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The Modified Fouling Index Ultrafiltration constant flux for assessing particulate/colloidal fouling of RO systems

Sergio G. Salinas-Rodriguez [a,*], Gary L. Amy [a,c], Jan C. Schippers [a], Maria D. Kennedy [a,b]

[a] UNESCO, IHE Institute for Water Education, Environmental Engineering and Water Technology Department, Westvest 7, 2611 AX Delft, The Netherlands
[b] Delft University of Technology, Faculty of Civil Engineering, Stevinweg 1, 2628 CN Delft, The Netherlands
[c] King Abdullah University of Science and Technology, Thuwal 23955-6900, Saudi Arabia

HIGHLIGHTS

- A safe MFI value has been defined for RO feed water.
- A new portable set-up can perform MFI-UF tests at constant flux filtration.
- Low filtration flux and membrane pore size are important variables in the test.
- The MFI-UF constant flux can assess pre-treatment of RO systems.
- Measuring the deposition factor is important to estimate the rate of RO fouling.

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ABSTRACT

Reliable methods for measuring and predicting the fouling potential of reverse osmosis (RO) feed water are important in preventing and diagnosing fouling at the design stage, and for monitoring pre-treatment performance during plant operation. The Modified Fouling Index Ultrafiltration (MFI-UF) constant flux is a significant development with respect to assessing the fouling potential of RO feed water. This research investigates (1) the variables influencing the MFI-UF test at constant flux filtration (membrane pore size, membrane material, flux rate); and (2) the application of MFI-UF into pre-treatment assessment and RO fouling estimation. The dependency of MFI on flux, means that to assess accurately particulate fouling in RO systems, the MFI should be measured at a flux similar to a RO system (close to 20 L/m^2/h) or extrapolated from higher fluxes. The two studied membrane materials showed reproducible results; 10% for PES membranes and 6.3% for RC membranes. Deposition factors (amount of particles that remain on the surface of membrane) were measured in a full-scale plant ranging between 0.2 and 0.5. The concept of "safe MFI" is presented as a guideline for assessing pre-treatment for RO systems.

1. Introduction

Particulate fouling in reverse osmosis (RO) and ultra-filtration (UF) systems is a phenomenon that has plagued operators since the earliest application of these technologies. A reliable index to predict the fouling potential of RO feed water is important in preventing and diagnosing fouling at the design stage of RO plants and for monitoring the performance of pre-treatment during plant operation. The Silt Density Index (SDI) is widely used to measure the fouling potential of RO feed water; however, fouling problems have been reported even with very low SDI values (i.e., SDI < 1).

The SDI, standardized by ASTM [3], is based on filtration of feed water through a 0.45 μm membrane in dead-end mode at constant pressure (207 kPa). The rate of plugging is measured and expressed as % flux decline per minute. As the SDI is simple to perform and cheap, it has been universally applied for the last 50 years as a tool to assess the particulate fouling tendency of a feed water, the effectiveness of pre-treatment processes, etc. and is often the basis of membrane guarantees and other plant performance contracts. However, increasingly the value of this test to predict the rate of fouling in RO systems due to particle deposition is being questioned. The limitations of the SDI test are well documented [1,2,5,21,23,29,33] and include: no correction for test water temperature; the result is heavily dependent on the test membrane permeability; not applicable for testing high fouling feed water e.g., raw water — ASTM recommends that turbidity should be <1 NTU;

* Corresponding author.
E-mail address: s.salinas@unesco-ihe.org (S.G. Salinas-Rodriguez).

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not applicable for testing UF permeate, which is increasingly being used in desalination pre-treatment; no linear relation with colloidal/suspended matter; fouling potential of particles smaller than 0.45 μm are not measured; and it is not based on any filtration mechanism.

The absence of temperature correction results in higher SDI values at higher temperatures. While, the non-linear relation between the measured SDI values with particle concentration means that water appears less fouling than it is, as the test filter becomes progressively plugged [5]. Theoretical prediction of flux decline in RO systems based on SDI results in extremely high fouling rates e.g., SDI = 3 effectively means a flux decline of 3% per minute [28]. Applying a direct correction between the SDI test flux (>1600 L/m^2/h at the start) and a typical RO flux (which is about 20 L/m^2/h), predicts a flux decline of 20% per hour. This rate of fouling is far outside the rates observed in practice.

To overcome these deficiencies, the Modified Fouling Index using the same 0.45 μm membrane filters (MFI-0.45) and MFI using ultrafiltration membranes and performed at constant flux (MFI-UF) were developed. The MFI-0.45 test uses the same equipment as the SDI test [29] and thus it also operates at constant pressure filtration. It takes into account that initially pore blocking occurs, followed by cake/gel filtration and finally cake/gel blocking and/or enhanced compression occurs. The obtained MFI value is corrected for temperature and pressure and shows a linear relation with colloidal/suspended matter concentration.

Predicting the rate of fouling in RO systems based on the MFI-0.45 is possible, assuming that cake/gel filtration is the dominant mechanism. However, the predicted rate of fouling turns out to be very low for MFI of 1 s/L^2 (equivalent to SDI$_{15}$ 1 to 3). A pressure increase of 1 bar is predicted to occur in more than 100 years with RO feed water with an MFI-0.45 = 1 s/L^2 [28].

Based on the mentioned above, it was concluded that particles much smaller than 0.45 μm were responsible for the fouling rate observed in practice. This was supported by the measurement of MFI at constant pressure with membranes of different pore sizes varying from 0.8 μm down to 0.05 μm for RO feed water which resulted is respective MFI values increasing from 4 to 4500 s/L^2. Consequently, the MFI-UF test with UF membranes was developed to capture these smaller particles. Brackish water measurements with the MFI-UF test using 13 kDa molecular weight cut-off (MWCO) UF membranes demonstrated that the cake/gel formed on the membrane surface was quite compressible [8]. Similar tests in seawater also showed cake compressibility [25,26]. Due to this compressibility, accurate prediction of fouling in RO systems was not possible using the MFI-UF test in constant pressure mode. Hence, the development of the MFI-UF test that focussed on operation in constant flux mode, whereby pressure increase to maintain constant flux over time is recorded. Furthermore, MFI-UF constant flux has the main advantage that allows the prediction of rate of fouling in nanofiltration (NF) and RO systems.

The goal of this study is to present the Modified Fouling Index with ultrafiltration membranes at constant flux filtration for assessing particulate and colloidal fouling potential of fresh and seawater samples. The objectives are the following: to describe the latest developments on MFI-UF constant flux set-up; to characterize the membranes used in the test; to investigate variables affecting the MFI-UF tests such as membrane pore size, membrane material and flux rate; to apply the MFI-UF test in seawater, in particle size distribution, and in effect of pretreatment for RO systems; and to apply the MFI-UF constant flux test to estimate the rate of fouling in RO systems.

2. Material and methods

2.1. Water samples

Ultrapure water (UPW) is deionized water with a resistance equal to 18.2 mΩ (0.055 μS/cm) and TOC of ~4 ppb. This water was used to clean membranes by soaking and flushing them before filtration, to measure the clean water flux, and to prepare synthetic seawater. Production of UPW from Delft tap water included softening, RO filtration, ion exchange, granular activated carbon filtration, 0.22 μm filtration, and finally a second pass RO filtration.

Synthetic seawater (SSW) with total dissolved salts of 33,000 mg/L was used during this research to simulate North Sea water according to the procedure specified by other researchers [32]. The preparation of SSW involved dissolving the salts in ultrapure water by increasing water temperature to 35 °C and by stirring the water at 900 rpm for at least 24 h. Then, checking the pH and the conductivity for the prepared SSW (pH should be around 8.0 and the conductivity should be about 50 mS/cm).

For the real water samples, the DOC and TDS concentrations are reported in Table 1. All sampling and testing campaigns took place in two years (2009 and 2010).

2.2. MFI constant pressure filtration

The filtration set-up to measure MFI-UF at constant pressure is illustrated in Fig. 1. The sample is placed in the sample reservoir (~3 L). Pressure is controlled with a control valve. Once filtration is started, the weight of the permeate is registered in an electronic balance and recorded in a computer. From the time and volume values, a graph of t/V vs. V is plotted and cake filtration is identified. The slope in this region is the fouling index (I).

2.3. Testing procedure and MFI-UF constant flux determination

Independently of the membrane's MWCO or the flux rate of the test, the MFI-UF testing procedure is the following:

1. The membrane resistance is measured with UPW at the same flux as the MFI-UF test to be performed.
2. Membrane filter is placed into the membrane holder. The active layer of the membrane is placed facing towards the water sample.
3. Filtration flux rate is controlled manually in the pump by defining the flow rate in mL/h. The effective membrane area (0.000346 m^2) must be considered when calculating the flux rate.
4. The software for recording the pressure and time values should be started. Both, pump and data logging must start simultaneously. Pressure readings (every second) were recorded with help of Hart OPC server from Endress + Hauser. Data was saved into MS Excel for further processing.
5. The fouling index (I) is calculated by dividing the slope of the *pressure* vs. *time* line over square flux and water viscosity.

$$I = \frac{\text{Slope}}{J^2 \cdot \eta} \tag{1}$$

6. Criteria to stop the test:
   a. When cake filtration is reached (linear trend between pressure and time or the slope of fouling index and time shows no change in time),
   b. When a minimum fouling index (I) value is observed;
   c. Change in MFI value in last 5 min filtration is less than 5% per minute.
   d. At least 35 min filtration occurred.

