



A novel configuration for an anaerobic submerged membrane bioreactor (AnSMBR). Long-term treatment of municipal wastewater under psychrophilic conditions



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HIGHLIGHTS

- Viability of the new membrane configuration for treating municipal wastewater.
- The configuration of the AnSMBR allows better distribution of the accumulated solid.
- Solid concentration and biogas sparging; main parameters for long-term operation.
- Three years of continuous operation without physical and chemical cleaning.

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ABSTRACT

A novel design for a pilot scale anaerobic submerged membrane bioreactor (AnSMBR) equipped with an ultrafiltration unit, treating municipal wastewater at 18 ± 2 °C, and inoculated with a mesophilic inoculum without acclimation, was implemented and evaluated over 3 years of stable operation. The AnSMBR operated with a volumetric loading rate between 1.6 to 2.0 kg COD/m³U_{ASB} d, 12.8 to 14.2 h hydraulic retention time, and reached a tCOD removal efficiency of around 90%. Biosolid production was between 0.05 and 0.083 g VS/g COD_{removed}. Dissolved methane oversaturation in the effluent was observed, reaching average values of 19.1 ± 0.84 mg CH₄/L. The permeate flow rate ranged from 10 to 14 L/m² h with trans-membrane pressure (TMP) values of 400–550 mbar, using cycles of 30 s backwash, 7.5 min filtration, and continuous biogas sparging (9–16 m/h). During the three years of continuous operation, the membrane was not physically or chemically cleaned.

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1. Introduction

The anaerobic treatment of wastewater has many advantages over aerobic treatment. It requires low energy consumption and low macro/micro-nutrients, while it provides low wastage of biological solids, and transforms the organic matter into valuable biogas. On the other hand, it has some disadvantages such as process sensitivity, vulnerability, odor problems, long start-up period, and post treatments in order to achieve discharge standards. In addition, during anaerobic treatments, greenhouse gas emissions are lower in comparison to aerobic technologies, if methane is used as an energy source (Bialek et al., 2014; Lew et al., 2009). On the contrary, the loss of dissolved methane in the effluent should be avoided, not only due to the loss of energy, but also because of

its higher global warming potential (Matsuura et al., 2015; Smith et al., 2013). Over the past years, anaerobic membrane technology has been of growing interest and has been investigated for the treatment of different wastewaters, including municipal and industrial wastewaters (Lin et al., 2013; Ozgun et al., 2013; Skouteris et al., 2012; Smith et al., 2012). Anaerobic membrane technology brings together the advantages of anaerobic processes with the production of solid free effluent, which provides an appropriate alternative to complete biomass retention, enabling an independent control of the hydraulic residence time (HRT) and the solid retention time (SRT). One of the goals of anaerobic treatment processes is to maintain a long SRT because of the slow growth rate of anaerobic microorganisms, especially when operating at psychrophilic conditions and with low strength wastewater, such as municipal wastewater (van Lier et al., 2001). The anaerobic membrane bioreactor (AnMBR) produces a better effluent quality in terms of suspended solids, chemical oxygen demand (COD), and

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pathogen count. Furthermore, there is a possibility of reusing and recycling the treated effluent for non-drinkable purposes and for agricultural irrigation. The anaerobic membrane technology is a viable technology for the treatment of municipal wastewater at psychrophilic conditions in cold countries (Smith et al., 2013, 2015). Nevertheless, due to the low temperature and the low strength wastewater, the recovery of dissolved methane is key to approaching energy-neutral operation for domestic wastewater treatment (Smith et al., 2012). The membrane fouling and the low permeate flux compared with the aerobic membrane bioreactor are the main disadvantages of anaerobic membrane technology. The AnMBR has two possible configurations, submerged and side-stream. In the former, the membrane is directly immersed in the mixed liquor, while in the latter the membrane is located outside the bioreactor and the mixed liquor circulates through it at an elevated flow, which is required to achieve a high cross flow velocity. Nowadays, submerged AnMBR is the more commonly applied configuration due to its lower energy consumption (Judd, 2006). In addition, the shear stress caused by the high velocity of the side-stream configuration has a negative effect on the microbial activity.

In the submerged configuration, the membrane is placed inside the reactor or inside an external tank. Depending on the type of the reactor or the degree of mixing, the placement of the membrane inside the reactor or inside an external tank could contribute in different ways to the fouling of the membrane, and therefore to the different energy consumption in long-term maintenance. In a previous study, Gouveia et al. (2015), operating a pilot scale AnMBR with a submerged membrane located in an external tank connected to the upflow anaerobic sludge blanket reactor (UASB), observed an accumulation of solids at the bottom of the membrane module. This suspension showed poor flow ability and sedimentability characteristics, impeding its recirculation to the UASB reactor, thus preventing adequate contact with the active biomass and consequently its possible biodegradation. In addition, the accumulation of this suspension contributes to the increase in solid concentration in the area surrounding the membrane, and therefore to the increased fouling of the membrane. In order to facilitate the circulation of solids between the membrane section and the biological section, and to try to reduce the fouling of the membrane, this paper proposes a novel configuration in which the membrane is placed on top of the UASB reactor. Thereby, the aim of this work was to experimentally study the general performance of a pilot scale AnSMBR with a novel membrane configuration, UASB reactor and filtration section in a single unit, and determine the long-term feasibility of the treatment of municipal wastewater under psychrophilic conditions.

