Comparison of external and submerged membranes used in Anaerobic Membrane Bioreactors. Fouling related issues and biological activity

Martinez[a], R.; Ruiz[a], M.O; Ramos[a], C.; Cámara[b], J.M.; Diez[a,*], V.

(a) Department of Biotechnology and Food Science, Chemical Engineering Division. University of Burgos, Plaza Misael Bañuelos, 09001 Burgos, Spain.

(b) Department of Electromechanical Engineering, Electronics Technology Division. University of Burgos, Avda. Cantabria s/n, 09006 Burgos, Spain.

ABSTRACT

Anaerobic sludge filtration tests were performed with two experimental set-ups equipped with either submerged hollow-fiber membranes or external tubular membranes operated with low crossflow velocity and gas sparging in a gas-lift mode. Particle size distributions, extracellular polymeric substance concentrations, methanogenic activity, reversible and irreversible fouling rates, and chemical cleaning efficiency were all studied and compared. An increase in the percentage of smaller particles and a decrease in the concentration of volatile suspended solids in the external membrane set-up were observed. Both soluble protein and soluble polysaccharide concentrations were also higher in the external filtration. Operating at 12 – 15 L/m^2·h for 7 days, the total resistance of the external membrane reached 9.3×10^{12} m^{-1}, while the resistance of submerged membrane reached 1.1×10^{12} m^{-1} despite operating at 15 – 25 L/m^2·h for 12 days. Finally, the type of fouling and the efficiency of chemical cleaning also differed in both configurations.

Keywords: Submerged filtration; External filtration; Anaerobic membrane bioreactor; Gas-lift mode; Shear stress; Fouling rate.

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[*]*Corresponding author. Tel.: +34 947497017 E-mail address: vdiezb@ubu.es

Department of Biotechnology and Food Science, Chemical Engineering Division. University of Burgos, Plaza Misael Bañuelos, 09001 Burgos, Spain

1 Introduction

A membrane bioreactor is an innovative and efficient technology used for wastewater treatment combining biological treatment and direct sludge separation by membrane filtration. Membrane fouling, one of the main bottlenecks for the successful operation of a membrane bioreactor, is a complex process that is affected by wastewater characteristics, sludge concentration and properties, membrane configuration, and reactor operating conditions. Fouling appears to be more severe in anaerobic environments, due in part to the lower anaerobic sludge filterability, when compared to aerobic sludge as a consequence of low sludge flocculation and an increase in the supernatant colloidal fraction [1,2]. Higher fouling propensity of the anaerobic sludge has led to low sustainable fluxes in anaerobic membrane bioreactors (AnMBR) even with high biogas sparging intensities [3].

Hydrodynamic conditions are strongly influenced by the mode in which anaerobic reactor and membrane are combined. Two types of membrane arrangements can be distinguished: external or side-stream AnMBR and internal or submerged AnMBR. The external AnMBRs (eAnMBR) commonly apply tubular membranes that operate with a high crossflow velocity for disrupting the formation of a filtration cake on the membrane surface, while submerged AnMBR (sAnMBR) usually apply hollow-fiber membranes or flat-sheet membranes, operating in a vacuum and sparging with biogas for fouling control. A Gas-lift AnMBR (Gl-AnMBR) is a particular type of reactor that combines the two previous technologies. It applies crossflow filtration in tubular membranes, in the same way as the eAnMBR, and low-pressure filtration and gas sparging, in the same way as the sAnMBR configuration, reducing the crossflow velocity that is required in the eAnMBR [4,5]. Each membrane configuration has its own advantages and disadvantages, depending on wastewater characteristics, operational goals, and space availability for the installation. External membrane configurations have well-defined hydrodynamic conditions that permit direct reversible fouling control by adjusting the crossflow velocity, and they are easy to clean and replace [6–8]. Chemical cleaning of submerged membranes is more difficult, if done in place, as the cleaning reagent might affect the biomass, and if done out of place, then the anaerobic system might be exposed to oxygen. That disadvantage can be limited by the combination of a side-stream approach and a submerged configuration in an AnMBR where the membrane is immersed in an external filtration tank [9]. Hollow-fiber membrane modules are often used in sAnMBR, because of their lower capital costs and lower energy consumption [6,7], from 0.038 to 5.68 kWh/m^3, as against the estimated consumption of 3 to 7.3 kWh/m^3 of an external membrane configuration [1,8,10]. On the other hand, optimizing filtration conditions directly affects the positive economic balance of AnMBR technology [11].

Particle Size Distribution (PSD), Extracellular Polymeric Substances (EPS) composition and even microbial activity can be affected by biomass shear stress, which will provoke differences in the fouling behavior between external and submerged membrane processes, due to their different hydrodynamic conditions. The recirculation in the external membrane configuration, required to reach high crossflow velocities that prevent deposition on the membrane surface, causes high shear forces that can lead to a decrease in biomass particle size [12] and the subsequent release of Soluble Microbial Products (SMP) [6,13,14]. Humic substances (HS) are the principal exopolymers in the air-sparged side-stream membrane configuration, and polysaccharides (PS), and proteins (PN) are prevalent in the submerged membrane configuration [15]. The accumulation of SMP on the membrane surface is related to the irreversible membrane fouling rate [16,17] and choosing the appropriate operating conditions could help to minimize major fouling mechanisms by reducing the release of high molecular weight substances [18]. Recent studies have shown that addition of powdered activated carbon in membrane bioreactors would reduce membrane fouling, and so the operating costs [19].

