# Anaerobic membrane bioreactor performance at different wastewater pre-concentration factors: An experimental and economic study

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## ABSTRACT

This research evaluated the performance of a lab-scale anaerobic membrane bioreactor (AnMBR) treating municipal sewage pre-concentrated by forward osmosis (FO). The organic loading rate (OLR) and sodium concentrations of the synthetic sewage stepwise increased from 0.3 to 2.0 g COD $L^{-1}$ $d^{-1}$ and from 0.28 to 2.30 g $Na^{+}$ $L^{-1}$ to simulate pre-concentration factors of 1, 2, 5 and 10. No major operational problems were observed during AnMBR operation, with COD removal efficiencies ranging between 90 and 96%. The methane yield progressively increased from 214 ± 79 to 322 ± 60 mL $CH_4$ $g^{-1}$ COD as the pre-concentration factor increased from 1 to 10. This was mainly attributed to the lower fraction of methane dissolved lost in the permeate at higher OLRs. Interestingly, at the highest pre-concentration factor (2.30 g $Na^{+}$ $L^{-1}$) the difference between the permeate and the digester soluble COD indicated that membrane biofilm also played a role in COD removal. Finally, a preliminary energy and economic analysis showed that, at a pre-concentration factor of 10, the AnMBR temperature could be increased 10 ºC and achieve a positive net present value (NPV) of 4 M € for a newly constructed AnMBR treating 10,000 $m^3$ $d^{-1}$ of pre-concentrated sewage with an AnMBR lifetime of 20 years.



**KEYWORDS**: Anaerobic digestion; Anaerobic membrane bioreactor (AnMBR); Forward osmosis (FO); Water resource recovery facility (WRRF); Municipal sewage treatment; Sodium inhibition

## 1. INTRODUCTION

Economic and environmental requirements are pushing a paradigm shift in municipal wastewater management. Wastewater is progressively being conceived as a source of resources rather than as a source of pollutants (Garrido-Baserba et al., 2018; Guest et al., 2009). Consequently, new technologies are being developed to maximise resource recovery from wastewater with the aim of converting wastewater treatment plants (WWTPs) into resource recovery facilities (RRF) (Puyol et al., 2017).

Anaerobic membrane bioreactor (AnMBR) technology is an emerging mainstream technology for municipal sewage treatment, which allows to produce renewable energy in the form of methane and to obtain high-quality effluents free of suspended solids and pathogens (Maaz et al., 2019). Additionally, the membrane separation process provides an excellent decoupling of the solids retention time (SRT) from the hydraulic retention time (HRT), which enables an excellent control on the active biomass in the digester (Robles et al., 2018). The complete biomass retention provided by the membrane is a distinctive feature of AnMBRs over other high-rate anaerobic technologies such as upflow anaerobic sludge blanket (UASB) and expanded granular sludge bed (EGSB) reactors (Ozgun et al., 2015a). Moreover, effluents free of suspended solids and with low residual organic matter facilitate the application of post-treatment technologies to remove dissolved methane and nutrients (Batstone et al., 2015).

AnMBR technology has experienced significant advances towards its implementation as mainstream treatment (Zhen et al., 2019). Many pilot-scale AnMBR plants for municipal sewage treatment have been operated with COD removal efficiencies above 85% and variable methane yields (0.07-0.31 mL $CH_4$ $g^{-1}$ COD) as reviewed by Shin and Bae (2018). However, some challenges need to be tackled to make AnMBR technology a reality. High volumetric flow rate is a critical challenge for mainstream AnMBR application since (i) it increases AnMBR capital and operating expenditures, (ii) it makes unviable to heat the influent, and (iii) it increases fugitive methane emissions (Ferrari et al., 2019a; Vinardell et al., 2020a; Wei et al., 2014). Sewage pre-concentration could overcome these limitations and improve the applicability of AnMBR as mainstream technology (Ozgun et al., 2013; Vinardell et al., 2020b).

Different membrane technologies have been tested for municipal sewage pre-concentration such as forward osmosis (FO), dynamic membrane filtration and direct membrane filtration (Guven et al., 2019; Nascimento et al., 2020). Among them, FO allows to concentrate sewage with a high rejection of organic matter, a low energy input and a low fouling potential (Ansari et al., 2017; Hube et al., 2020). FO is a spontaneous process where water permeation is driven by the osmotic difference between the sewage and the draw solution. Therefore, water permeates from the higher chemical potential solution (sewage) to the lower chemical potential solution (draw solution) (Cath et al., 2006).

FO pre-concentration provides four conceivable advantages for AnMBR: (i) it reduces the AnMBR volume, (ii) it increases the methane energy recovered per $m^3$ of sewage, (iii) it minimises the losses of methane dissolved in the permeate, and (iv) it reduces the volume of post-treatment units required to remove or recover dissolved methane and nutrients. Additionally, the diluted draw solution can be re-generated by reverse osmosis whilst producing reclaimed water (Blandin et al., 2016; Lee and Hsieh, 2019). However, the integration of FO and AnMBR technologies for municipal sewage treatment presents some challenges such as (i) the low water fluxes of FO membranes, (ii) the presence of suspended solids in municipal sewage which may lead to FO membrane fouling, (iii) the high energy required to regenerate the draw solution, and (iv) the high salinity in the

AnMBR influent after FO pre-concentration (Ferrari et al., 2019b; Ozgun et al., 2013; Vinardell et al., 2020b).

