Membrane materials for microalgae dewatering

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Abstract

A strategy to reduce costs in the microalgae dewatering process via membrane cross-flow filtration was investigated in this work. It consisted in assessing the possibilities of reduce membrane material cost. Several membrane materials, from commonly used in membrane technology (ceramic, polysulfone and polyacrylonitrile) to generally employed in packaging industry (acrylonitrile butadiene styrene, glycol-modified polyethylene terephthalate and polylactide) were tested. Where, last ones are considerably cheaper than common ones.

Experiments carried out showed interesting results in terms of permeabilities for polysulfone-Pluronic® F127 blended membranes and polyacrylonitrile membranes, but with high related costs. Polylactide membranes had good price, mechanical properties results and biodegradability, but low permeability values. Glycol-modified polyethylene terephthalate

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membranes showed attractive results in terms of costs and permeability, but poor mechanical properties. Finally, the polymer that gave the best results altogether was the acrylonitrile butadiene styrene that reached membrane permeabilities of $19 \pm 0.9 \text{ L/h/m}^2/\text{bar}$, really close to ceramic membranes performance ($21 \pm 2.9 \text{ L/h/m}^2/\text{bar}$). Thus, a novel functionality was found for these not common polymers in water membrane filtration. Additionally, the biodegradable polylactide polymer introduces a new concept of cheap and environmental friendly membrane in this application.

**Keywords**: microalgae, microfiltration, material, poly(lactic acid), acrylonitrile-butadiene-styrene.

1. Introduction

In order to be cost-competitive with petroleum fuels, all the efforts to make feasible the production of biodiesel from microalgae are directed to the reduction of the price that it entails [1-3]. Many studies are addressed to the investigation of microalgae cultivation process and reactors design, but microalgae dewatering is still a major limiting factor for the economic point of view [4]. Consequently, a strategy to reduce its cost is being considered in this paper.

Since now, most used methods are flocculation, flotation, filtration and centrifugation, in general combined [5]. Pros and cons are well described in other articles [6, 7], and it is clear that improvements are still necessary in this way. Membrane filtration is a promising technique in a large number of processes and in particular in this application [8]. Nevertheless, there is a necessity of technological development in this field to reduce process
cost. Several approaches can be considered as dynamic filtration to reduce fouling [8], or membrane material optimization, which is the work being presented in this paper.

So in this work, a comparison between performances of various membrane materials was carried-out. Membrane performance is mainly assessed by its permeability that usually decreases significantly with time due to fouling and concentration polarization [9-13]. Ceramic membranes offer good performances in terms of flow, reproducibility and use, although they are more expensive than polymeric ones [14]. In order to find a membrane with performances like ceramic ones or better but cheaper, membrane synthesis, characterization and testing from different polymeric materials were experimented. A common method to produce membranes is phase inversion, which is the method acquired in this work for membrane synthesis. Where a polymer is dissolved to a non solvent and the solution is introduced in a coagulation bath. Here a mass transfer occurs: the solvent pass to the non solvent and conversely, giving way to the formation of the membrane.

According to several authors polyacrylonitrile (PAN) membranes are chemical stable and they show good performances and versatility in aqueous applications [15, 16], whereas polysulfone (PSf) membranes are the most commonly used and studied [17-19]. For this reason both were investigated in this research. Furthermore PSf was chemically modified to let it be more hydrophilic and to decrease fouling and concentration polarization effects with Pluronic® F127 blending, chosen because of its antifouling [20], biodegradable and biocompatible characteristics.

As the aim of this investigation was to find cheapest way to process microalgae in a biorefinery concept, the attention was addressed not only to polymers commonly used for membrane production (PSf and PAN), but also three polymers not common in this field:
PolyLactic Acid (PLA), GlycolModified Polyethylene Terephthalate (PETG) and Acrylonitrile Butadiene Styrene (ABS).

PLA is a polymer biodegradable coming from acid lactic. It is extracted from 100% renewable resources rich in amides (corn, sugar cane and beet) and its qualities are equivalents or, sometimes, betters than many plastics becoming from crude oil. In addition to be a biodegradable polymer it is also a biocompatible and bioabsorbable one, so it can be assimilated from a biologic system (i.e. human body). Therefore it is a type of polymer introduced in membrane production especially in biomedical applications [21, 22]. Mechanics characteristics, bioadsorption and degradation velocity of PLA can be controlled by modifying chemical composition, molecular weight and crystalline structure [23]. Studies about ABS membranes can be only found in gas permeation and it is reported to have good filtration and mechanical characteristics [24, 25].