Shear stress can result in decreased microbial activity, due to possible disruption of syntrophic relationships between the different groups of microorganisms, and can even induce cell disruption that lowers biogas production and decreases chemical oxygen demand (COD) removal efficiency in side-stream processes [20,21]. Ghyoot and Verstraete [22] filtered sludge from an anaerobic digestor checking that the shear stress disrupted the interactions between the different species in the anaerobic consortia, and that the negative effect on the microbial communities depended on the frequency of anaerobic sludge displacement throughout the filtration circuit, due to shear stress in its mechanical parts. However, the effect of side-stream pumping and shear on methanogenic activity is still unclear [23], as it also depends on the type of pump. Brockmann and Seyfried [24] studied the effect of different types of pumps (centrifugal pumps with rough or polished surfaces, mono-pumps, peristaltic pumps, lobular pumps, and centrifugal screw pumps) on the aerobic sludge filtration and found a significant influence of the recirculation pump type on the disintegration of sludge flocs.

The literature includes some comparative studies of the behavior of external and submerged configurations operating under the same conditions, but the results were in some cases contradictory because there were relevant differences in the characteristics of the bioreactors. Le-Clech et al. [25] compared the performance of a tubular membrane bioreactor configured both as a submerged and as a side-stream MBR. They observed a lower fouling propensity in the submerged configuration, due to the prevalent slug flow hydrodynamics regime resulting from the air-liquid flow rates that were employed. Chen et al. [26] compared the performance of external and submerged granular AnMBR configurations operating in parallel to treat municipal wastewater. For the external granular AnMBR, a hollow-fiber membrane was immersed in an external filtration tank fed with the effluent of an upflow anaerobic granular bioreactor, while in the submerged granular AnMBR, an identical hollow-fiber membrane module was directly immersed in the mixed liquor in the settling zone of the bioreactor. The submerged configuration demonstrated higher fouling propensity, higher cake layer resistance and more deposition of EPS in the cake layer, as a consequence of the deteriorated granular sludge properties with granule fragmentation and reduced granule settleability. Martin-García et al. [1] compared submerged hollow fiber and external tubular membrane AnMBR configurations, operating the latter in both pumped and gas-lift mode. The filtration performance of flocculated and granulated AnMBR treating domestic wastewater was compared. Higher critical fluxes and lower fouling rates were achieved for the granular anaerobic sludge using either submerged hollow fibers or external tubular membranes operated in pumped side-stream mode; however, the specific energy demand was significantly lower for the submerged hollow fibers membranes than for the pumped side-stream configuration.

It is difficult to compare the behavior of external and submerged membrane bioreactors when there are differences in reactor configuration, sludge characteristics, wastewater composition, and operating conditions. Likewise, few studies have compared fouling in tubular membranes and hollow-fiber membranes filtering anaerobic sludge under the same conditions, in order to assess the impact of hydrodynamic conditions imposed by each configuration. The aim of this work is therefore to provide a direct comparison of the anaerobic sludge filtration behavior with external tubular membranes and submerged hollow-fiber membranes under the same conditions: biomass concentration filtration and backwash flux and duration, and biogas sparging flow per unit of membrane area. Thus, an integrated study is performed, of the factors relating to shear stress, such as PSD, EPS and SMP concentrations, and biomass methanogenic activity, in addition to reversible and irreversible fouling rates, and the efficiency of physical and chemical cleaning processes.

2 Material and Methods

2.1 Filtration set-ups and operating conditions

Two filtration set-ups, equipped with an external tubular membrane (eM) and a submerged hollow-fiber membrane (sM), were simultaneously operated under the same conditions (Table 1). A simplified diagram of the eM and sM set-ups is shown in Fig. 1. Each of the two set-ups had a 20 L tank, 160 mm in diameter, that was loaded with sludge from an anaerobic digestor of food industries biowastes diluted with effluent from a pilot scale AnMBR, to obtain an initial total suspended solids concentration of 4 g/L.

Table 1. Membrane characteristics and operating conditions of each one.

Membrane	External	Submerged
Type	multitube	hollow fiber
Material	PVDF	PVDF
Filtration mode	in-out	out-in
Nominal pore diameter (μm)	0.04	0.04
Filtration area (m^2)	0.31	0.93
Effective membrane length (mm)	1000	692
Membrane diameter (mm)	8	2
Number of tubes	13	n/a
Cross flow velocity (m/s)	0.51	n/a
Gas flow (m^3/h)	0.3 – 0.4	1.0 – 1.2
Specific gas demand ($m^3/m^2 \cdot h$)	1.2±0.1	1.2±0.1
Superficial gas velocity (m/s)	0.15±0.02	0.015±0.001
Filtration flow (L/h)	4.65	14.0 – 18.5 – 23.0
Filtration flux ($L/m^2 \cdot h$)	15	15 – 20 – 25

