



Flux dependency of particulate/colloidal fouling in seawater reverse osmosis systems

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ABSTRACT

Fouling is the main operational problem in seawater reverse osmosis systems (SWRO). Particulate fouling is traditionally measured through the silt density index (SDI) and through the modified fouling index (MFI). In recent years, ultrafiltration membranes were used successfully at constant flux – MFI-UF – to measure particulate/colloidal fouling potential and tested in sea water applications. Furthermore, constant flux operation allows predicting the rate of fouling in RO systems. The objectives of this study are: (1) to measure the flux effect in MFI-UF with different membranes (100, 30 and 10 kDa) for raw seawater and pre-treated water before reverse osmosis in three different locations; (2) to study the particulate and colloidal fouling potential of seawater in reverse osmosis systems; (3) to project the increase in pressure due to cake resistance in reverse osmosis systems. In this research, flat ultrafiltration membranes (100, 50, 30 and 10 kDa) are used in a constant flux filtration mode to test and compare real seawaters from various locations (North and Mediterranean Sea) and from various full scale facilities including different pre-treatments (i.e., ultrafiltration and coagulation + dual media filtration). The operated fluxes range from 350 down to values close to real RO operation, 15 l (m² h)⁻¹. After each filtration test, the MFI-UF is calculated to assess the particulate fouling potential. The obtained results showed that: (1) the particulate and colloidal fouling potential is directly proportional to the applied flux during filtration. This proportionality is related to the compression of the cake deposit occurring at high flux values; (2) the higher the flux, the higher the required pressure, the less porous the cake and therefore the higher the specific cake resistance; (3) particulate and colloidal fouling potential of seawater is site specific and is influenced by pre-treatment.

Keywords: Particulate fouling; Seawater; MFI-UF; Constant flux; Reverse osmosis; Pre-treatment

1. Introduction

Reverse osmosis systems foul. Fouling can be classified according to its origin in: particulate, organic, inorganic and bio-fouling. Cleaning of the membranes help

to restore the production capacity of the membranes. The more efficient the pre-treatment, the less the cleaning frequency in the system. To assess pre-treatment, fouling indices are used in practice in a daily base and even, membrane manufacturers use this indices to guarantee operation and life time of the membranes. Fouling indices are a measure for fouling potential due to particles and colloids.

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The most common fouling index is the silt density index (SDI); however, fouling problems have been reported even with very low SDI values, that is, SDI < 1. Currently, the SDI method has the support of the ASTM. One other fouling index is the modified fouling index (MFI). Currently its use is expanding. It was developed by Schippers and Verdouw in 1980, has many advantages over the SDI including: (1) a linear relation between the concentration of colloidal particles and MFI, (2) cake filtration is assumed to be the dominant filtration mechanism when employing the MFI while the SDI is not based on any filtration mechanism, and (3) since the MFI is based on the occurrence of cake filtration, flux decline or increase in net driving pressure in an RO plant can be predicted using cake formation.

Both the SDI and MFI operate at constant pressure and make use of membrane with nominal pore size of 0.45 μm [1]. However, particles smaller than 0.45 μm are not captured by the 0.45 μm membrane and thus are not measured in the MFI test. It is widely believed that particles smaller than 0.45 μm are responsible for fouling of RO membranes. Consequently, the MFI using membranes with a pore size of 0.05 μm was introduced in 1981 [2]. At the end of the nineties, Boerlage et al. developed the MFI-UF test to capture smaller particles using a polyacrylonitrile UF membrane with a MWCO of 13 kDa. Furthermore, Boerlage et al. also introduced the MFI at constant flux with a manual set-up [3,4].

Since the introduction of the MFI-UF by Boerlage et al. in 2003, applications have mainly been limited to fresh water sources. In recent years with the increase of desalination plants, the MFI-UF test at constant flux is being tested on seawater reverse osmosis plants.

Constant flux test is superior to constant pressure test. First, it resembles the operation of RO systems, and secondly it can be used to predict the rate of fouling in SWRO systems.

Reverse osmosis systems operate in cross flow and most of them operate at constant flux and at constant recovery. When membranes foul, the pressure has to be increased to keep the capacity (and flux) constant. Cross flow indicates that water passes tangentially to the membranes. At constant flux filtration, the flux value has a dominant effect on the development of feed pressure. To double the flux, it is required four times the feed pressure. This comes from the equation ruling particulate fouling. This phenomenon explains why manufacturers of spiral wound elements recommend lower design fluxes at higher fouling potential of feed waters.

