

Anaerobic treatment of urban wastewater in membrane bioreactors: evaluation of seasonal temperature variations

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ABSTRACT

The objective of this study was to evaluate the effect of seasonal temperature variations on the anaerobic treatment of urban wastewater in membrane bioreactors (MBRs). To this aim, sludge production, energy recovery potential, chemical oxygen demand (COD) removal and membrane permeability were evaluated in a submerged anaerobic MBR fitted with industrial-scale membrane units. The plant was operated for 172 days, between summer and winter seasons. Sludge production increased and energy recovery potential decreased when temperature decreased. COD removal and membrane permeability remained nearby stable throughout the whole experimental period.

Key words | ambient temperature, anaerobic MBR (AnMBR), industrial-scale membrane, urban wastewater

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NOMENCLATURE

AnMBR	anaerobic MBR	TSS	total suspended solids
CH ₃ COOH	acetic acid	UWW	urban wastewater
COD	chemical oxygen demand	VFA	volatile fatty acid
COD _{Removed}	COD removed	VS	volatile solids
EPS	extracellular polymeric substances	VSS	volatile suspended solids
HRT	hydraulic retention time	VS _{Wasted}	amount of VS wasted
J ₂₀	20 °C-standardised transmembrane flux	WSP	waste sludge production
K ₂₀	20 °C-standardised membrane permeability	WWTP	wastewater treatment plant
LMH	litre per square metre of membrane per hour	$\eta_{\text{Recovery}}^{\text{CH}_4}$	methane recovery efficiency
MBR	membrane bioreactor		
MT	membrane tank		
OLR	organic loading rate		
S ²⁻	total sulphide expressed as S ²⁻		
S-COD	soluble chemical oxygen demand		
SGD _m	specific gas demand per square metre of membrane area		
SRT	sludge retention time		
SO ₄ -S	sulphate measured as sulphur		
T-COD	total chemical oxygen demand		
TS	total solids		

INTRODUCTION

The world's population growth and an increasing standard of living are pushing human use of natural resources beyond sustainable limits (Wallace 2005). Therefore, the current approach for urban wastewater (UWW) treatment, focused on what must be removed, is becoming increasingly unsustainable. Sustainability demands are shifting UWW treatment from its current approach towards a new

paradigm focusing on resource recovering (e.g. water, materials, and energy) from UWW (Kleerebezem & van Loosdrecht 2007; Daigger 2008; Larsen *et al.* 2009; Li & Yu 2011).

Compared to aerobic processes, anaerobic ones require less energy input, since no aeration is required, and produce a fraction of the residuals, given the lower biomass yields (Ho & Sung 2010). Furthermore, anaerobic biotechnology allows for the energetic valuation of the organic matter in the wastewater through conversion to methane-rich biogas that can offset or even outweigh process energy requirements. However, Martín *et al.* (2011) reported that influent chemical oxygen demand (COD) concentrations higher than 4–5 g L⁻¹ would be necessary to generate enough biogas to heat the bioreactor up to mesophilic temperature conditions. Therefore, given the typical low COD concentration of UWW, operation at ambient temperature is essential for sustainable anaerobic treatment.

Operational temperature certainly affects some metabolic pathways during anaerobic treatment, although no evidence of low-temperature inhibition has been reported so far (Smith *et al.* 2012). Overall, hydrolysis and microorganism growth rate decrease with temperature, making necessary high sludge retention times (SRTs) to counterbalance the low microbial activity. On the other hand, the high per-capita UWW production makes necessary the use of low hydraulic retention times (HRTs). Recent advances in membrane technology have led to a drop in membrane costs (Furukawa 2008), which have in turn broadened out its applicability. Membrane technology allows for complete retention of biomass and particulate organics whilst uncoupling SRT and HRT, overcoming the main constraints of anaerobic processes at low temperature. Several authors have studied the anaerobic treatment of UWW in membrane bioreactors (MBRs) at low temperature (Martínez-Sosa *et al.* 2011, 2012; Smith *et al.* 2012). However, the effect of all-year-round temperature variation in middle-latitude temperate climates on the performance of an anaerobic MBR (AnMBR) treating UWW has yet to be assessed.