Table 1
Water samples DOC and TDS.

Origin	DOC, mg/L	TDS, mg/L
Delft canal water (DCW)	18.5	700
Mediterranean Sea	0.95 ± 0.03	35,600
North Sea (Scheveningen) batch 1	2.8	30,030
North Sea (Scheveningen) batch 2	2.1	30,030
North Sea (Kamperland)	1.46 ± 0.18	31,500

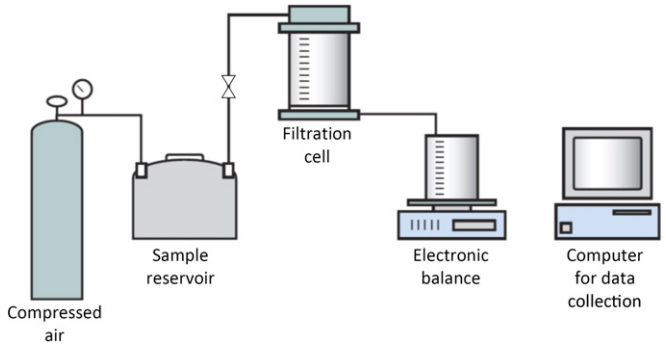

Fig. 1. Scheme of filtration set-up for MFI measurements at constant pressure.

7. MFI-UF is calculated considering the minimum I values.
8. In order to keep MFI-UF values comparable with MFI-0.45, the MFI-UF values are standardized to reference conditions namely: viscosity at temperature of 20 °C ($\eta_{20\,°C} = 0.001$ N s/m^2), pressure of 2 bar (ΔP_0) and surface of area of a 0.45 μm microfilter ($A_0 = 0.00138$ m^2) as shown in Eq. (2).

$$\mathrm{MFI} = \frac{\eta_0 \cdot I}{2 \cdot \Delta P_0 \cdot A_0^2} \tag{2}$$

3. Part I: development of the MFI-UF constant flux filtration set-up

A filtration set-up was developed to work at constant flux (Fig. 2). The key components of the set-up are: pump, membrane holder, pressure sensor, temperature meter, computer, and a three-way valve. The set-up was verified for correct pressure readings, constant flux, no leakages, and air trapped in the system. The readings from the pressure sensor were verified with a second manometer (pressure gauge) by filtering ultra-pure water (UPW) at various flux rates. A maximum 2% difference was observed, which is considered acceptable.

The flux rate was verified by monitoring the permeate weight over time with help of an electronic balance (Sartorius TE6101). Several flux rates (10–400 L/m^2/h) were tested and a maximum difference of 2.8% was observed between the expected and measured fluxes at the lower flux rate.

Verification of leakages in the set-up was performed by pressurizing the system (up to 4 bar) without allowing filtration and monitoring the pressure change over time. No leaks were observed at pressures less than 4 bar over time. However, after stopping the pump a back pulse was observed in the piston pump that yielded a slight decrease in pressure (0.1 bar pressure loss over 40 min).

The presence of air in the system is not desirable. To verify the effect of air trapped in the system, air was intentionally introduced and filtration was allowed. Erroneous high pressure values were observed by the effect of air; this could be related to the bubble point of the membranes or related to the compression of air that will produce erratic pressure development.

Two different piston-pumps were tested (Fig. 2). The maximum pressure at which they can operate is the main difference. Pump 1 (small one, Fresenius Injectomat Agilia) has a maximum capacity of 1.2 bar while pump 2 (Harvard Apparatus, Pump 33 Twin Syringe Pump) has a capacity of 3.5 bar.

The chosen pressure transmitter is corrosion resistant and commercially available (Cerabar M HART PMC41, Endress & Hauser). The operational pressure range is 0–4 bar with a maximum deviation of 0.036%. A three-way valve is used to connect the syringe (water sample) with the membrane holder and at the same time with the pressure transmitter.

For data logging of the pressure transmitter, a computer with HART OPC Server (Endress + Hauser) and RENSEN OPC office link were used. The pressure transmitter was connected to the computer via a modem (Endress + Hauser, FXA195 HART modem) with a USB connection.

3.1. Membranes

Two commercial membrane materials which are available in various pore sizes (or MWCO) were investigated. The materials were polyether sulfone (PES) and regenerated cellulose (RC) both from Millipore. Both membrane filters are circular flat sheets (25 mm diameter). The average pressure to filter UPW and the nominal membrane molecular weight cut-off (MWCO) as rated by the manufacturer are summarized in Table 2. All membranes tested were new. The stable pressure to filter UPW was measured at 100 L/m^2/h in the MFI-UF equipment, then normalized to 20 °C.

Membrane filters were obtained from the supplier in packages each containing ten filters. Each package is numbered with a batch code and manufacturing date.

In addition to PES and RC membranes, PVDF 0.1 μm membranes (Durapore) and CA 0.45 μm (Whatman) filters were used to measure MFI values with other pore sizes than available in PES and RC.

3.1.1. Membrane cleaning and conditioning

Membrane filters must be clean and pores and surface be wet before performing the MFI-UF test. A surface that is not clean may affect the way that the fouling cake is formed on the membrane and a membrane that is not wet will required more pressure during filtration.

According to the operating instructions provided by the membrane manufacturer, the membranes (PES and RC) are coated with glycerine to prevent the membrane drying out and also sodium azide (NaN_3) to

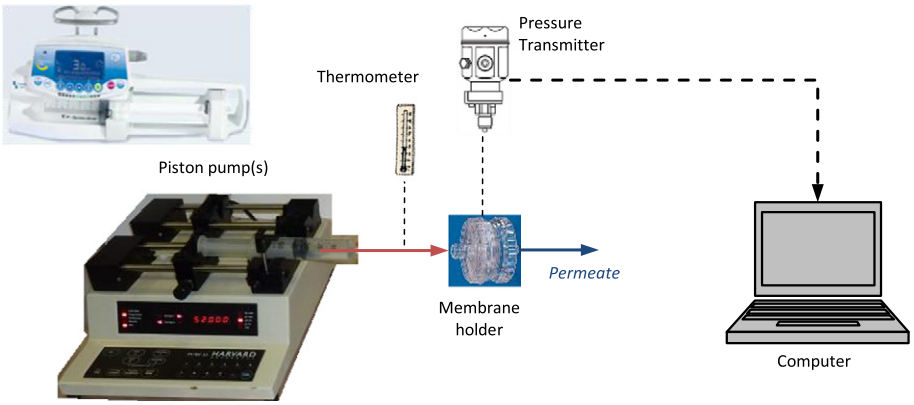

Fig. 2. Constant flux filtration set-up.

Table 2
Specifications of the ultrafiltration membranes.

Material	MWCO, kDa	Clean water pressure (bar) at 20 °C and 100 L/m^2/h
PES	5	3.4
	10	0.29
	30	0.23
	50	0.18
	100	0.09
RC	10	4.29
	30	0.51
	100	0.14

preserve the membrane. To clean the membranes, a 24 h soaking and 200 mL flushing with ultra-pure water (UPW) was effective for reducing the DOC to concentrations less than 0.1 mg/L. In addition, the membrane resistance was measured before the MFI tests by filtering UPW through the membrane.

3.2. Membrane holder

The membrane holder is the place where the membrane filter is placed for the filtration test. It should avoid leakages, and not damage the membrane at all. In this research, a holder for 25 mm diameter membranes was used. Several types were tested (Sterlitech — 316 stainless steel, Whatman GE — polypropylene, Schleicher & Schuell — polypropylene) with the Schleicher & Schuell membrane holder chosen. The chosen membrane holder was selected due to its material, uniform flow distribution to the membrane surface, transparent color that allows to see if air is trapped, and no leakages up to a pressure of 4 bar.

This membrane holder was slightly modified by removing the upper inner wall of the membrane holder, so in this way the flow distribution towards the membrane is uniform and only the membrane captures all the particles in the sample water. In the set-up, the filter holder is connected with a three-way valve that connects with the syringe (sample water) and with the pressure sensor.

Nahrstedt and Camargo Schmale [21] studied the effect of the membrane holder in SDI and MFI results. They tested three filter holders: Millipore inline 47 mm (external diameter), Sartorius SM 47 mm (external diameter), and Sartorius SM 25 mm (external diameter). They measured up to 90% different SDI values and 20% MFI values for the three membrane holders when filtering the same solution. The differences were attributed to different flow distributions inside the filter holder and attributed to the effective or real filter area affected by the holder support.

3.3. Membrane characterization

3.3.1. Scanning electron microscopy

Clean membranes were randomly selected from the package (3 membranes from each package containing 10 specimens), then samples were gold coated using a sputtering coater and scanned on with a field emission-scanning electron microscope (Jeol JSM-7500 F) at various accelerating voltages and at magnifications of up to 100,000×.