PLA, PETG and ABS are generally used in the packaging industry. Although high technical materials need to be used in common membrane application, it is not necessary in this purpose, because large particles in aqueous media are to be concentrated, at low transmembrane pressures. To allow microalgae treatment process being feasible there is a necessity to decrease the costs and here is one of the key. These three polymers are extremely cheap compared to technical polymers normally used in membrane production. And if additionally it is possible to use an environmental friendly polymer the advantages duplicate. There are three order of magnitude differences in price between technical polymers as PSf that as a cost of around 480 US$/kg (Sigma Aldrich) or PAN that has a cost of 1,850 US$/kg (Sigma Aldrich) and not technical ones as PLA that costs 2.2 US$/kg [26] or ABS with a cost of 850-1850 US $/ Ton and PETG with a cost of 800-1800 US $/ Ton.
Even if it is still more expensive than ABS and PETG, PLA polymer has the opportunity to become even cheaper, since the continuous increase of crude oil price per barrel.

Despite all the efforts did still now in membrane research, fouling problems are not completely solved yet. In this sense PLA membranes, thanks to its biodegradability, can be substituted with new ones more often without causing a big environmental impact.

2. Experimental

2.1. Materials and biological biomass

2.1.1 Microalgae

Experiments were carried out with *Phaeodactylum Tricornutum* microalgae. Algal strain was kindly provided by the Institut de Recerca i Technologia Agrolimentaries in Sant Carles de la Ràpita (Tarragona, Spain). Algae were grown in 6L round volumetric flasks with seawater (37 g·L⁻¹ salinity) filtered through 25, 10, 5 and 1 μm pore size filters (polyKLEAN, MICRO-KLEAN, 3M/ Cuno), UV sterilized and enriched with commercial fertilizer (Walne’s medium plus vitamins B2 and B12). Cultures were kept at 25°C (±2), aerated and illuminated (16:8 light: dark cycle) with daylight fluorescents which gave an irradiance of ca. 200 μmol photon·m⁻²·s⁻¹ at the flask surface.

First studies showed that there was a great dependence of duration of the experiment and the microalgae concentration and especially species [8]. For this reason, it was decided to repeat all experiments with constant microalgae concentration and only one specie. For all experiments performed, cultures were harvested in stationary phase and the concentration was measured with a microscope Carle Zeiss AxioScope A1 by hemocytometer and it resulted equal to 21 ± 2.6·10⁶ cell/ml (mean and standard deviation, as in the entire article).
2.1.2 Membranes

Commercial ceramic, commercial polymeric and own produced polymeric membranes were investigated in this work. Previous studies showed that microfiltration is the best range for this microalgae specie [8, 14]. Therefore ceramic membranes (ZrO₂/TiO₂) with membrane cut-off equal to 1.4 microns were purchased from TAMI Industry and polymeric membranes of different cut-offs (1.2, 3.0, 5.0 and 8.0 micrometers) were purchased from GE OSMONICS.

For membrane synthesis PSf (Mw=35,000), PAN (Mw=150,000) and Pluronic® F127 were purchased from Sigma-Aldrich. N,N-dimethylformamide, DMF (99.9%, Multisolvent®), Dimetilacetamida, DMA (99.5%) and 1-Methyl-2-pyrrolidone, NMP (99.5%) were purchased from Scharlab. PLA 2002 D was kindly donated from AIMPLAS.

ABS copolymer Terluran® GP-22 from BASF was employed with a density of 1.04 g/cm³, processing temperatures between 220 and 260 °C and a Tensile Strength at Yield of 45 MPa. PETG resin SKYGREEN K2012 manufactured by SK Chemicals with a density of 1.27 g/cm³, a Glass Transition Temperature of 80°C and Tensile Strength at Yield of 50MPa.

