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Nanofiltration vs. reverse osmosis for the removal of emerging organic contaminants in water reuse

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ABSTRACT

Reverse osmosis (RO) in existing water reuse facilities is a water industry standard. However, that approach may be questioned taking into consideration that “tight” NF can be equal or “better” than RO. NF can achieve the same removals of RO membranes when dealing with emerging organic contaminants (pharmaceuticals, pesticides, endocrine disruptors and others). Experiments using 18 emerging contaminants were performed using membranes NF200 and NF90 at bench-scale units, and for a more complete study, results of NF and RO pilot and full-scale experiments where compared to our experimental results. The removal results showed that NF can remove many emerging contaminants. The average removal by tight NF was 82% for neutral contaminants and 97% for ionic contaminants. The average removal by RO was 85% for neutral contaminants and 99% for ionic contaminants. Aquifer recharge and recovery (ARR) followed by NF can effectively remove emerging contaminants with removals over 90% when loose NF membranes are used.

Keywords: Water reuse; Nanofiltration; Reverse osmosis; Emerging contaminants

1. Introduction

Water stress in many regions of the world is leading to an increase of water reuse projects. In principle municipal wastewater is being reclaimed by using membrane technology in the Netherlands [1], Kuwait [2], Australia [3], Singapore [4], just to name a few cases worldwide. The municipal wastewater of the city of Terneuzen (Zeeland, Nederland) is providing Dow Benelux B.V. (Terneuzen) of water treated after microfiltration (MF) and reverse osmosis (RO) [1]. The booming of water reuse projects is expected to increase mainly driven by population growth, climate change and urbanization in areas where water scarcity can pose a serious problem. For instance, in Spain, the Spanish government is setting up a new plan for water reuse and recycling in 2010; as a result, reuse and recycling is expected to increase from 368 million m^3/yr by the end of 2009 to one billion m^3/yr by 2015 [5–6]. The benefit of water reuse is undeniable; however there are concerns of contamination or prevalence of emerging organic contaminants in the product. Those emerging organic contaminants enclose pharmaceutically active compounds (PhACs), endocrine disrupting compounds (EDCs), personal care products (PCPs) and other organic compounds used in the industry. The organic contaminants are either only moderately or not removed during conventional wastewater treatment as well as subsequent conventional drinking water treatment [7–9]. As a result, organic compounds have been detected in many surface waters in

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the Netherlands [10], China [11], and many other countries. Moreover, a recent study reported the presence of PhACs, EDCs and other unregulated organic contaminants in U.S. drinking waters [12]. Nanofiltration (NF) and RO are membrane technologies that remove most of the emerging organic contaminants [13–18]. Although many of these studies have been conducted at laboratory scale, they have identified the main mechanisms of removal occurring through interactions between organic solutes and membranes: size/steric exclusion, electrostatic repulsion, and hydrophobic adsorption and partitioning. Only a few studies have reported full-scale results of NF and RO for removal of emerging organic contaminants during water treatment for groundwater recharge or drinking water treatment [19–21]. The trend in practical implementation of water reuse technology has favoured RO instead of NF. We investigate and compare both technologies in terms of advantages and disadvantages of their capabilities for reclaiming wastewater achieving an acceptable removal of emerging organic contaminants, and make conclusions about which one should be chosen when considering water reuse projects.

Table 1 describes a comparison of NF and RO for water treatment. It can be seen that NF is advantageous after a rapid evaluation in terms of energy consumption and water quality. NF membranes are designed to operate at pressures lower than RO membranes; this translates into less operating costs in terms of energy consumption. Another advantage of NF that sometimes does not receive much attention is its capability to allow the necessary passage of salts that water needs to contain in many applications. This fact also translates in less operating costs of post-treatment. Nonetheless, the actual market favours the popularity and therefore the use of RO membranes. RO applications in brackish water treatment and desalination are well-known.