Fig. 3 shows the SEM photos for PES. Pores could only be identified down to 50 kDa. In the case of PES 100 kDa different pore diameters could be observed from 8.5 nm to 38 nm. However, a pore size distribution could not be estimated. The RC membrane is less porous than PES membrane and has a rougher surface. Fig. 4 shows the SEM pictures for RC 100 kDa membrane.

Fig. 5 shows the cross-section for a PES membrane. It can be observed that this is an asymmetrical membrane with a porous support layer. Cross-section for RC membranes was not performed.

3.3.2. Contact angle

The contact angle between a membrane and a droplet of an aqueous phase (e.g., water) is an indication of the overall hydrophobicity or hydrophilicity of the membrane. The lower the angle means a more hydrophilic membrane. Hydrophobicity was estimated by the sessile drop method using a CAM 100 goniometer (KSV Instruments). The goniometer with help of a video camera and software measures the left and right angle of a droplet of 2 μL of pure water on a membrane surface.

Before measurement, membrane filters were soaked in ultra-pure water for at least 1 h, and then rinsed. Three soaking and rinsing cycles were performed to remove membrane-coating materials. Rinsed membranes were dried in a desiccator for a day and kept in Petri dishes. To measure contact angle, a membrane sample piece (~1 cm^2) was mounted on a glass support. A 2.0 μL volume of lab water was dropped onto the membrane. Contact angle was measured within 10 s after the water droplet was applied. Similar testing procedure was reported by other researchers [10,15]. The results of the measurements are presented in Table 3.

Both membrane materials were found to be hydrophilic; however, RC membranes are more hydrophilic than PES membranes. Similar results were reported in a recent study, the PES 100 kDa (56° ± 3°) was reported to be more hydrophobic than RC 100 kDa (26° ± 3°) [16,22].

3.3.3. Fourier transformed infrared spectroscopy

An ATR–FTIR Spectrum 100 instrument (Perkin Elmer) was used to measure a Fourier transform infrared (FTIR) spectrum of the surface of the clean PES and RC membranes. The system was used to determine the functional group characteristics of the membrane surface materials. Before the test, clean membranes were dried in a desiccator at room temperature for three days, and then cut into a ~1 cm^2 piece. The results are presented in Fig. 6.

The indicative peaks of RC were seen at 3400 and 1650 nm (amide carbonyl group), 2915, 1430, 1380, 1180, and 1100 (aromatic double bond carbons), and 1050, 1000, 930, 850, and 675 (hydrocarbon, benzene ring).

The indicative peaks of PES were seen at 1300–1100 nm (ether group), 1420–1490 nm (alkanes), 1480–1580 nm (amide), 750–800 nm (ethyl group), and 1325 ± 25 and 1140 ± 20 nm (sulfone group).

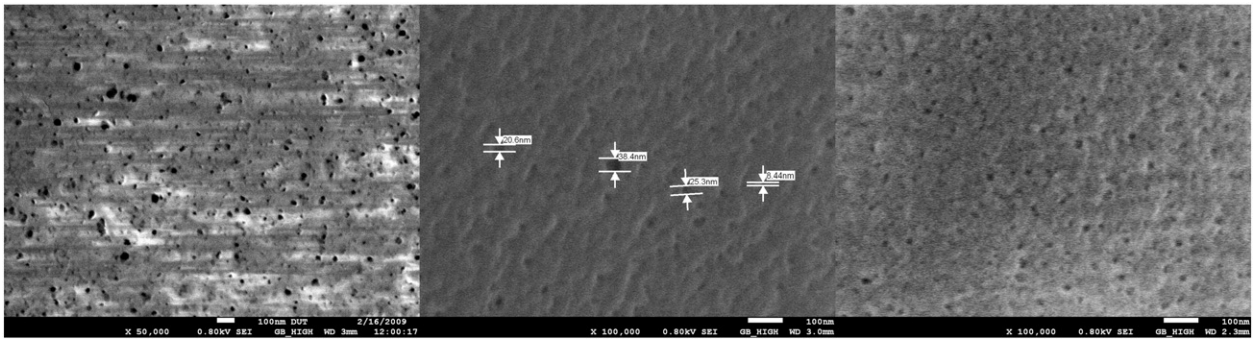

Fig. 3. FE-SEM pictures PES 100 kDa (left — 50,000×, and middle — 100,000×) and PES 50 kDa (right — 100,000×).

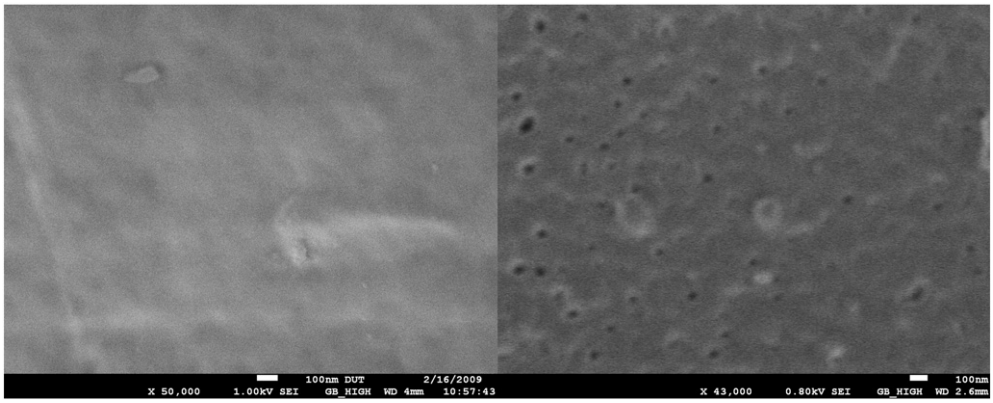

Fig. 4. FE-SEM picture for RC 100 kDa membrane (left — 50,000×, right — 43,000×).

3.3.4. Zeta potential

Zeta potential was measured using a SurPASS electrokinetic analyzer apparatus (Anton Paar GmbH, Austria). Membrane specimens (PES & RC, 100 & 30 kDa) were cut to fit the measurement cell and then wetted in 0.05 mM KCl solution. The zeta potentials of the membranes were determined over a wide range of pH (2.5–12). The zeta potential was measured four times for each pH value. In all cases the correlation coefficient was more than 0.9.

The results are presented in Fig. 7. In general PES is more negatively charged than RC membranes. For the same material, different MWCO produced different zeta potential values. For PES, the 100 kDa membrane was more negative than the 30 kDa. For RC, the 30 kDa membrane was more negative than the 100 kDa. The iso-electric point for PES and RC membranes was found at acidic pH values; for RC at pH < 3, and for PES at pH < 4.5. At basic pH values of > 10.5, the measured zeta potential values increased in all cases.

In a recent study the zeta potential for a PES 100 kDa membrane was reported as −16.1 ± 1.0 at pH 5.4 [16,17]. Zeta potentials for most membranes have been observed in many studies to become increasingly more negative as pH is increased and functional groups deprotonate [9,19]. In most seawater and freshwater samples, the pH range of the water is between 7.5 and 8.2. In this pH range, the zeta potential of the membrane is not expected to play a role.

3.3.5. Membrane resistance

The measurement of membrane resistance (R_m) is performed for every MFI test before measuring the sample. The R_m value is used to identify membranes with too high or too low R_m (identification of outliers according to the Dixon's Q test) in comparison with the average value of the manufacturing batch. UPW is filtered through a membrane and the clean water pressure is obtained. Furthermore, the R_m value is calculated by using the Eq. (3).

$$R_m = \frac{\Delta P}{\eta \cdot J} \tag{3}$$

Membrane resistance values and their variations used in the experiments and collected during this research are shown in Table 4.

As can be observed in Table 4, the smaller the MWCO the higher the membrane resistance and therefore higher pressure required (shown in Table 2). For RC membranes, the standard deviation in the R_m values ranged from 7% for 10 kDa up to 17% for 100 kDa. For PES membranes, where more membranes were considered in the average, the standard deviation ranged from 10.5% for 50 kDa up to 13.8% for 10 and 30 kDa. The average R_m value for a package (10 membranes) is in general more homogeneous than the average considering several packages. This may be due to a lot-to-lot manufacturing differences while producing membranes. The measured variation in R_m might be due to a non-uniform pore size distribution and non-uniform surface porosity.

