Removal of bisphenol A by a nanofiltration membrane in view of drinking water production

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ABSTRACT

The efficiency with which a nanofiltration membrane (Desal 5 DK) removes bisphenol A (BPA) was investigated, together with the mechanisms involved. Whereas high retention ($>90\%$) was obtained at the beginning of the filtration, the observed retention coefficient (R_{obs}) decreased to around 50% when the membrane became saturated, due to adsorption of BPA onto the membrane structure. The presence of ions (Na^+, Cl^-) affects the R_{obs}, this effect being attributed to a change in BPA hydrodynamic radius. Moreover, in our operating conditions, the presence of natural organic matter (1 mg/L) in the feed solution does not lead to variation in BPA retention at steady state.

1.   Introduction

Recently, evidence has emerged that some chemicals (e.g. estradiol, nonylphenol, pharmaceuticals and some pesticides) described as endocrine disrupting chemicals (EDCs), can interfere with endocrine systems if they are present in drinking water at a certain concentration. Bisphenol A (BPA, CASRN: 80-05-7), one of these EDCs, is a white solid which is used as an intermediate in the manufacture of epoxy, polycarbonate, polysulfone and certain polyester resins. Since 1993, when Krishnan documented that BPA was released from polycarbonate flasks during autoclaving and had estrogenic activity, the effects of BPA on health have become a controversial issue (Krishnan et al., 1993; Kagawa et al., 2004).

Studies of the removal of EDCs such as BPA during water treatment have been limited due to the low concentration of these components in water sources and the associated difficulties in analysis. Nanofiltration (NF) and reverse osmosis (RO), as promising membrane technologies, could be alternative methods for removing low molecular weight organic micropollutants, particularly pesticides (Van der Bruggen et al., 1999; Kiso et al., 2000) and pharmaceutically active compounds (PhACs) (Kimura et al., 2004) and EDCs (Nghiem et al., 2004).

Some researchers consider that the retention of uncharged organic molecules by NF membranes is mainly determined by the sieving mechanism and that solute transport takes place by convection due to a pressure difference and by diffusion due to a concentration gradient across the membrane

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(Kosutic and Kunst, 2002; Schaep et al., 1998). Others consider that as NF membranes span the gap between UF and RO membranes, the sorption–diffusion mechanism in a non-porous structure can contribute to the separation process. According to the Stokes–Einstein law, there is an inverse proportionality between the diffusion constant and the size of a component, so the diffusion rate will be smaller for a larger component, resulting in a decrease in solute transfer. A similar tendency (decrease in transfer as solute size increases) is obtained by considering a size exclusion mechanism. However, in some cases, pollutants with molecular weights greater than the membrane MWCO were still detected in the permeate during the NF/RO process (Van der Bruggen et al., 1999). Physico-chemical parameters such as molecular size, hydrophobicity, polarity or feed solution composition are then considered important and their effects on NF efficiency investigated (e.g. Van der Bruggen et al., 1999) but in most cases their individual contributions to membrane retention, not to mention their combined effects, are not well identified. Moreover, most of the studies already published have been conducted with pollutant concentrations higher than those that can be found in water sources. When evaluating the performance of membranes in removing organic micropollutants such as EDCs/PhACs, experiments should be conducted at a realistically low concentration. Finally, the solution matrix can also affect the efficiency of the process. Various papers published report the effect of NOM on retention of pollutants. In some cases the retention is reduced in the presence of NOM (Nghiem et al., 2002), in some cases it is raised (Devitt et al., 1998) and in other work almost no effect on total pollutant retention was observed (Taylor et al., 2000).

Concerning the elimination of BPA, there are very few studies on its removal from water sources, except those concerning an electrochemical process (Kuramitza et al., 2004), photo-oxidation (Zhou et al., 2004) or sorption (Ying et al., 2003). Few publications have reported the retention of BPA by NF/RO membranes (Kimura et al., 2004; Agenson et al., 2003), the first paper cited using a contaminant concentration of 100 ppb ($\mu\text{g L}^{-1}$) and the second, a concentration of 50 ppb.