2. Methods

2.1. AnSMBR configuration

Fig. 1 shows the experimental pilot plant set up. Raw sewage is pumped from the municipal sewer to a rotary sieve (defender TR-40/25 Toro Wastewater Equipment Industries), pre-treated in a circular primary settler (with a total volume of 25 L and HRT between 1 and 3 h) and then pumped to the UASB reactor with a submerged ultrafiltration membrane placed on top of the reactor. In order to improve settling ability, two baffles are placed between the three-phase separator and the ultrafiltration section. The total volume of the UASB reactor was 326 L, (useful volume of 284 L). The filtration section (total volume = 175 L) was equipped with two ultrafiltration membrane modules (ZW-10 Zenon, GE), with a mean pore size of 0.045 μm , and a filtration area of 0.93 m^2 /module. Both settler and reactor were placed in a room provided with an

air-conditioned system in order to control the temperature of the UASB reactor at 18 °C. The pilot plant was equipped with biogas flow meters, pressure meters, and two temperature sensors. One sensor was placed at the bottom of the UASB reactor (1 m from the bottom), and the other one was placed on top, in the filtration section (1 m from the top). Temperature and pressure filtration were stored online using a data acquisition system by Pico Technology.

In order to control the membrane fouling and to maintain the TMP biogas sparging, relaxation time, and permeate back-flush were used. The initial operation cycle was fixed at 15 min filtration, 10 s relaxation time, 1 min back-flush and a further 10 s relaxation time. The biogas was continuously sparged, (coarse bubbles) at the bottom of the hollow fibers with a superficial velocity of between 8 and 16 m/h. The operation of the AnSMBR was divided into two periods; Period I, before reinoculation (day 0–308), and Period II, after reinoculation (day 309–1060). During Period II, the AnSMBR was operated without recirculation from the filtration section to the bottom of the UASB reactor (Period II.1 from day 309 to day 491) and with recirculation (Period II.2 from day 492 to day 1060). The upflow velocity operating without recirculation was 0.12 ± 0.03 m/h, and with recirculation 0.34 ± 0.03 m/h.

2.2. Inoculum and feed wastewater

The reactor was inoculated with granular sludge (6.6 kg of volatile solid (VS)) from a mesophilic anaerobic reactor treating wastewater from a paper recycling factory without any previous acclimatization to psychrophilic conditions. After 10 months of operation, the reactor was reinoculated with granular sludge (7.5 kg VS) from the same mesophilic anaerobic reactor also without acclimation. The pilot plant was fed with raw municipal wastewater from the city of Valladolid obtained from a nearby sewer.

2.3. Chemical assays and sampling

Samples were taken twice a week from the pilot plant to monitor the process performance. The sampling points (S.p.) 0, 1, 2, 3, 5 and effluent (4), are shown in Fig. 1. Alkalinity, tCOD, sCOD, total solids (TS), volatile solids (VS), total suspended solids (TSS), volatile suspended solids (VSS), biological oxygen demand (BOD_5), total nitrogen (N-TKN) and ammonia nitrogen (N-NH_4^+) were determined according to the Standard methods for the examination of water and wastewater (APHA. et al., 2005). sCOD was determined following sample filtration through 0.45 μm . The concentrations of volatile fatty acids (VFAs) were determined by gas chromatography using Agilent 7820A GC-FID equipped with a G4513A autosampler and a Chromosorb WAW packed column (2 m \times 1/8" \times 2.1 mm SS) (10% SP 1000, 1% H_3PO_4 , WAW 100/120) (Teknokroma, Spain). Nitrate nitrogen (NO_3^- -N), nitrite nitrogen (NO_2^- -N), chloride (Cl^-), sulfate (SO_4^{2-}) and soluble phosphorus (P-PO_4^{3-}) concentrations were analyzed by HPLC-IC using a Waters 515 HPLC pump (Waters, Milford, USA) coupled with an ion conductivity detector (Waters 432, Milford, USA) and equipped with an IC-Pak Anion Guard-Pak column (Waters, Milford, USA) and an IC-Pak Anion HC (150 mm \times 4.6 mm) column (Waters, Milford, USA). Biogas composition was analyzed using a gas chromatograph (Varian CP-3800, Palo Alto, CA, USA) coupled with a thermal conductivity detector and equipped with a CP-Molsieve 5A (15 m \times 0.53 mm \times 15 μm) and a CP-Pora BOND Q (25 m \times 0.53 mm \times 15 μm) columns. Helium was used as the carrier gas.

2.4. Determination of dissolved CH_4

The methodology used for the sampling and determination of dissolved methane in the reactor and in the effluent of the AnSMBR was as follows:

