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# Seawater pretreatment by dead-end micro and ultrafiltration in pressure-driven inside feed

Julie Guilbaud[a], Anthony Massé[a,*], François-Charles Wolff[b], Pascal Jaouen[a]

[a]*LUNAM, Université de Nantes, CNRS, Laboratoire GEPEA UMR 6144, 37 Bd de l'Université, BP 406, 44602 Saint-Nazaire Cédex, France*
*Email: anthony.masse@univ-nantes.fr*
[b]*Laboratoire d'Economie et de Management de Nantes Atlantique (LEMNA), EA 4272, Chemin de la Censive du Tertre - BP 52231 44322 Nantes Cedex 3 France*

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ABSTRACT

Low pressure microfiltration (MF) and ultrafiltration (UF) is used as seawater pretreatment before reverse osmosis membranes for capacity that ranged from 6,700 to 140,000 $m^3$ per day at Colakoglu Steel Mill (Turkey), Yu-Han (China), Kindasa (Saudi Arabia), Fukuoka (Japan), and Ad Dur (Bahrain). Among all modes of porous membrane filtration, pressure-driven inside feed configuration accounts for about 30% of all membrane configurations used for water and wastewater treatment. The present study deals with the MF and UF with hollow fiber membranes (polyacrylonitrile [PAN] 50 kDa, polyethersulfone [PES] 100 kDa, and polyvinylidene fluoride [PVDF] 0.1 μm) of seawater in pressure-driven inside feed configuration. Several cycles of filtration have been carried out at $100\,L\,h^{-1}\,m^{-2}$ during 30 min for each followed by 30 s of permeate backwash at $250\,L\,h^{-1}\,m^{-2}$. Microalgae-rich seawater has been prepared at laboratory which contained 30 g of salt, $1.2\times10^8$ $(+/-0.25\times10^8)$ of cells (*Nannochloropsis oculata* and *Skeletonema costatum*) per liter. The highest fouling resistance ranging from 1.57 to $3.25\times10^{11}\,m^{-1}$ has been found for the PES membrane with an increase of the resistance value along filtration cycles. Whatever the used membrane, all microalgae have been retained and the backwash efficiencies to microalgae removal from membrane increased along filtration cycles. On the basis of these results, the 0.1 μm PVDF membrane seems to be more suitable to seawater membrane pretreatment.

*Keywords:* Seawater; Ultrafiltration; Microfiltration; Microalgae; Natural organic matter

## 1. Introduction

Seawater treatment capacity at industrial scale tends to increase with the continuous increase of fresh water demand. For example, 17,000 $m^3/d$ of ultrafiltered seawater are produced by PES membrane (100 kDa Norit X-Flow—Pressure Driven Inside) at Colakoglu Steel Mill in Turkey for 2008. $SDI_{15}$ and turbidity were inferior to 3% min and 0.1 NTU, respectively. At Yu-Han in China (2006), an installation of immerged hollow fiber ultrafiltration (UF) membrane generates 70,000 $m^3/d$ of ultrafiltered water (PVDF 100 kDa Zenon—submerged membrane) [2]. In Perth (Australia), a project was commissioned in 2011

*Corresponding author.

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for microfiltration (MF) pretreatment system extension to 316,000 $m^3/d$ [3].

MF and UF with hollow fibers membrane are more and more frequently used to pretreat seawater [4–6] and particularly before reverse osmosis (RO) in the place of conventional pretreatment [7] because they are more compact [8,9] and more efficient to protect RO from fouling by removing phytoplankton, silica, and organic matter. Indeed, the MF/UF membranes produce permeate with almost constant quality contrary to the water filtered with sand filter, in particular, during bloom periods [10,11].

UF membrane setup allows a decrease in the chemical volumes needed to clean the unit. Consequently, the cost associated to pretreatment does not increase and is around 10–40% of the total specific energy consumption of desalination units [12].

For seawater pretreatment, dead-end filtration is the most useful mode of filtration. Three kinds of dead-end filtration exist: the feeding is driven inside the fiber lumen (PDI), outside the fibers (PDO), or membrane is submerged inside the seawater to be treated (SUB). PDI configuration represents 30% of all membrane configurations used for water and wastewater treatment [1]. With PDI configuration, chemical volumes of cleaning are often lower than for the other configurations.