In comparison with fouling indices, the RO cross flow operation means that: (1) not all of the particles are deposited on the surface of the membranes, and (2) the cake formed in RO has different characteristics than the

cake formed in dead-end, for example, porosity, and so on. These differences were respectively translated by Schippers and Kostense (1980) in (1) the particle deposition factor “ Ω ” ($\Omega < 1$ for cross flow) and (2) the cake factor “ Ψ ” [5]. The cake ratio factor (Ψ) is considered by correcting the measured fouling index at a certain flux to a MFI value that would be obtained at similar RO operation, that is, $15 \text{ l} (\text{m}^2 \text{ h})^{-1}$.

Fouling is characterized by increase in resistance due to the growth of a cake deposit on the surface of the membrane. The characteristics of the formed cake such as its thickness and porosity control the filtration performance. During constant flux filtration, the pressure increases due to both cake growth and cake compression.

The specific cake resistance equation for cake filtration [$\alpha = 180 \cdot (1 - \epsilon) / (\rho_p \cdot d_p^2 \cdot \epsilon^3)$] is based on the assumption that the average cake resistance is constant over time. For incompressible materials, the specific cake resistance is constant with time and pressure. In compressible cake filtration, layers of the cake compress under the pressure of material above as a result of fluid drag. The eventual extent of compression depends on the pressure exerted. The resulting change in specific cake resistance through the filtration process is one of the major reasons why non-linear TMP versus time relationships are typically observed in (compressible) cake filtration over time. In addition to the time-dependent changes in specific cake resistance, it is important to note that, for highly compressible cakes, the average specific cake resistance does not adequately describe the permeability of the cake as there is significant variation in the cake properties within the cake.

A new automatic and portable set-up has been developed to perform MFI-UF tests at constant flux. The set-up is described in Section 4. This set-up has been transported to various locations to perform the tests on-site and in this way to avoid any possible influence of transport and preservation of the samples.

This study focuses in measuring the particulate and colloidal fouling of seawaters before and after pre-treatment in SWRO systems.

2. Goals and objectives

The objectives of this research were:

- To measure the flux effect in MFI-UF with different membranes (100, 30 and 10 kDa) for Raw seawater and pre-treated water before reverse osmosis in three different locations.
- To study the particulate and colloidal fouling potential of seawater in reverse osmosis systems.

3. Background

This section shortly presents the calculation of the MFI at constant flux. This calculation has been mentioned several times during the development of the MFI-UF at constant flux [3,4,6].

3.1. Filtration mechanisms

The current equations describing cake formation assume that the average specific cake resistance is constant over time.

3.2. The MFI measured at constant flux

In this mode of filtration the production capacity is kept constant by increasing the pressure when resistance increases due to fouling. Now, taking the standard equation describing the flux through a membrane:

$$\frac{dV}{A \cdot dt} = \frac{\Delta P}{\eta \cdot (R_m + R_c)} \quad (1)$$

and substituting J for flux and R_c by:

$$R_c = \frac{V}{A} \cdot I \quad (2)$$

the following equation is obtained:

$$J = \frac{\Delta P}{\eta \cdot \left(\frac{V}{A} \cdot I + R_m \right)} \quad (3)$$

rewriting V/A as $J \cdot t$ and rearranging gives:

$$\Delta P = J \cdot \eta \cdot R_m + J^2 \cdot \eta \cdot I \cdot t \quad (4)$$

Eq. (4) is valid when no compression occurs. The fouling index I can then be determined from the slope of the linear region in a plot of ΔP versus time which corresponds to cake filtration or by manipulation of Eq. (4). The MFI can be calculated using I (from Eq. (4)) for standard reference conditions as follows [6]:

$$\text{MFI} = \frac{\eta_{20\text{C}} \cdot I}{2 \cdot \Delta P_0 \cdot A_0^2} \quad (5)$$

3.3. Cake compression

One of filter cake behaviour is its compressibility, which describe the compaction of cake structure

in relation to changes in physicochemical properties of the filtration system [7]. According to Coulson and Richardson [8] filter cakes are divided into two classes, incompressible cakes and compressible cakes, which can be distinguished by the specific cake resistance:

- For incompressible filter cake, the specific cake resistance is not affected by the pressure differences across the cake or by the deposition of solid during filtration. The specific cake resistance is constant with time and pressure [9,10].
- For compressible filter cake, the specific cake resistance of cake is affected by the pressure difference across the cake. As the pressure increase, the porosity of compressible cake layer will decrease because of particle deformation in the cake [11].