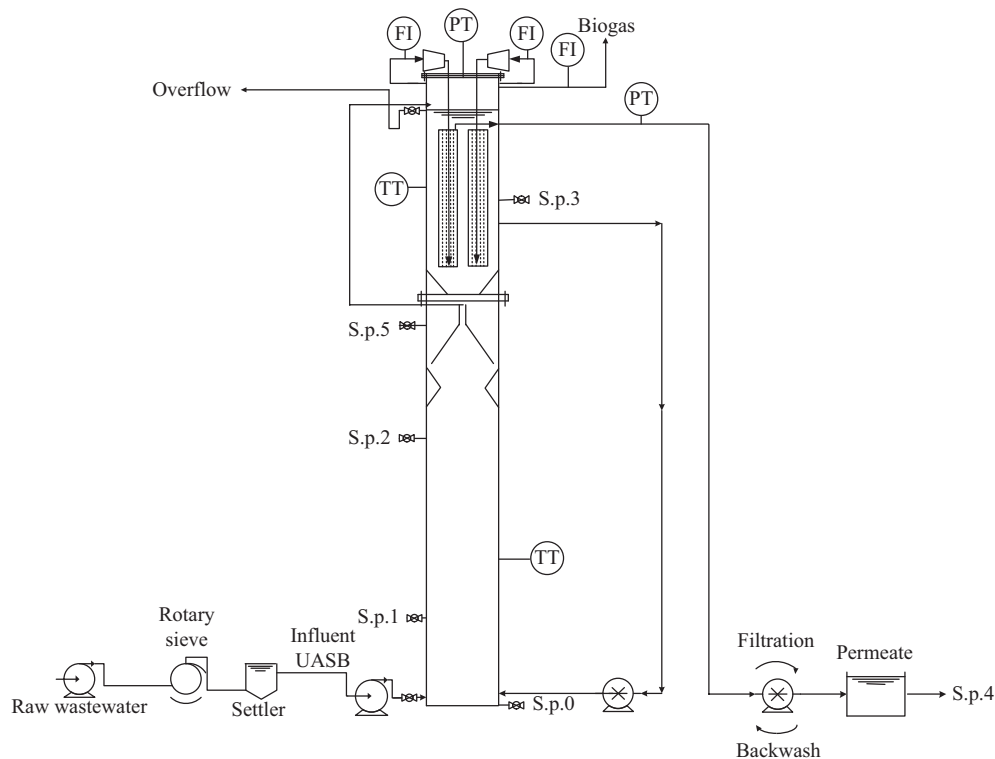


Fig. 1. AnSMBR pilot plant flow scheme (FI – flow-rate indicator; PT – pressure transmitter; TT – temperature transmitter; S.p. – sampling points).

1. A known volume of liquid was carefully collected in 2.15 L glass bottles avoiding any turbulence. The bottles were immediately closed with rubber stoppers and sealed with aluminum caps.
2. After filling, the bottle was manually agitated for 3 min in order to desorb the dissolved methane in the liquid phase.
3. Next, the pressure in the head space was measured and the gas contained was sampled and analyzed to determine the gas composition. The amount of desorbed methane was calculated according to the ideal gas behavior. (At the end of the experiment, the volume of the gas phase was assessed by calculating the difference between the total volume of the bottle and the exact measured volume of the liquid phase.)
4. The bottles were kept from 3 to 4 h at a controlled temperature in order to reach equilibrium between the gas phase and liquid phase.
5. Finally, the head space of the bottle was again sampled and analyzed to determine the gas composition. The remaining methane dissolved in the liquid phase was calculated according to Henry's Law, with a constant value of $1.48 \times 10^{-3} \text{ mol atm}^{-1} \text{ L}^{-1}$ at 20 °C for water (Metcalf & Eddy, 2003).

The total amount of dissolved methane in each sample was calculated taking into account the desorbed methane plus the methane in the liquid phase when in equilibrium with the desorbed gas phase. The percentage of oversaturation was calculated on the basis of the theoretical value of methane dissolved in the liquid phase, calculated according to Henry's Law using the constant formerly mentioned for clean water. It was calculated considering local atmospheric pressure for the effluent, the local atmospheric pressure plus the water column (1.9 m) for sampling point 2, and taking into account a methane composition in the biogas phase of 80% for both samples. The conductivity of the wastewater was around 950–1400 $\mu\text{S/cm}$.

2.5. Biochemical methane potential (BMP) assays

Biochemical methane potential assays at mesophilic conditions were carried out from the solid waste purged from the filtration section. The substrate/inoculum ratio selected for the BMP assays was between 0.23 and 0.40 g VS/g VS. The methodology used for the BMP assay is described in detail in a previous work (Gouveia et al., 2015).

3. Results and discussion

The UASB was continuously fed with municipal wastewater, pre-treated in a rotary sieve (1 mm mesh) and a primary settler. The main characteristics of the wastewater feeding the UASB are listed in Table 1. Particulate COD represents between 28% and 46% of tCOD. The pH of the wastewater was constant throughout the operation with values around 7.2 ± 0.6 . The alkalinity of the wastewater reached values of $494.7 \pm 40.4 \text{ mg CaCO}_3/\text{L}$. As shown in the figures, the reactor operated continuously for 1060 days. The whole operation is divided into three periods: I. Start-up, II.1 operation without recirculation and II.2 operation with recirculation.

Table 1
Pretreated wastewater characteristics fed to the UASB (average values).

Parameter	Influent (mg/L)
tCOD	978 ± 210
sCOD	610 ± 146
tBOD ₅	474 ± 203
sBOD ₅	329 ± 146
TS	83.0 ± 8.63
VS	51.2 ± 8.13
N-TNK	92 ± 10
N-NH ₄ ⁺	75 ± 16
P-PO ₄	10 ± 2
SO ₄ ²⁻	47 ± 25