n/a not applicable

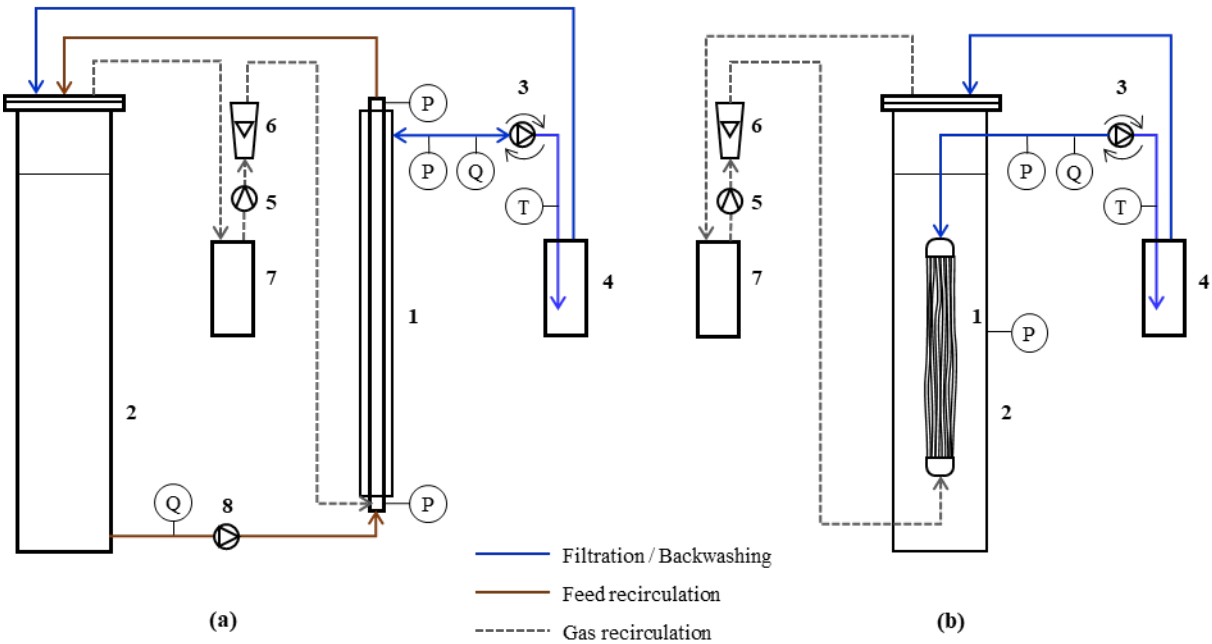


Fig. 1. Experimental set-ups: (a) external membrane set-up, and (b) submerged membrane set-up. (1.a) tubular membrane, (1.b) hollow-fiber membrane, (2) feed tank, (3) reversible filtration/backwashing pump, (4) permeate vessel, (5) biogas compressor, (6) rotameter, (7) condensate trap, (8) recirculation pump, P, Q, T pressure, flow, and temperature gauges.

In eM, Fig. 1(a), a backwashable ultrafiltration multi-tubular membrane is detailed (Berghof 63.03I8) with a surface area of 0.31 m^2, a bore size of 8 mm and a nominal pore size of 0.04 µm. It runs a recirculation pump with a low maximum rotational velocity (950 rpm) and a two-blade open impeller for wastewater with high levels of suspended particles. A frequency converter is used for crossflow control, avoiding flow control valves. The eM set-up, had a biogas recirculation circuit to operate as a gas-lift side-stream filtration system for increasing shear over the membrane surface and reducing pumping requirements for recirculation. In sM, Fig. 1(b), an ultrafiltration hollow-fiber membrane module is detailed (ZeeWeed ZW-10) with a surface area of 0.93 m^2 and a nominal pore size of 0.04 µm that is directly submerged in the filtration tank.

Reversible wear pumps (Micropump Eagle Drivef GJ-N21) were used for filtration and backwashing and membrane scouring by biogas recirculation was performed with Secoh SV50 (Kantauri) compressors in each set-up. Electronic pressure gauges (PN2069, IFM Electronics), electronic-inductive flowmeter (MIK 5NA, Kobold Mesura), and digital temperature sensors (TR2432, IFM Electronics) were applied for filtration monitoring. Ardunino-based PLCs (M-Duino 42, Industrial Shields) were programmed for flux, filtration/backwash cycle, biogas sparging, and crossflow control. A desktop application running on a PC connected to the PLCs collected the data relayed from the sensors and calculated the filtration and backwashing resistances and the fouling rates on-line.

The filtration cycle duration was 15 minutes with 30 seconds for backwash and 30 seconds for relaxation before and after the backwash, the same for both membranes. The specific gas demand per square meter of membrane area (SGD_m) was 1.2±0.1 $Nm^3/m^2 \cdot h$, also identical, apparently high for full-scale membrane modules, but not so high for lab-scale modules [27,28]. Crossflow velocity was maintained at 0.51 m/s during filtration and backwash in eM, in the order of Gl-AnMBR, between 0.3 and 1.0 m/s [29]. Backwashing flux was 20 $L/m^2 \cdot h$ for both membranes. Filtration flux was set at 15 $L/m^2 \cdot h$ over the first 7 days and was then increased up to 25 $L/m^2 \cdot h$ in the sM.

2.2 *Filtration and fouling characterization*

Filtration performance is characterized by total resistance at the beginning of the filtration, R_0, the resistance during the backwash, R_{bw}, the reversible fouling rate, $(dTMP/dt)_{rev}$, from the increase in transmembrane pressure, during each filtration cycle and irreversible fouling rate, $(dR/dt)_{irr}$, from the increase in the initial filtration resistance throughout the assay.

Transmembrane pressure, TMP (Pa), for a flux J ($m^3 \cdot m^{-2} \cdot s^{-1}$), was calculated for the external membrane as:

$$TMP = \left(\frac{P_i + P_o}{2} - P_f\right)_J - P_{J=0} \tag{1}$$

where, P_i and P_o are the pressure at the membrane inlet and outlet, respectively, P_f is the pressure in the filtration line, and $P_{J=0}$ is the previous difference for J = 0, which value depends of the relative height of the pressure gauges, neglecting the head loss in the filtration line. TMP for the submerged membrane was calculated as:

$$TMP = \left(P_t - P_f\right)_J - P_{J=0} \tag{2}$$

where, P_t is the pressure in the filtration tank.

Total resistances, R (m^{-1}), were determined according to Darcy's law

$$R = \frac{TMP}{J \cdot \mu} \tag{3}$$

where, μ is the viscosity of permeate (Pa·s) approximately represented by the viscosity of tap water, dependent on its temperature , T (°C), in accordance with the correlation [30]:

$$\mu = \frac{479 \cdot 10^{-3}}{(T + 42.5)^{1.5}} \tag{4}$$

From the irreversible fouling rate $(dR/dt)_{irr}$, the irreversible increase in resistance over the filtered volume per unit of area, $(dR/dV)_{irr}$ (m^{-2}), was determined as:

$$\left(\frac{dR}{dV}\right)_{irr} = \frac{(dR/dt)_{irr}}{J} \tag{5}$$

where, V is the filtrated volume per unit of membrane surface area ($m^3\ m^{-2}$).