4. Material and methods

4.1. Filtration set-up

In this study the filtration tests were performed at constant flux where the pressure increase over time was recorded. At the beginning of every test the temperature of the water sample was measured. The set-up is shown in Fig. 1.

The experimental procedure for each test is described below:

1. The membrane resistance was measured in advance for all the membranes and then soaked in ultra pure water (UPW) before the test.
2. The membrane was placed into the membrane holder (porous support).
3. The required flux was controlled manually in the pump by defining the flow rate in ml h⁻¹.
4. All the tests were stopped after cake filtration was reached, a minimum I value was observed or the P vs. $t(I)$ slope showed no change in time; and let run at least for 35 min.
5. I is calculated by dividing the slope of the P vs. t line over square flux and water viscosity ($I = \text{Slope}/J^2\eta$). MFI-UF is calculated using Eq. (4) considering the minimum I values.

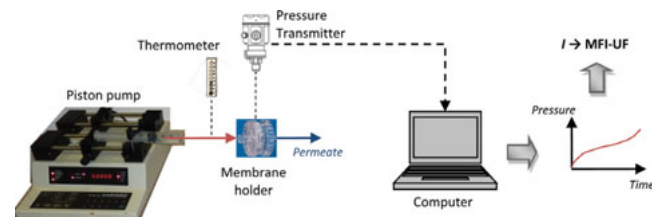


Fig. 1. Scheme of the MFI-UF constant flux set-up.

6. In order to keep MFI-UF values comparable with $MFI_{0.45}$ (standard reference conditions of temperature in terms of water viscosity, surface area and pressure), the MFI-UF values were referred to reference conditions namely; viscosity at temperature of 20°C ($\eta_{20^\circ\text{C}}$), pressure of 2 bar (ΔP_o) and surface of area of a MFI 0.45 μm micro filter (A_o) as shown in Eq. (5).

4.2. Membranes

Membranes have been described as the Achilles heel of MFI-UF filtration tests. The membrane non-uniformity (surface porosity, thickness, tortuosity) even in the same manufactured batch has been reported in literature.

In advance of the on-site testing, all the membranes were first cleaned and characterized by measuring the membrane resistance (R_m) with UPW. The UPW was produced from Delft tap water fed to a RO unit, then passed through a Millipore system including UV, GAC and 0.2 μm filter and finally a second pass through a

second RO unit. In this way the number of particles, ions, organic matter was limited in the UPW.

A 100, 50, 30 and 10 kDa, polyethersulfone (PES), 25 mm diameter membrane filters were used to measure the particulate/colloidal fouling potential of the water. The average membrane resistances for the filters used in the testing are presented on Table 1. The manufacturer provides sets/packages of 10 membranes. For the complete sets (1 set for 100 kDa and 2 sets for 10 and 30 kDa) of membranes the R_m s were measured and the average and standard deviation calculated. The membranes with R_m values higher or lower than 9% (10 kDa) and 5% (30 and 100 kDa) the average R_m value were no used for the testing.

4.3. Water samples

Three different locations were selected for the tests. Two are place along the Mediterranean sea and one is located on the North sea. The water samples were taken from the intakes of each desalination plant. The locations and pretreatments are briefly described on Table 2.