In this study, the performance of a demonstration-scale submerged AnMBR working at ambient temperature has been evaluated for 172 days, between summer and winter seasons. Sludge production, energy recovery potential and COD removal have been selected as indicators of the biological process performance due to its representativeness of the main advantages claimed by AnMBR technology. The 20 °C-standardised membrane permeability (K_{20}) has been selected as an indicator of the filtration process performance.

MATERIAL AND METHODS

AnMBR plant description

Experiments were carried out in an AnMBR plant, fed with the effluent from the pre-treatment of a full-scale wastewater treatment plant (WWTP) located in Valencia (Carraixet WWTP). The AnMBR mainly consists of one 1.3 m³ anaerobic reactor connected to two 0.8 m³ separation tanks (MT1 and MT2). Each membrane tank includes one commercial ultrafiltration hollow-fibre membrane system (PURON[®], Koch Membrane Systems, 0.05 µm pore size, 30 m² total filtering area). A rotfilter of 0.5 mm screen size has been installed as a pre-treatment system. One equalisation tank (0.3 m³) and one clean-in-place tank (0.2 m³) are also included as main elements of the pilot plant.

To improve the stirring conditions in the anaerobic reactor and to favour the stripping of the produced gases from the liquid phase, a fraction of the produced biogas is continuously recycled to this reactor. In order to minimise the cake layer formation, another fraction of the produced biogas is also continuously recycled to the membrane tanks through the bottom of the hollow-fibre membranes. This biogas sparging for membrane scouring is measured in this study as specific gas demand per square metre of membrane area (SGD_m). To recover the bubbles of biogas in the permeate leaving the membrane tank, two degasification vessels were installed: each one between the respective membrane tank and the vacuum pump. The funnel-shaped section of the conduit makes the biogas accumulate at the top of the degasification vessel.

The filtration process was studied from experimental data obtained from MT1 (operated with continuous recycling of the obtained permeate to the system), whilst the biological process was studied from experimental data obtained from MT2 (operated for the biological process without recycling the obtained permeate). Hence, different 20 °C-standardised transmembrane flux (J_{20}) values (i.e. permeate flow per square metre of membrane area) were tested in MT1, without affecting HRT.

Further details on this AnMBR plant can be found in Giménez *et al.* (2011).

Analytical monitoring

Analytical methods (i.e. total COD (T-COD), soluble COD (S-COD), total suspended solids (TSS), volatile suspended solids (VSS), total solids (TS), volatile solids (VS), SO₄-S and S²⁻) were performed according to *Standard Methods* (APHA 2005). Volatile fatty acids (VFAs) were determined by titration

according to the method proposed by WRC (1992). Biogas composition was monitored with an Emerson Process Analytical GmbH 'X-stream multiple purpose' multiparametric gas analyser after sample conditioning. Non-dispersive infrared and ultraviolet, and thermoconductivity analysers were used for CH₄ and CO₂, H₂, and H₂S, respectively. Dissolved CH₄ was indirectly determined with the static-headspace gas chromatography analysis technique. Further details on this technique can be found in Giménez *et al.* (2012). The IBM® SPSS® Statistics v.19 software suite was used for experimental data statistical analysis. Extracellular polymeric substances (EPS) were extracted according to the cation exchange resin method proposed by Frølund *et al.* (1996). The carbohydrates and proteins of EPS were determined by colorimetry according to the methodology proposed by Dubois *et al.* (1956) and Lowry *et al.* (1951), respectively.

Experimental design

In order to assess the influence of all-year-round ambient-temperature variations in middle-latitude temperate climates on the AnMBR performance, an experimental period comprising 172 days of continuous operation was sub-divided into three different operating periods: summer, fall and winter. Figure 1 depicts the evolution of the main operational parameters (i.e. SRT, HRT and temperature) throughout the different operating periods. In this sense, temperature has been considered an operational parameter rather than a process variable.