2.3 Analytical methods

Proteins (PN), Polysaccharides (PS), and Humic Substances (HS) were analyzed in the mixed liquor and in the permeate. Soluble Microbial Products (SMP) in the sludge were determined from the supernatant obtained by centrifugation at 4500 rpm. From the solid fraction, loosely bound extracellular polymeric substances (LB-EPS) were determined after water extraction at room temperature.

PN were determined using the modified Bradford method [31], PS were measured using the method suggested by Dubois [32], and HS were determined using Lowry's modified method [33] and a Hitachi U-2000 UV/vis spectrophotometer. Bovine serum albumin (VWR Prolabo Chemicals), humic acid (SIGMA-ALDRICH), and D(+)-anhydrous glucose (VWR Prolabo Chemicals) were used as the calibration standards. Each sample was analyzed in triplicate.

Anaerobic activity tests were performed in glass bottles of 2.1L. 400 ml of initial anaerobic sludge were fed with pet food at an S/X ratio of 1 g COD/g VSS Bicarbonate, macro and micronutrients were also added according to Angelidaki et al. [34]. After removing O_2 with N_2, the samples were incubated at 35°C in a temperature controlled room, and softly agitated in a Wheaton roller culture apparatus. Biogas production was monitored by a digital pressure sensor throughout the assay. A blank sample with the initial anaerobic sludge was prepared. The assays were performed in duplicate and the biogas production of blank assays without substrate was subtracted from the fed assay. The methane concentration was determined with a multi-gas measuring device, mobile MULTITEC 545 (Sewerin).

Analyses of Total Suspended Solids (TSS) and Volatile Suspended Solids (VSS) were completed in accordance with the protocols described in the Standard Methods for the Examination of Water and Wastewater [35]. PSD was obtained using a Mastersizer 2000 (Malvern Instruments) coupled to a Hydro 2000SM with a detection range of 0.02–2000 μm.

2.4 *Membrane cleaning*

The cleaning of both membranes was performed sequentially under increasingly strong conditions. The cleaning protocol was divided into 4 steps: physical rinsing with tap water, chemical rinsing with diluted sodium hypochlorite, 100 mg/L of NaClO, oxidizing cleaning with 500 mg/L of NaClO, and acid cleaning with 100 mg/L of oxalic acid. The length of each step was 2 hours during which filtration and backwash resistances were measured. After each cleaning stage the membranes were rinsed with tap water to remove detached materials. The efficiency of each cleaning step was evaluated on the basis of the total and backwashing resistances at the end of the stage, before the removal of the detached solids with tap water [36].

3 Results and Discussion

3.1 *Particle size distribution*

Fig. 2 shows the PSD of the initial anaerobic sludge, and the PSD of the samples taken on days 1 and 7 from the eM tank (a), and from the sM tank (b). In the case of eM, the PSD of a sample taken from the outlet of the pump after 1hour of operation is also represented.

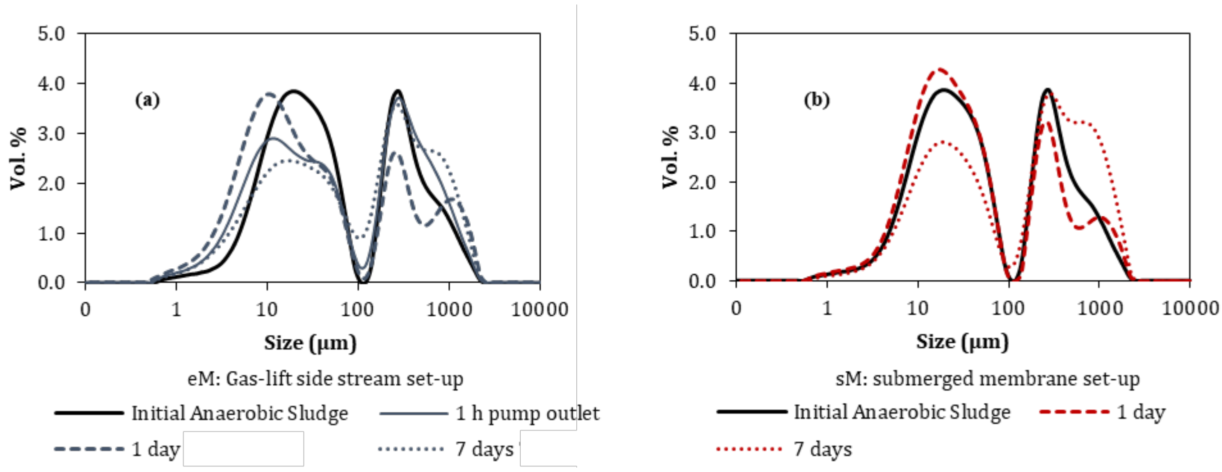


Fig. 2. Particle size distribution of the initial anaerobic sludge and (a) samples taken from the gas-lift side-stream membrane set-up and (b) from the submerged membrane tank.

The PSD of the initial anaerobic sludge shows a bimodal curve with central peaks at 20 µm and 120 µm. This type of distribution is maintained throughout the whole experience in both membrane set-ups with decreasing percentages of the larger size particles in favor of the smaller ones.

The difference in the concentration of particles of around 100 μm between eM and sM is noticeable. 100 μm particles were neither found in the initial anaerobic sludge nor in the samples taken from the sM tank, and the presence of those particles in the eM tank was clear evidence of the breakup of the larger particles when using the side-stream configuration. However, with regard to membrane fouling, the smallest particles had a higher contribution in the fouling process [12,37–40], because their back-transport from the membrane is less effective and they form a less porous cake [41], and it is therefore of greater importance to highlight the different size distributions of the particles smaller than 100 μm.