Table 1
Average R_m for the PES membranes used in the testing on site A

Location	Membranes	Complete set(s)		#Total/ # excluded	Partial set(s)	
		R_m, m^{-1}	Std Dev, %		R_m, m^{-1}	Std Dev, %
A	100 kDa PES	2.99×10^{11}	10	10/2	2.89×10^{11}	3
	30 kDa PES	7.43×10^{11}	3.4	10/1	7.48×10^{11}	3
	10 kDa PES	1.01×10^{12}	9	19/4	9.80×10^{11}	4
B	100 kDa PES	3.21×10^{11}	9	10/2	3.10×10^{11}	5
	50 kDa PES	6.59×10^{11}	5.4	8/2	6.58×10^{11}	3.3
	30 kDa PES	7.57×10^{11}	5.9	17/3	7.48×10^{11}	3.4
	10 kDa PES	1.03×10^{12}	8	20/5	9.97×10^{11}	5
C	100 kDa PES	2.76×10^{11}	18	–	–	–
	50 kDa PES	6.77×10^{11}	10	–	–	–
	30 kDa PES	1.38×10^{12}	22	–	–	–
	10 kDa PES	1.49×10^{12}	14	–	–	–

Table 2
Description of the tested locations

Location	Intake	Pre-treatment	Comment
A (Northern Mediterranean water)	Submerged pipe (next to the shore)	Strainer – UF (0.01 μm)	Flux $\approx 57 \text{ l (m}^2 \text{ h)}^{-1}$
A (Northern Mediterranean water)	Submerged pipe (next to the shore)	Strainer – Coagulation + Dual media filter	DMF flow rate $\approx 0.9 \text{ m}^3 \text{ h}^{-1}$ 2 mgFe ³⁺ l ⁻¹ + 0.2 mg Polymer l ⁻¹ DMF = Anthracite and Sand
B (North-Western Mediterranean water)	Submerged pump (L = 2.5 km)	UF (0.02 μm)	Flux $\approx 50 \text{ l (m}^2 \text{ h)}^{-1}$
C (North sea water)	Submerged pipe (L = 300 m)	Strainer – UF ($\approx 300 \text{ kDa}$)	Flux $\approx 60 \text{ l (m}^2 \text{ h)}^{-1}$

Table 3
Summary of water characteristics

Sample	Location	DOC, mg l ⁻¹	pH	T, °C	SUVA (l (mg m) ⁻¹)	EC, mS cm ⁻¹	NTU
Raw water	A	1.2	8.21	18	0.8	57.1	–
UF permeate	A	0.85	8.2	18	0.7	57.1	–
Coag + DMF effluent	A	0.77	7.8–8.0	18	0.6	57.1	–
Raw water	B	0.75	8.1	16.4	0.55	57–58	0.5–1.5
UF permeate	B	0.72	8.0	16.4	0.45	57–58	–
Raw water	C	1.45	8.1	13.5	2.3	48.5	8–12
UF permeate	C	1.3	6.5	13.5	2.15	48.5	–

A summary of the water properties is presented on Table 3. Location C has the higher DOC concentration, while the location B has the lower DOC concentration.

To reduce the impact of variations in water quality, as many tests as possible were performed in the same day.

5. Results and discussion

In this section the several water samples were tested at various fluxes with different membrane MWCOs.

5.1. Flux effect on different locations

5.1.1. Location A

The raw water sample was tested with 100 and 10 kDa membranes at various fluxes with the purpose of measuring the relation flux—MFI value. The results are presented in Table 4 and plotted in Fig. 2.

For the raw sea water, with 100 and 10 kDa membranes, the MFI-UF values as a function of flux showed a linear trend for the range of fluxes tested (50–350 l (m² h)⁻¹). The regression coefficients are $R^2 = 0.97$ in both cases.

Table 4
Raw seawater “A” MFI-UF values at various fluxes

Flux, l (m ² h) ⁻¹	100 kDa MFI-UF, s l ⁻²	10 kDa MFI-UF, s l ⁻²
349	6300	21,500
251	3700	16,650
150	2550	12,500
52	1000	3900
15 ^a	203	3000

^aProjected value from the linear equations in Fig. 2.

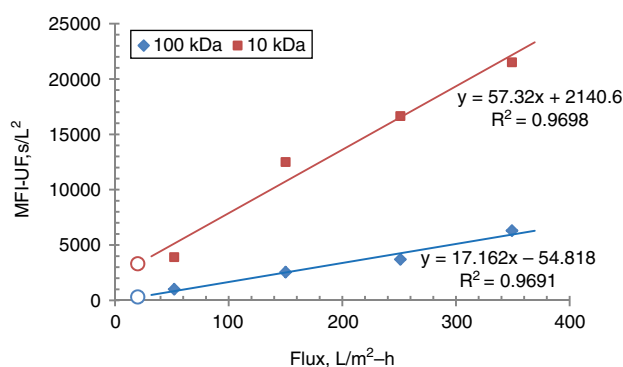


Fig. 2. MFI-UF values for RSW-A at various fluxes and projected MFI-UF values for 15 l (m² h)⁻¹ (Circles).