Membrane resistance as expressed by Poiseuille's equation (Eq. (4)) depends on thickness (Δx), tortuosity (τ), porosity (ε) and pore size (r_p), as follows:

$$R_m = \frac{8 \cdot \Delta x \cdot \tau}{\varepsilon \cdot r_p^2}. \tag{4}$$

RC membranes showed a higher membrane resistance than PES membranes for the same MWCO. This is an indication than RC membranes most likely have a higher thickness, lower surface porosity, and/or higher tortuosity and/or smaller pore size, hence, leading to

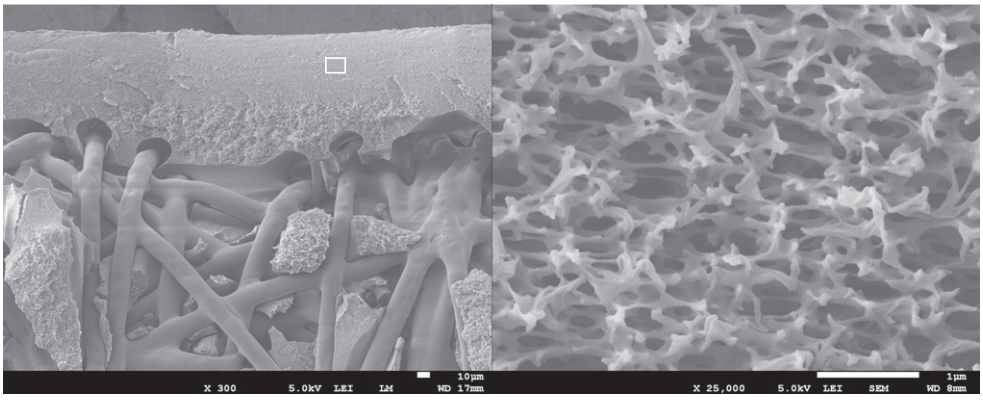

Fig. 5. Cross-section of PES 100 kDa membrane (left — 300×, right — 25,000×).

Table 3
Contact angle values for PES 100 kDa (3 samples) and RC 10 kDa (5 samples) membranes.

Material	Pore size	Left angle	Right angle	Average	Wettability
PES	100 kDa	64° ± 3.9°	65° ± 4.6°	64° ± 4.2°	Hydrophilic +
RC	10 kDa	41° ± 0.5°	40° ± 2.6°	41° ± 1.5°	Hydrophilic ++

higher pressure through the membrane at same flux (shown in Table 2). According to Mulder [20], a uniform molecular weight of membrane polymer does not exist but rather a molecular weight average. Hence, even though the MWCOs are the same, this does not mean that the pore size is the same as most manufacturers measure the MWCO in different ways.

4. Part II: variables in the MFI-UF test

4.1. Membrane material

There are several materials used in ultrafiltration such as: PES, RC, PAN, and PVDF. PVDF is produced mainly for tight MF membranes. PAN and PES are more likely for hollow fiber membranes and in various pore sizes. For this research, PES and RC membranes were tested as the range of pore size available was wider.

Fig. 8 shows the measured MFI values for the same solution (Delft canal water) using a whole package of new membranes (containing 10 specimens). For the PES membranes, the average was 3880 s/L^2 ± 395 (10.3%), and for the RC membranes the average was 3800 s/L^2 ± 235 (6.3%). Both membrane materials have an average value close to each other. The relative standard deviation for RC membranes was 6.3% while for PES membranes was 10.3%.

4.2. Membrane pore size

The pore size range of ultrafiltration membranes is large. Pore sizes can vary from a few micrometers to nanometres. As the MFI-UF test works in a dead-end configuration, all the particles larger than the pore size of the membranes are retained. This means that the smaller the membrane pore size the more particles will be captured, thus creating a thicker and less porous cake. At the same time, the fouling potential of the water is proportional to the concentration of particles in the water; this means that the MFI value with a smaller pore size membrane will be higher that with a looser membrane. This is illustrated in Eq. (5) [5,27].

$$\mathrm{MFI} = \frac{\eta_{20\ ^\circ\mathrm{C}} \cdot 90 \cdot (1-\varepsilon) \cdot C_b}{\rho_p \cdot d_p^2 \cdot \varepsilon^3 \cdot \Delta P_0 \cdot A_0^2}. \tag{5}$$

The above formula considers the ideal case that particles are spherical. Where: ρ_p = particle density forming the cake, kg/m^3; ε = porosity of cake; d_p = particle diameter, m; η_{20} = viscosity at 20 °C, N · s/m^2; ΔP_0 = trans-membrane pressure of 2 bar as reference at 20 °C; A_0 = membrane surface area of 13.8×10^{-4} m^2.

This trend is illustrated in Fig. 9 where North Sea water was tested with various pore sizes.

It can be observed that the measured MFI value depends strongly on the pore size (MWCO) of the membrane used in the test. 10 kDa membranes gave MFI values 3 to 4 times higher than 100 kDa which clearly indicate that small particles dominate the fouling potential. This finding is supported by the equation of Carman–Kozeny where the specific cake resistance in inversely proportional to the particle size.

To define a membrane pore size for the MFI-UF test with the criterion "one size fits all" is incorrect as feed waters are unique (concentration of

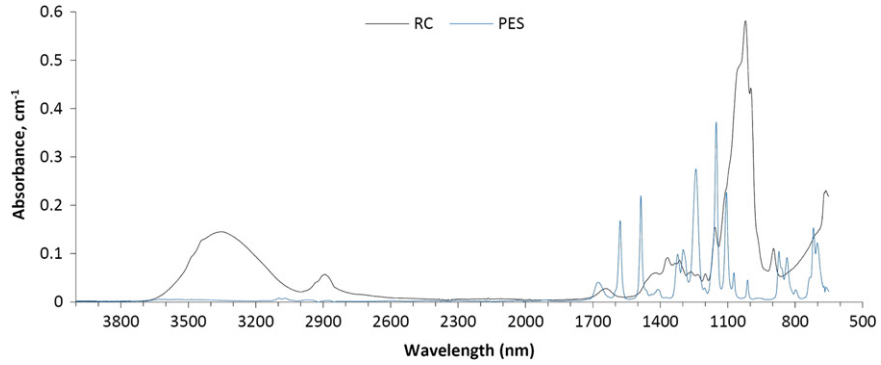

Fig. 6. FTIR characterization of clean PES 100 kDa and RC 10 kDa membranes.

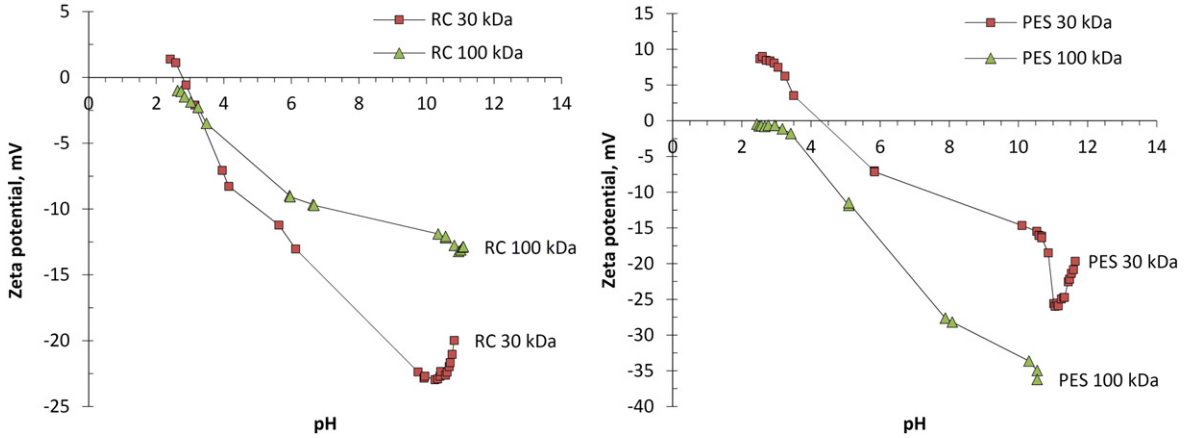

Fig. 7. Zeta potential measurements for RC and PES membranes.

Table 4
Membrane resistance values for PES and RC membranes.

Material	MWCO, kDa	Nr. filters	Avg. R_m, 1/m	Max	Min	Rel. Std Dev.
PES	5	4	1.22E + 13	1.30E + 13	1.12E + 13	6.2%
	10	39	1.05E + 12	1.65E + 12	9.10E + 11	13.8%
	30	56	8.38E + 11	7.02E + 11	7.02E + 11	13.8%
	50	16	6.49E + 11	7.50E + 11	5.55E + 11	10.5%
	100	43	3.30E + 11	4.96E + 11	2.84E + 11	12.9%
RC	5	4	3.21E + 13	3.47E + 13	3.05E + 13	7.1%
	10	6	1.54E + 13	1.64E + 13	1.39E + 13	6.9%
	30	7	1.83E + 12	2.06E + 12	1.37E + 12	12.3%
	100	45	5.01E + 11	6.47E + 11	3.57E + 11	16.8%

particles, type/nature of particles and colloids, etc.). The proper membrane pore size should be selected by projecting the increase in net driving pressure and comparing with actual RO performance. Nevertheless, depending on the purpose of the measurements, some guidelines can be given. The applications of the MFI-UF measurements can be: i) monitor water quality before and after pre-treatment for RO systems, and ii) predict the rate of RO particulate fouling.

4.3. Flux rate

The applied flux in the MFI test affects the MFI value of certain water. Fig. 10 shows the MFI-UF values for Mediterranean raw seawater (left) and for UF permeate (0.02 μm pore size) and dual media filtration (DMF) effluent (right) measured at various flux rates from ~50 L/m^2/h up to 350 L/m^2/h.

