rejection of inorganic ions resulting in higher feed pressure requirements to maintain the same permeate flow than NF270. Furthermore, permeate water from ESNA1LF2 showed the lowest bicarbonate content which increases its corrosive power.

Both membranes showed high rejections of THM precursor material, which resulted in very low THMFP in the permeate water. A blending ratio of 0.5 between filtered and feed water ensured a THMFP lower than $100\,\mu\mathrm{g\,THM\,l}^{-1}$ in all cases regardless of the seasonal and operational conditions variation (TMF and total water recovery).

Feed water showed higher THMFP in the period between June and September. Seasonal variation considering higher temperature, biological activity, and NOM concentration in the warm period could explain this increase. SUVA values were closely related to the THMFP of the feed water, which demonstrated the ability of this parameter to indicate the THMFP of the studied water. Furthermore, residual THMFP in the filtered samples could be related to the presence of permeable low molecular weight THM precursor material.

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