3.1. Start-up

The UASB reactor was started-up with a food/microorganism ratio of 0.06 g tCOD/g VS d and gradually increased by decreasing the hydraulic residence time. As shown in Fig. 2 during Period I (from day 0 to day 308), after 3 months of operation with tCOD removal efficiencies of around 70%, the coincidence of two negative factors (two weeks out of operation due to pumping problems from the sewer, as well as a very low temperature of 12 °C contributed to VFA accumulation (370 mg AcH) and the subsequent loss of COD removal efficiency (<50%). In order to recover the removal efficiency and to avoid VFA accumulation, the action undertaken was to decrease the influent flow rate, while simultaneously increasing the inlet temperature to 18 °C. This action was sufficient to improve the performance of the COD removal when operating at a high hydraulic residence time (HRT = 56–28 h). Nevertheless, the removal efficiency decreased markedly with increasing organic loading rates, even after 75–100 days of the episode of acidification. Methanogenic activity tests, using the sludge from the bottom of the UASB reactor, were carried out at 18 °C, obtaining values of 0.004 g COD/g SV d and thus indicating that at low temperature it is very difficult to recover normal conditions after a severe episode of VFA inhibition. In order to recover this activity, part of the sludge was purged and the reactor was reinoculated (day 308) with granular sludge. As a result of this reinoculation, the reactor could be operated with higher

volumetric loading rates, although with a slow but progressive decrease in the hydraulic residence time in order to obtain a higher COD removal efficiency. This behavior indicates a slow adaptation to psychrophilic conditions. The slow adaptation to psychrophilic conditions was also observed in a previous work of Gouveia et al. (2015) operating an AnMBR and inoculated with flocculent sludge from a mesophilic anaerobic digester from the municipal wastewater treatment plant of Valladolid. A long start-up period is needed to obtain higher tCOD removal when mesophilic sludge is used to inoculate a psychrophilic reactor. Shin et al. (2014), worked with an anaerobic fluidized membrane bioreactor (SAF-MBR) at temperatures of at 8–15 °C during the first winter. At this point, before acclimation, removal efficiencies of 81% and 85% were obtained for COD and BOD₅ respectively. After becoming fully acclimated, during the following summer and winter the COD removals increased to 94% and 90%. Nevertheless, McKeown et al. (2009), working with a laboratory-scale expanded granular sludge bed-anaerobic filter (EGSB-AF) hybrid bioreactor between 15 °C and 4 °C, and treating synthetic, volatile fatty acid (VFA)-based wastewater, concluded that mesophilic inocula can physiologically adapt to sub-optimal operational temperatures, reaching efficiencies and sludge loading rates at 4 °C (day 1243) which are comparable to those achieved at 15 °C (day 0). Bowen et al. (2014) indicate that one of the strategies for seeding low-temperature anaerobic reactors is the use of mesophilic sludge acclimated to low temperatures. They conclude that the sludge sourced from a

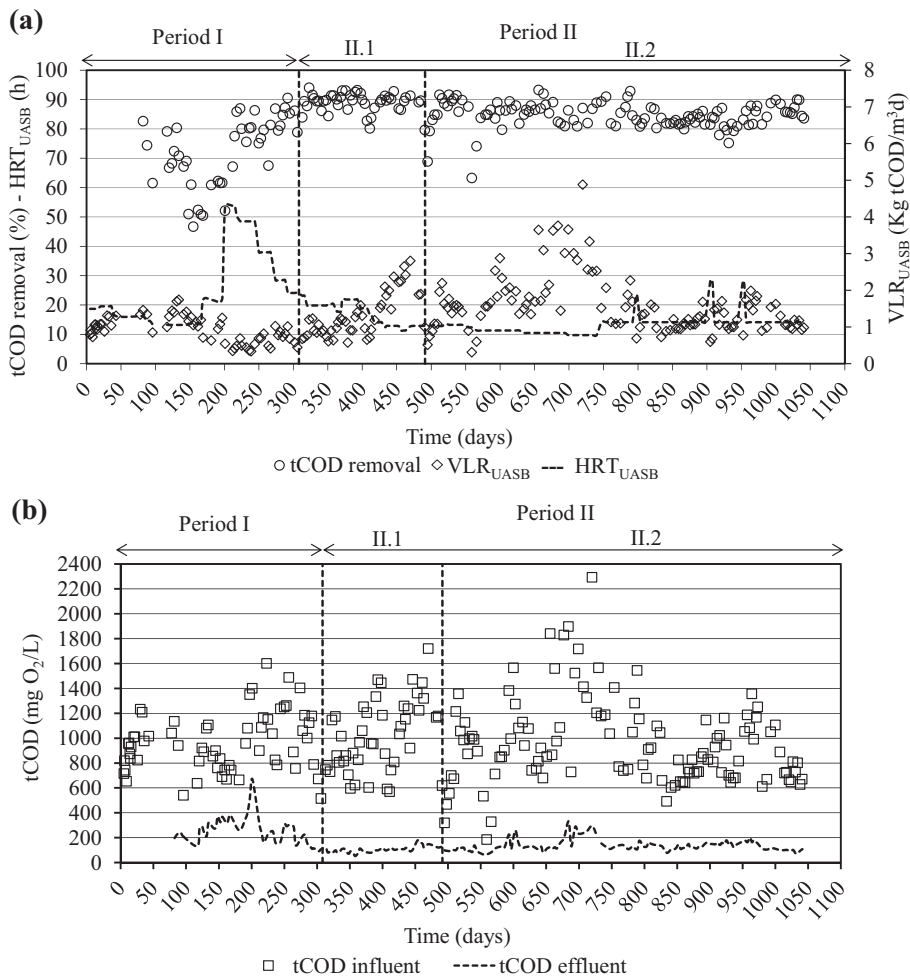


Fig. 2. (a) Evolution of tCOD removal efficiency of the AnSMBR, VLR_{UASB} and HRT_{UASB}. (b) Evolution of tCOD in the influent and effluent of AnSMBR during the period of operation.

bioreactor acclimated to treating domestic wastewater at 15 °C would not be able to function below 8 °C.