The linear increase of the MFI value proportional to the applied flux is most likely due to the cake compression effect. The specific cake resistance is not a function of pressure only but also depends on filtration rate and velocity of solid deposition. With increasing linear velocity of solid deposition, specific cake resistance increases [14,24]. In general, regarding the equation derived by Carman [34] specific cake resistance (or MFI-UF value, see Eq. (6)) is inversely proportional to porosity and size of deposited particles formed the cake layer on membrane, but the tests shown in Fig. 10 were carried out using same feed water, therefore, cake resistance is only related to cake porosity.

The dependency of MFI-UF on flux, means that to assess accurately particulate fouling in RO systems, the MFI-UF should be measured at a flux similar to a RO system (close to 20 L/m^2/h) or extrapolated from higher fluxes. Boerlage et al. [6] also observed a linear trend when measuring MFI-UF values with 13 kDa polyacrylonitrile (PAN) membranes for tap water and canal water for flux rates between 70 and 110 L/m^2/h.

Karabelas and Sioutopoulos [18] examined the effect of flux on specific resistance of sodium alginate solutions (10 mg/L and TDS = 500 mg/L and 2000 mg/L) for a narrower and lower flux range (10–30 L/m^2/h) under constant flux and constant pressure filtration. The authors also reported a direct relation between these two variables and recommended the use of the specific cake resistance as indicator of fouling propensity.

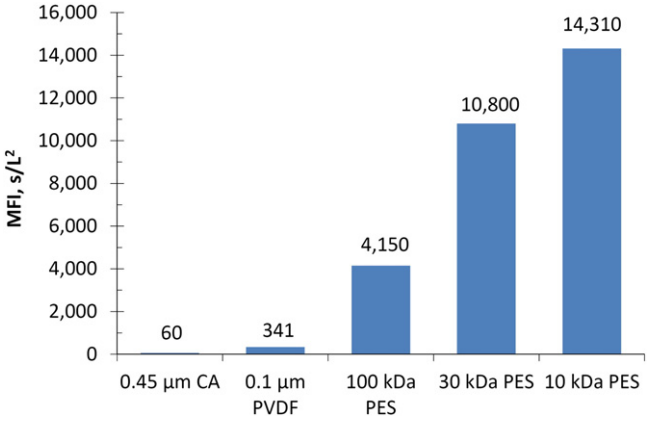

Fig. 9. MFI values for North Sea water for various MWCOs. Values obtained at constant pressure filtration.

4.4. Concentration of particles

Further evidence that cake filtration occurs during the MFI-UF test can be observed in the results of the MFI-UF as a function of particle concentration in the feed water [8]. This premise is based on the fouling index, I, being directly related to the concentration of particles C_b (Eq. (6)).

$$I = \alpha \cdot C_b. \tag{6}$$

Thus, I will decrease directly in proportion to an increase in the dilution factor of C_b while the specific cake resistance component (α), characteristic of a feed water type and independent of concentration, remains constant.

In Fig. 11, the results of the MFI-UF with dilutions of Delft canal water at an applied flux of 100 L/m^2/h are presented. 50% concentration means that the canal water was diluted 1:1. 75% concentration means that 3/4 of the total volume corresponds to canal water and 1/4 corresponds to ultra-pure water. Dilutions were prepared with ultra-pure water.

Linearity was found for the feed water, with the regression coefficient calculated as 0.989. These results demonstrate that the MFI-UF is

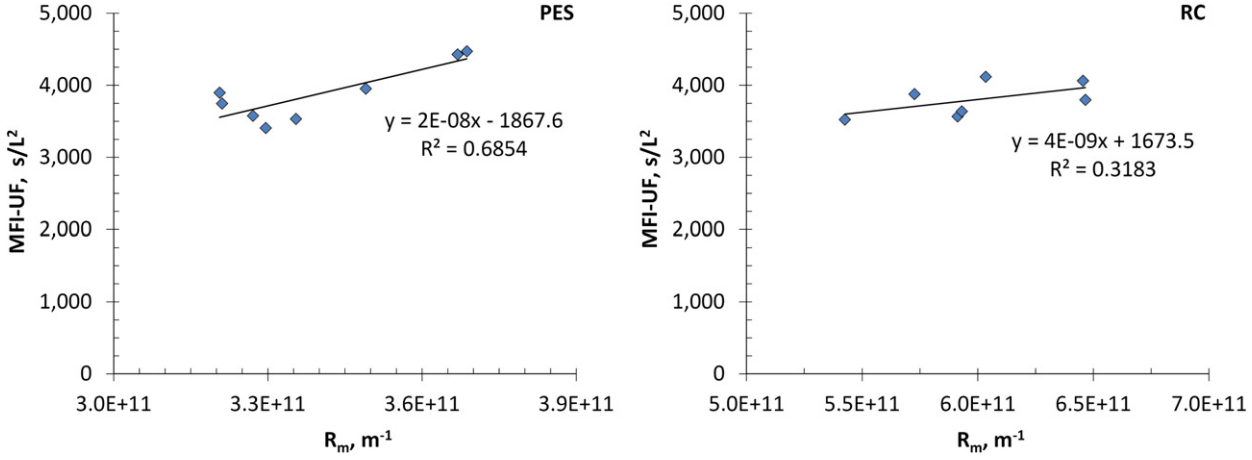

Fig. 8. MFI values for Delft canal water measured with 100 kDa PES (left) and 100 kDa RC (right) at 100 L/m^2/h.

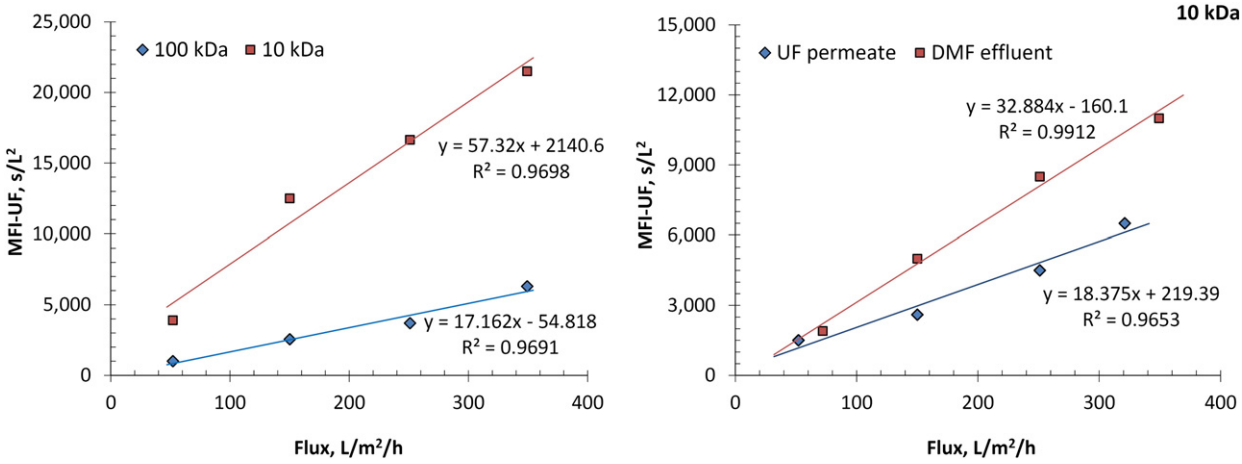

Fig. 10. MFI-UF values as function of filtration flux for Mediterranean seawater. Left as function of membrane MWCO, and right as function of pre-treatment. PES membranes.

proportional with the concentration and verifies that cake filtration occurs in the test during constant flux filtration.

Boerlage et al. [6] reported a linear correlation for four different freshwater samples between MFI-UF constant flux and concentration. Schippers and Verdouw [29] reported, after filtering formazine solutions, that the MFI 0.45 is linear with concentration. A 1 mg/L of formazine had a MFI value of ~1 s/L^2.

4.4.1. Other effects

4.4.1.1. Effect of pressure on membrane material. Compaction of the membranes due to the applied pressure during filtration may occur and it may influence the MFI-UF test as, for instance, the membrane resistance in compacted membranes increases. Membrane compaction is defined as mechanical deformation of a polymeric membrane under pressure causing the porous structure to densify and consequently the flux to decline [20].

To evaluate the effect of pressure on membrane compaction (increase in R_m), ultra-pure water was filtered through PES and RC membranes (100, 30, and 10 kDa). The constant pressure set-up was used for these tests. The pressure was varied between 0.5 and 3.5/4.0 bar in 0.5 bar intervals. The temperature of the feed water was maintained constant throughout the experiments ranging from 20.5 to 22.2 °C. The flux and membrane resistance at each pressure value were measured and calculated according to Eq. (4).