The objective of this study was to acquire a better understanding of the transfer mechanisms of BPA through a NF membrane. With this aim in view, we have evaluated the removal efficiency of BPA by the membrane and investigated the factors influencing retention, such as transmembrane pressure, feed composition and BPA concentration, this last parameter being chosen in the range of concentrations that are found in water sources (lower than 10 ppb).

2. Materials and methods

2.1. Membrane and chemicals

The NF membrane Desal 5 DK (supplied by GE Osmonics) used in this study is a three-layer thin-film polysulfone-based membrane with a polyamide top layer.

The endocrine disrupter selected, BPA, is hydrophobic (n-octanol/water partition coefficient $K_{OW} > 2$). In order to use low contaminant concentrations (ranging from 1 to 10 ppb), molecules radio-labeled with ^{14}C were used. Scintillation liquid was added to the samples before analysis with a scintillation counter (1500 Tricares Packard). The detection limit was 0.02 ppb. The BPA concentrations were determined with an accuracy of ± 0.02 ppb.

All the solutions were prepared from ultra pure water (Milli-Q). They were filtered at their natural pH ranging from 5 to 6 (stable during experiments) for which the molecules were neutral and the membrane carried negative charges (Hagmeyer and Gimbel, 1999). General information on the membrane and the BPA molecule is reported in Table 1.

To study the influence of solution ionic strength on the removal of BPA, NaCl (Aldrich) was added to the Milli-Q water to reach the ionic strength 10, 50 or 100 mM, and this solution was spiked with ^{14}C-BPA up to a concentration of 1 ppb. To investigate the effect of the presence of NOM, a solution containing 1 ppm (mg/L) of NOM (Suwannee River 1R101, IHSS) and 1 ppb of BPA was prepared and left to stand for 24 h in order to allow contact between the NOM and BPA before filtration.
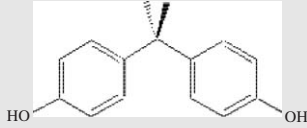
2.2. Equipment and filtration protocol

All experiments were carried out in a dead-end batch filtration cell (Berghof), with magnetic stirring (see Causserand et al., 2005). The stirring rate was maintained constant over all the experiments. The trans-membrane pressure was set by air pressurization of the cell (in the range $5 \times 10^{+5}$–$20 \times 10^{+5}$ Pa). The filtrate flux J ($\text{m}^3\,\text{m}^{-2}\,\text{s}^{-1}$) was measured with an accuracy of 0.5% by timed collection using an electronic balance (± 0.01 g). All experiments were performed at room temperature ($20 \pm 2\,^\circ$C).

A new membrane (effective area $3.52 \times 10^{-3}\,\text{m}^2$) and a new O-ring gasket were used for each experiment. Membranes were compacted for 2 h at $20 \times 10^{+5}$ Pa, prior to the determination of permeability for water, both steps using Milli-Q water. The stirred cell was then emptied and filled again with 370 ml of BPA solution. For each condition (pressure, concentration)

Table 1 – General characteristics of the nanofiltration membrane (provided by the manufacturer) and the molecule used in this study

Membrane: Desal 5 DK (GE Osmonics, USA)		Molecule: ^{14}C-BPA	
MWCO (g/mol)	200	Molecular weight (g/mol)	228
r_{pore} (nm)	0.47	Water solubility (mg/L)	120–300
Max pressure (Pa)	$40 \times 10^{+5}$	pKa	9.6–10.2 (Staples et al., 1998)
Surface charge (neutral pH)	Negative	K_{ow}	3.40
Lp (L/m² h bar)	3.2	Molecular width (nm)	0.325 (Agenson et al., 2003)
		Molecular structure and formula	$C_{15}H_{16}O_2$ or $(CH_3)_2C(C_6H_4OH)_2$

retentate and filtrate samples were collected for subsequent analysis both during and at the end of the run in order to monitor the evolution of contaminant concentration.