The selection of the draw solute is critical for FO technology since it affects the salinity of the AnMBR influent. Sodium chloride (NaCl) is a widely used draw solute in both natural (seawater) and synthetic draw solutions (Awad et al., 2019; Coday et al., 2013). NaCl has been reported as a suitable solute to achieve high FO fluxes since its high diffusivity allows to reduce the impact that dilutive internal concentration polarization (ICP) on the support layer has over FO fluxes (Ansari et al., 2015; Shaffer et al., 2015). However, the high diffusivity of NaCl is also counterproductive for the operability of an FO+AnMBR process. The reverse solute flux (RSF) of NaCl from the draw solution to the sewage through the FO membrane is a drawback of using a NaCl solution as draw solution since it increases the salinity of the AnMBR influent (Corzo et al., 2017; Itliong et al., 2019).

Sodium is a well-known inhibitor of the anaerobic digestion process since high sodium concentrations disintegrate cellular material by generating an osmotic pressure difference between both sides of the membrane cell (Muñoz Sierra et al., 2018, 2019). Inhibitory sodium concentrations have been reported to start at 2-3 g $Na^+$ $L^{-1}$ (Astals et al., 2015; Feijoo et al., 1995), despite strong inhibition typically occurring at sodium concentrations above 8 g $Na^+$ $L^{-1}$ (Chen et al., 2008; McCarty, 1964). The potential of sodium to inhibit anaerobic biomass varies depending on several factors such as substrate load, environmental conditions, microbial community or biomass acclimation (Astals et al., 2015). However, the impact of sodium inhibition appears more important during the acclimation of the anaerobic biomass to high and moderate inhibitory concentrations (Chen et al., 2008). Chen et al. (2003) reported that, after biomass acclimation, the sodium concentration that causes total inhibition of methane production increased from 12.7 to

22.8 g $Na^+$ $L^{-1}$. Accordingly, biomass acclimation stands as a critical process to develop a microbial community able to work under high sodium concentrations and prevent digester failure during the AnMBR start-up and long-term operation (Appels et al., 2008; Basset et al., 2016).

Several publications have evaluated the effect that progressive salinity increases have on AnMBR performance (Chen et al., 2019; Muñoz Sierra et al., 2018; Song et al., 2016). To the best of our knowledge, only Ferrari et al. (2019a) have evaluated the performance of an AnMBR treating sewage pre-concentrated by FO (four-fold sewage pre-concentration, 1.72 g COD $L^{-1}$) in a study devoted to assessing the effects of temperature variations on AnMBR performance. However, Ferrari et al. (2019a) did not consider the salinity increase in sewage due to RSF in the FO membrane. The effect of RSF is important since higher influent sodium concentrations can compromise the long-term performance of an AnMBR treating sewage pre-concentrated by FO. Accordingly, evaluating the combined increase in OLR and salinity is needed to better understand the implications of combining FO and AnMBR technologies for municipal sewage treatment.

The present article investigates the performance of an AnMBR treating pre-concentrated municipal sewage by FO. To this aim, different pre-concentration factors were applied to evaluate the effects and operational implications that the progressive increase in organic matter and sodium concentrations have on the AnMBR performance. Finally, an energy-economic analysis was conducted to evaluate the opportunities that methane production offers at different FO pre-concentration scenarios.

## 2. MATERIALS AND METHODS

### 2.1 Feedstock composition

Synthetic municipal sewage was used as feedstock for the AnMBR. Synthetic sewage was used due to the difficulty to consistently obtain the amount of concentrated sewage needed to feed the AnMBR unit. The composition of synthetic sewage was adapted from Huang et al. (2011): $C_6H_{12}O_6$ = 407 mg $L^{-1}$, $CH_3COONa$ = 229 mg $L^{-1}$, $NH_4Cl$ = 95 mg $L^{-1}$, $K_2HPO_4$ = 28 mg $L^{-1}$, $NaHCO_3$ = 600 mg $L^{-1}$, $MgCl_2 \cdot 4H_2O$ = 4.12 mg $L^{-1}$, $CaCl_2 \cdot 2H_2O$ = 19.34 mg $L^{-1}$, $FeCl_3 \cdot 6H_2O$ = 22.5 mg $L^{-1}$, $MnCl_2 \cdot 4H_2O$ = 0.14 mg $L^{-1}$, $Na_2MoO_4 \cdot 2H_2O$ = 1.45 mg $L^{-1}$, $ZnSO_4 \cdot 7H_2O$ = 0.002 mg $L^{-1}$, $H_3BO_3$ = 0.002 mg $L^{-1}$, KI = 0.002 mg $L^{-1}$. The feedstock was prepared three times a week to minimise organic matter degradation in the feed tank.

Sewage composition was adjusted to simulate four different pre-concentration scenarios based on the pre-concentration factor. The pre-concentration factor is determined by the FO recovery, which can be defined as the percentage of water that permeates the FO membrane. This parameter is particularly relevant for the integration of FO and AnMBR technologies since organic and salinity concentrations increase as FO recovery increases. The increase of salinity in sewage is caused by (i) the reduction in the sewage volumetric flow rate and (ii) the RSF of draw solute through FO membrane. The final sodium concentration in synthetic sewage due to RSF was estimated using Eq. (1) (Ansari et al., 2015):

$$C_f = \frac{1}{J_W/J_S} \cdot \frac{FO_{recovery}}{100 - FO_{recovery}} \qquad \text{Eq. (1)}$$

where $J_w$ is the FO water flux (L $m^{-2}$ $h^{-1}$), $J_S$ is the RSF (g $m^{-2}$ $h^{-1}$), $C_f$ is the draw solute concentration in the influent sewage (g $L^{-1}$), and $FO_{recovery}$ is the water recovery in the FO membrane (%).

## 2.2 AnMBR set-up and operation

The AnMBR set-up consisted of a jacked completely stirred tank reactor (CSTR) of 5.5 L connected to an external membrane module. The membrane module was a flat sheet polyvinylidene difluoride (PVDF) ultrafiltration module (Rayflow Module, Orelis Environment, France) with a membrane area of 0.02 $m^2$ and a pore size of 0.05 µm. The membrane system was kept at a constant transmembrane pressure (TMP) of approximately 0.1 bar. The membrane was physically and chemically cleaned (see Figure 2 for further information about cleaning periodicity). The physical cleaning consisted in manually flushing the membrane with distilled water. The chemical cleaning consisted in submerging the membrane into a solution of sodium hypochlorite (0.3% of chlorine) for 2 hours.