2.2. Methods

2.2.1 Membrane synthesis

Synthesized polymeric membranes were obtained by phase inversion with immersion precipitation method in a coagulation bath of 100% demineralised water. This is a classic technique to prepare selective films, well known and described in the bibliography [27]. The polymer was dissolved in the solvent, by stirring it for 24 hours at room temperature. Then the solution was deposited onto a glass plate using a casting knife. The gap thickness of the casting knife could be adjusted by using the incorporated a micrometer. To allow proper
comparison between the commercial and synthesised membranes, the last ones were produced with a similar thickness of 100 micrometers than commercial ones. In consequence, the casting knife was adjusted to 300, 250, 200, 200 and 150 micrometers for PLA, PSf, PAN, PETG and ABS membranes respectively. The knife was pushed over the glass thanks with an automatic film applicator at constant velocity rate of 110 mm/s (BYK-Gardner Automatic Film Applicator L). Phase inversion occurred after immersing the polymeric solution (attached to the glass) in a precipitation bath. After synthesizing the membrane the final thickness was measured by the digimatic micrometer IP65 from Mitutoyo Corporation. Polymeric membranes from different casting solutions with several polymers were synthesized. Below the different polymer-solvent systems are listed for the ternary groups studied: PSf-DMF, PAN-NMP, PLA-DMF, PETG-DMA and ABS-DMA. Demineralised water was always used as non solvent in the ternary systems. The polymer concentration was 10 wt % in all cases except for ABS that was 25 wt % to guarantee total microalgae rejection. Tests were performed also with the additive Pluronic® F127 (3 wt %) in the solution of PSf-DMF.

2.2.2 Membrane morphological characterization

The Morphology of the membranes was investigated by using the Scanning Electron Microscopy (SEM) and an Environmental Scanning Electron Microscopy (ESEM). The SEM used was a JEOL JSM-6400 Scanning Microscopy Series, with a working voltage of 15 kV. The ESEM used was a FEI Quanta 600, with a voltage between 15 and 20 kV and with low vacuum pressure, since the samples are not conductive and no sputtering was applied.
2.2.3 Membrane contact angle

Contact angles were determined by sessile drop technique using the automatic video-based analysis system OCA 35 (Dataphysics). Measurements were carried-out dispensing 2 µL drops on the surface of the membranes, absorption was recorded and the measurement was taken at 31-32 seconds after the drop contacted the surface (time at which the steady state angles were typically reached). More than six measurements were performed for each membrane.

2.2.4 Membrane permeability

Membrane permeability values were obtained by measuring the mass flow rate collected in a tank over a scale and recorded by a computer. In order to know membrane irreversible fouling resistance and its recovery, water permeabilities were measured before and after all experiments. Furthermore the volumetric flux reduction was calculated (VFR) in order to evaluate the membrane fouling. The system was washed with water after each microalgal biomass permeability recording. Again, the water permeability was measured to determine the irreversible fouling resistance. Permeability values are reported at the steady state time, generally observed after two hours or more.

2.2.5 Membrane mechanical characterization

The mechanical properties from commercial and synthesised membranes were evaluated through uniaxial tensile tests following the procedure suggested by the ISO 527 standard. Because of the membrane dimensions, small test specimens were cut using an ISO 527 type 1BA tensile cutting die. The gage length and the width of narrow portion were 25 and 5 mm respectively. The thickness of the membranes was measured using a coating thickness meter MEGA-CHECK 5F-ST to avoid damages on the specimens.
The samples were tested in a universal testing machine (Galdabini Sun 2500) equipped with a 1 kN load cell and data processing settings. The tests were performed at a crosshead rate of 1 mm/min and at room temperature (23 ± 2°C). Young’s modulus (E) and yield at break (σb) were obtained from the engineering stress versus strain curves, and the elastic deformation was measured using a video extensometer (Mintron OS-65D). When the curves showed a diffusive yielding, the Considère’s criterion was applied [28].

2.3 Equipment

Experiments were carried-out using a setup containing two commercial tangential cross-flow membrane modules (Figure 1). One for polymeric membranes (SEPA CFII, GE Osmonics) and another for ceramic ones (ISDRAM TAMI Industries). By using three-way valves, the module used could be chosen. Trans-membrane pressure (TMP) was set to 1 bar and controlled by using a TESCOM back-pressure. Broth temperature was also set to 21 ºC and controlled by using a Huber, K6-cc-NR equipment. Recalculating flow rate was maintained at 55 ± 3 L/h in all experiments. Permeate was collected in a tank located over a scale that was connected to a computer in order to calculate the actual mass flow rate in a one second frequency.