3.2. COD removal efficiency

Fig. 2 shows the tCOD removal efficiencies, volumetric loading rate (VLR), HRT and the tCOD of the influent and effluent of the AnSMBR during the operation of the pilot plant. The parameters VLR and HRT are both calculated considering the useful volume of the biological section. Table 2 summarizes the average results obtained for pseudo-steady state periods with different HRTs. During the operation without recirculation (Period II.1) the VLR was gradually increased from 1 to 2.4 kg tCOD/m³ d by decreasing the HRT in the biological section from 20 to 11 h, leading to a decrease in the quality of the effluent from 93.7 ± 11.2 mg/L to 147 ± 17.5 mg/L. The respective BOD₅ increased from 30 mg/L to 75 ± 5 mg/L.

As can be seen in Table 2, the results obtained during the recirculation period (Period II.2) were similar to those obtained during Period II.1. By operating with VLR between 1.84 ± 0.27 kg tCOD/m³ d (HRT = 14 h) and 2.7 ± 0.85 kg tCOD/m³ d (HRT = 10.5 h) the tCOD of the effluent increased from 134 ± 26 mg/L to 176 ± 80 mg/L. When the VLR increased to 3.18 kg tCOD/m³ d (HRT = 9.8 h), the tCOD of the effluent increased to 225 ± 58 mg/L.

The results obtained in this work confirm that the AnSMBR treating municipal wastewater at psychrophilic temperature (18 ± 2 °C) achieves a removal efficiency of 89.6 ± 2%, reaching values of the tCOD in the effluent around 120 mg tCOD/L at HRT of 13–14 h and VLR between 1.5–2 kg tCOD/m³ d. Comparing these results with those obtained in a previous work (Gouveia et al., 2015) with a different AnMBR configuration and fed with identical wastewater but inoculated with flocculent sludge, it was observed that the current work obtained similar percentages of tCOD removal, but operating with higher HRT, between 13–14 h, as opposed to 8–10 h in the previous work. This difference could be due to the different adaptation and evolution of each inoculum to the psychrophilic conditions. The specific methanogenic activity

at 18 °C of the sludge from the bottom of the UASB reactor was slightly lower in the present work than in the previous work, between 0.04–0.06 g COD/g VS d as opposed to 0.05–0.11 g COD/g VS d respectively. Similar tCOD removal was found in the literature consulted. Dong (2015), working with a pilot AnMBR fed with sewage from the Burlington Skyway Wastewater Treatment Plant, at 23 ± 1 °C, with HRT of 8.5 h and VLR between 0.858–1.09 kg tCOD/m³ d, obtained COD removal efficiencies between 88 ± 3.9%–91.8 ± 1.9%. Huang et al. (2013) with three submerged anaerobic membrane bioreactors (SAnMBRs), at 25–30 °C, VLR of 1.02 ± 0.14 kg tCOD/m³ d, achieved total COD removal efficiencies of 84 ± 6%, 85 ± 3%, 86 ± 3%, respectively. Martin-Garcia et al. (2011) working with a granular-AnMBR, HRT of 16 h, VLR of 0.51 kg tCOD/m³ d, upflow velocity of 1 m/h, and fed with real domestic wastewater and without external temperature control, obtained a similar COD removal of 86%.

VFA in the effluent of the AnSMBR were practically not detected for VLR lower than 1 kg COD/m³ d and it was around 22 ± 15.4 mg/L for VLR between 2–2.2 kg COD/m³ d, operating without recirculation (Period II.1). The VFA composition was acetic, propionic and butyric acid at 56 ± 35.8%, 30.4 ± 28.4%, and 13.5 ± 6.8%, respectively. Similar results were observed operating with recirculation. At VLR of 1.84–1.91 kg COD/m³ d, the VFA concentration in the effluent was 29.5 ± 24.3 mg/L (composed by acetic and propionic acid at 54 ± 44.4%, 46 ± 38.1%, respectively). When the VLR increased to 3.18 kg COD/m³ d, the tCOD removal decreased and the VFAs concentration in the effluent increased to values of around 59 ± 31 mg/L, being mainly composed of acetic acid and propionic acid (49.6 ± 27.4%, 40.9 ± 24.8%).

3.3. Nitrogen and phosphorus removal

The N-TKN and N-NH₄⁺ were determined in the soluble phase of the influent, at sampling points S.p.2, S.p.3 and at the effluent of the AnSMBR. According to Table 1, most of the N-TKN in the influent was present in the form of N-NH₄⁺ (80.7 ± 7.2% of the total N-TKN). There was no significant increase in the concentration of

Table 2
Principal parameters of operation of AnSMBR (average values).