The configuration used to feed the AnMBR was similar to the one described in Basset et al. (2016) and consisted of a 500 mL cylinder vessel kept at constant volume and connected to the digester (Figure S1, supplementary material). This configuration (based on communicating vessels) allows to keep the digester volume constant despite oscillations in membrane flux. The digester was kept at 35 ºC by recirculating water from a heated water bath (HUBER 118A-E) through the digester external jacket. The digester was stirred at 80 rpm using an overhead paddle stirrer. The headspace of the AnMBR was connected to a sodium hydroxide solution trap to absorb the $CO_2$ from biogas. A phenolphthalein indicator was added to ensure that the sodium hydroxide solution was not neutralised. A Ritter MGC-1 gas counter was used to measure the produced volume of methane. All the methane yields reported in this publication refer to the fed COD. The AnMBR was operated at an HRT of 3.1 ± 0.8 d while the SRT was not controlled since biomass was only purged during the sampling events.

The digested sewage sludge used to inoculate the AnMBR was collected from a mesophilic anaerobic digester in a municipal WWTP, which treats a mixed sewage sludge at a solid concentration of 10 g TS $L^{-1}$ and a pH of 7.2. The full-scale WWTP has a treatment capacity of approximately 400,000 population equivalent (Barcelona Metropolitan Area, Spain). The AnMBR was inoculated with a 1:3 dilution of the digested sewage sludge with deionised water to achieve an initial suspended solids concentration of about 3 g $L^{-1}$. Inoculum dilution aimed to reduce membrane fouling and cleaning events during the AnMBR start-up.

The COD and the sodium concentration of the AnMBR influent varied according to the sewage flow rate reduction and the RSF. Specifically, sewage COD and sodium concentrations were progressively increased to simulate the different pre-concentration factors: (i) without pre-concentration (Period 1), (ii) pre-concentration factor of 2 (Period 2, 50% FO recovery), (iii) pre-concentration factor of 5 (Period 3, 80% FO recovery) and (iv) pre-concentration factor of 10 (Period 4, 90% FO recovery) (see Table 1). The progressive increase in COD and sodium concentrations aimed to favour the acclimation of the anaerobic biomass to harsher conditions (Basset et al., 2016). Each period was operated for a minimum of 5-HRT equivalents.

**2.3 Analytical methods**

Chemical oxygen demand (COD), total suspended solids (TSS) and volatile suspended solids (VSS) analysis were performed following the Standard Methods 5220C, 2540D and 2540G, respectively (APHA, 2017). Total ammonium nitrogen (TAN) was analysed using a Thermo Fisher Scientific ammonium ion-selective electrode (Orion 9512HPBNWP), following the Standard Methods procedure 4500-NH3D. The pH was analysed with a Crison pH electrode (pH series 52-04). Volatile fatty acids (VFA, i.e. acetic, propionic, i-butyric, n-butyric, i-valeric, n-valeric, i-caproic, n-caproic, heptanoic acid) were analysed using a gas chromatograph (Shimadzu GC-2010 Plus) equipped with a Nukol$^{TM}$ column (15m x 0.53mm) and a flame ionisation detector (see Astals et al. (2012) for gas chromatograph configuration and procedure).

### 2.4 Energy-economic analysis

The energy-economic analysis of the AnMBR process under different FO pre-concentration scenarios was done to evaluate the opportunities that methane production offers to the AnMBR technology. Specifically, the energy-economic analysis evaluated: (i) the sewage temperature increment that could be achieved for each pre-concentration factor and (ii) the impact of sewage pre-concentration on the AnMBR economic balance. Four different scenarios were included in the analysis (i.e. pre-concentration factor of 1, 2, 5 and 10). Energy production was calculated with the average experimental methane yields obtained from each period. It was considered that methane dissolved in the permeate was not recovered.

Two alternatives were considered for on-site energy valorisation: (i) thermal energy valorisation (without methane cogeneration) and (ii) thermal and electrical energy valorisation (with methane cogeneration). A methane calorific value of 38,800 kJ Nm$^{-3}$ was considered for both alternatives. A combined heat and power (CHP) unit was used for energy production with an electricity and heat efficiency of 33 and 55% according to common literature values (Appels et al., 2011; Batstone et al., 2015; Cogert et al., 2019; Pöschl et al., 2010; Ruiz-Hernando et al., 2014). Eq. (2) was used to calculate the potential temperature increase:

$$\Delta T = \frac{q \cdot \eta}{Q \cdot \rho \cdot c_p} \qquad \text{Eq. (2)}$$

where $\Delta T$ is the temperature increment of the influent sewage (°C), q is the heat energy (kJ d$^{-1}$), $\eta$ is the heat exchange efficiency (90%), $\rho$ is the water density (1000 kg m$^{-3}$), Q is the sewage flow rate (m$^3$ d$^{-1}$) and $c_p$ is the water specific heat (4.18 kJ kg$^{-1}$ °C$^{-1}$).

The AnMBR capital and operating costs and the revenue from electricity generation were considered for the economic evaluation. The AnMBR capital and operating costs were adapted from Vinardell et al. (2020a) while the electricity was assumed to be sold at a unit price of 0.1149 € $kWh^{-1}$ (Eurostat, 2019) The AnMBR influent flow rate was calculated for each pre-concentration factor considering an influent flow rate before pre-concentration of 100,000 $m^3$ $day^{-1}$ (see Table 2).