3. Results and discussions

3.1. Membrane characterization

3.1.1. Contact angle

Contact angle measurements showed a hydrophilic behaviour for most of the membranes analyzed, except for the PSf and ABS synthesized ones (Table 1). Between all, the most hydrophilic membranes were the commercial ones. Between the synthesized membranes PAN
and PLA showed the most hydrophilic behaviour. Under our knowledge, PLA membranes contact angles are reported for the first time. Pluronic® F127 gave more hydrophilicity to the PSf membranes, which caused a permeability increase, as it is reported in the following sections.

3.1.2. Morphology. Scanning electron microscopy micrographs

SEM micrographs of commercial ceramic membrane (Figure 2 a, b) showed a thin selective layer (ca. 0.9 µm) as opposed to a thick non-selective one (ca. 2.5 mm). This structure guarantees a proper selectivity and higher strength. On the other hand, PSf commercial membranes showed a symmetric structure (Figure 2 c) really similar than other PSf membranes with different cut-off.

PSf, PLA, ABS and PAN membranes (Figure 2 d, e, f, g and h) showed similar structures, possessing long channels in the proximity of the selective layer and macro voids in the proximity of the micro-porous side. The achievement of similar structures was important for the comparison of the membranes permeabilities, as well as their thickness. PAN membrane (Figure 2 d) showed a smooth texture that, among other characteristics, could be used to explain its higher permeability, as explained next.

Furthermore, it can be observed that Pluronic® F127 is completely integrated to PSf membrane matrix. Moreover, this additive gives more homogeneity to the structure, without the presence of a thin layer in the selective side (Figure 2 e, f).

3.1.3 Mechanical properties

Because of their biodegradability, mechanical properties of PLA membranes were evaluated and compared with commercial ones. Moreover, additional tensile tests were conducted on the PLA membranes after five hours of filtration. The mechanical parameters are listed in Table 4.
The engineering stress versus strain curves obtained from tensile tests of PLA and commercial membranes are shown in Figure 4. The curves showed similar behaviour with a diffuse yielding and slight strain hardening, which indicates that the plastic region is not absolutely flat, before rupture. Physically, the specimens did not present traces of plastic deformation mechanisms like necking, shear bandings or whitening. Additionally, PETG membrane was also tested showing a distinct mechanical response because it broke at low values of strain (approximately 3%, Table 4), hence the PETG was not possible to compare.

The mechanical parameters ie Young’s modulus (E), yield strength (σ_y), tensile and elongation at break (σ_b and ε_b respectively) presented in Table 4 indicate the commercial membrane has values on Young’s modulus and strength lower than the PLA membrane. Furthermore, the filtration process applied to the synthetized membrane affected some mechanical properties of the PLA because the stiffness and the strength were notoriously reduced without significant variations on ductility. The previous could be attributed to some physical damages on the membrane structure, probably caused by the water molecules during the filtration process.

3.2 Filtrations experiments

3.2.1 Commercial membranes

Filtration performance of commercial ceramic and commercial polymeric membranes were studied.

Ceramic membranes showed a typical permeability curve fall (Figure 3), with high permeability in water filtration (1275 ± 356 L/m²/h/bar). It decreased two orders of magnitude when microalgae were filtrated (21 ± 3 L/m²/h/bar). Several commercial PSf membranes with different molecular weight cut-off were tested, starting from 1.2 microns (close to the ceramic
Permeability studies performed with water before each microalgae experiments showed the same values for all of them ($4100 \pm 180$ L/h/m$^2$/bar) and it resulted higher than ceramic one, but as for the ceramic membranes microalgae permeability decreased two orders of magnitude when microalgae were filtrated. The plateau (steady state) value was reached in all cases and a slight augmentation of permeability was observed with the increase of pore size (Table 2), getting closer to ceramic value (Figure 3).

In both cases, for ceramic and for PSf commercial membranes the permeability fall was important with VFR values of 98% and 99% respectively.