Table 1 summarises the main operational parameters which were set for the different operating periods, as well as the evolution of ambient temperature. The median has been selected as the position statistic, since data were not normally distributed. Furthermore, Q₁ and Q₃ (which

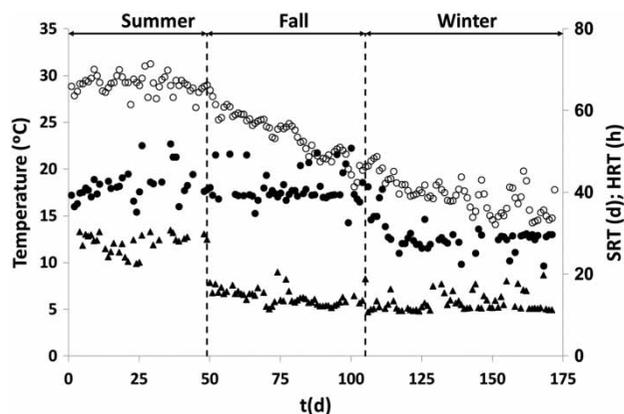


Figure 1 | Temperature (○), SRT (●) and HRT (▲) variation during the studied experimental period.

Table 1 | Main operational conditions during the different operating periods evaluated

Period	Parameter	n	Median	(Q ₁ -Q ₃)	Range
Summer	SRT (d)	33	41.1	(39.9-42.8)	16.6
	HRT (h)	31	28.4	(26.6-29.5)	8.1
	Temperature (°C)	49	29.2	(28.7-29.6)	4.0
Fall	SRT (d)	49	39.5	(39.2-42.0)	18.3
	HRT (h)	51	14.0	(13.1-15.5)	9.0
	Temperature (°C)	56	24.2	(21.6-25.3)	10.3
Winter	SRT (d)	42	28.6	(27.4-29.5)	11.4
	HRT (h)	54	12.1	(11.6-14.3)	8.8
	Temperature (°C)	60	17.1	(15.6-17.9)	6.0

stand for first and third quartile, respectively) and the range between the maximum and the minimum values were selected as dispersion statistics.

As regards filtration, the main operating conditions were as follows: J₂₀ from 7 to 11 litre per square metre of membrane per hour (LMH) (set in MT1); TS entering the membrane tank from approx. 10 to 25 g L⁻¹; sludge recycling flow in membrane tank of 2.7 m³ h⁻¹; and SGD_m of 0.23 m³ h⁻¹ m⁻².

Influent characterisation

Table 2 shows the influent characterisation for the three operating periods evaluated in this study. Influent organic load and derivative features (T-COD, TSS, VFA, organic loading rate (OLR), and T-COD/S-SO₄) were log-normally distributed due to regular high-loaded discharges to the full-scale WWTP, which right-skewed both influent particulate and soluble organic-load features. Therefore, geometric mean and standard deviation (S.D.) rather than their arithmetic analogues were used for log-normally distributed features as position and dispersion statistical descriptors, respectively.

Sludge production and energy recovery potential assessment

In this study, waste sludge production (WSP) has been defined as the ratio between VS_{Wasted} and COD_{Removed} during a given period:

$$WSP = \frac{VS_{Wasted}}{COD_{Removed}}$$

Energy recovery potential was evaluated by means of the total methane production (i.e. dissolved and biogas CH₄) per COD removed and methane recovery efficiency

Table 2 | Influent characterisation for the different operating periods evaluated