Table 1
A general comparison between NF vs. RO

Situation	NF	RO
Effective pore size (range)	1–2 nm	< 1 nm
Energy consumption	Low to moderate	High
Removal of salts	Moderate	High
Post-treatment for addition of salts (ions)	Not necessary	Necessary
Removal of contaminants (micropollutants)	Low to high, depending on “tight” or “loose” membrane and type of contaminant	Low to high, depending on type of contaminant
Membrane availability	Low to moderate	Plentiful
Types of membranes by manufacturer	Few	Many

2. Experimental and methodology

The list of emerging contaminants with their respective physicochemical properties is presented in Table 2; the selection of organic contaminants was based on their occurrence in water environments impacted by wastewater treatment plant effluents. Compounds were classified as ionic or neutral according to presence or absence of ionic species at pH 7. Size descriptors included in Table 2 are: molar volume (MV), molecular length, and equivalent molecular width. The molecular length is defined as the distance between the two most distant atoms of a particular molecule. The molecular width and molecular depth (width > depth) are measured by projecting the molecule on a plane perpendicular to the length axis. The equivalent molecular width is defined as the geometric mean of width and depth [22]. The octanol-water partition coefficient (log Kow), often used to describe hydrophobicity, is a measure of the equilibrium concentration of a compound between octanol and water. The ratio of the equilibrium concentrations of all species (ionised and unionised) of a particular molecule in the octanol phase and in the water phase is expressed as log D; it differs from log Kow in that ionised species as well as the neutral form of the molecule are considered. Values of log D were calculated by ADME/Tox web software. Values of log Kow were obtained from SRC Physprop experimental database. The dipole moment was calculated by Chem3D Ultra 7.0 software (Cambridgesoft). Molecular Modeling Pro (ChemSW) was used to compute molar volume, molecular length and equivalent width.

The pharmaceutical compounds (fenoprofen, ketoprofen, gemfibrozil, caffeine, sulfamethoxazole, acetaminophen, phenacetine, phenazone, carbamazepine, naproxen, ibuprofen, metronidazole), endocrine disrupting compounds (17α-ethynilestradiol, 17β-estradiol, estrone, bisphenol A, atrazine) and 1,4-dioxane were purchased from Sigma-Aldrich (Schnelldorf, Germany). Potassium chloride, sodium hydroxide, hydrochloric acid and magnesium sulphate anhydrous were purchased from J.T. Baker (Deventer, Netherlands). The organic contaminants were analyzed by Technologiezentrum Wasser (TZW, Karlsruhe, Germany). The detection limit was 10 ng/l per compound. The uncertainty of estimates was ±15% according to a validation method of the analytical

Table 2
List of organic contaminants in feed water

Name	Name ID	MW (g/mol)	log K_{ow} [a]	log D[b] (pH 7)	Dipole moment (debye)[c]	Molar volume[d] (cm^3/mol)	Molec. length (nm)[d]	Equiv. width (nm)[d]	pK_a [b]	Classification
Acetaminophen	ACT	151	0.46	0.23	4.55	120.90	1.14	0.53	10.2	neutral
Phenacetine	PHN	179	1.58	1.68	4.05	163.00	1.35	0.54	n.a.	neutral
Caffeine	CFN	194	–0.07	–0.45	3.71	133.30	0.98	0.70	n.a.	neutral
Metronidazole	MTR	171	–0.02	–0.27	6.30	117.80	0.93	0.66	n.a.	neutral
Phenazone	PHZ	188	0.38	0.54	4.44	162.70	1.17	0.66	n.a.	neutral
1,4-dioxane*	DIX	88	–0.27	–0.17	0.00	89.10	0.71	0.59	n.a.	neutral
Carbamazepine	CBM	236	2.45	2.58	3.66	186.50	1.20	0.73	n.a.	neutral
17β-estradiol	E2	272	4.01	3.94	1.56	232.60	1.39	0.74	10.3	neutral
Estrone	E1	270	3.13	3.46	3.45	232.10	1.39	0.76	10.3	neutral
Bisphenol A	BPA	228	3.32	3.86	2.13	199.50	1.25	0.79	10.3	neutral
Atrazine	ATZ	216	2.61	2.52	3.43	160.07	1.26	0.74	n.a.	neutral
17α-ethynilestradiol	EE2	296	3.67	3.98	1.27	225.60	1.48	0.85	10.3	neutral
Sulfamethoxazole	SFM	253	0.89	–0.45	7.34	173.10	1.33	0.64	5.7	ionic
Fenoprofen	FNP	242	–0.02	0.38	1.88	180.90	1.16	0.83	4.3	ionic
Ketoprofen	KTP	254	–0.52	–0.13	3.42	187.90	1.16	0.83	4.3	ionic
Naproxen	NPN	230	3.18	0.34	2.55	192.20	1.37	0.76	4.3	ionic
Ibuprofen	IBF	206	3.97	0.77	4.95	200.30	1.39	0.64	4.3	ionic
Gemfibrozil	GFB	250	4.77	2.30	0.95	221.90	1.58	0.78	4.9	ionic