Polyethersulfone (PES) and polyvinylidene fluoride (PVDF) are traditionally used membrane materials for seawater pretreatment before RO. PES membranes are usually used in PDI configuration contrary to PVDF membranes which are used in PDO and SUB configurations [1,13,14]. Polyacrylonitrile (PAN) is not a common material used at industrial scale, but Rossi et al. [15] showed that, PAN 50 kDa allowed to ultrafiltrate a microalgae suspension without lot of fouling. PAN membrane has a moderate hydrophilicity, requires low cleaning frequency, and is less susceptible to fouling with water and wastewater [14].

MF and UF for seawater pretreatment at industrial scale are more often conducted at constant permeate flux. The imposed permeate flux ranges from 60 to 140 $L\,h^{-1}\,m^{-2}$ and depends on feed quality. Membranes are generally backwashed every 15-60 min of filtration [16], during 20–60 s with a flux 2.5–3 times higher than permeate flux [17]. The aim of this study is to choose the membrane which induces the lowest specific energy consumption and a total rejection of microalgae during microalgae blooms.

In this study, operating conditions of filtration and backwash correspond to those usually imposed at industrial scale: 30 min of filtration at 100 $L\,h^{-1}\,m^{-2}$, followed by b1ackflush fixed at 250 $L\,h^{-1}\,m^{-2}$ during 30 s. Three membranes were used: PAN 50 kDa, PES 150 kDa, and PVDF 0.1 μm with PDI configuration.

## 2. Material and methods

### 2.1. Synthetic seawater reconstitution

Because physicochemical and biological characteristics of natural seawater vary with tide, season, climate, and locality, synthetic seawater was prepared in this study.

According to bibliographic study, it appears that maximal phytoplankton concentration is around $1.2 \times 10^8$ cells/L during seawater bloom periods.

Phytoplankton is generally composed in majority by diatoms and dinoflagellates. One of the smallest toxic dinoflagellate listed and present in natural blooms is *Azadinium spinosum* [18], its ovoid size is around 5–12 μm. If these microalgae are retained by membrane process, all microalgae will be retained too. As these microalgae are toxic, it has not been used for seawater synthesis in lab for security reason. A microalgae with closed size (around 2–6 μm): *Nannochloropsis oculata*, was selected and cultivated in airlift with controlled conditions (fed-batch, pH = 8, in f/2 media [19,20]). *Skeletonema costatum* was chosen to simulate the diatoms naturally present in many coastal seas. Diatoms are cultivated in raceway feeding by natural drilling seawater.

Synthesized seawater rich in microalgae was composed of 75% of *S. costatum* and 25% of *N. oculata*. After cells counting, microalgae cultures were diluted with osmosed water containing 30 g/L of NaCl to obtain reconstituted seawater with $1.2 \times 10^8$ cells/L.

The characteristics of synthetic seawater are summarized in Table 1. Mean diameter (in number) equal to 4.5 μm was determined with image analysis sensor (Sympatech—QicPic). Conductivity was observed to be equal to 44 mS/cm at 20˚C.

### 2.2. MF–UF pilot plant

Novel synthetic seawater was prepared before each micro or UF.

The pilot plant used for filtration is described in Fig. 1.

This pilot is equipped with a volumetric membrane pump (Grundfos DMX 221) which allows to feed fibers lumen with seawater. Pressure and temperature captors (Wika—accuracy: 0.5–1% of span) are located in the module (0–2.5 bar) and in the permeate

Table 1
Characteristics of prepared seawater

| TSS (mg/L) | Turbidity (NTU) | Chlorophyll a (µg/L) | TOC (mg/L) | DOC (mg/L) |
|---|---|---|---|---|
| 2.5 ± 0.3 | 1.86 ± 0.30 | 75.8 ± 11.7 | 1.771 ± 0.346 | 0.551 ± 0.050 |

Note: TSS—total suspensed solid; TOC—total organic carbon; DOC—dissolved organic carbon.