Parameter	Period	n	Mean	S.D.	Q ₁ -Q ₃	Range
T-COD (mg O ₂ · L ⁻¹)	Summer	22	468	2	(300–685)	854
	Fall	30	543	2	(354–737)	969
	Winter	38	598	2	(449–889)	1,210
% S-COD	Summer	6	15.3	7.2	(8.2–21.5)	19.1
	Fall	8	14.6	4.9	(10.8–20.2)	12.3
	Winter	9	9.2	3.6	(6.1–12.3)	10.9
TSS (mg TSS · L ⁻¹)	Summer	22	233	2	(133–397)	620
	Fall	29	267	2	(170–428)	738
	Winter	38	308	2	(218–517)	802
% VSS	Summer	21	82.4	6.7	(78.5–84.5)	29.0
	Fall	21	78.3	7.7	(74.5–84.0)	33.0
	Winter	33	82.0	8.8	(78.0–86.0)	43.0
OLR (g O ₂ · d ⁻¹ · m ⁻³)	Summer	21	343	2	(249–503)	635
	Fall	29	870	2	(600–1,237)	1,857
	Winter	37	1060	2	(767–1,410)	1,547
SO ₄ -S (mg S · L ⁻¹)	Summer	8	342.7	31.3	(311.1–364.8)	90.9
	Fall	13	322.1	29.0	(305.0–335.7)	113.5
	Winter	14	300.3	24.8	(275.5–319.6)	78.2
T-COD/ SO ₄ -S (mg S · mg ⁻¹ O ₂)	Summer	4	5.16	1.46	(3.65–7.25)	3.74
	Fall	7	5.18	1.50	(3.61–6.91)	6.24
	Winter	9	7.31	1.69	(4.55–10.99)	11.12
VFA (mg CH ₃ COOH · L ⁻¹)	Summer	15	4.2	1.9	(0.6–5.0)	16.5
	Fall	28	4.2	1.7	(2.8–6.3)	10.4
	Winter	36	7.3	2.6	(3.5–19.7)	31.2

($\eta_{\text{Recovery}}^{\text{CH}_4}$), which is defined as

$$\eta_{\text{Recovery}}^{\text{CH}_4} = \frac{\text{CH}_4^{\text{Biogas}}}{\text{CH}_4^{\text{Biogas}} + \text{CH}_4^{\text{Dissolved}}} \cdot 100$$

RESULTS AND DISCUSSION

COD removal

To assess the system performance in terms of COD removal, statistical inferences were based on logarithms of T-COD and OLR data instead of on raw data, given that logarithms of log-normally distributed data are normally distributed. The Kolmogorov–Smirnov–Lilliefors test and Levene's test were performed to assess normality and equality of variances, respectively, prior to the analysis of variance (ANOVA). The ANOVA test stated that influent T-COD concentration was similar in the different operating periods considered (P -value = 0.105). Figure 2(a) shows both influent and effluent COD concentration.

Influent COD exhibited similar dispersion in the different operating periods. However, there were differences in OLR between the different periods (P -value = 0.000). Bonferroni's correction showed that OLR in summer was different from OLR in fall and winter, but no significant differences were found between OLR in fall and winter. Therefore, differences in OLR between summer and fall, and between summer and winter, can be mainly attributed to the lower HRT in fall and winter than in summer.

Figure 2(b) shows the COD removal percentage and the VFA concentration in the effluent. Overall, COD removal percentage slightly increased as the HRT decreased (89.7 ± 4.3 , 92.4 ± 4.2 , and 94.0 ± 5.5 , for summer, fall and winter periods, respectively), since an increasing particulate-matter fraction was retained in the system (due to increasing OLR). COD removal percentage was not dependent on the filtration process since membranes promoted total particulate COD retention in the system. Therefore, variations in COD removal percentage were mostly linked to the biological process performance due to variations in the removal of soluble COD. Nevertheless, an adequate