Time (d)	HRT _{UASB} (h)	VLR (kg tCOD/m ³ UASB d)	F/M (kg tCOD/kg VS _{UASB} d)	tCOD effluent (mg/L)	BOD ₅ effluent (mg/L)	CH ₄ ^c (L/g tCOD _{red})	tCOD removal efficiency (%)
320–344 ^a	19.9	0.97 ± 0.16	0.060 ± 0.010	93.7 ± 11.2	n.a.	0.22 ± 0.02	89.7 ± 2.2
348–358 ^a	20.3	0.72 ± 0.11	0.045 ± 0.007	73.7 ± 13.8	n.a.	0.19 ± 0.04	89.2 ± 2.8
411–418 ^a	15.9	0.94 ± 0.19	0.081 ± 0.016	113 ± 6.9	34.3 ± 0.2	0.26 ± 0.01	83.7 ± 2.8
420–428 ^a	14.2	1.56 ± 0.04	0.134 ± 0.003	112 ± 1.5	30 ± 5	0.22 ± 0.01	89.5 ± 0.4
433–453 ^a	12.8	2.00 ± 0.29	0.131 ± 0.018	120 ± 27.8	34 ± 9	0.20 ± 0.01	90.2 ± 1.8
456–463 ^a	11.4	2.44 ± 0.17	0.160 ± 0.011	147 ± 17.5	75 ± 5	0.18 ± 0.01	88.9 ± 1.7
470–485 ^a	12.9	2.12 ± 0.43	0.144 ± 0.028	133 ± 12.2	n.a.	0.18 ± 0.01	90.0 ± 0.9
	13.1	0.88 ± 0.20	0.058 ± 0.013	104 ± 10.5	34.3 ± 8.4	0.21 ± 0.02	80.1 ± 5.5
491–509 ^b							
519–545 ^b	13.3	1.55 ± 0.12	0.091 ± 0.007	105 ± 18.0	37 ± 9	0.21 ± 0.03	89.4 ± 1.8
554–566 ^b	12.0	0.60 ± 0.24	0.035 ± 0.013	73 ± 8.5	n.a.	0.19 ± 0.02	74.9 ± 9.9
572–628 ^b	11.4	1.91 ± 0.44	0.112 ± 0.025	151 ± 46.9	60 ± 20	0.17 ± 0.02	85.8 ± 2.2
635–651 ^b	10.5	1.59 ± 0.16	0.097 ± 0.009	117 ± 7.6	n.a.	0.18 ± 0.03	86.3 ± 0.9
656–694 ^b	10.5	2.70 ± 0.85	0.179 ± 0.059	176 ± 79.9	88 ± 49.9	0.19 ± 0.02	86.6 ± 4.1
699–740 ^b	9.8	3.18 ± 0.80	0.238 ± 0.050	225 ± 58.3	111.8 ± 52.8	0.18 ± 0.01	85.3 ± 2.9
761–775 ^b	14.2	1.10 ± 0.02	0.087 ± 0.002	130 ± 14.8	46.4 ± 5.6	0.18 ± 0.01	82.7 ± 1.9
782–792 ^b	14.2	1.84 ± 0.27	0.145 ± 0.021	134 ± 26.6	n.a.	0.14 ± 0.01	88.9 ± 3.2
803–908 ^b	15.3	1.11 ± 0.27	0.066 ± 0.015	132 ± 18.9	54.2 ± 5.9	0.19 ± 0.01	80.9 ± 1.9

n.a. – not analyzed.

^a Without recirculation (Period II.1).

^b With recirculation (Period II.2)

^c Produced + desorbed.

N-NH₄⁺ in the effluent of the AnSMBR as a result of the treatment process (83.2 ± 9.0% of the total N-TKN). Soluble phosphorous concentration underwent a similar trend, with no significant difference between the concentration of P at the soluble phase of the influent and effluent of the AnSMBR being recorded.

3.4. Biogas

The biogas composition in Period II.1 and Period II.2, without and with recirculation, respectively is reported in Table 3. These results showed that the recirculation had no significant effect on the composition of the biogas. There was no difference either in the hydraulic residence time, as shown in the work of Souza et al. (2011), where a clear increase in the methane composition was obtained by increasing the HRT. The N₂ composition was lower compared with the values from the literature. Matsuura et al. (2015) working with an UASB reactor, treating municipal sewage at ambient temperatures (10–28 °C), obtained nitrogen concentrations in biogas of between 25% and 33%.

The dissolved methane was estimated in order to determine the loss of methane in the effluent of the AnSMBR. Table 4 summarizes the methane desorbed (mg CH₄/L of wastewater treated) from the effluent of the AnSMBR (S.p.4) and from the UASB section (S.p.2) together with the percentage of oversaturation. As can be seen in Table 4, the mean value of desorbed methane from the biological section (S.p.2) was 39.2 ± 6.9 mg CH₄/L, while from the effluent of the AnSMBR it was 19.1 ± 0.9 mg CH₄/L. The results showed that the mean value of oversaturation in the UASB (S.p.2) was 109.1 ± 17.7%, while for the effluent of the AnSMBR it was 15.3 ± 5.3%. This significant difference could be because of a higher turbulence in the filtration section than in the biological section, due to the biogas sparging which contributes to the desorption of the dissolved methane and decreases the loss of methane in the effluent of the AnSMBR. Giménez et al. (2012) reported that biogas-assisted mixing avoided super-saturation and guaranteed the minimum concentration of dissolved methane at the effluent of the AnMBR. Nevertheless, Smith et al. (2015) reported average values of oversaturation of 2.2 ± 0.74 when treating domestic wastewater in an AnMBR at 15 °C, and similar results at 12 °C. In addition, significant differences are observed in the dissolved methane at the effluent from the UASB reactors working at psychrophilic conditions. Souza et al. (2011), when working at 25 °C, reported a degree of oversaturation of between 40% and 70% in the effluent of different UASB reactors. Urban et al. (2007), in a study carried out with municipal wastewater in a UASB pilot plant, obtained values of between 20 and 25 mg CH₄ dissolved/L when working at temperatures between 20–25 °C. Matsuura et al. (2015) described a supersaturated effluent in a pilot-scale UASB with an average of 13%.