With the aim of specifically determining the behavior of the particles with the highest impact on the fouling process, particles larger than 100 μm were excluded in the PSD analysis. To that end, the cumulative volume of particles under d_p in the 0-100 μm interval, were recalculated as:

$$Vol.\%\ under\ d_{p[0-100]} = \frac{\sum_0^{d_p}(Vol.\%)_d}{\sum_0^{100}(Vol.\%)_d}\cdot 100 \quad (6)$$

where, $\sum_0^{d_p}(Vol.\%)_d$ is the cumulative volume of particles under d_p and $\sum_0^{100}(Vol.\%)_d$ is the cumulative volume of particles under 100 μm.

Fig. 3 presents the cumulative volume curves of the initial anaerobic sludge, and samples taken on the days 1 and 7 in both the eM and the sM tanks. It is worth noting that the cumulative curve of particles under 100 μm in the sM tank after 7 days practically matched the initial value. However, the cumulative curve of the sludge was notably altered in the eM tank. A significant increase in the fine particles in the eM tank was observed; for instance, the percentage of particles smaller than 10 μm increased from 25.1% to 34.6% within only 24 hours and to 44.8% after 7 days, while the increase in the sM tank (negligible on the first day, not represented) reached only 31.5% on day 7. These results indicated that the hydrodynamic conditions in the eM set-up had promoted a sharp increase in the breakup rate of the anaerobic biomass over time that was barely observable in the sM set-up. The breakage of particles has already been reported as a consequence of shear stress [6,13,21,42,43], which is higher in the external AnMBRs, because the sludge recirculation required to maintain an adequate crossflow velocity. Jeison et al. [20] highlighted the need to establish the recirculation rate under which surface shear would be enhanced, but avoiding exposure of the sludge to shear stress levels that could reduce the size of the flocs and even induce cell disruption.

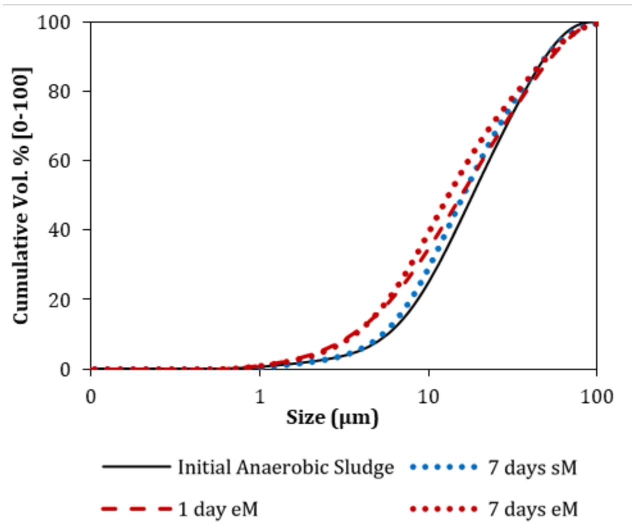


Fig. 3. Cumulative volume of particles under 100 µm of initial anaerobic sludge, after 1 day in the external membrane set-up and after 7 days in both set-ups.

3.2 *EPS development*

LB-EPS and SMP were analyzed from initial anaerobic sludge and samples from the filtration set-ups on day 7. PN, PS, and HS were also analyzed in the permeate samples after 1 hour and on day 7. The membrane rejection of SMP was calculated from the concentration of SMP in the initial sludge and in the permeate samples and is summarized in Table 2.

Table 2. PN, PS, and HS content in LB-EPS, SMP, SMP in permeate and percentage of retention of SMP in eM and sM at day 0, after 1 hour and after 7 days.

Set-up	Parameter	Time (days)	LB-EPS (mg/g VSS)	SMP (mg/L)	$SMP_{PERMEATE}$ (mg/L)	Retention (%)
eM	PN	Initial sludge	5.62 ± 0.03	25.0 ± 1.0		
		1 hour			10.6 ± 0.8	57 ± 4
		7 days	6.62 ± 0.09	42.4 ± 1.0	4.9 ± 1.3	88 ± 3
	PS	Initial sludge	5.00 ± 2.51	31.1 ± 0.9		
		1 hour			23.7 ± 0.4	24 ± 3
		7 days	5.53 ± 0.12	59.5 ± 3.1	30.3 ± 0.4	49 ± 2
	HS	Initial sludge	18.88 ± 0.53	183.0 ± 2.0		
		1 hour			63.1 ± 2.4	66 ± 1
		7 days	2.00 ± 0.21	151.1 ± 13.6	88.6 ± 8.9	41 ± 10
sM	PN	Initial sludge	5.62 ± 0.03	25.0 ± 1.0		
		1 hour			12.5 ± 0.1	50 ± 2
		7 days	6.64 ± 0.03	35.4 ± 1.1	10.6 ± 0.5	70 ± 2
	PS	Initial sludge	5.00 ± 2.51	31.1 ± 0.9		
		1 hour			23.1 ± 0.2	26 ± 2
		7 days	6.26 ± 0.26	43.3 ± 1.4	23.8 ± 4.8	45 ± 9
	HS	Initial sludge	18.88 ± 0.53	183.0 ± 2.0		
		1 hour			77.3 ± 3.6	58 ± 2
		7 days	1.79 ± 0.21	145.3 ± 9.3	105.9 ± 11.3	27 ± 11

The LB-EPS behavior was similar in both the eM and the sM set-ups, meaning that no significant difference in LB-EPS behavior were induced by differences in the hydrodynamic conditions. Loosely bound PN and PS remained constant or slightly increased, however loosely bound HS significantly decreased by 89% in the eM set-up and by 91% in the sM, due to its release in the liquid phase, which also increased the levels of HS in the permeate.