The net present value (NPV) method was used for the AnMBR economic evaluation (Garrido-Baserba et al., 2018; Verrecht et al., 2010) (Eq. (3)).

$$\mathrm{NPV}\ (€) = \sum_{t=1}^{T} \frac{I_t - OPEX_t}{(1+i)^t} - CAPEX \qquad \text{Eq. (3)}$$

where $I_t$ is the electricity revenue at year t (€), $OPEX_t$ is the AnMBR operating expenditures at year t (€), CAPEX is the AnMBR capital expenditures (€), i is the discount rate (5%) and t is the plant lifetime (20 years).

## 3. RESULTS AND DISCUSSION

### 3.1 AnMBR performance and operation

The lab-scale AnMBR was successfully operated for 80 days under four different sewage pre-concentration factors. The COD and the sodium concentrations of the AnMBR influent were increased at each operational period to simulate different pre-concentration factors. Table 1 summarises the main operating conditions and results for the four operational periods.

Figure 1 shows the OLR, influent sodium concentration, permeate COD concentration, VFA concentration and COD removal efficiency for the four operational periods. Fluctuations in COD removal efficiency with values ranging between 79 and 98 % were observed during Period 1, probably caused by the ongoing acclimation of the anaerobic biomass to the AnMBR conditions (Figure 1). Despite these fluctuations, the average COD removal efficiency was above 90% and the permeate COD met the EU regulations concerning municipal sewage treatment (< 125 mg COD $L^{-1}$) (CEC, 1991). Period 2 (two-fold sewage pre-concentration) was characterised by a stable AnMBR performance with COD removal efficiencies above 95% and permeate COD concentrations below 60 mg COD $L^{-1}$.

The performance of the AnMBR decreased during Period 3 (five-fold sewage pre-concentration) since permeate COD concentration progressively increased from 50 to 350 mg COD $L^{-1}$ (day 58). However, the average COD removal efficiency remained high (94 ± 4%) due to the higher influent COD concentration (ca. 3,200 mg COD $L^{-1}$). The increase in the permeate COD concentration could be attributed to the increased OLR rather than the sodium concentration (1.14 g $Na^{+}$ $L^{-1}$) since sodium concentrations below 2 g $Na^{+}$ $L^{-1}$ have been reported as not inhibitory for anaerobic microbes (Astals et al., 2015; Wang et al., 2017).

FO pre-concentration reduces influent volumetric flow rate and increases influent COD concentration. However, these experimental results illustrate that while sewage pre-concentration can provide conceivable advantages for municipal sewage treatment, it can also compromise the compliances with COD concentration limits. In this regard, COD removal efficiencies above 96% would be required to meet the EU discharge limits for five-fold pre-concentrated sewage. Furthermore, high effluent COD concentrations could negatively affect nutrient removal post-treatments such as the autotrophic partial nitritation/anammox process (Dai et al., 2015; Giustinianovich et al., 2016).

High COD removal efficiencies (95 ± 5%) were also achieved during Period 4 (ten-fold pre-concentrated sewage), despite the instability occurred between day 66 and 68. On day 65, membrane was chemically cleaned and, therefore, membrane flux significantly increased from 2.1 to 5.6 L $m^{-2}$ $h^{-1}$ (LMH) (see Figure 2). Consequently, an OLR shock above 3 g COD $L^{-1}$ $d^{-1}$ occurred on day 66, which led to permeate COD concentrations above 900 mg COD $L^{-1}$ (Figure 1). Moreover, COD removal efficiency was worsened by the removal of the biofilm layer on the membrane which also played a role in COD removal efficiency (see Section 3.2). During this instability period, VFAs concentration increased from 10 to 706 mg COD $L^{-1}$, mainly acetate (55%) and propionate (38%) (Figure S2, supplementary material). To recover the system and prevent further accumulation of VFAs, the membrane system was switched off until the VFA concentration decreased below 100 mg COD $L^{-1}$. This accumulation of VFA showed that the AnMBR performance is susceptible to OLR shock loads. On day 71, AnMBR performance reached previous operational values and COD removal efficiencies above 95% were sustained until the end of the operational period with COD permeate concentrations below 100 mg COD $L^{-1}$.

Methane yields progressively increased with the pre-concentration factor (Table 1). Specifically, the methane yield increased from 214 ± 79 to 322 ± 60 mL $CH_4$ $g^{-1}$ COD as the pre-concentration factor increased from 1 to 10, respectively. The differences in methane yields were primarily attributed to the lower fraction of dissolved methane lost in the permeate with respect to the total fraction of methane produced (i.e. dissolved methane + gas methane) as the OLR increases. Note that the dissolved methane concentration in the permeate is expected to be similar regardless of the influent COD concentration since its equilibrium mainly depends on the temperature (ca. 13.7 mg $CH_4$ $L^{-1}$ at 35ºC and saturation level). This finding is in agreement with Yeo et al. (2015), who attributed the lower fraction of dissolved methane to the higher methane production and mass transfer rate at higher OLRs.

Figure 2 shows the membrane flux of the AnMBR for the four operational periods. The membrane system was operated at constant transmembrane pressure (TMP) of 0.1 bar and, consequently, membrane flux progressively decreased between cleaning events. The maximum membrane flux (7.5 LMH) was obtained after the first chemical cleaning (day 5 in Period 1). Physical membrane cleanings were carried out in day 26, 39 and 52 when membrane flux decreased below 3-4 LMH (green vertical lines in Figure 2). In day 61, membrane flux sharply decreased below 2 LMH. At the early stages of Period 4, MLSS concentration had increased from 4.2 to 5.6 g TSS $L^{-1}$ which probably exacerbated membrane fouling and decreased membrane flux. The increase in MLSS concentration could be attributed to (i) the non-controlled SRT and (ii) the higher biomass growth due to the higher influent COD concentration (6,510 mg COD $L^{-1}$). On day 65, after a membrane chemical cleaning event, the membrane fluxes increased above 5 LMH. However, a 5 LMH membrane flux was lower than the obtained after the first chemical cleaning in Period 1 (ca. 7.5 LMH). The different response after both chemical cleanings could be explained by (i) the higher MLSS concentration in Period 4 and (ii) the membrane fouling caused by compounds that cannot be removed through chemical cleanings (Basset et al., 2016; Dong et al., 2016).