n.a. data not applicable because the compound does not ionize.
[a] Experimental database: SRC PhysProp Database.
[b] ADME/Tox Web Software.
[c] Chem3D Ultra 7.0.
[d] Molecular Modeling Pro.
*only present in NF90 feed water.

protocol. Recoveries were between 70 and 100%. For group A (acetaminophen, caffeine, carbamazepine, ibuprofen, naproxen, phenacetine, phenazone, fenoprofen, gemfibrozil and ketoprofen), the analysis was conducted by a HPLC-MS-MS method. More information about the analytical protocol of group A had been published [23]. For group B (metronidazole and sulfamethoxazole), the analysis was conducted by a HPLC-MS-MS method. More details about the method for group B can be found elsewhere [24]. For group C (17β estradiol, estrone, bisphenol A, 1,4-dioxane and 17α-ethynilestradiol), the analysis was conducted by GC-MS. For group C, more analytical details are described in a previous publication [25].

A bench-scale cross-flow filtration apparatus (Fig. 1, SEPA CF II, GE Osmonics) was used for experiments with Filmtec NF membranes NF200 and NF90. A detailed description of the NF experimental apparatus and analytical equipment used for monitoring and membrane characterisation has been previously published by the author [26–27]. Micropollutants were spiked to the feed water as a cocktail, with individual concentrations ranging from 8 to 17 µg/l In addition, the laboratory-scale soil column that simulated river bank filtration removal experiments also referred as aquifer recharge and recovery, has been described by Maeng et al. in a previous publication [28].

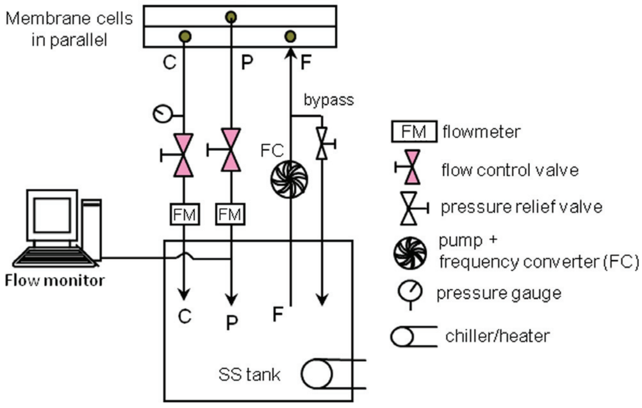

Fig. 1. Piping and instrumentation diagram of filtration apparatus.