Based on the linear relations, the MFI-UF values at 15 l (m² h)⁻¹ (similar to SWRO operation) were projected. The projected value with 10 kDa membrane is 15 times higher than with the 100 kDa as shown in Table 4.

The MFI-UF values with a 10 kDa PES membrane were measured for DMF effluent and for UF permeate at different fluxes. The results are shown in Table 5 and plotted in Fig. 3.

Table 5
MFI-UF values for Coag + DMF effluent and for UF permeate at various fluxes with 10 kDa membrane

Flux, l (m ² h) ⁻¹	Coag + DMF effluent MFI-UF, s l ⁻²	Flux, l (m ² h) ⁻¹	UF permeate MFI-UF, s l ⁻²
349.3	11,000	321	6500
251	8500	251	4500
150	5000	150	2600
72	1900	52	1500
15 ^a	333	15 ^a	495

^aProjected values from the linear equations in Fig. 3.

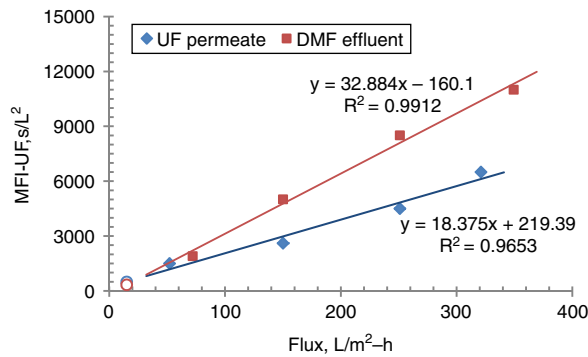


Fig. 3. MFI-UF values for Coag + DMF effluent and for UF permeate at various fluxes with 10 kDa membrane.

The MFI-UF values for DMF effluent are higher than the MFI-UF values for UF permeate. The obtained MFI-UF values fitted linear equation with good regression coefficients (R^2). In the case of UF permeate the $R^2 = 0.97$ and in the case of DMF effluent the $R^2 = 0.99$. The slopes of the equations are important to notice, as the rate of MFI-UF change with flux is much higher with DMF effluent than with UF permeate (1.78 times).

With the obtained equations it was projected the MFI-UF value corresponding to a flux similar to SWRO systems operation ($15 \text{ l (m}^2 \text{ h)}^{-1}$). The projections are presented in Table 5.

Even though the measured MFI UF values for DMF effluent were higher than the UF permeate values, the projected MFI-UF values at $15 \text{ l (m}^2 \text{ h)}^{-1}$ showed the opposite; DMF water's particulate fouling is lower than the one of UF permeate.

5.1.2. Location B

The raw water sample was tested with 30 and 10 kDa membranes at various fluxes with the purpose of measuring the relation flux—MFI value. The results are presented in Table 4 and plotted in Fig. 2.

For the raw sea water, with 30 and 10 kDa membranes, the MFI-UF values as a function of flux showed a linear trend for the range of fluxes tested ($72\text{--}350 \text{ l (m}^2 \text{ h)}^{-1}$). The regression coefficients (R^2) are above 0.98 in both cases (Fig. 4).

Based on the linear relations, the MFI-UF values at $15 \text{ l (m}^2 \text{ h)}^{-1}$ (similar to SWRO operation) were projected. In this case, for both 30 and 10 kDa membranes the projection shows a negative MFI value meaning no particulate/colloidal fouling potential of the water at $15 \text{ l (m}^2 \text{ h)}^{-1}$ flux (Table 6). This can be observed already at fluxes less than 72 s l^{-2} .