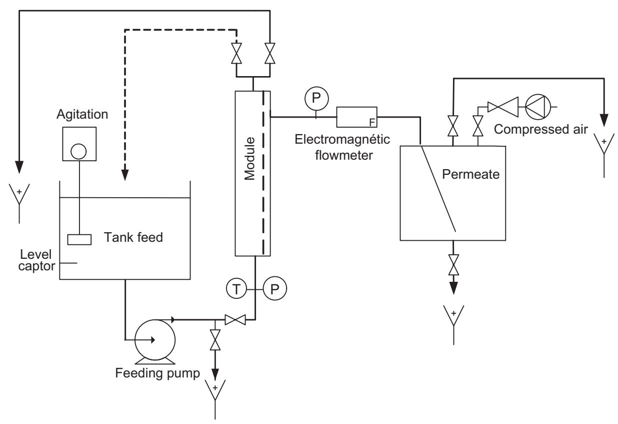


Fig. 1. Pilot plant scheme.

exit (0–4 bar). An electromagnetic flow meter (Techfluid-Flomid 0 FX) connected to computer allows to maintain a constant permeate flux. All data were recorded. Resistances and permeability are calculated with Darcy Law (Eq. (1)) and permeate flux is corrected at 20°C.

$$J_p = \frac{TMP}{\mu \cdot R} \quad (1)$$

With,
TMP: transmembrane pressure (Pa);
$J_p$: permeate flux ($m^3\,m^{-2}\,s^{-1}$);
$\mu$: viscosity at 20°C (Pa s);
$R$: resistance ($m^{-1}$).
Each filtration sequences are composed of:

- 30 min of filtration at 100 $L\,h^{-1}\,m^{-2}$.
- 5 s of relaxation.
- 30 s of backwash at 250 $L\,h^{-1}\,m^{-2}$.
- 5 s of relaxation.

During this study, seawater was filtered at constant flux of permeate: 100 $L\,h^{-1}\,m^{-2}$ during 30 min i.e. 50 L of seawater filtered per square meter of membrane.

The characteristics of used membranes are summarized in Table 2.

### 2.3. Sampling and analyses

Synthetic seawater was sampled before each trial to analyze chlorophyll a concentration, turbidity, total suspended solid (TSS), dissolved organic carbon (DOC), and total organic carbon (TOC). DOC was analyzed with Shimazu T5000A after the filtration of sample (MWCO: 0.7 µm) acidified with hydrochloric acid (pH 2) and purging of inorganic carbon with nitrogen. Turbidity was measured with HACH 2100AN Turbidimeter. Chlorophyll a concentration was analyzed with AFNOR method NF T 90-117 [21].

At the end of filtration sequence (filtration + backwash), sample of the whole permeate produced during all the cycles of filtration was taken. The total volume produced during backwash was sampled and analyzed.

Some samples are taken during backwash: solutions produced during 5th–10th, from 15th to 20th,

Table 2
Characteristics of membranes used in dead-end filtration

| Type of membrane | UF | UF | MF |
|---|---|---|---|
| Material | PES | PAN | PVDF |
| MWCO UF (kDa), MF (µm) | 150 | 50 | 0.1 |
| Membrane surface ($m^2$) | 0.25 | 0.19 | 0.12 |
| Module dimension (mm) | 380 × 50 | 374 × 42 | 374 × 42 |
| Fibers internal/external diameter (mm) | 0.9/–[a] | 0.8/1.4 | 1.4/2.2 |
| $Lp_0$ at 20°C ($L\,h^{-1}\,m^{-2}\,bar^{-1}$) | 572 | 536 | 1,463 |

[a]Multibore membrane containing seven capillaries, external diameter of the fiber is 4.3 mm.


Fig. 2. Selected samples during backwash time.

and from 25th to 30th second of backwash (Fig. 2) were analyzed.

## 3. Results

### 3.1. Comparison of TMP and backwashes efficiency on four filtration sequences for the three tested membranes

#### 3.1.1. Hydraulic efficiency

Fig. 3 presents the evolution of TMP in function of time. TMP during filtration for the PVDF 0.1 µm and PAN 50 kDa membranes was 0.08 and 0.2 bar, respectively. Consequently, TMP measure did not show a membrane fouling.