3. Results and discussion

3.1. Removal of emerging organic contaminants by NF

The removal of neutral contaminants by NF membranes is shown in Fig. 2. First of all, NF200 results are shown in the front and are compared to the results of the NF90 membrane. The NF200 membrane is defined as a "loose" membrane having a reported (from the manufacturer) MWCO of approx. 300 Da. It can be seen that removals by NF200 were always lower than the removals achieved by the NF90 membrane. The removals of neutral contaminants by NF200 varied in the range of 23% (for ACT) to 90% (for EE2). On the other hand, removals of neutral contaminants by NF90 were between 47–97%, with DIX being the compound with lowest removal. The NF90 membrane removed contaminants more efficiently than NF200 due to his reduced (compared to NF200) effective pore size (~1 nm); which can be translated or equivalent to a MWCO of 200 Da (as reported by the manufacturer). The removals of a third membrane, Desal HL (DeHL), are also shown in Fig. 2. The data was taken from a recent publication by Verliefde et al. [29] that produced experimental data in a pilot-scale NF unit for the construction and validation of a full-scale model able to model rejection of organic micropollutants (emerging organic contaminants) by NF membranes. The investigation of Verliefde et al. [29] used additional contaminants (pharmaceuticals and pesticides) as shown in Fig. 2: aminopyrine (APY), cyclophosphamide (CYP), pentoxifylline (PXF), metribuzim (MEB), pirimicarb (PIR) and dimetheamid (DMA). The NF membrane Desal HL is reported to have a MWCO between 150-300 Da. This fact may explain why the removals by DeHL for PHN (82% vs. 93% by NF90), CBM (84% vs. 91% by NF90) and ATZ (70% vs. 97% by NF90) were lower (as shown in percentages) than the removals achieved by NF90. This is just part of the explanation, certainly other factors, such as the different operating conditions: pilot scale at 75% recovery vs. bench-scale at 8% recovery, influenced the differences in removal. Nonetheless, the mechanisms of removal were: i) steric hindrance, with increased removals by increased length and equivalent width; ii) hydrophobic partitioning, expressed as log K_{ow} or log D, with decreased removals due to adsorption and subsequent partitioning mechanisms. These removal mechanisms are in accordance with findings from the literature [14,16–18,30–37] and our previous publications [26,38]. The problem of a not well defined membrane such as Desal HL (tight or loose, or something in between) for effects of removal prediction can be resolved when considering salt rejection of magnesium sulphate as a parameter incorporating steric/size hindrance instead of a MWCO [38]. The removal of BPA by NF200 is particularly interesting because hydrophobic and partitioning interactions were identified, which diminished its removal. In a different way, for the NF90 membrane, the effect of hydrophobicity of contaminants and dipole moment had no impact on increase/decrease of rejection, mainly due to a tighter effective pore size that did not allow partitioning interaction of the compound within the top layer of the membrane.

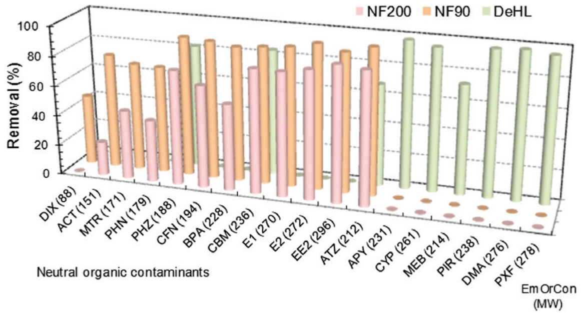

Fig. 2. Removal of neutral contaminants by NF membranes (empty spots mean no data available).

The removals of ionic contaminants are reported in Figs. 3 and 4 for negatively charged ionic contaminants and positively charged ionic contaminants, respectively. The study of Verliefde et al. [29] used ionic-positive contaminants, the removals by the Desal HL membrane were higher than 81% (Fig. 4), in this case mainly due to effects of steric hindrance. The sizes of terbutaline (TER),

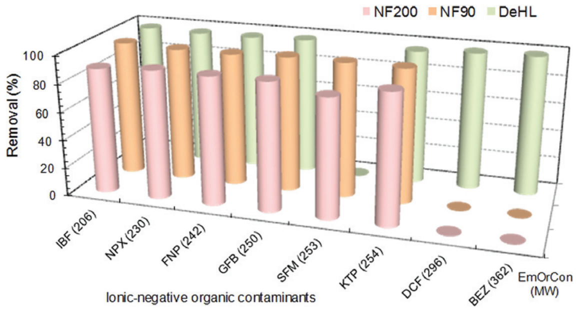

Fig. 3. Removal of ionic (negative) contaminants by NF membranes (empty spots mean no data available).