In Sections 3.1 and 3.2.1, in order to avoid the possible effect of the volume reduction in the cell and maintain BPA concentration in the retentate almost constant, permeate was periodically recycled into the filtration cell.

The observed retention coefficient of BPA defined as $R_{obs} = (1 - C_p/C_r) \times 100\%$ ($\pm 2\%$ in the range of concentrations experimented in this work) was calculated from concentrations measured in the permeate C_p and the retentate C_r. The amount of BPA adsorbed on the membrane, expressed in µg per m² of membrane area, was calculated by mass balance ($\pm 7\%$).

At the end of each run, the feed solution in the cell was replaced by Milli-Q water that was then filtered under the same applied pressure as the BPA solution, to evaluate the reversible and irreversible parts of the adsorption.

3. Results and discussion

3.1. Adsorption and retention of BPA by the membrane Desal 5 DK

The R_{obs} and the cumulated adsorbed BPA (1 ppb) by the membrane Desal 5 DK as function of volume filtered at $20 \times 10^{+5}$ Pa are shown in Fig. 1. It can be seen that R_{obs} decreased significantly, from 89% at the beginning of filtration to 47%, when an almost stable value was reached after 200 mL filtered, while the cumulated quantity adsorbed reached the value 30 µg m^{-2}. Obviously, as already observed with other pollutant-NF membrane systems (Van der Bruggen et al., 1999), the MWCO of the membrane (200 g mol^{-1} for Desal 5 DK) is not a reliable parameter for accurately predicting retention of EDCs such as BPA with MW 228 g mol^{-1}. This is easily understood by the fact that the physico-chemical properties of a standard solute (e.g., PEG or sugar) used for determination of MWCO are often totally different from those

of organic compounds like BPA. In this study, the decrease in apparent retention versus volume filtered can be explained by the adsorption of BPA onto the membrane surface or structure at the beginning of each run. As a consequence, an accurate evaluation of the membrane in terms of BPA retention is not possible until the membrane has been completely saturated (Kimura et al., 2003). The high concentrations used by other researchers are probably the reason why membrane saturation is seldom observed in their results as it happens unrealistically fast.

At steady state (corresponding approximately to equilibrium in terms of BPA adsorption onto the membrane) a low retention coefficient was obtained. Because BPA is hydrophobic in nature, it is probably smaller in size in aqueous solution than a hydrophilic molecule of same molecular weight (Braeken et al., 2005). The molecule width of BPA deduced from calculation (Agenson et al., 2003) compared to average pore radius (Table 1) would allow a Ferry retention coefficient to be calculated at 80% when experimental retention ranges from 40% to 60%. That is to say, in pollutant-membrane system for which the retention is mainly controlled by size exclusion, the size parameter of the molecule that should be considered is the hydrodynamic radius, especially for molecules whose size is in the range of membrane pore radii. This is confirmed in Section 3.2.2.