### 3.2 The relative importance of suspended biomass and membrane biofilm on AnMBR performance under saline conditions

The AnMBR operation was satisfactory accomplished with sodium concentrations up to 2.3 g $Na^{+}$ $L^{-1}$. The progressive increase of OLR and sodium concentration allowed to acclimatise the anaerobic biomass to higher saline concentrations without major disturbances. It has been reported that sodium concentrations below 3.9 g $Na^{+}$ $L^{-1}$ do not significantly affect AnMBR performance (Chen et al., 2019, 2014; Song et al., 2016). Song et al. (2016) reported that, after biomass acclimation, high TOC removal (98%)

efficiencies can be achieved at sodium concentrations of 2 g $Na^+$ $L^{-1}$. Similarly, Chen et al. (2019) achieved a 94% COD removal efficiency at 2 g $Na^+$ $L^{-1}$. These experimental results agree with the results obtained in this study, where AnMBR performance was sustained for sodium concentrations up to 2.3 g $Na^+$ $L^{-1}$. However, the increase in sodium and concentration and OLR had a direct impact on the role of biofilm in process performance. Figure 3 shows the differences between digester and permeate soluble COD (sCOD) concentrations for the four operational periods. The sCOD concentration in the digester was consistently higher than in permeate for all the operational periods, clearly indicating that membrane biofilm played a role in COD removal.

Differences between permeate and digester sCOD have been reported in previous AnMBR studies (Martinez-Sosa et al., 2011; Smith et al., 2013). The difference in Period 4 (550 mg COD $L^{-1}$) was significantly higher than the difference in Period 1, 2 and 3 (80-120 mg COD $L^{-1}$). These results indicate that the role of biofilm in AnMBR performance is higher under less favourable conditions (2.3 g $Na^+$ $L^{-1}$), although the sodium concentration was below the reported strong inhibitory concentrations. It is well-known that many bacteria form biofilm as a survival strategy under stress conditions (e.g. chemical, biological or physical) or non-optimal growth conditions (Jefferson, 2004). Smith et al. (2015) reported that the contribution of membrane biofilm in COD removal efficiency increased from ~40% to ~90% when AnMBR temperature decreased from 15 to 3 ºC. In addition, membrane biofilm has been reported to increase dissolved methane supersaturation in the permeate due to the methanisation of acetate and hydrogen in the biofilm (Smith et al., 2013, 2015). However, further studies are needed to evaluate the role that membrane biofilm has under high salinity conditions and the underpinning microbial community changes in both AnMBR mixed liquor and membrane biofilm.

**3.3 The role of reverse solute flux (RSF) in the operation of an FO+AnMBR system**

Experimental results showed that sodium inhibition did not occur at 2.3 g $Na^+$ $L^{-1}$ (Figure 1). This is relevant for FO+AnMBR system since it indicates that high process performance can be sustained despite the RSF of sodium through FO membrane. However, sodium RSF may have a direct impact on the performance and profitability of AnMBR process. Besides the changes in membrane biofilm activity and development (Figure 3), the generation of an AnMBR permeate with 2.3 g $Na^+$ $L^{-1}$ significantly hinders its application in agriculture. The use of high saline effluents for agricultural irrigation can negatively affect crop growth and soil structure (Beletse et al., 2008; Foglia et al., 2020).

The diffusion of salt has also negative connotations for FO process since it reduces the effective osmotic pressure difference and increases operational costs in areas where natural draw solutions (e.g. seawater) are not available (Blandin et al., 2015; Corzo et al., 2017). The RSF depends on many factors such as FO membrane properties, operational conditions and solute characteristics (Zou et al., 2019). The development of new FO membranes has gained special attention to improve FO membrane performance (Blandin et al., 2015; Lee and Hsieh, 2019; Zhao et al., 2012). The development of new FO membranes has mainly focused on improving water flux. However, little attention has been given to develop FO membranes able to achieve high water fluxes while minimising the RSF (Zou et al., 2019). Most research efforts have focused on (i) reducing ICP effects by modifying the porosity, tortuosity and hydrophilicity of the support layer and (ii) increasing water permeability by modifying membrane characteristics of the active layer (Blandin et al., 2015; Tiraferri et al., 2013). However, these modifications do not necessarily mitigate RSF and the associated increase of sewage salinity. Consequently, the development of FO membranes with limited RSF is important for the success of the FO+AnMBR process.

### 3.4 Energy-economic analysis

Temperature can limit the application of AnMBR technology in cold and temperate climates since uncontrolled psychrophilic temperatures will be required due to the impossibility to heat the digester (Dev et al., 2019). Psychrophilic temperatures have a direct impact on AnMBR performance and fugitive methane emissions (Martin Garcia et al., 2013; Ozgun et al., 2015b; Smith et al., 2013). Therefore, the increment of sewage temperature has been explored as an option in FO pre-concentration scenarios to improve AnMBR performance and broad the applicability of AnMBR to cooler regions. It is worth highlighting that, although this study focused on pre-concentrated municipal sewage reaching COD concentrations up to 6,500 mg $L^{-1}$ (ten-fold sewage pre-concentration), the operational and energy-economic results of the present work could be extendible to high-strength industrial wastewaters.