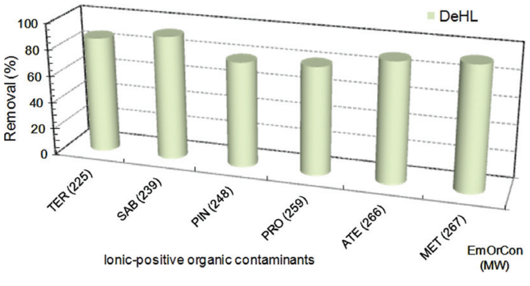

Fig. 4. Removal of ionic (positive) contaminants by NF membranes (data is available only for Desal HL).

salbutamol (SAB), pindolol (PIN), propanolol (PRO), atenolol (ATE), and metoprolol (MET) are greater than 1.34 nm and 0.73 nm in terms of length and equivalent width, respectively. Contrarily, for ionic-negative contaminants the removal mechanism is different, the existence of electrostatic interactions between the negative charge of the membrane surface and the negative charge of the ionic compounds results in high removals even for a loose membrane such as NF200 (Fig. 3) and for small compounds in size, such as ibuprofen and naproxen (with eqwidth 0.64 and 0.76), or fenoprofen (FNP) and ketoprofen (KTP), both with a length of 1.16 nm. Previous studies have also identified that electrostatic repulsion is an important rejection mechanism of ionic-negative organic contaminants [33,39–41].

This study also evaluated the behaviour of NF after aquifer recharge and recovery (ARR) experiments conducted to remove "naturally" organic contaminants during passage of water through soil columns. Biodegradation of organic contaminants occurred during soil column experiments. Biodegradation was confirmed after adding a biocide (sodium azide) and determining a negative effect on removals. As shown in Fig. 5, removal results of contaminants for ARR followed by NF were very effective. ARR combined with NF200 removed contaminants at least with an efficiency of 97%. And ANN combined with NF90 removed contaminants at least with an efficiency of 99%. Carbamazepine (CBM) a compound not removed by ARR (0%) was well removed by NF200 (82%) and NF90 (91%), which means that a combination of ARR and NF results beneficial.

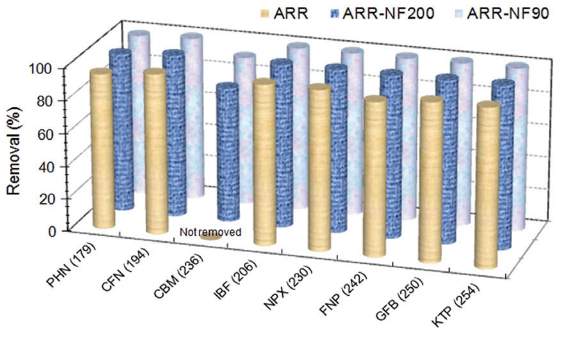

Fig. 5. Removal of contaminants by a combination of ARR-NF (removal by ARR for CBM was 0%).

3.2. *NF vs. RO for removal of contaminants*

The removals of the NF90 membrane determined at bench-scale were compared with removals of NF90 and RO membranes at pilot and full-scale configurations. For that purpose, removals of three other studies [19–21] that included RO membranes and the NF90 membrane during pilot and full-scale tests were used. A summary of the membrane characteristics and experimental conditions is shown in Table 3.

Table 3
List of membranes and operating conditions used for comparison of NF and RO