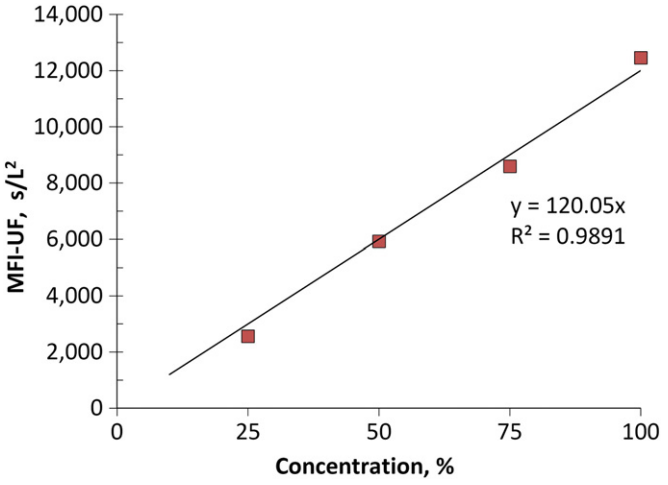

Fig. 11. MFI values for dilutions of Delft canal water measured with 100 kDa RC membrane at 100 L/m^2/h.

Fig. 12 (left) shows the results of flux as function of pressure. The results indicate that 100, 30, and 10 kDa PES membranes are stable over the pressure range of 0.5–3.5 bar, and a linear relationship was obtained between flux and ΔP ($R^2 = 0.99$).

In the case of RC membranes, for 30 and 10 kDa no significant effect of pressure on membrane compressibility was observed ($R^2 = 0.99$ linear). In contrast, the RC 100 kDa membrane showed signs of compaction as the pressure increased from 0.5 to 3.5 bar; the flux did not increase linearly, but started to level-off above a pressure of 1 bar. Moreover, the initial R_m was increased by 38% from 4.9 to 7.9 × 10^{11} m^{-1} as shown in Fig. 12 (right).

The membrane compaction coefficient was calculated by using Eq. (7).

$$R_m = R_{m0} \cdot \Delta P^h \tag{7}$$

where: R_m is the membrane resistance (m^{-1}), R_{m0} is the membrane resistance at zero compressive pressure, ΔP is the trans-membrane pressure (bar) and h is the membrane compaction coefficient.

For the 100 kDa RC membrane, a power law relationship between membrane resistance and pressure, with a *compaction coefficient* of 0.25, was observed for the range of applied pressure (0.5 and 3.5 bar). These results suggest that 100 kDa RC membranes should be used with pressure up to 1 bar. Above this bar, membrane compaction may influence membrane surface properties and thus influence the pressure readings during filtration.

Boerlage [4] also found a power law relationship between membrane resistance and pressure for the PAN 13 kDa. A compaction coefficient of 0.058 and 0.052 was estimated for new and used membranes, respectively. In her study, the initial membrane resistance increased by 8% and 7% for new and used membranes, respectively, while the applied pressure increased from 0.5 to 2 bar using RO permeate water.

4.4.1.2. Effect of salinity on membrane permeability. The adsorption of solutes has a negative influence on the flux because the adsorbed layer presents an extra resistance towards mass transfer and consequently contributes to a decline in flux [20].

Cho et al. [11] studied the influence of ionic strength (10 mM NaCl, and 4 mM Ca^{2+}) on PEG rejection and found higher PEG rejection with higher ionic strength, thus indicating that the pore radii of the membranes are decreased by higher ionic strength. Braghetta et al. [9] studied the permeability of a negatively charged sulfonated polysulfone NF membrane with 1 kDa MWCO and found that the permeability decreased when using ultra-pure water with different amounts of NaCl (93–4380 mg/L) at pH 7. The reduction of permeability was attributed

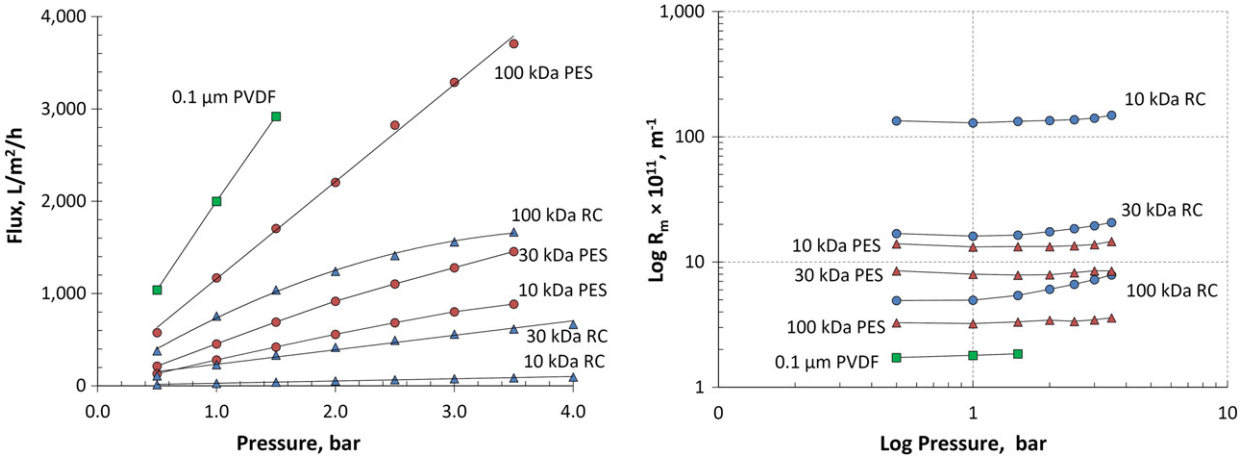

Fig. 12. Flux vs. pressure (left) and log R_m vs. log pressure (right).

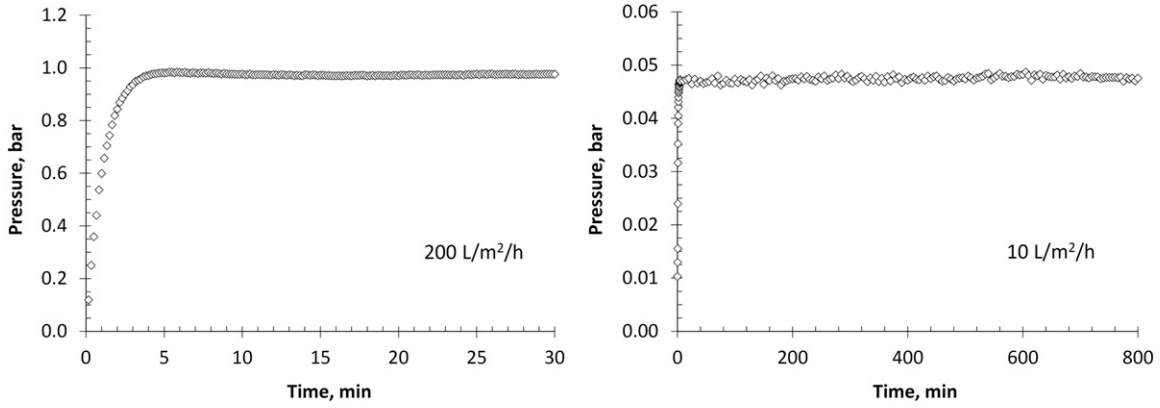

Fig. 13. Filtration of synthetic seawater solution (TDS = 33,000 mg/L) through a PES 10 kDa membrane. Left figure $J = 200$ L/m^2/h. Right figure $J = 10$ L/m^2/h.

to a compaction of the membrane matrix resulting from charge neutralization at the membrane surface and electric double layer compression.

The effect of salinity on the membrane was studied by measuring the MFI-UF value of synthetic seawater solution. Fig. 13 shows two of the filtration tests with a 10 kDa membrane at 200 and 10 L/m^2/h.

Fig. 13 presents the pressure development over time for two flux rates: 200 and 10 L/m^2/h. The slope (pressure over time) in the graphs is horizontal which corresponds to a MFI-UF value equal to zero. As the pressure reading remain constant over the filtration time, the effect of salinity on membrane permeability can be neglected in MFI-UF tests.

4.4.1.3. Effect of salinity on particles. Guéguen et al. [12] cited that increasing ionic strength is known to decrease the effective molecular size of organic molecules in solution, potentially increasing their adsorption

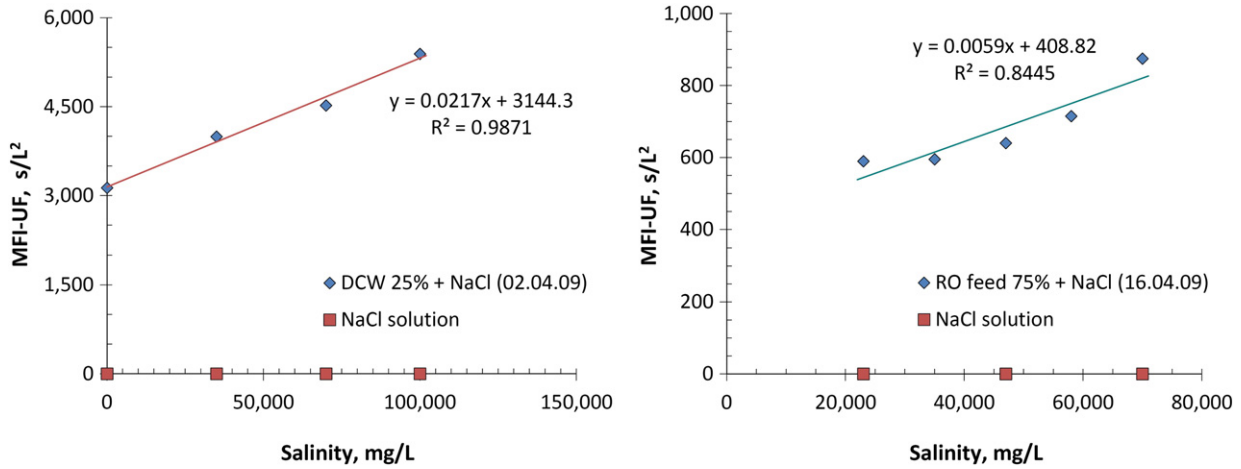

Fig. 14. Salinity effect on particles — MFI-UF of Delft canal water diluted to 25% (left) and SWRO feed (right). PES 10 kDa membranes at flux = 250 L/m^2/h.