The energy-economic analysis was conducted (i) to calculate the sewage temperature increments that could be achieved at each FO pre-concentration scenarios and (ii) to determine if FO pre-concentration can make an AnMBR economically self-sufficient. The experimental average methane yields (i.e. 214, 259, 317 and 322 mL $CH_4$ $g^{-1}$ COD) for each FO pre-concentration scenario (i.e. pre-concentration factor of 1, 2, 5 and 10) were used for the energy-economic analysis.

Table 2 shows the energy and economic results for the four scenarios under study. Scenarios with low pre-concentration factors (≤ 2) do not allow to heat the influent sewage more than 2.4 ºC and, therefore, increasing the influent temperature is considered unviable. Considering only thermal energy valorisation, a pre-concentration factor of 10 allows to increase sewage temperature up to 16.3 ºC, which would approach municipal sewage treatment to mesophilic conditions. Wei et al. (2014) also reported that mesophilic conditions could be achievable at pre-concentration factors above 5. Operating at mesophilic conditions has three relevant positive connotations: (i) it improves anaerobic digestion kinetics, (ii) it reduces methane solubility, and (iii) it improves effluent post-treatments performance, which are sensitive to temperature such as partial nitrification/anammox (Dev et al., 2019; Morales et al., 2015). However, under the circular economy framework, this scheme is not conceivable since it fails to recover renewable energy (e.g. electricity, biomethane). The combination of electrical and thermal energy valorisation (i.e. cogeneration) limits sewage temperature increase to 4.8 and 10 ºC for pre-concentration factors of 5 and 10, respectively. However, it allows to produce renewable electrical energy from biogas (Table 2).

FO pre-concentration decreases AnMBR influent flow rate and increases the energy recovered per $m^3$ of sewage, which shows the importance of FO pre-concentration on AnMBR economics (Table 2). High pre-concentration factors (i.e. 5 and 10) allow to increase electricity revenue and reduce AnMBR costs. Therefore, NPV increases from -163 to 4 M€ as the pre-concentration factor increases from 1 to 10, respectively (Table 2). This analysis shows that the economic self-sufficiency of the AnMBR is only achieved with a pre-concentration factor of 10. The potential of AnMBRs to achieve economic and energy self-sufficiency when treating high-strength sewage has also been reported in other studies (Galib et al., 2016; Van Zyl et al., 2008). It should be noted that the methane produced in the scenarios with a pre-concentration factor of 2 and 5 would be enough to offset the AnMBR OPEX. However, economic self-sufficiency is not achieved in these scenarios, mainly due to the high membrane CAPEX.

The reduction in the AnMBR volumetric flow rate also allows to reduce the amount of dissolved methane leaving the permeate, which (i) increases energy production, (ii) reduces the size of the methane recovery device (iii) and reduces fugitive methane emission. The latter is especially relevant owing to the high methane global warming potential (Crone et al., 2016; Huete et al., 2018). Smith et al. (2014) showed that dissolved methane accounted for 75% of the global warming impact of an AnMBR. Accordingly, sewage pre-concentration would allow to reduce the environmental impacts related to dissolved methane, which makes this approach particularly relevant for mainstream AnMBR application.

Finally, it is worth mentioning that this economic evaluation has not included AnMBR post-treatments nor FO pre-concentration which could significantly increase the overall costs. Indeed, FO pre-concentration has been reported as the main cost contributor of the FO+AnMBR treatment due to the low FO water fluxes (Vinardell et al., 2020a). This is particularly critical at high FO recoveries where the progressive decrease of the driving force leads to lower FO water fluxes and larger FO membrane areas that can compromise the economic feasibility of FO+AnMBR system. Indeed, as discussed above, the economic and technical feasibility of FO+AnMBR requires the development of FO membranes featuring high water fluxes and low sodium RSF from which renewable methane energy production can be maximised in the AnMBR process.

## 4. CONCLUSIONS

The performance of an AnMBR at different pre-concentration factors was investigated. OLR and sodium concentration progressively increased from 0.3 to 2.0 g COD $L^{-1}$ $d^{-1}$ and from 0.28 to 2.30 g $Na^{+}$ $L^{-1}$, to simulate pre-concentration factors of 1, 2, 5 and 10. The AnMBR was successfully operated achieving COD removal efficiencies above 90% regardless of the pre-concentration factor. The methane yield at 35 ºC progressively increased from 214 ± 79 to 322 ± 60 mL $CH_4$ $g^{-1}$ COD as the pre-concentration factor increased from 1 to 10. These results were attributed to the lower fraction of dissolved methane lost in the permeate as the OLR increases. Experimental results showed that membrane biofilm plays a role in COD removal efficiency particularly at the highest pre-concentration factor (2.30 g $Na^+$ $L^{-1}$). Finally, an energy-economic analysis estimated that, at a pre-concentration factor of 10, the combination of pre-concentration and AnMBR technologies allows to increase sewage temperature 10 ºC and achieve a positive net present value (NPV) of 4 M€ for a newly constructed AnMBR with a lifetime of 20 years and treating 10,000 $m^3$ $d^{-1}$ of pre-concentrated sewage. These results show that sewage pre-concentration stands as an option to make AnMBR economic self-sufficient.

**Declaration of competing interests**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The authors also declare that this manuscript reflects only the authors' view and that the Executive Agency for SME/EU Commission are not responsible for any use that may be made of the information it contains.