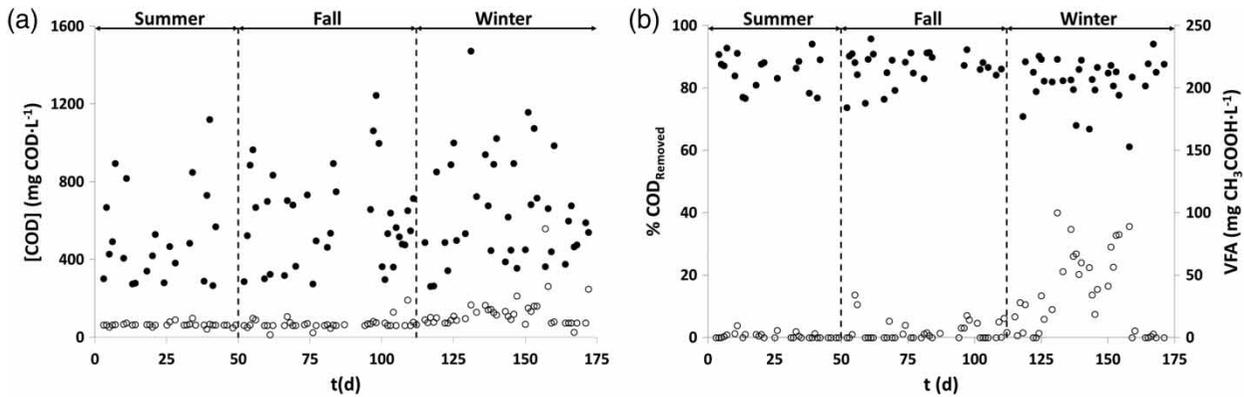


Figure 2 | (a) Influent (●) and effluent (○) COD during the experimental period. (b) % of COD removal (●) and effluent VFA (○) during the experimental period.

equilibrium between the different stages of the anaerobic organic-matter degradation prevented intermediary products from accumulating during summer and fall periods, resulting in an effluent VFA concentration close to zero. However, the operational conditions during the winter period (SRT = 28.6 d; HRT = 12.1 h, and $T = 17.1$ °C) promoted a temporary VFA accumulation in the system, which resulted in an effluent VFA concentration increase between days 125 and 158. As can be seen in Figure 2(b), VFA accumulation significantly affected the COD removal percentage. After day 158, the effluent VFA concentration decreased, indicating that the equilibrium condition between the different stages of anaerobic degradation was recovered. Therefore, COD removal was also high for the winter period. The performance indicators were calculated in this study for the last part of each operating period, where TS and VS concentration remained nearby stable. These pseudo-steady conditions showed that hydrolysis counterbalanced particulate matter accumulation.

Sludge production

Previously, COD removal has been stated to be virtually total in AnMBR systems due to membrane retention, as long as VFAs are prevented from accumulating. Furthermore, low HRTs have been suggested to promote high COD removals since large amounts of particulate matter are retained. On the other hand, the amount of particulate matter increases in the system as long as it is neither hydrolysed nor wasted. Therefore, besides HRT, hydrolysis rate and SRT will define the particulate-fraction features of the mixed liquor that determine the VS wastage.

Figure 3(a) shows the evolution of TS and VS concentration, and the %VS in the system throughout the different operating periods. VS percentage exhibited an opposite trend compared to temperature (see Figure 1 for temperature trend). This relation can be explained by the decrease with temperature of hydrolysis and microorganism growth rate (Smith *et al.* 2012). Therefore, a linear regression

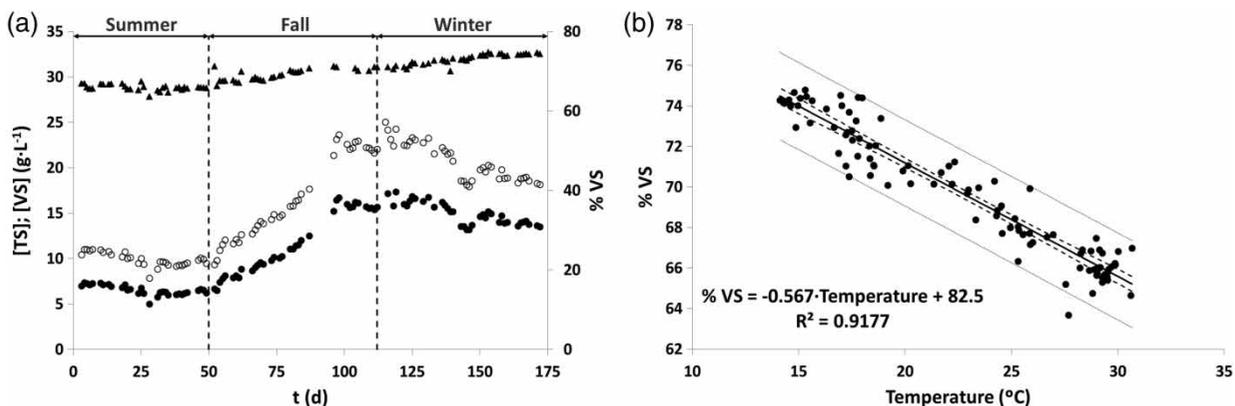


Figure 3 | (a) TS (○) and VS (●) concentration, and % of VS (▲) during the experimental period. (b) Linear regression for temperature–%VS pairs of values. Continuous line represents the fitted linear model. Dashed and dotted lines represent confidence and prediction bands (95%), respectively.