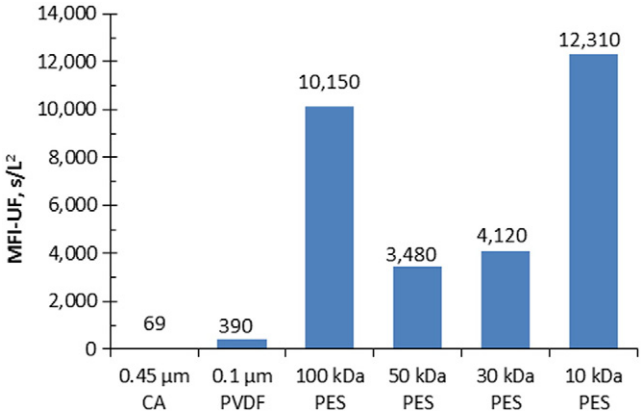

Fig. 15. MFI-UF results for serial fractionation of North Sea water batch 1.

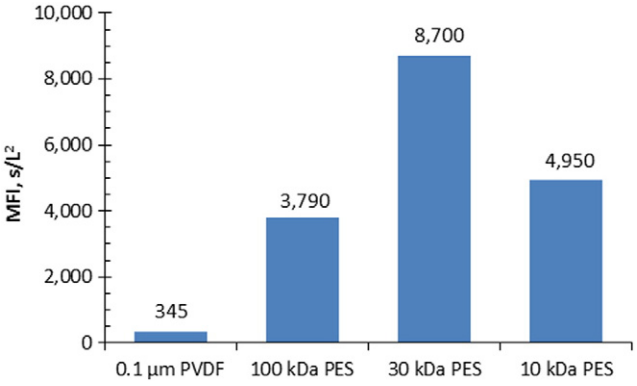

Fig. 17. MFI-UF results of serial fractionation for North Sea water batch 2.

properties on membrane sites. High ionic strength may also favor cake formation in cross-flow filtration as explained below.

Typically, surface water particles are negatively charged and stable due their high zeta potential. In addition, the membrane surface and pores have a negative charge and, when contacted with water, cause a polar medium which develops a double layer. Therefore, an increase of ionic strength may cause compression of the double layer around the particles and membrane surface which lead to an increase of specific cake resistance [7]. These considerations might be valid for hydrophobic particles which get their stability from the charge. Nevertheless, hydrophilic particle, get their stability from the fact that they are surrounded and/or consists mainly of water. The stability comes from the fact that Van der Waals forces are here very weak since the attraction comes from the interaction of water molecules mainly.

In Fig. 14, the MFI-UF values for Delft canal water (DCW, diluted 4 times) and for SWRO feed water (North Sea, diluted from 35 to 23 g/L) are presented. The salinity of the first solution was altered by adding concentrated solution of NaCl (99.9999% purity). For both samples an increase of MFI-UF with salinity was observed.

For a salinity level and an increase similar to a 40% SWRO recovery, the increase was about 10% in the case of canal water and, in the case of SWRO feed water, the increase was about 20%.

The observed effects suggest that salinity may play a role when comparing MFI values of waters with different salinity, as the MFI values of RO feed and RO concentrate. This comparison is of relevance when calculating the deposition factor.

Boerlage et al. [7] tested the effect of salinity on tap water in the range of 0 to 0.2 mol/L, and observed a peak value at 0.1 mol/L. Boerlage explained that ionic strength causes an initial increase in specific cake resistance due to a reduction in cake porosity which is caused by a decrease in the inter-particle distance between particles in cake filtration.

5. Part III: applications

5.1. Particle size and fouling

To investigate the relation between particle size and MFI-UF, North Sea water batch 1 (NSW 1) was tested in series. Serial filtration consisted of using the permeate water of the first filtration step as a feed water for the next filtration test with smaller MWCO than the previous one.

Results in Fig. 15 show an irregular trend. The 10 kDa and 100 kDa MFI-UF values (for particle range 0.1 µm–100 kDa and 30–10 kDa) are of the same order of magnitude and 3–4 times higher than the 50 kDa and 30 kDa MFI values (for fractions 100–50 kDa and 50–30 kDa). These results illustrate the fouling potential of various fractions in the sample water. In the same way, the MFI-UF has a linear relationship with the particle concentration where MFI-UF value increases as particle concentration increases.

A sample was taken after each filtration step and was analyzed by liquid chromatography with organic carbon detection (LC-OCD). Results are presented in Fig. 16 left and right, where: DOC = chromatographic dissolved organic carbon, BP = biopolymers, HS = humic substances, BB = building blocks, and N = neutrals.

With respect to the feed water, a total (chromatographic-) DOC removal of ~9% was found in the permeate of the 10 kDa membrane and the partial DOC removals were 0.2%, 5.7%, 8.3%, 8.5% and 8.7% for 0.1 µm, 100 kDa, 50 kDa, 30 kDa and 10 kDa, respectively. The biopolymers were the organic matter fraction that was mainly retained by the filters (73% in total). The partial removals of biopolymers were 1%, 52%, 26%, 6%, and 16% for 0.1 µm, 100 kDa, 50 kDa, 30 kDa, and 10 kDa, respectively. Humic substances were slightly (~6% in total) removed after the 10 kDa membrane with respect to the raw water.

A second serial fractionation with a different sample from the North Sea batch 2 (NSW 2) was tested as shown in Fig. 17. This feed water indicated that the particles retained by a MWCO of 30 kDa were the most

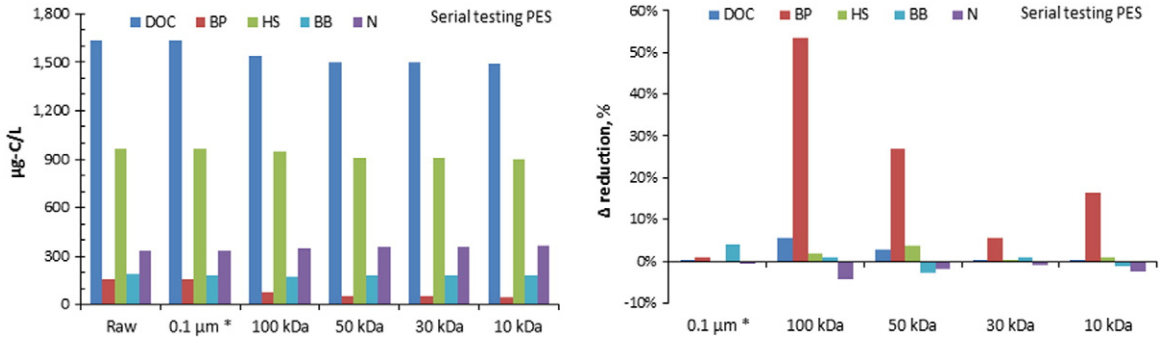

Fig. 16. LC-OCD results — North Sea water batch 1 — serial fractionation.

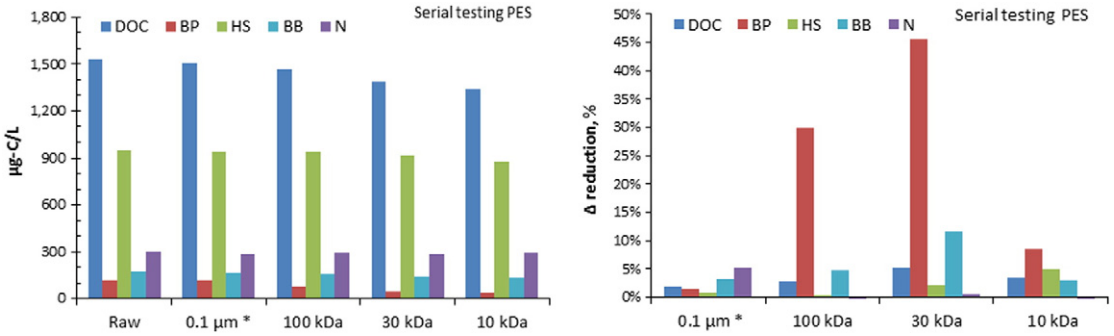

Fig. 18. LC-OCD results for North Sea water batch 2 serial fractionation.

foulant particles. Nevertheless, the water after 30 kDa membrane still has particles which produced a similar MFI-UF value as the 100 kDa membrane.