The adsorption of pollutant onto the membrane can be physical or chemical in nature or both. The former is a completely reversible process, while the latter can be irreversible for strong chemical bonds such as polymerisation or reversible for weak secondary chemical bonds such as hydrogen bonding and complexation. In a membrane filtration process involving trace organics it is possible that both chemical (hydrogen bonding) and physical (hydrophobic interactions) adsorption occurs. In our experiments, whatever the initial concentration of the BPA solution filtered, 30% of total BPA in the filtration cell can adsorb onto the membrane. When, in a second step, the BPA solution in the cell is replaced by water, significant desorption to the permeate side can be observed as shown in Fig. 2. Around 30% of the
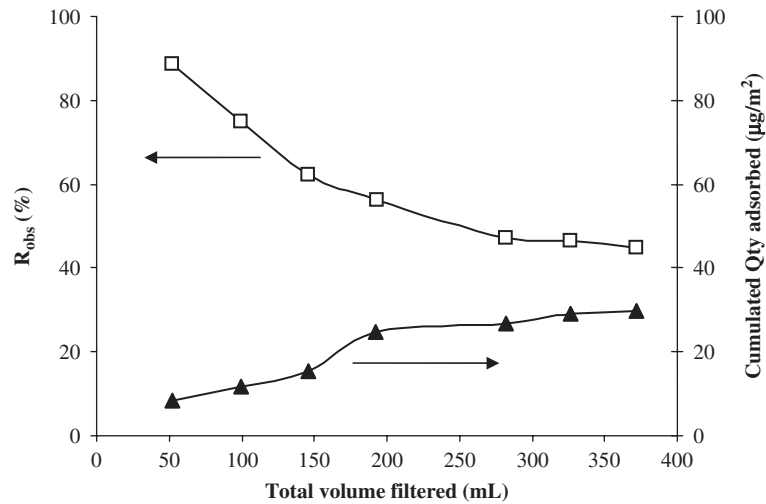
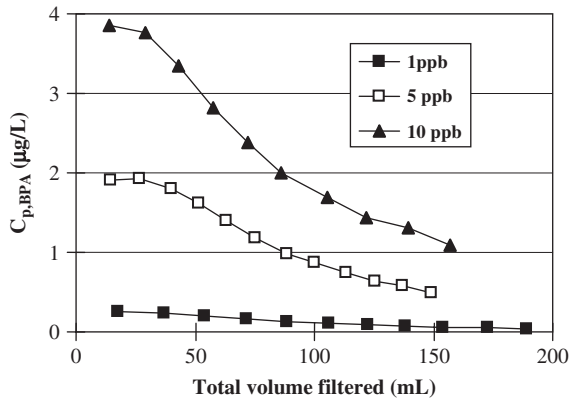**Fig. 1 – R_{obs} and cumulated quantity of BPA (1 ppb) adsorbed by the membrane Desal 5 DK versus total volume filtered $(20 \times 10^{+5}\,Pa)$.**

Fig. 2 – BPA concentration in the permeate versus volume of water filtered during desorption step $(20 \times 10^{+5}\,Pa)$ as a function of BPA concentration in bulk solution in the first filtration step.
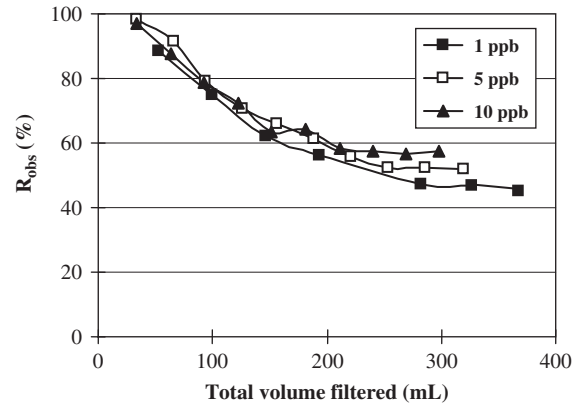

Fig. 3 – Effect of BPA concentration on the evolution of R_{obs} by the membrane Desal 5 DK $(20 \times 10^{+5}\,Pa)$.

cumulated quantity of BPA adsorbed was eluted from the membrane after filtering a volume of 200 mL. The maximum concentration in the eluted permeate corresponds to approximately 40% of the initial concentration in BPA solution used in the first step of the experiment. This result suggests that BPA-polyamide bonds are not very strong. It also means that when the water source is of good quality after a period of impaired quality, pollutants could be released from the membrane structure into the permeate, leading to a contamination over a short period of operation.