With regard to SMP in the sludge, soluble PN and PS increased significantly in both set-ups on day 7. However, the prevalent SMP, HS, decreased from 183.0 mg/L in the initial anaerobic sludge to 151.1 mg/L and 145.3 mg/L in the eM and sM, respectively. The concentration of the three compounds was higher in the eM than in the sM set-up, with a more significant difference for PN than for PS, which brings new evidence of the difference between HS versus PN and PS behavior. The higher increase in soluble PN and PS in the eM sludge indicated that the shear stress imposed on the biomass could have caused the release of microbial polymeric substances. Yu et al. [44] observed an increase of soluble COD in the sludge of an AnMBR as a consequence of SMP release, due to shear forces caused by a crossflow velocity of 1.0 m/s. Xiong et al. [45] detected a significant increase in PN and PS concentrations in the soluble EPS of an AnMBR fed with synthetic wastewater, however they attributed it to accumulation in the retentate of higher molecular weight SMP produced in the microbial process.

It was found that PN was always higher than PS rejection, which is probably explained by the higher propensity of PN than PS to attach to the membrane surface, due to electrostatic interactions with the polymeric membrane [46]. PN and PS rejection rates were higher over time. The increased retention of PN and PS was also observed by Chen et al. [26] who assumed that the TMP increase, due to the fouling, enhanced SMP retention by the cake layer, which might explain why SMP rejection was higher in the eM.

The HS in the permeate showed an unexpected behavior over time. The HS rejection decreased on day 7, from 66% to 41% in the eM set-up and from 58% to 27% in the sM set-up. Two phenomena could explain these results, on the one hand the above-mentioned loosely-bound HS release, and on the other hand the likely breakdown of the HS complex molecules that made possible its permeation.

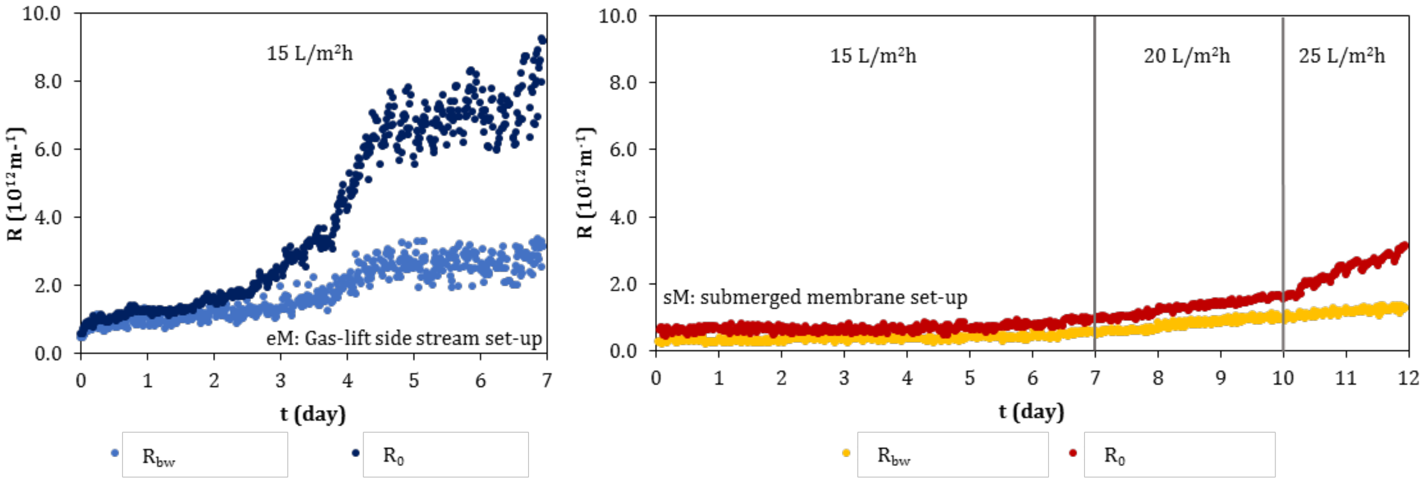
Chen et al. [26] on the contrary, when comparing submerged and external granular AnMBR, reported fewer microbial products, better biomass granule quality, and less fouling propensity in the external AnMBR. However, it should be considered that these authors compared a membrane submerged in the mixed liquor with an external membrane fed with the biomass-free effluent of an upflow anaerobic granular bioreactor.

3.3 Membrane fouling

Fouling development was very different in the eM and the sM filtration set-ups. Under the same filtration conditions, fouling of the gas-lift side-stream set-up was faster and higher than that of the submerged membrane set-up. Fig. 4 shows the behavior of filtration and backwashing resistance of (a) the eM and (b) the sM. The eM filtration resistance quickly increased reaching a value of $9.3\pm0.6\times10^{12}$ m^{-1} in just 7 days. The most pronounced fouling appeared on days 3 and 4, in which the TMP exceeded 300 mbar. At that point, the filtration pump flow control system was unable to maintain the desired flux rate, which decreased throughout each cycle from 15 L/m^2·h to values around 12 L/m^2·h, reducing the fouling rate and increasing the resistance data dispersion. The fouling rate of sM under the same conditions was notably lower than the eM, reaching, over the same period, a resistance of 1.1×10^{12} m^{-1}, so that between day 7 and 10 the flow rose to 20 L/m^2·h, thereafter increasing to 25 L/m^2·h. Under those conditions, the fouling rate was significantly higher and the filtration resistance reached $3.6\pm0.2\times10^{12}$ m^{-1} on day 12, clearly lower than the eM resistance.

The irreversible increase in resistance over the filtered volume per unit of area, $(dR/dV)_{irr}$, rose rapidly from 1.4×10^{12} m^{-2} to 11.5×10^{12} m^{-2} during the first four days of eM operation. During the same period, the irreversible fouling rate in the sM was held between 0.3 and 0.8 $\times10^{12}$ m^{-2}. The $(dR/dV)_{irr}$ in the sM increased with the flux, however it remained below 1.2 $\times10^{12}$ m^{-2} at 25 L/m^2·h.