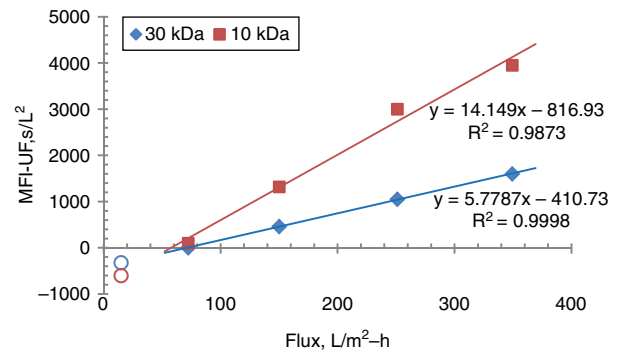


Fig. 4. MFI-UF values for raw seawater at various fluxes and projected MFI-UF values for $15 \text{ l (m}^2 \text{ h)}^{-1}$ (Circles).

Table 6

Raw water MFI-UF values at various fluxes

Flux, $\text{l (m}^2 \text{ h)}^{-1}$	30 kDa MFI-UF, s l^{-2}	10 kDa MFI-UF, s l^{-2}
349	1600	3950
251	1050	3000
150	460	1320
72.2	0	100
15 ^a	<0	<0

^aProjected values from the linear equations in Fig. 2.

The MFI-UF values with a 10 kDa PES membrane were measured for UF-B permeate at different fluxes. The results are shown in Table 5 and plotted in Fig. 3. As UF-B has a nominal pore size of $0.02 \mu\text{m}$, then the colloids in UF-B are smaller than $0.02 \mu\text{m}$ and have a narrower particles size distribution in comparison with raw water.

Additionally to the 10 kDa results, in Fig. 3 are included the punctual values for 30 and 100 kDa (640 and 230 s l^{-2} , respectively).

The obtained MFI-UF values with the 10 kDa membrane fitted a linear equation with good regression coefficient (R^2). In the case of UF-B permeate the $R^2 = 0.98$. With the obtained equation, $\text{MFI} = 3.23 * \text{Flux} + 389$, it was projected the MFI value corresponding to a flux similar to SWRO systems operation ($15 \text{ l (m}^2 \text{ h)}^{-1}$) (Fig. 5). The projection is presented in Table 5.

In comparison with the observed effect for the raw water in the previous section, for the UF-B permeate at $15 \text{ l (m}^2 \text{ h)}^{-1}$ the MFI-UF value is positive and around 440 s l^{-2} (Table 7). This may be attributed to the narrower particle size distribution present in UF permeate that may create a less porous cake and therefore higher specific cake resistance.

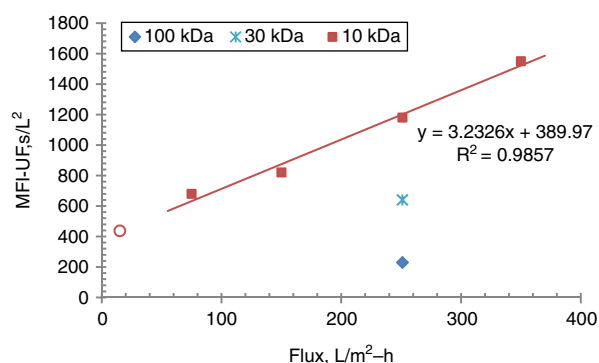


Fig. 5. MFI-UF values for UF-B permeate at various fluxes with 10 kDa PES membrane and projected MFI value at $15 \text{ l (m}^2 \text{ h)}^{-1}$ (Circle).

Table 7

MFI-UF values for UF permeate at various fluxes with 10 kDa membrane

Flux, $\text{l (m}^2 \text{ h)}^{-1}$	UF permeate MFI-UF, s l^{-2}
349	1550
251	1180
150	820
75	680
15 ^a	438

^aProjected value from the linear equation in Fig. 3.

5.1.3. Location C

The RO feed water sample was tested with 100, 50, 30 and 10 kDa membranes at various fluxes with the purpose of measuring the relation flux—MFI value. The results are presented in Table 8 and plotted in Fig. 6.

For the RO feed water, the MFI-UF values as a function of flux showed a linear trend for the range of fluxes tested ($72\text{--}400 \text{ l (m}^2 \text{ h)}^{-1}$). The regression coefficients (R^2) are above 0.87 in all cases.

Based on the linear relations, the MFI-UF values at $15 \text{ l (m}^2 \text{ h)}^{-1}$ (similar to SWRO operation) were projected.