TMP of PES 150 kDa membrane was around 0.19 bar at the beginning of the first cycle whereas TMP was around 0.25 bar at the end of the 4th cycle. The fouling resistance ranged from 1.57 to $3.25 \times 10^{11}\,m^{-1}$. While the initial water of the both membranes is close, PES 150 kDa seems more sensitive to fouling than PAN 50 kDa. So, UF with PES 150 kDa requires a higher pressure to maintain the flux at $100\,L\,h^{-1}\,m^{-2}$, and this pressure increases with the number of cycles.

With the same conditions of filtration and backwash, a residual fouling remains with PES membrane after each backwash. For the trials with the PVDF and PAN membranes, no residual resistance is visible after backwash. With unchanged MF/UF conditions, the mean TMP is 2.5 times lower for PVDF 0.1 µm than for PAN 50 kDa. Indeed, membrane resistance of PVDF 0.1 µm ($2.45 \times 10^{11}\,m^{-1}$) is around 2.7 times lower than for PAN 50 kDa ($6.7 \times 10^{11}\,m^{-1}$).

The specific energy consumption ($E$ in $Wh\,m^{-3}$) is defined by the Eq. (2). The head loss through the feed pump ($\Delta P$) is related with pressure upstream the membrane. So, specific energy consumption in MF with PVDF 0.1 µm would be 33.5% lower than with PAN membrane.

$$E = \left(\frac{Q_p \cdot \overline{\Delta P_F} \cdot \Delta t_F}{3600 \cdot \eta} + \frac{Q_{BW} \cdot \overline{\Delta P_{BW}} \cdot \Delta t_{BW}}{3600 \cdot \eta}\right) / (Q_p \cdot \Delta t_F - Q_{BW} \cdot \Delta t_{BW}) \quad (2)$$

With,
$Q_{P,\,BW}$: permeate and backwash flow rate ($m^3\,s^{-1}$);
$\Delta P_{F,\,BW}$: mean pressure head loss during a filtration or backwash cycle (Pa); $\Delta t_{F,\,BW}$: filtration or backwash time (s);
$\eta$: pump yield.

Seawater contains silica colloids which can be introduced in MF membrane pores [22] and induce more fouling at long term. So, MF and UF must be driven on long term to choose the more appropriate membrane for the application.

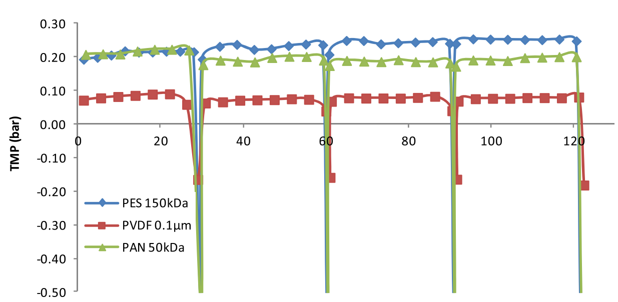


Fig. 3. TMP comparison for three membranes (PAN 50 kDa, PES 150 kDa, and PVDF 0.1 µm): filtration at $100\,L\,h^{-1}\,m^{-2}$ during 30 min and backwash at 250 $L\,h^{-1}\,m^{-2}$ during 30 s.

#### 3.1.2. Permeate quality and microalgae rejection

Some revival tests were made in the laboratory: 500 mL of permeate were filtered on sterile membrane discs (0.2 µm) then were put on a Petri box with f/2 medium with 9 g/L of agar. The boxes were placed in an illuminated incubator at 16℃. After four weeks of culture, growth of microalgae was not observed.

Whatever the membrane, microalgae are retained in totality. The chlorophyll a concentration in permeate was inferior to 0.1 µg/L for all cycles with all membranes.

DOC contained in permeate is inferior to 0.100 mg/L with both UF membranes. DOC in permeate during MF decreases from 0.394 for the first cycle to less than 0.100 mg/L at the last cycle.