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**Table 1.** Operating conditions and performance of the lab-scale AnMBR.

| | Period 1 | Period 2 | Period 3 | Period 4 |
|---|---|---|---|---|
| Pre-concentration factor | 1 | 2 | 5 | 10 |
| FO recovery (%) | 0 | 50 | 80 | 90 |
| Influent COD (mg COD $L^{-1}$) | 576 ± 22 | 1,176 ± 9 | 3,187 ± 98 | 6,510 ± 43 |
| COD removal (%) | 90.9 ± 6.0 | 95.9 ± 0.7 | 94.2 ± 3.7 | 95.8 ± 5.6 |
| OLR (g COD $L^{-1}$ $d^{-1}$) | 0.25 ± 0.06 | 0.36 ± 0.04 | 1.04 ± 0.26 | 1.96 ± 0.51 |
| HRT (d) | 2.4 ± 0.6 | 3.3 ± 0.4 | 3.4 ± 0.3 | 3.6 ± 1.1 |
| Membrane flux (L $m^{-2}$ $h^{-1}$) | 4.8 ± 1.5 | 3.5 ± 0.4 | 3.5 ± 0.4 | 3.5 ± 0.9 |
| MLSS (g $L^{-1}$) | 3.3 | 3.4 | 4.2 | 5.6 |
| pH | 7.6 ± 0.3 | 8.2 ± 0.1 | 8.4 ± 0.2 | 8.4 ± 0.2 |
| Methane yield (mL $CH_4$ $g^{-1}$ COD) | 214 ± 79 | 259 ± 15 | 317 ± 61 | 322 ± 60 |
| Permeate COD (mg COD $L^{-1}$) | 53 ± 34 | 47 ± 7 | 131 ± 107 | 254 ± 344 |
| Permeate VFA (mg COD $L^{-1}$) | 10 ± 12 | 12 ± 10 | 59 ± 88 | 113 ± 240 |

**Table 2.** Energy production and economic results of AnMBR treating pre-concentrated municipal sewage.

| | Scenario 1 | Scenario 2 | Scenario 3 | Scenario 4 |
|---|---|---|---|---|
| Pre-concentration factor | 1 | 2 | 5 | 10 |
| FO recovery (%) | 0 | 50 | 80 | 90 |
| AnMBR sewage flow rate ($m^3$ $d^{-1}$) | 100,000 | 50,000 | 20,000 | 10,000 |
| Energy production (kWh $m^{-3}$) | 1.2 | 3.1 | 10.2 | 21.1 |
| $\Delta$T without cogeneration (ºC) | 0.9 | 2.4 | 7.8 | 16.3 |
| $\Delta$T with cogeneration (ºC) | 0.6 | 1.4 | 4.8 | 10.0 |
| Electricity production (kWh $m^{-3}$) | 0.4 | 1.0 | 3.4 | 6.9 |
| NPV (M€) | -163 | -68 | -9 | 4 |

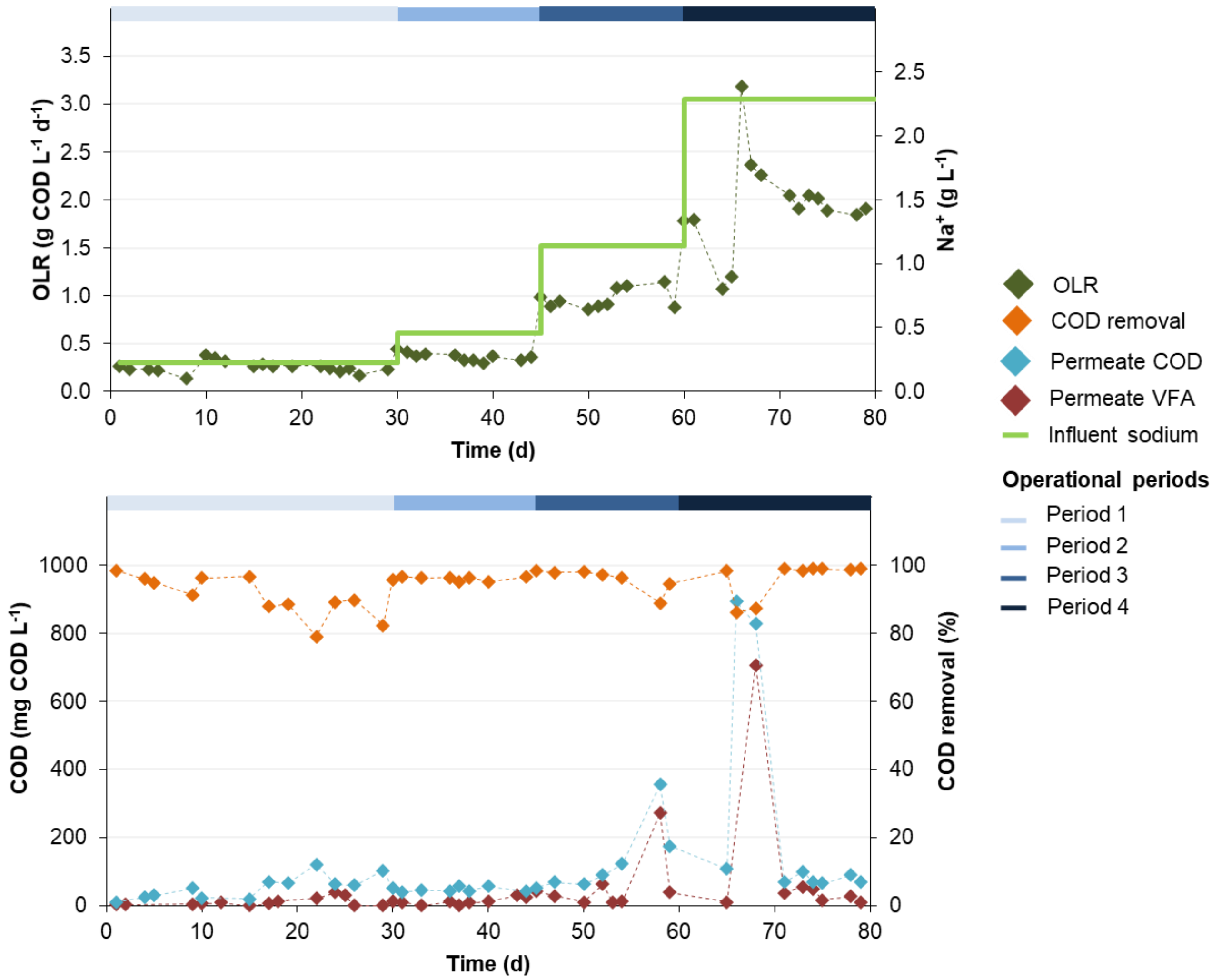


**Figure 1.** (top) Influent sodium concentration and OLR; (bottom) permeate VFAs, permeate COD and COD removal efficiency for the four operational periods.