3.2.2 Synthesized membranes

After finding the commercial polymeric membrane that better approached ceramic efficiency, it was investigated the possibility to use a material cheaper than PSf. First tests were carried out to reproduce PSf membrane performances. Several polymer concentrations were experimented (20%, 15%, 12% and 10%) till to obtain the maximum permeability within total rejection, with 10 wt %. Permeability results showed low values both with water and with microalgae (Table 3). Commercial membranes usually contain some additive to improve their performances. For this reason, Pluronic® F127 was added to the ternary solution and, even if the addition was modest (3 wt %) the increment of permeability values were important. Water permeability was one order of magnitude higher than PSf membranes (Table 3) and when microalgae were filtrated permeabilities increased till to reach
commercial ones, with $11 \pm 0.7$ L/h/m$^2$/bar (Figure 3). Its VFR one of the best resulting equal to 86%.

Another well-known polymer in membrane technology is PAN, which was chosen for its good characteristics (specially hydrophilicity) and for the possibility to be turned biodegradable [29]. It achieved, without any additive, high permeability values in water filtration and the same permeability that ceramic one when microalgae were filtrated. PAN has higher costs than PSf, but lower than ceramic, and its permeability fall resulted slighter then both of them (VFR of 95%), so it resulted a good candidate for this application with *Phaeodactylum tricornutum* microalgae.

To further decrease microalgae concentration costs other three polymers, not common in water filtration, were studied: ABS, PETG and PLA.

For these polymers preliminary experiments were carried out to screen the appropriate parameters and to determine the experimental conditions (including different kinds of suitable solvents: 1.4 dioxane, tetrahydrofuran, dichloromethane, NMP, DMF and DMA; temperature addition, till to 80º when possible, and change of polymer weight percentages). Among the solvents tested the one that showed the better results for PLA polymer was the DMF, on the other hand for PETG and ABS was the DMA. They showed a whitish colour that suggested that they were porous, which was confirmed by SEM micrographs, as follows.

PETG homogeneous membranes were of difficult achievement and they showed poor mechanical properties. More polymer weight percentage didn’t allow the pellets to dissolve and less didn’t allow the membrane formation. Furthermore, for PETG-DMA solution only 4 hours were waited after first mixing, because waiting 24 hours (as others) it became too dense and no homogeneous membrane were attain. Nevertheless, some membranes were obtained and even the poor mechanical properties filtration was possible reaching a permeability of 23
L/h/m²/bar, with a VFR of 97%. Result that deserve more investigation on the production of
these membranes.

PLA membrane gave similar permeability efficiency than PSf one, with values equal to $5 \pm 1.4$ L/h/m²/bar. On the other hand, it showed better efficiency in term of VFR (of only 29%)
and mechanical properties (as showed previously).

ABS membranes gave the most interesting results. Even if with water the permeability was
lower than ceramic ones, with microalgae it increased till to $19 \pm 1.9$ L/h/m²/bar, value really
close to the higher ones obtained with PAN and ceramic membranes (Figure 3). Its VFR was
also appreciable equal to 93%. Value that could be probably improved, as well as the
permeability, with surface modification using for example some additive, as Pluronic® F127,
or other techniques, like surface-coating [30].

4. Conclusions

A screening of various membrane materials to concentrate microalgae has been performed in
this study. From commercial and well known materials in membrane technology (ceramic and
PSf) to not common polymers as PLA or ABS.

Results confirmed good performances of ceramic membranes ($21 \pm 2.9$ L/h/m²/bar). The
study of commercial PSf membranes with different pore sizes showed an increment of
permeability from $10.5 \pm 0.7$ L/h/m²/bar of the 1.2 microns membrane to $14.7 \pm 1.2$
L/h/m²/bar of the 8.0 microns membrane. An important increment of permeability was
obtained as well with the addition of Pluronic® F127 in the PSf membranes synthesized in the
laboratory, reaching $11 \pm 0.7$ L/h/m²/bar versus the $2 \pm 2.0$ L/h/m²/bar of PSf one. Interesting
permeability values were obtained using PAN polymer of $22 \pm 0.6$ L/h/m²/bar, reaching
ceramic performances.
Regarding the not common polymers in this field, PLA membranes gave better permeability results (5± 1.4 L/h/m\(^2\)/bar) and mechanical properties than PSf synthesized membranes. Moreover they are good candidates in term of costs and they have lower VFR (28% versus 60% of PSf membranes).