#### 3.1.3. Backwash efficiency to remove fouling from membranes

The mass of microalgae eliminated by backwash per mass of microalgae brought to the membrane during one filtration cycle ($\%^w_{BW}$) was calculated according to the following Eq. (3):

$$\%^w_{BW} = 100 \times m^n_{BW} / m^n_F \quad (3)$$

with,
$m^n_{BW}$: mass of chlorophyll a recovered in the $n$th backwash drain during cycle I of filtration.$m^n_F$: mass of

chlorophyll a in feed brought to the membrane during the filtration cycle.

It is worth noting that all microalgae have been retained by membranes used in the present study. The mass of microalgae eliminated by backwash per mass of microalgae brought to the membrane during the $n$ filtration cycle and the remaining after backwash on membrane during $n-1$ filtration cycles $\%^w_{\mathrm{AC-BW}}$ was calculated with the Eq. (4):

$$\%^w_{\mathrm{AC-BW}} = \frac{m^n_{\mathrm{BW}}}{\sum_0^{n-1}(m^i_{\mathrm{F}} - m^i_{\mathrm{BW}}) + m^n_{\mathrm{F}}} \quad (4)$$

with $\sum_0^{n-1}(m^i_{\mathrm{F}} - m^i_{\mathrm{BW}})$ the sum of the residual mass of chlorophyll a remaining on membrane after backwash during the previous filtration cycle(s).

Table 3 shows the percentages of chlorophyll a recovered in backwash drain at each filtration cycle for each membrane. Whatever the membrane, the increase in number of the cycles induces a better removal of particles (microalgae). For example, 27.6% of microalgae have been recovered for PVDF membrane after the 1st cycle against 88.1% after the 3rd for the PVDF membrane.

$\%^w_{\mathrm{BW}}$ with PAN membrane, the recovery is 26–35% less than those of PVDF and PES membrane at 3rd cycle whereas the figure of TMP evolution in function of the time did not show the membrane fouling. $\%^w_{\mathrm{BW}}$ increased from 40.4 to 97.0% for the first to the third cycle for experiment with PES membrane.

The 4th backwash was selectively sampled as explained by Fig. 2. From the 5th to the 10th second of backwash, the biomass is mainly eliminated during the first 10 s of backwash (Fig. 4). Thus, between the 5th and the 10th second, 63.9, 53.4, and 29.3% of biomass brought to the membrane during the fourth filtration cycle are recovered inside backwash drain, for PAN, PES, and PVDF membrane, respectively. From 25 to 30 s, only 1 to 3% were removed. Consequently, the increase of backwash duration is not necessary to eliminate microalgae.

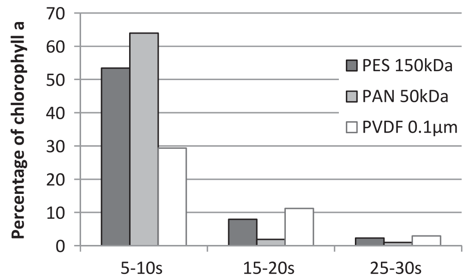


Fig. 4. Percentage of chlorophyll a rejected in the fourth fractionated backwash.

For PAN membrane, more microalgae were recovered inside backwash drain during the time 5–10 s compared to PVDF and PES membrane. Microalgae seem to adhere more highly to the PVDF and PES membrane than PAN membrane.

### 3.2. *Effect of phytoplankton concentration increase on PVDF membrane fouling during 12 h of filtration*

In this part, filtrations have been carried out during 12 h. MF and UF with PVDF and PAN membranes were done at the same conditions as previously presented: $J_{\mathrm{P}} = 100\,\mathrm{L\,h^{-1}\,m^{-2}}$, $J_{\mathrm{BF}} = 250\,\mathrm{L\,h^{-1}\,m^{-2}}$, and filtered volume per cycle = $50\,\mathrm{L\,m^{-2}}$.

The concentration of biomass for the essay with PVDF was doubled in order to show the influence of concentration on the performance of the PVDF membrane. Consequently, feed suspension from trial with PVDF contained $4.5\mathrm{mg_{TSS}/L}$, a turbidity of 3.20 NTU, 148.4 µg/L of chlorophyll a, and 1.428 $\mathrm{mg_{DOC}/L}$. Permeate and retentate were sampled at the end of the 2nd, 3rd, 9th, 10th, 23rd and 24th filtration sequences. The 10th and 24th backwashes were fractionated samples as explained in Fig. 2.