Table 8

RO feed water MFI-UF values at various fluxes

Flux, $\text{l (m}^2 \text{ h)}^{-1}$	100 kDa MFI-UF, s l^{-2}	50 kDa MFI-UF, s l^{-2}	30 kDa MFI-UF, s l^{-2}	10 kDa MFI-UF, s l^{-2}
350/430/350/250	235	4688	10,959	35,453
250/350/250/200	179	4906	–	17,686
200/250/150/150	50	1601	7981	13,372
150/150/84/84	0	631	4058	2455
15 ^a	<0	<0	0	<0

^aProjected values from the linear equations in Fig. 6.

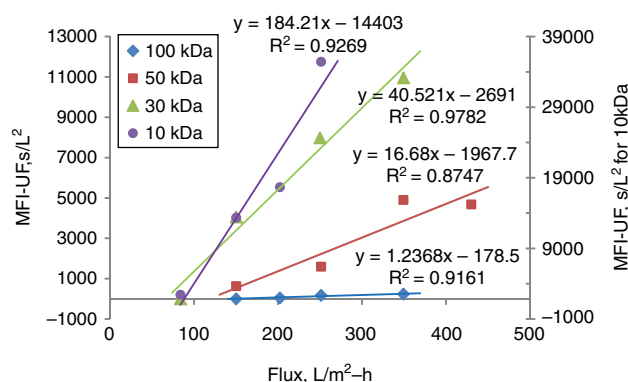


Fig. 6. Flux effect on RO feed.

In all cases, for 100, 50, 30 and 10 kDa membranes the projection shows a negative or zero MFI value meaning the particulate/colloidal fouling potential is below detection limit.

6. Conclusions

- The particulate and colloidal fouling potential is directly proportional to the applied flux during filtration. This proportionality is related to the compression of the cake deposit occurring at high flux values. The higher the flux, the higher the required pressure, the less porous the cake and therefore the higher the specific cake resistance.
- Particulate and colloidal fouling potential of seawater is site specific and is influenced by pre-treatment. In general for seawater applications this fouling potential is low at flux rates similar to full scale reverse osmosis operation.

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Symbols

DMF	—	Dual media filtration
kDa	—	Kilo Dalton
MFI-UF	—	Modified fouling index—ultra filtration
MWCO	—	Molecular weight cut off
PES	—	Polyethersulfone
RC	—	Regenerated cellulose
RO	—	Reverse osmosis
SDI	—	Silt density index
SWRO	—	Seawater reverse osmosis
UF	—	Ultra filtration
A	—	Effective membrane surface area [m^2]
A_o	—	Standard reference area of the MFI 0.45 μm membrane (13.8×10^{-4}) [m^2]
C_b	—	Concentration of particles in a feed water [kg m^{-3}]
d_p	—	Diameter of particles forming the cake [m]
I	—	Fouling index of particles in water to form a layer with hydraulic resistance [m^{-2}]
J	—	Permeate water flux [$\text{m}^3 \text{m}^{-2} \text{s}^{-1}$]
R_c	—	Cake formation resistance [m^{-1}]
R_m	—	Membrane resistance [m^{-1}]
r_p	—	Pore radius [m]
R_t	—	Total resistance [m^{-1}]
T	—	Filtration time (second)
T	—	Temperature of feed water ($^{\circ}\text{C}$)
V	—	Filtrate volume [m^3]
ΔP	—	Applied trans-membrane pressure [bar or N m^{-2}]
ΔP_o	—	Standard reference applied trans-membrane pressure [bar or N m^{-2}]
ΔP_c	—	Pressure drop over the cake [bar or N m^{-2}]
Δx	—	Membrane thickness [m]
α	—	(Average) specific cake resistance [m kg^{-1}]

α_o	—	Initial specific cake resistance [m kg^{-1}]
Ω	—	Deposition factor [-]
ε	—	Cake/membrane surface porosity [-]
$\eta_{20^{\circ}\text{C}}$	—	Water viscosity at 20°C [N s m^{-2}]
η_T	—	Water viscosity at temperature T [N s m^{-2}]
ρ_p	—	Density of particles forming the cake [kg m^{-3}]
τ	—	Tortuosity of membrane pores
ω	—	Compressibility coefficient [-]
Ψ	—	Cake ratio [-]

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