3.5. Specific methane yield

The specific methane yield (SMY) was calculated by the ratio between the methane production and the tCOD removed. The tCOD removed was determined by subtracting the tCOD of the influent, and the tCOD of the effluent of the AnSMBR. The methane production was calculated taking into account the methane production

measured plus the methane dissolved in the effluent, considering a mean value of 18.8 ± 1.7 mg CH₄/L of wastewater treated.

No significant difference was observed in the SMY when operating without or with recirculation, the respective values obtained were 0.226 and 0.216 N m³ CH₄/g tCOD_{removed}. This would mean that the reactor operating without recirculation appears to be adequately mixed. This slight difference could indicate a good distribution of the accumulated particulate material in the new configuration. The value obtained indicates that around 60% of the tCOD is biologically removed and around 40% is accumulated in the system due to the ultrafiltration process, however it is not biodegraded. In a previous work by Gouveia et al. (2015), working with identical wastewater and temperature but with a different membrane configuration, obtained SMY values of 0.187 N m³ CH₄/g tCOD_{removed} without recirculation and 0.235 N m³ CH₄/g tCOD_{removed} with recirculation. In both configurations the SMY were below the theoretical value. The SMY value obtained in the current work is similar to that reported in literature. Martínez-Sosa et al. (2012), using an AnSMBR and treating low-strength wastewater under psychrophilic conditions (20 °C), obtained an average SMY of 0.24 N m³ CH₄/kg COD_{removed} and 0.29 L CH₄/g tCOD_{removed}, considering the theoretical dissolved methane. Lin et al. (2011) reported SMY of 0.26 L CH₄/g tCOD_{removed} by treating domestic wastewater in a submerged anaerobic membrane bioreactor. Gao et al. (2014) obtained a SMY of 0.19 L CH₄/g tCOD_{removed} working at 35 and 25 °C, and a value of 0.14 L CH₄/g tCOD_{removed} at 15 °C.

3.6. Accumulation of particulate COD

Fig. 3 shows the evolution of the sCOD inside the filtration section (S.p.3) and the tCOD of the effluent (S.p.4). As can be seen, there was a great difference between the tCOD of the effluent and the sCOD inside the filtration section. This difference was due to the ultrafiltration membrane, which fully retained inside the system any particulate/colloidal matter larger than 0.045 μm. This retention caused the accumulation of particulate matter. The accumulated materials were inorganic and organic solids that were non-biodegradable or slowly biodegradable from the wastewater, as well as cellular debris and biological organic material. The municipal wastewater fed to the UASB reactor had a particulate COD content at around 26–48% of the tCOD. The accumulation rate depends on the particulate material content of the wastewater, its biodegradability, the volumetric loading rate, the purges carried out, the recirculation between the filtration section and the biological section, and the configuration between the membrane and the reactor. As can be seen in Fig. 3, during the continuous purge, the sCOD inside the filtration section (S.p.3) remained almost constant, while it was seen to increase during the rest of the operating conditions, except in those cases when purges were performed or the feeding pump was stopped due to sewage failures. When operating without recirculation (Period II.1), working at VLRs of between 0.94 and 2.4 kg tCOD/m³UASB d, an increase of the tCOD accumulation rate was observed in the filtration section of 239–702 mg tCOD/L d respectively, while the accumulation rate of the soluble COD was practically constant between 82–126 mg sCOD/L d. Nevertheless, operating with recirculation (Period II.2) and working with VLRs of between 1 and 1.55 kg tCOD/m³UASB d, the accumulation rate in the filtration section was between 90–119 mg tCOD/L d and the soluble COD was around 14–18 mg/L d. This accumulation effect of particulate/colloidal material caused by the membrane has been reported in recent literature (Gouveia et al., 2015; Bae et al., 2014; Martínez-Sosa et al., 2012).

As previously mentioned, there was no significant effect of the recirculation on the specific methane yield. Nevertheless, the recirculation improved the degree of mixing. As Fig. 4 shows, tCOD at the filtration section (S.p.3) decreased and remained below the

Table 3
Biogas composition during Period II.1 and Period II.2.

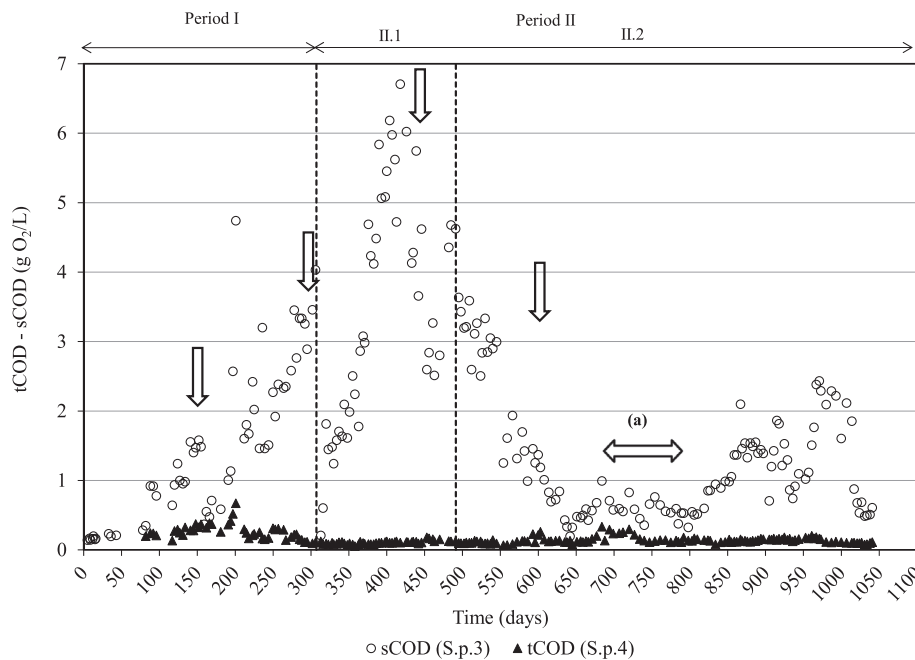
	Period II.1 (%)	Period II.2 (%)
CO ₂	9–12	5–10
H ₂ S	0.2–0.3	0.1–0.3
N ₂	6–9	7–12
CH ₄	80–83	81–83