3.2. Factors influencing R_{obs} of BPA

3.2.1. Effect of BPA concentration

BPA solutions at 1, 5 and 10 ppb were prepared and filtered at $20 \times 10^{+5}\,Pa$ to study the effects of feed concentration on R_{obs}. The results reported in Fig. 3 show only a slight effect of initial concentration of BPA on R_{obs} values at steady state. A similar conclusion can be drawn from results (not shown here) at the

pressure of $5 \times 10^{+5}\,Pa$. These results are consistent with previous studies on pesticide removal conducted in a higher concentration range 300–2500 µg/L (Van der Bruggen et al., 1998).

The results presented in the above section confirm that in order to reach saturation of the membrane in our experimental set-up, 200 mL needs to be filtered whatever the concentration and pressure used. This volume corresponds to 5.7 L/m² of membrane effective area.

3.2.2. Effect of presence of salt

The effect of the presence of salt in the feed solution on the R_{obs} of BPA by Desal 5 DK was determined. Filtration was performed here under dead-end conditions (no recycling of the permeate into the cell). The retention coefficient and the filtration flux $J_{stabilized}$ were determined under steady-state conditions (after 200 mL filtered). The pressures applied to obtain these data were: 5, 10, 15 and $20 \times 10^{+5}\,Pa$. The results obtained with feeds containing BPA (1 ppb) dissolved in pure water or in NaCl solutions (IS 10, 50 and 100 mM) are
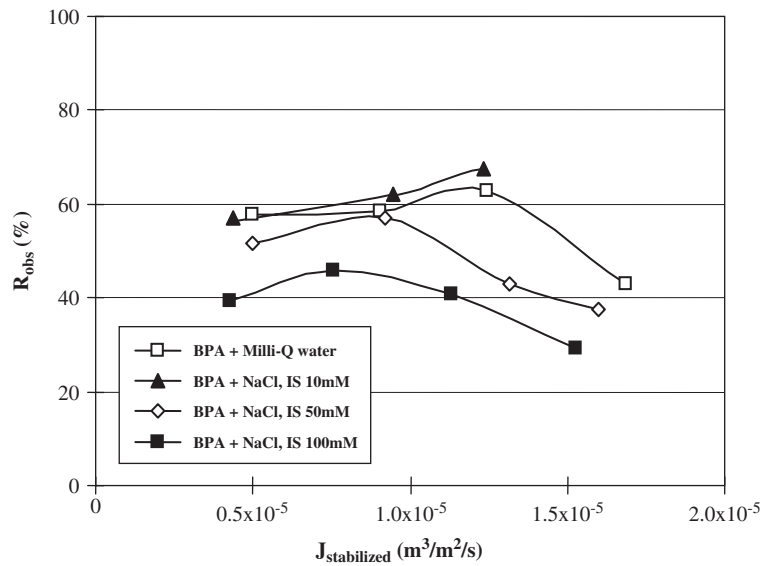
Fig. 4 – R_{obs} of BPA (1 ppb) versus stabilized permeation flux in absence and in presence of NaCl at various ionic strengths.

presented in Fig. 4 as a function of NF flux, so as to compare results at the same concentration polarization.

These results show that R_{obs} decreases beyond IS 50 mM, whereas for a lower salt concentration almost no effect on retention is observed. This decrease in retention at sufficiently high salt concentration could be explained by a swelling of the membrane pores, a decrease of the BPA hydrodynamic radius or a combination of the two phenomena (Bargeman et al., 2005; Bouchoux et al., 2005). At constant pH (that is the case in this study) the charge density of NF membranes depends on the presence of salts. Adding a salt in solution induces a greater counter-ion concentration in the electrical double-layer at the surface of the pores, this effect results in pore swelling and in compaction of the electrical double-layer. As a consequence the apparent pore radius would increase. On the other hand, in a mixed solution of BPA and salt, water preferentially solvates the salt to the detriment of BPA ("salting-out" effect). So the less hydrated BPA may present a lower apparent volume than in the absence of salt and could permeate more freely through the membrane. This effect becomes stronger when salt concentration increases, so it could explain a decrease in BPA retention when the ionic strength increases.