Name	Test	MWCO (Da) SR, NaCl (%)	Feed conc. (µg/l)	pH	Operating flux (l/m^2-h)	Recovery (%)	Pressure (kPa)	Zeta potential @ pH 7 Ionic strength 10mM KCl (mV)	Pure water perm. @ 20°C (l/day-m^2-kPa)
NF200*	Bench-scale	~300 72.0	5–18	7	13	8	483	–28	1.01
NF90*	Bench-scale	~200 90.0	5–18	7	13	8	345	–32	2.23
NF90[*,a,b]	Pilot-scale	~200 90.0	0.01–2.6	6–7	20[a]	10[b], 80[a]	n.a.	n.a.	2.23
NF90[*,c]	Full-scale	~200 90.0	0.008–0.14	6	23	65	n.a.	n.a.	2.23
BW30LE[*,b]	Pilot-scale	<200 99.0	0.01–0.5	7	n.a.	10	n.a.	n.a.	<2
BW30LE[*,c]	Full-scale	<200 99.0	0.008–0.14	6	23	73	n.a.	n.a.	<2
ESPA2[¤,a]	Full-scale	<200 99.5	0.05–2.6	6–7	17	80	n.a.	n.a.	<2

*Dow-Filmtec, [¤]Hydranautics; SR (salt rejection, 2,000 mg/l, 480 kPa, 25°C and 15% recovery).

[a] Bellona et al., 2008 [20].

[b] Schrotter et al., 2009 [21].

[c] Radjenovic et al., 2008 [19].

The average removal efficiencies of tests with bench, pilot and full-scale experiments using NF90 were compared to average removal efficiencies of tests with bench, pilot and full-scale experiments with two RO membranes (BW30LE and ESPA2). The results are shown in Fig. 6 for neutral contaminants. The removal of ACT was of 72% during a full-scale test [19], NF90 removed ACT by 67%, a small difference. Similarly, the difference in rejection for PHN was only 10% between NF90 and ESPA2. The pilot-scale investigation of Bellona et al. [20] using NDMA revealed a 45% removal by NF90, while a full-scale test showed NDMA removal of 35% with ESPA2, an RO membrane. More importantly, all contaminants with a MW>200 were removed by NF90, BW30LE and ESPA2 with rejections over 90%.

Regarding ionic contaminants, Fig. 7 demonstrates that NF and RO membranes are almost equally effective for most of the contaminants. Moreover, low molecular weight ionic contaminants such as salicylic acid (SAC) can also be removed by NF or RO. The average removal by tight NF was 82% for neutral contaminants and 97% for ionic contaminants. The average removal by RO was 85% for neutral contaminants and 99% for ionic contaminants. Therefore, NF compares in removal efficiency of contaminants to RO. But in addition, NF can be more efficient if other advantages are taken in consideration, as previously discussed in section 3.2.

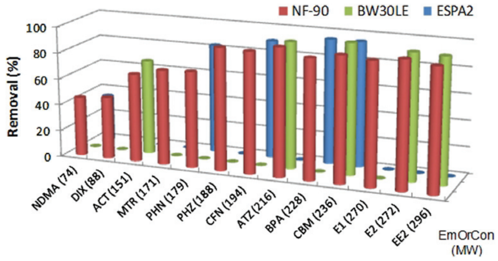

Fig. 6. Removal of neutral contaminants by NF vs. RO.

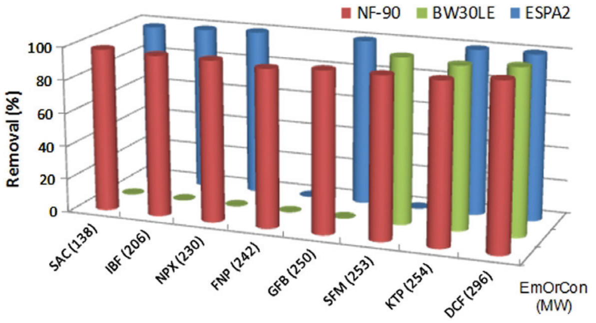

Fig. 7. Removal of ionic contaminants by NF vs. RO.

4. Conclusions

- NF vs. RO for the removal of emerging organic contaminants is a debaTable topic. This study showed that NF may be a winning option when applied properly.
- One of the advantages of NF is its low energy consumption, and less expenditure in post-treatment.
- The removal efficiencies of NF are very close to those achieved by RO membranes, which is beneficial when operating costs are considered instead of (not existing) water quality regulations for emerging contaminants.
- NF after aquifer recharge and recovery can be a more cost-effective solution than expensive RO systems used in water reuse projects.

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