In Fig. 5, mean pressure during 12 h is around 0.11 and 0.14 bar for experiment with PVDF, respectively, with initial (Cx1) and doubled (Cx2) concentration against 0.23 bar for PAN membrane. During 12 h, mean TMP with MF membrane is 40% inferior to those with PAN membrane to maintain the flux at

Table 3
Percentage of chlorophyll a recovered by backwash

| Essays number | | $\%^w_{\mathrm{BW}}$ | | | $\%^w_{\mathrm{AC-BW}}$ | | |
|---|---|---|---|---|---|---|---|
| | Cycles | 1 | 2 | 3 | 1 | 2 | 3 |
| 1 | PAN 50 kDa | 25.9% | 45.5% | 61.6% | 25.9% | 26.1% | 20.3% |
| 2 | PES 150 kDa | 40.4% | 86.3% | 97.0% | 40.4% | 54.1% | 41.6% |
| 3 | PVDF 0.1 µm | 27.6% | 69.4% | 88.1% | 27.6% | 40.2% | 32.0% |

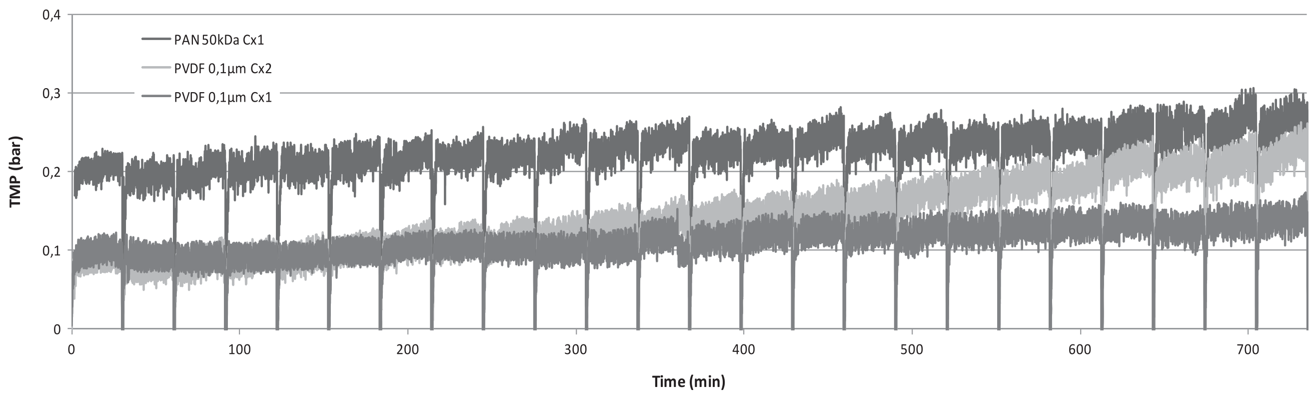


Fig. 5. TMP evolution vs. time for MF/UF of synthetic seawater for 24 filtration cycles at $100\,L\,h^{-1}\,m^{-2}$. Cellular concentration in experiment: $2.4 \times 10^8$ cells/L (Cx2) and $1.2 \times 10^8$ cell/L (Cx1).

Table 4
Chlorophyll a percentages recovered by backwash

| Cycles | 2 | 3 | 9 | 10 (5–10 s) | 10 (15–20 s | 10 (25–30 s) | 23 | 24 (5–10 s) | 24 (15–20 s) | 24 (25–30 s) |
|---|---|---|---|---|---|---|---|---|---|---|
| PAN 50 kDa Cx1 | 52.1 | 68.1 | 79.6 | 57.3 | 3.5 | 0.9 | 95.5 | 43.8 | 5.2 | 1.7 |
| PVDF 0.1 µm Cx2 | 61.0 | 75.3 | 91.8 | 9.5 | 21.8 | 14.3 | 96.6 | 8.3 | 25.5 | 9.4 |
| PVDF 0.1 µm Cx1 | 56.3 | 85.0 | 86.9 | 0.9 | 51.4 | 5.9 | 94.1 | 1.6 | 43.1 | 7.4 |

$100\,L\,h^{-1}\,m^{-2}$ in spite of increase in phytoplankton concentration. From the 10th hour to the end, the pressure was closed from 0.21 to 0.22 bar for PVDF (Cx2) and from 0.25 to 0.26 bar for PAN (mean pressure on one filtration cycle).