In order to quantify the extent of pore swelling as a function of the operating conditions used, it is shown in Fig. 5 the evolution of $J_{stabilized}$ versus effective trans-membrane pressure during the filtration of Milli-Q water and NaCl solution at IS 100 mM. The effective pressure is obtained, in the case of salt, by subtracting from the applied trans-membrane pressure the calculated osmotic pressure due to the difference in salt concentration between retentate and permeate (by taking into account the NaCl transmission determined around 70% and by using the van't Hoff law). The results show that the presence of NaCl does not modify the slope of the straight line obtained by plotting $J_{stabilized}$ vs. effective pressure, in comparison with data obtained with pure water: this indicates absence of variation in membrane permeability. As a
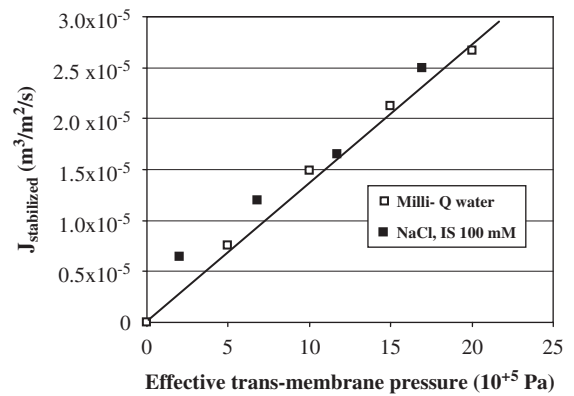

Fig. 5 – Permeation flux at steady state of Milli-Q water and solution of NaCl at IS 100 mM as a function of effective pressure.

conclusion, increase in the apparent pore radius due to the presence of NaCl of IS 100 mM seems negligible under our conditions.

Concerning the variation of R_{obs} versus $J_{stabilized}$, we observe a maximum in R_{obs}, in both cases (BPA dissolved in pure water and in NaCl solution). This behavior is characteristic of a retention mainly controlled by the sieving mechanism, where solute transport takes place by convection and by diffusion with a likely contribution from concentration polarization. The R_{obs} maximum value corresponds to the situation where concentration polarization and dispersion by diffusion in the porous medium are both negligible.

These results show that the effect of ions (NaCl) on the R_{obs} of BPA depends on ion concentration. This effect is most likely due to a change in BPA hydrodynamic radius. Moreover, the efficiency of the process in retaining EDCs such as BPA is dependent on the filtration flux with a maximum in the curve R_{obs} vs. $J_{stabilized}$, whose location is characteristic of the solute-membrane system studied. As a comparison, in a previous

study (Causserand et al., 2005) performed under the same operating conditions (same membrane, pressure and concentration ranges) with a pesticide (dichloroaniline), we obtained a range of fluxes for which R_{obs} gradually increased as $J_{stabilized}$ was decreased. The maximum in retention was not reached.

3.2.3. Effect of presence of NOM

In order to investigate the effect of fouling caused by the presence of NOM on BPA retention, two series of experiments were conducted. For the first series, the membrane permeability was measured before and after the filtration of a NOM solution (1 ppm). Then the filtration cell was filled with the BPA solution (1 ppb) for the filtration. The second series consisted in filtering the solution of BPA (1 ppb) and NOM (1 ppm), and measuring the membrane water flux before and after filtration. The effect of NOM on water flux and on BPA retention is shown in Figs. 6 and 7, respectively.