With respect to the feed water, a total DOC removal of 13% was found in the permeate of the 10 kDa membrane. The total removal of biopolymers after 10 kDa membrane was 66%. In addition, with respect to the feed water, the total removals of biopolymers were 1%, 31%, 62% and 66% for the 0.1, 100 kDa, 30 kDa and 10 kDa, respectively. The partial biopolymer removals were 1%, 30%, 46% and 8% for 0.1 µm, 100 kDa, 30 kDa and 10 kDa respectively. In the LC-OCD test, the low molecular weight acids were not detected (Fig. 18).

In both cases, NSW1 and NSW2, the organic matter fraction that was mainly removed by the filters was the biopolymers. For the NSW 2, there is a more clear relation between the biopolymer and humic substances removal and MFI-UF values at 100, 30 and 10 kDa; while for NSW 1, there is a high biopolymer removal and high MFI value in the 100 kDa membrane. The combination of MFI-UF and LC-OCD results illustrate that smaller particles have high fouling potential even at low concentrations.

5.2. Assessment of pre-treatment

Fig. 19 shows the MFI-UF values measured with 100, 50, and 10 kDa membranes at 250 L/m^2/h along a SWRO plant treating water from the North Sea. The plant is located in Kamperland (The Netherlands) and has been described extensively by other researchers [30].

The performance of pre-treatment processes at the Jacobahaven SWRO demonstration plant was assessed using MFI-UF measured with 100, 50, and 10 kDa test membranes as shown in Fig. 19. The Amiad strainer showed only a small reduction in MFI-UF as expected with a relatively large aperture size of 50 µm. Whereas, the reduction in MFI-UF (and fouling) observed following UF (nominal MWCO of

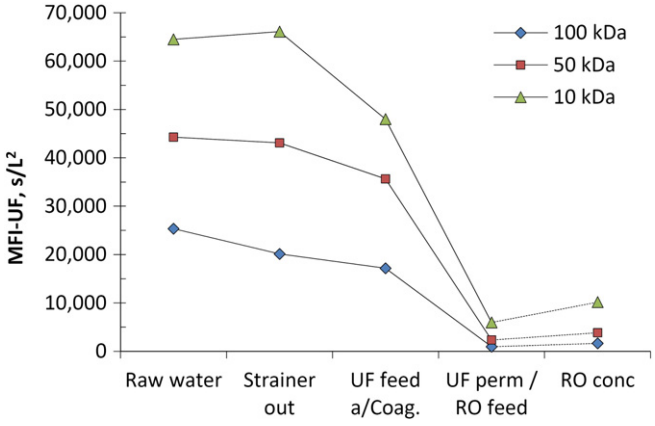

Fig. 19. Effect of pre-treatment on MFI-UF in a seawater pilot plant using PES membranes of 100, 50, and 10 kDa at 250 L/m^2/h.

150 kDa) was much larger i.e., of 94%, 93%, and 88% reduction for 100 kDa, 50 kDa, and 10 kDa MFI-UF test membranes, respectively.

In addition, in Table 5 the MFI measurements are presented for various dates. Although the raw water values varied with time, the percentage decrease of MFI-UF values was, in all cases, more than 90%. These results clearly illustrate that the MFI-UF can be used (at any temperature) to measure low and high fouling feed water and for UF permeates.

5.3. Predicting pressure increase in RO systems

Most RO desalination plants operate at constant flux to meet production requirements. Changes in feed water temperature are compensated for by adjusting feed pressure. Similarly, fouling resulting in an increase in membrane resistance is compensated for by increasing the feed pressure and hence net driving pressure (NDP). In this case, increase in the NDP can be predicted through Eq. (8). However, for accurate prediction a correction factor, deposition factor Ω, has to be incorporated. The deposition factor takes into account that not all particles passing the membrane surface (in cross-flow) deposit and remain attached on the membrane surface.

$$P_t = \eta \cdot R_m \cdot J + \eta \cdot \Omega \cdot I \cdot J^2 \cdot t \tag{8}$$

where: P_t = NDP at time "t" to maintain constant flux (N/m^2); and Ω = deposition factor (−). Note: Osmotic pressure enhanced fouling is not accounted for in this equation. Consequently the pressure development might be under predicted [13,31].

An indication of the deposition factor can be obtained by measuring the MFI_{feed} in feed water and MFI_{conc} in the concentrate and applying Eq. (9) [28]:

$$\Omega = \frac{1}{R} + \frac{MFI_{conc}}{MFI_{feed}} \cdot \left(1 - \frac{1}{R}\right) \tag{9}$$

where: R = recovery (−). A deposition factor equal to zero indicates no particle accumulation while a deposition factor equal to 1 indicates that all particles retained by the RO membranes remain on their surface and contribute to pressure increase.

Based on Eq. (9), a theoretical "safe MFI" can be calculated assuming e.g., an allowable increase in NDP of 1 bar in 6 months. Fig. 20 illustrates

Table 5
MFI-UF (100 kDa) values in s/L^2 and percentage decrease of MFI-UF after UF.

Date	Raw water	UF feed	UF perm	Decrease of MFI-UF value
23 April	4310	2935	190	94%
28 April	4840	4295	125	97%
16 June	3800	3650	395	89%
02 July	2950	2285	203	91%
06 July	2840	2450	200	92%
10 May	25,340	17,190	980	94%

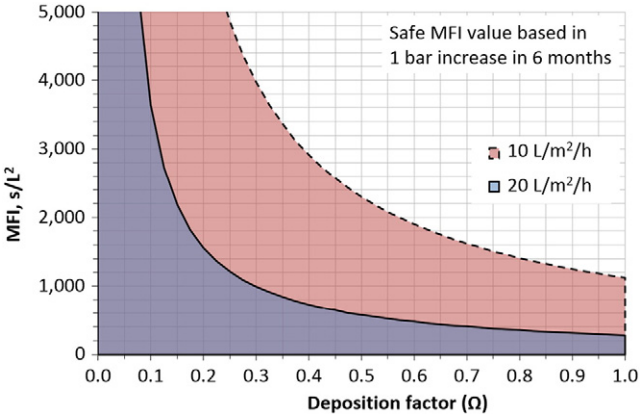

Fig. 20. "Safe MFI value" as a function of deposition factor for 10 L/m^2/h and 20 L/m^2/h.

MFI calculated as a function of the deposition factor Ω at a flux of 10 to 20 L/m^2/h, which is commonly applied in seawater RO.

"Safe MFI" values are heavily dependent on the deposition factor, emphasizing the need to determine deposition factors in full-scale and pilot plants. Deposition factors were measured using membranes of various sizes (100 kDa, 50 kDa, 10 kDa, and 5 kDa) in a pilot SWRO plant in Kamperland (Netherlands). The pilot SWRO makes use of UF as pre-treatment (Pentair, Xiga UF). The RO concentrate samples were diluted to same TDS concentration as the RO feed water to consider the effect of salinity as presented in Fig. 14.

The results showed deposition factor values ranging between 0.20 and 0.45 (Table 6).

The deposition factors were positive and suggest the partial accumulation of particles on the surface of the RO membranes. Measuring the deposition factor in a multitude of full-scale plants to define the "safe MFI" needs to be addressed to further develop the MFI-UF tool and spread its application. This requires operational data on fouling rates in RO systems for validation.

6. Conclusions and recommendations

- A new portable set-up has been developed to perform MFI-UF tests at constant flux filtration. The set-up has been used for on-site testing and for testing in laboratory.
- Two membrane materials (PES and RC) and various MWCOs (100, 50, 30, and 10 kDa) were investigated for MFI-UF tests. PES membranes have much lower resistance than RC membranes. It was measured up to 14% variation in R_m in the same batch of membranes.
- The MFI-UF constant flux is a promising tool for assessing particulate and colloidal fouling potential of fresh and seawater. It can be used to assess pre-treatment efficacy in controlling particulate fouling and in estimating the rate of RO fouling.
- Pore size of the membranes and filtration flux are the most important variables in the test. UF membranes with low MWCO are promising in predicting rate of RO fouling. Filtration flux similar to the average flux rate in a pressure vessel is recommended to measure the fouling potential of RO feed water.
- A safe MFI value has been defined for RO feed water. Considering 1 bar of pressure increase over a six month period, and a deposition factor equal to one (worst case), the maximum MFI value is equal to 280 s/L^2 for flux equal to 20 L/m^2/h, or 1120 s/L^2 for flux 10 L/m^2/h.

Table 6
Deposition factor measured with various MFI-UF test membranes (PES) at a SWRO pilot plant.

Membrane, MWCO	100 kDa	50 kDa	10 kDa	5 kDa[a]
Deposition factor Ω	0.23	0.29	0.20	0.45

[a] 5 kDa membranes were tested at flux = 10 L/m^2/h. Other tests were performed at 250 L/m^2/h.

Three major aspects need to be addressed in further developing the MFI test: i) MFI test membranes with even smaller pores, ideally close to the NF range, and preferably down to 5, and 1 kDa. ii) Measuring the deposition factor in as many full-scale plants as possible to validate the "safe MFI". This requires operational data on fouling rates in RO systems. Differentiation between particulate fouling and bio-fouling is also required. iii) Measuring the enhanced osmotic pressure effect due to fouling in full-scale plants.

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