was fitted to temperature–%VS pairs of values. Figure 3(b) shows the fitted regression model. Every single linear-regression model assumption (i.e. linearity, independence and normality of residuals, and homoscedasticity) was successfully fulfilled.

According to Figure 3(b), temperature drop between summer and winter successfully accounted for the %VS increase shown in Figure 3(a). Therefore, %VS can be suitably estimated in this study by means of temperature, according to the following equation:

$$\%VS = 82.5 - 0.567 \cdot \text{Temperature} \pm 1.648 \cdot \sqrt{1 + \frac{1}{54} + \frac{(\text{Temperature} - 22.3)^2}{1634.9}}$$

Furthermore, as can be seen in Figure 3(a), VS concentration remained stable at around 6.5 g L^{-1} during the summer period, but it increased during the fall period and subsequently decreased during the winter period. Besides the increase in %VS due to the temperature drop from summer to winter periods, the HRT reduction from summer to fall periods brought about further particulate matter accumulation. On the other hand, the reduction in SRT from fall to winter periods brought about a decrease in both TS and VS, given the higher waste-sludge flow rate.

Figure 4 shows the amounts of COD removed and VS wasted, and the WSP. The increase in the amount of VS wasted from summer to fall periods was related to the VS concentration increase throughout the mentioned periods, whereas the increase in COD removal was related to the HRT reduction. On the other hand, the SRT reduction

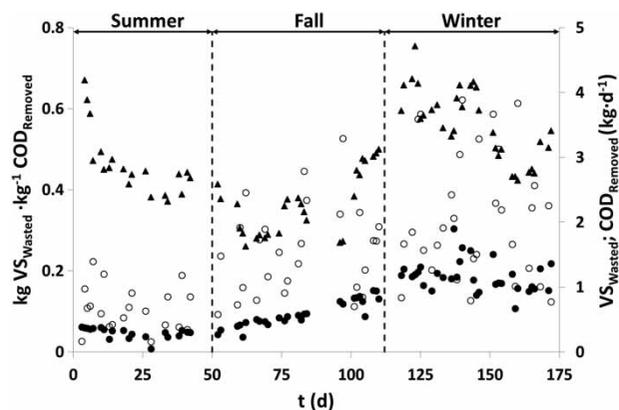


Figure 4 | WSP (▲), and COD removed (○) and VS wasted (●) during the experimental period.

from fall to winter periods contributed to firstly increasing the amount of VS wasted as a result of a waste-sludge flow rate increase. As mentioned before, the higher waste-sludge flow rate brought about a TS and VS concentration drop that finally reduced the amount of VS wasted. Bonferroni post-hoc correction for the amount of COD removed in each period showed that the average COD removed during winter and fall periods was not significantly different (P -value = 0.576).

The average values for WSP were 0.37 ± 0.01 , 0.42 ± 0.01 , and $0.46 \pm 0.02 \text{ kg VS}_{\text{Wasted}} \text{ kg}^{-1} \text{ COD}_{\text{Removed}}$ for summer, fall, and winter periods, respectively. WSP experienced a sharp decrease at the early fall period, given that the HRT reduction promoted an immediate COD removal increase, whilst VS wastage was gradually increased as particulate matter accumulation increased. In contrast, a sudden increase in WSP was observed at the early winter period. The SRT reduction brought about a higher waste-sludge flow rate than in summer and fall periods, which increased the sludge wastage. The higher sludge wastage promoted a particulate matter reduction in the system, which contributed to gradual reduction of the WSP.