A slight decrease of 24% in membrane water permeability is observed in Fig. 6 after the filtration of NOM which means that Desal 5 DK shows good resistance to fouling by NOM
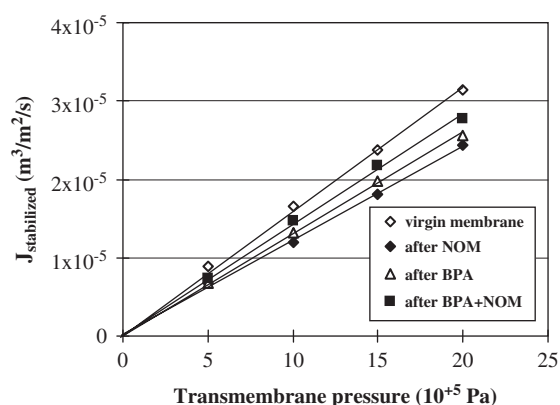
under the operating conditions used in the present study. This could be explained by a slight in-depth membrane fouling due to a 70% NOM retention. On the other hand, it could be expected that NOM adsorption onto the membrane surface is the major contribution to the decrease in water permeability.

Fig. 7 shows that the presence of NOM causes a faster decrease in BPA retention over the first 200 mL filtered in comparison with R_{obs} obtained in the absence of NOM, whereas the level of stabilized retention is not NOM dependent. Concerning the first part of the curve, the adsorption sites of the membrane surface being partly occupied by NOM molecules, the membrane exhibits a lower adsorption capacity for BPA which is detected in the permeate at a lower volume filtered. The fact that the retention at steady state is almost unchanged seems to show that NOM adsorption has no impact on membrane selectivity towards BPA and that the adsorption of BPA on NOM molecules is negligible, whatever the location of NOM molecules, in solution or adsorbed onto the membrane surface.

These results illustrate the complexity of the adsorption and retention mechanism in the presence of NOM that needs further investigations to be better understood. Many factors, such as membrane properties, pollutant characteristics, distribution of NOM components can all affect the process efficiency.

4. Conclusion

This study reveals that an accurate evaluation of a given membrane in terms of the retention of a contaminant is not possible until the saturation of the membrane with the component of interest is accomplished.

For a molecule similar in size to the pore radii, the initial decrease in retention versus volume filtered follows a slope that is characteristic of the affinity between the component and the membrane material.

The retention coefficient obtained at steady state is partly controlled by the composition of the water matrix. The presence of ions could affect the degree of hydration of the component: the R_{obs} is then dependent on ion concentration. In the present study, addition of NOM (1 ppm) caused negligible membrane fouling and did not modify the BPA retention of the membrane.

The efficiency of NF membranes in retaining small chemicals has to be determined with particular care, because of the quite complex nature of the mechanisms involved. Moreover, due to the adsorption–desorption balance, the membrane can be considered as a reservoir of EDCs and retained compounds can be released into the permeate if the pollutant concentration in the raw water has an erratic behavior.

Fig. 6 – Water flux at steady state determined on a fresh membrane, on a membrane after filtration of BPA (1 ppb), of NOM (1 ppm) and of the mixture BPA+NOM.
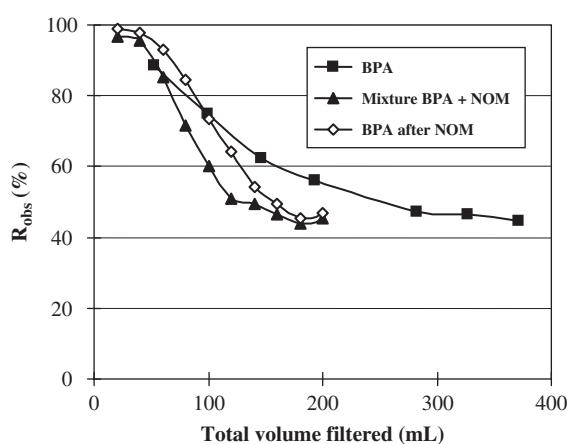

Fig. 7 – Effect of NOM (1 ppm) on R_{obs} of BPA (1 ppb) by the membrane Desal 5 DK ($20 \times 10^{+5}$ Pa).

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