The development of the backwash resistance, R_{bw}, related to irreversible fouling by pore-blocking [47], followed the same pattern as for filtration resistance. Starting with similar backwashing resistances, 0.5×10^{12} m^{-1} and 0.3×10^{12} m^{-1} for the eM and the sM, respectively, the increase in R_{bw} for eM was notably higher than for sM, up to $3.3\pm0.4\times10^{12}$ m^{-1} for the first one and only $1.4\pm0.1\times10^{12}$ m^{-1} for the second one, despite higher operating fluxes.



(a) (b)

Fig. 4. Filtration and backwash resistance behavior: (a) external; and, (b) submerged membrane.

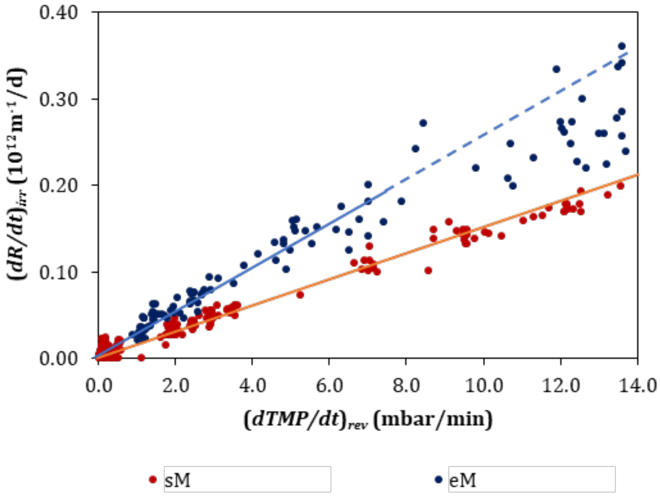


Fig. 5. Development of irreversible fouling, dR/dt, with regard to reversible fouling, dTMP/dt, in the external and in the submerged membranes.

Fig. 5 shows the irreversible fouling rate $(dR/dt)_{irr}$ versus the reversible fouling rate $(dTMP/dt)_{rev}$. A quasilinear relationship between both fouling rates can be observed, with proportionality constants of 0.027 and 0.014×10^{12} $m^{-1}\cdot min/(mbar\cdot d)$, for both the eM and the sM, respectively. The eM worked in a stable manner with a reversible fouling rate of up to 7 mbar/min. The flow control problems that took place from day 4 explain the data dispersion for the highest reversible fouling rate. The sM operated without any difficulty with a reversible fouling rate of up to 14 mbar/min, reaching an irreversible fouling rate of 0.2×10^{12} m^{-1} d^{-1}.

Ersahin et al. [21] using dynamic membrane AnMBRs equipped with flat sheet membrane modules operating in gas-lift mode, observed that filtration resistance in the eAnMBR was lower than in the sAnMBR. Nevertheless, as the authors indicated, the observed differences could be due to the gas diffusers in the sAnMBR that were placed inside the mixed liquor, which implied a lower efficiency of the gas bubbles in the control of the dynamic membrane thickness.

Stricot et. al. [48] studied the impact of hydrodynamic stress on sludge in two different side-stream MBRs, demonstrating that high shear stresses associated with a crossflow velocity induced significant modifications of the sludge, floc breakage, and release of polymeric substances, which increased its fouling potential drastically from 5×10^{14} m^{-2} to 50×10^{14} m^{-2}.

Choo and Lee [39], during a continuous long-term experiment with an anaerobic bioreactor coupled to a plate and frame membrane by a gear-type pump, also observed that the permeation flux dropped sharply from 100 L/m^2·h to 10 L/m^2·h within less than 20 days.

3.4 *Physical and chemical cleaning*

The cleaning efficiency as the membrane fouling behavior could be influenced by operating conditions [49]. A comparative study of physical and chemical cleaning processes on both membrane set-ups, was performed, determining the efficiency of a series of cleaning steps: rinsing with tap water, and chemical cleaning with sodium hypochlorite solutions of 100 and 500 mg/L, and an oxalic acid of 100 mg $H_2C_2O_4$/L. Table 3 summarizes the filtration and backwash resistances values at the end of each cleaning step for both membranes.

Table 3. Initial resistance, R_0, and backwash resistance, R_{bw}, at the end of each cleaning step for both membranes.

Cleaning step	**eM**		**sM**	
	R_0 (10^{12} m^{-1})	**R_{bw}** (10^{12} m^{-1})	**R_0** (10^{12} m^{-1})	**R_{bw}** (10^{12} m^{-1})
Initial conditions	7.4	3.3	3.6	1.3
Tap water	6.9	3.3	1.4	0.9
NaClO (100 mg/L)	2.7	1.9	1.1	0.6
NaClO (500 mg/L)	1.6	1.6	0.6	0.6
$H_2C_2O_4$ (100 mg/L)	0.8	0.5	0.6	0.3

Given that, as stated in the preceding section, the fouling of the eM was worse than the fouling of the sM, the resistances of both membranes were not initially equal. It is worth highlighting the different resistances to backwash, which were 3.3 and 1.3×10^{12} m^{-1}, for the eM and the sM, respectively, suggesting that the effectiveness of the cleaning stages might also foreseeably be different, because of their relation with the internal fouling of the membrane [47].

Rinsing the eM with tap water showed a very low efficiency, under 7% of the filtration resistance and without any effect on backwash resistance. On the contrary, rinsing the sM with tap water reduced its resistance to around 60% in the filtering stage and to 36% in the backwash stage.