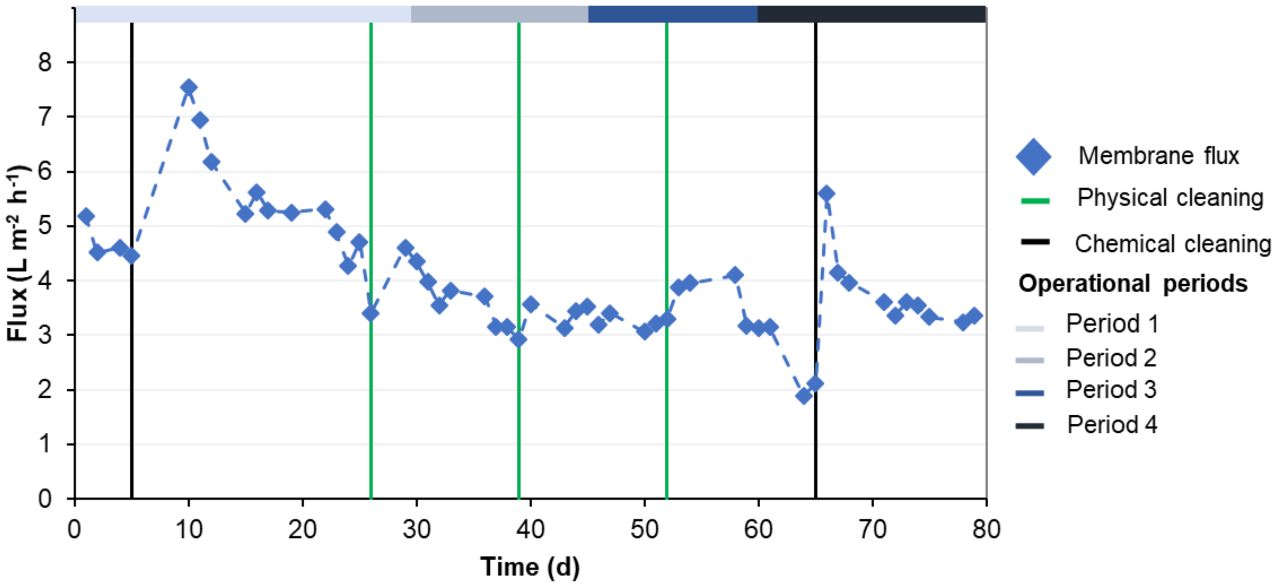


**Figure 2.** Membrane flux of the AnMBR for the four operational periods.

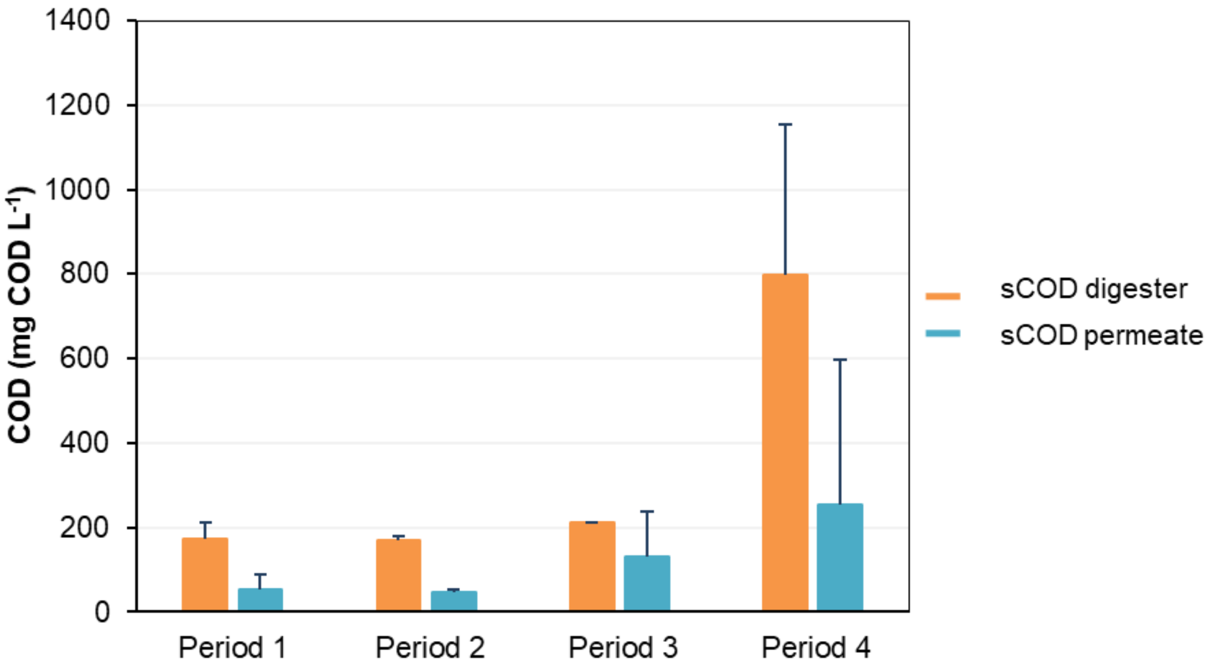


**Figure 3.** Soluble COD in permeate and mixed liquor for the four operational periods.