Energy recovery potential

Figure 5 shows the evolution of both the total methane production per COD removed, and the methane recovery efficiency. As opposed to WSP, both total methane production and methane recovery efficiency decreased from summer to winter periods. On the one hand, the temperature drop promoted a hydrolysis-rate slowing down, which in turn reduced the substrate availability for fermentative

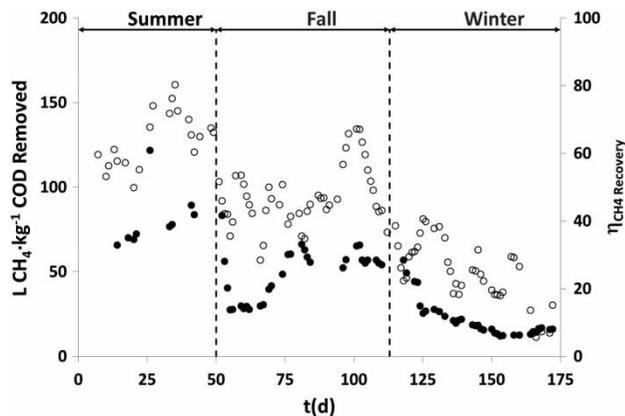


Figure 5 | Total CH_4 production (●) and CH_4 recovery efficiency (○) during the experimental period.

and methanogenic microorganisms. On the other hand, methane solubility inversely depends on temperature. Therefore, the dissolved methane fraction increased as the temperature dropped.

The average values for the total methane production per COD removed were 82.1 ± 5.8 , 60.1 ± 5.1 , and 15.4 ± 1.3 L CH₄ kg⁻¹ COD_{Removed} for summer, fall, and winter periods, respectively. On the other hand, the methane recovery efficiency was 71.0 ± 6.6 , 62.5 ± 5.2 , and $9.1 \pm 3.7\%$ for summer, fall, and winter periods, respectively.

Membrane performance

Despite the dynamics in the TS concentration entering the membrane tank, K₂₀ remained stable at around 100 LMH bar⁻¹ during the experimental period shown in this study. Therefore, no significant irreversible fouling was observed. Nevertheless, since different SRTs and temperatures were evaluated (see Table 1) different propensities for irreversible fouling were expected. In this respect, EPS seem to be a key factor affecting irreversible fouling in MBRs (Le-Clech *et al.* 2006), which is directly dependent on temperature (Robles *et al.* 2013) and SRT (Meng *et al.* 2009). Usually, EPS decrease as SRT increases, whilst EPS increase as temperature increases due to greater microbial activity. Therefore, irreversible fouling may be assumed to be a function of both temperature and SRT. Nonetheless, irreversible fouling rate remained nearby stable in this study (dK₂₀/dt of approx. -0.1 LMH bar⁻¹ d⁻¹). This was mainly attributed to the presence of similar amounts of EPS in the mixed liquor throughout the whole experimental period (around 35 ± 3 mg g⁻¹ VS and 126 ± 10 mg g⁻¹ VS measured as carbohydrate and protein, respectively). Since the operating period spanning the highest SRT (41.1 days) was conducted at the highest temperature (29.2 °C), the operating period spanning the medium SRT (39.5 days) was conducted at the medium temperature (24.2 °C) and the period spanning the lowest SRT (28.6 days) was conducted at the lowest temperature (17.1 °C), it was expected that the impact of both SRT and temperature on irreversible fouling would offset each other.

CONCLUSIONS

The effect of seasonal temperature variations on sludge production, energy recovery potential, COD removal and K₂₀ was evaluated in an AnMBR fitted with industrial-scale membrane units. The percentage of VS showed an opposite trend compared to temperature mainly due to a decrease with

temperature of hydrolysis and microorganism growth rate. Hence, sludge production rose as temperature dropped due to increasing VS concentration. Energy recovery potential decreased from summer to winter periods since the temperature drop promoted a hydrolysis-rate slowing down and increased the methane dissolved in the effluent. COD removal remained more or less stable throughout the whole experimental period. Irreversible fouling rate remained stable due to similar amounts of EPS in the mixed liquor.

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