This difference might be related to the degree of consolidation of the fouling layer [43]. The TMP of almost 300 mbar that was reached in the eM consolidated the fouling layer by compression, which reduced the effectiveness of the rinsing. On the contrary, operating at under 50 mbar, a relaxed fouling layer was maintained in the sM, which facilitated its elimination in the rinsing stage.

Cleaning with low concentrations of NaClO, 100 mg/L can be considered as a form of "chemical rinsing", which permits the foulants to be removed from the system. The low concentration of NaClO enable the withdrawal of the detached foulants avoiding the higher membrane fouling potential of the sludge exposed to NaClO [50]. In this way, the effectiveness of the subsequent stages was increased, in which the cleaning agent detached the materials that adhered most firmly to the membrane.

Chemical rinsing permitted the removal of over 60% of the eM resistance, both in filtration and in backwash, although its effectiveness was lower in the sM, due to the weakly attached materials that could be removed by rinsing with tap water.

The oxidizing cleaning with 500 mg/L NaClO was capable of eliminating over 40% of the resistance to filtration from both membranes. Finally, an acid-based cleaner with 100 mg/L of oxalic acid was used. The sM practically recovered its initial permeability with the acid cleaning, however, the eM never recovered its original resistance to filtration, 0.5×10^{12} m^{-1}, staying at 0.8×10^{12} m^{-1}. The cause of the different behaviors of both membranes through the chemical cleaning stages has to be looked for in the different chemical compositions both of the caked layer and of the materials that caused the pore blocking, which in the final analysis was a consequence of the shear stress to which the biomass of the eM was subjected.

3.5 Biomass concentration and methanogenic activity

Samples from the initial anaerobic sludge were taken after 1 day and 7 days from both experimental set-ups for methanogenic activity control.

Specific methanogenic activity was barely affected by shear stress, remaining at values of 11.5±0.5 ml CH_4/g VSS·d in the eAnMBR and at values of 12.3±0.6 ml CH_4/g VSS·d in the sAnMBR. However, a decrease was observed in mixed liquor VSS in the eM tank over time, from 3.3 g VSS/L in the initial anaerobic sludge, to 2.8 g VSS/L within 1 day and to 1.2 g VSS/L within 7 days, a period in which VSS concentration only decreased to 2.7 g VSS/L in the sM tank. Beaubien et al. [51] demonstrated that the high shear rates generated by the circulation pump in a side-stream anaerobic reactor were not detrimental to methanogens that maintained their specific methanogenic activity. Jeison et al. [20] operating a side-stream thermophilic AnMBR also observed a biomass concentration decrease, although lower, from 17 g VSS/L to 15.3 g VSS/L that was attributed to the low apparent yield of thermophilic anaerobic sludge and the washout of active thermophilic microorganisms. Ersahin et al. [21] comparing side stream and submerged anaerobic dynamic membrane configurations, detected less biogas production and less methane concentration in biogas for eAnMBR, however, those same authors also observed a decrease in methanogenic activity of 25% from 0.20 g CH_4-COD/g VS·d in sAnMBR to 0.15 g CH_4-COD/g VS·d in eAnMBR. It may be noted that both authors determined the activity from the concentration of Volatile Solids (VS), so no solid breakdown can eventually be corrected, while methanogenic activity in this work was determined on the basis of the suspended fraction.

Choo and Lee [39] observed an increase in supernatant turbidity and a decrease in biomass from 3000 mg VSS/L to 300 mgVSS/L in an anaerobic bioreactor coupled to a UF membrane when a gear-type recycling pump was used for a CFV between 0.67-0.95 m/s, and from

2410 mgVSS/L to 920 mgVSS/L for a lower mechanical shear pump (mono-pump) and 0.5 m/s in sludge velocity. The authors attributed their findings to cell lysis caused by mechanical shear stress and biomass displacement from the bioreactor onto the membrane surface.

4 Conclusions

Two filtration set-ups respectively equipped with an external tubular membrane and a submerged hollow-fiber membrane, fed with anaerobic sludge, were simultaneously operated to discern the effects of the hydrodynamic conditions on the characteristics of the sludge and on the fouling of the membranes.

Operating the external membrane set-up with gas sparging and low crossflow, the shear stress observed in a series of batch assays has been shown to cause a reduction in the particle size, increasing the concentration of the particles under 10 μm, from 25.1% to 34.6%, within only one day.

After 7 days, the concentrations of soluble proteins and polysaccharides in the sludge were 20% and 37%, respectively, greater in the external than in the submerged membrane set-up. The degree of polysaccharide retention was similar in both membranes, however, the retention of proteins and humic substsances was notably greater in the external membrane.

The concentration of anaerobic sludge in the eM tank was reduced from 3.3 g VSS/L to 1.2 g VSS/L, over 7 days, but the specific methanogenic activity remained nearly constant.

By operating both set-ups under the same filtration cycle (15 minutes), flux (15 $L/m^2 \cdot h$), SGD_m (1.2±0.1 $Nm^3/m^2 \cdot h$), and at a CFV of 0.51 m/s for the external membrane, filtration resistance after 7 days reached 9.3×10^{12} m^{-1} in the eM and 1.1×10^{12} m^{-1} in the sM. The irreversible fouling rate on the filtered volume basis reached 11.5×10^{12} m^{-2} in the eM, in so far as the irreversible fouling rate of the sM was maintained at 0.3×10^{12} m^{-2}, which increased to 1.2×10^{12} m^{-2} when the flux was raised to 25 $L/m^2 \cdot h$.

The effectiveness of the membrane cleaning processes was also different. Rinsing with tap water followed by chemical rinsing of the sM with 100 mg/L of NaClO decreased the resistance to filtration to 1.1×10^{12} m^{-1}. Under the same conditions, the resistance of the eM remained at

2.7×10^{12} m^{-1} and a chemical cleaning with 500 mg/L of NaClO was insufficient to recover its original permeability.

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