Best results were obtained with the ABS membrane that achieved a permeability of 19 ± 1.9 L/h/m\(^2\)/bar, really close to the best ones (of ceramic and PAN membranes).

The results of this study shows the potentiality of introduce cheap materials (not common in membrane technology) in membrane dewatering. Further experiments should be performed to assess whether they can be used alone or blended. In addition, other membrane applications may use these kind of materials.

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References


Figure captions

Figure 1. Experimental set-up.
Figure 2. Scanning electron microscopy micrographs of membranes cross sections: a) Ceramic membrane; b) Ceramic membrane selective layer; c) Commercial PSf membrane; d) PAN-NMP membrane; e) PSf membrane; f) PSf-Pluronic® F127 membrane; g) PLA-DMF membrane; h) ABS-DMA membrane; i) PETG-DMA membrane.
Figure 3. Microalgae permeability fall of ceramic commercial membrane.
Figure 4. Representative curves $\sigma - \varepsilon$ of PLA, PETG and commercial PSf membranes.

Table captions

Table 1. Membrane contact angle measurement results.

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Contact angle ($^\circ$)</th>
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<tr>
<td>Commercial membranes</td>
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<tr>
<td>Ceramic</td>
<td>38 ± 3</td>
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<td>Polymeric</td>
<td>60 ± 1</td>
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<td>Polysulfone</td>
<td>92 ± 3</td>
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<td>Polymethacrylonitrile</td>
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<td>Acrylonitrile butadiene styrene</td>
<td>94 ± 3</td>
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<td>Synthesized membranes</td>
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Table 2. Permeability results of commercial membranes.

<table>
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<th>Membrane</th>
<th>$E$ (MPa)</th>
<th>$\sigma_l$ (MPa)</th>
<th>$\sigma_s$ (MPa)</th>
<th>$e_b$ (%)</th>
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<td>Commercial polysulfone</td>
<td>0.88 ± 0.08</td>
<td>2.06 ± 0.04</td>
<td>3.43 ± 0.025</td>
<td>21.60 ± 5.90</td>
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<td>PETG</td>
<td>2.33 ± 0.31</td>
<td>2.44 ± 0.03</td>
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<td>PLA before filtration</td>
<td>2.69 ± 0.92</td>
<td>4.23 ± 0.26</td>
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<td>18.35 ± 4.53</td>
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<td>PLA after filtration</td>
<td>1.76 ± 0.27</td>
<td>4.25 ± 0.24</td>
<td>4.53 ± 0.065</td>
<td>22.95 ± 4.63</td>
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Table 3. Permeability results of synthesized membranes.

<table>
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<tr>
<th>Membrane</th>
<th>Membrane pore size (µm)</th>
<th>Water permeability (L/h/m²/bar)</th>
<th>Microalgae permeability (L/h/m²/bar)</th>
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<tr>
<td>Ceramic</td>
<td>1.4</td>
<td>1275 ± 356</td>
<td>21 ± 3</td>
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<td>Polysulfone</td>
<td>1.2</td>
<td>4025 ± 126</td>
<td>10 ± 1</td>
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<td>3</td>
<td>4050 ± 140</td>
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<td>5</td>
<td>4182 ± 212</td>
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<tr>
<td></td>
<td>8</td>
<td>4250 ± 71</td>
<td>15 ± 1</td>
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Table 4. Mechanical parameters.

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Water permeability (L/h/m²/bar)</th>
<th>Microalgae permeability (L/h/m²/bar)</th>
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<tr>
<td>Polysulfone</td>
<td>5 ± 2</td>
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<td>Polysulfone–Pluronic® F127</td>
<td>80 ± 28</td>
<td>11 ± 1</td>
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<td>Polyacrylonitrile</td>
<td>400 ± 61</td>
<td>22 ± 1</td>
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<td>Acrylonitrile butadiene styrene</td>
<td>260 ± 173</td>
<td>19 ± 2</td>
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<td>Polylactic acid</td>
<td>7 ± 0</td>
<td>5 ± 1</td>
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