Chlorophyll a was not detected in permeates of PAN experiment. For the experiment with PVDF, the chlorophyll a concentration in permeate ranged from 0.1 to 0.3 µg/L from the 2nd to the 24th cycle. Detected chlorophyll a could be due to microalgae breakage. Cell fragments or intracellular compounds are probably the cause of the pore plugging. A small TMP increase is visible on PAN membrane during 12 h of filtration from 0.20 to 0.26 bar.

Table 4 presents mass of chlorophyll a eliminated by backwash per mass of chlorophyll a brought to the membrane during one cycle ($\%^{w}_{BW}$). For MF with doubled phytoplankton concentration, the increase in number of cycles induced a higher percentage of removed chlorophyll a by backwash (from 61.0 to 96.6%) as for PAN experiment (from 52.1 to 95.5%). This evolution seems to confirm that adhesion between membrane and biomass is stronger than between biomass layers. DOC was not detected in total purge of backwash and in permeate with PAN membrane. Consequently, DOC was accumulated on membrane surface. As membrane pores' size is smaller than particles' size (microalgae), fouling could be induced by dissolved organic matter initially present in seawater or coming from cells breakage.

For experiment with PVDF membrane (C82), 1.318–2.213 mg/L of DOC were removed by backwash at each cycle but no DOC was detected in permeate water after the third cycle. Cellular concentration increase seems to induce a constant DOC removal.

Percentages of biomass rejected by backwash on filtrated biomass during the 10th and 24th cycle are presented on Fig. 6. The 10th and 24th backwashes were fractionated. For UF with PAN membrane, 5–10th, 15–20th, and 25–30th seconds of backwash

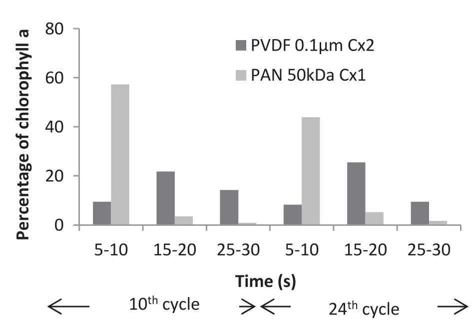


Fig. 6. Percentage of biomass rejected by backwash on biomass filtrated during the 10th and 24th cycle.

induced the removal of 57.3, 3.5, and 0.9% of the biomass filtrated for the 10th backwash and 43.8, 5.2, and 1.7% for the 24th backwash. As in first part of this study, majority of microalgae concentration was removed from the 5th to the 10th second of filtration for the experiments with PAN membrane.

With PVDF membrane, the chlorophyll a concentration was 9.5, 21.8, and 14.3% from the 5–10th, 15–20th, and 25–30th second of the 10th backwash and 8.3, 25.5, and 9.4% for the 24th backwash (Fig. 6).

So, a higher volume of backwash was required to remove fouling layer deposed on membrane surface at 10th and 24th cycle for a doubled biomass concentration. As TMP was multiplied by 2.7 in 12 h of filtration, cake layer on membrane surface was probably compressed making removal by backwash more difficult.

## 4. Conclusion

Microalgae are retained by three tested membranes. MF membranes are more sensitive to blocking pore on long term than UF because particles have size more closed to the size of pore. Consequently, essay on 12 h of filtration shows the advantages of UF. In spite of the advantage of UF, this publication underlines that mean pressure for 12 h needed to maintain permeate flux at $100\,L\,h^{-1}\,m^{-2}$ is 33.5% lower for PVDF than PAN membrane although MF has been realized with doubled biomass concentration. So, in term of energy consumption, PVDF seems more adapted on seawater pretreatment.

To conclude, PVDF membrane is retained for the following studies which will aim to determine the hydrodynamic optimization for the synthetic seawater MF and describe the type of fouling [23,24]. Obtained results will allow to study the techno-economy of the process.

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