Table 4

Desorbed methane to the gas phase and methane dissolved in the liquid phase after desorption, in samples from the UASB reactor (S.p.2) and from the effluent (S.p.4).

Day	UASB reactor (S.p.2)			Effluent AnSMBR (S.p.4)		
	Gas phase (mg CH ₄ /L _{H₂O})	Liquid phase (mg CH ₄ /L _{H₂O})	Oversaturation (%)	Gas phase (mg CH ₄ /L _{H₂O})	Liquid phase (mg CH ₄ /L _{H₂O})	Oversaturation (%)
517	39.4 ± 1.7	2.71 ± 0.11	98.5 ± 8.2	n.a.	n.a.	n.a.
590	43.7 ± 1.5	3.53 ± 0.12	122.7 ± 7.1	n.a.	n.a.	n.a.
609	45.2 ± 1.6	3.47 ± 0.01	129.5 ± 7.4	19.4 ± 1.5	1.53 ± 0.12	18.1 ± 8.5
624	44.7 ± 1.6	3.43 ± 0.11	126.9 ± 7.3	19.5 ± 1.6	1.45 ± 0.12	18.2 ± 8.9
636	43.6 ± 1.6	3.24 ± 0.11	120.8 ± 7.5	18.6 ± 1.4	1.58 ± 0.12	13.8 ± 7.8
723	22.8 ± 1.5	1.90 ± 0.12	16.5 ± 7.1	18.9 ± 1.6	1.42 ± 0.12	14.6 ± 9.1
855	38.0 ± 1.5	2.83 ± 0.11	92.5 ± 7.3	17.2 ± 1.5	1.32 ± 0.11	4.5 ± 8.4
881	36.2 ± 1.5	2.80 ± 0.11	83.9 ± 7.1	n.a.	n.a.	n.a.
939	39.4 ± 1.7	2.65 ± 0.11	98.3 ± 8.1	20.1 ± 1.4	1.65 ± 0.11	22.7 ± 7.9
1267	n.a.	n.a.	n.a.	19.8 ± 2.9	0.77 ± 0.11	16.9 ± 16.3
1314	n.a.	n.a.	n.a.	19.1 ± 2.1	0.98 ± 0.11	13.3 ± 12.1

n.a. – not analyzed.

**Fig. 3.** Evolution of tCOD of the effluent (S.p.4), sCOD in the filtration section (S.p.3) during the entire period of operation (arrows indicate the purges carried out; (a) – continuous purge).

tCOD values of the upper part of the UASB reactor (S.p.2) during the recirculation period, while sCOD remained almost similar during that period in both sections. Comparing this behavior with that obtained in a previous work (Gouveia et al., 2015), it seems that the new membrane configuration when operating with recirculation allows for a better distribution of the accumulated material through the system.

3.7. Biosolids production

The VS concentration was measured in the biological section (S.p.0, 1 and 2) and in the filtration section (S.p.3 and 5). In the biological section, the VS concentration was maintained between 58.3 ± 2.3 g VS/L, 55.5 ± 7.8 g VS/L and 6.5 ± 3.5 g VS/L respectively. No degranulation or loss of granular sludge integrity was observed throughout the operation. Taking into account the volume of each zone, the amount of VS in the biological section was maintained between 4.5 kg and 5.7 kg throughout the whole operation of the pilot plant. No biomass wastage of the biological section was carried out, except for the samples taken to monitor the process. However, in the filtration section, four periodical purges were

carried out approximately every 150 days, and a continuous purge from day 684 to day 792. Table 5 summarizes the purges carried out throughout the operation. The biosolid production (BS) was calculated taking into account the volume removed from the filtration section (V_{purged}) and the concentration of VS contained therein (g VS/L) at the moment of the purge, considering the following equations:

$$\text{WFR} = \frac{V_{\text{purged}}}{\text{days between purges}} \quad (1)$$

$$\text{MFR} = \frac{V_{\text{purged}} * [\text{VS}]}{\text{days between purges}} \quad (2)$$

$$\text{BS} = \frac{\text{MFR}}{[\text{tCOD}_{\text{removed}}/\text{d}]} \quad (3)$$

$$\text{WR} = \frac{\text{WFR}}{Q_{\text{fed}}} * 100 \quad (4)$$

where WFR is the wasting flow rate (L/d), MFR is the mass flow rate of volatile solids wasted (g VS/d), BS is the biosolid production

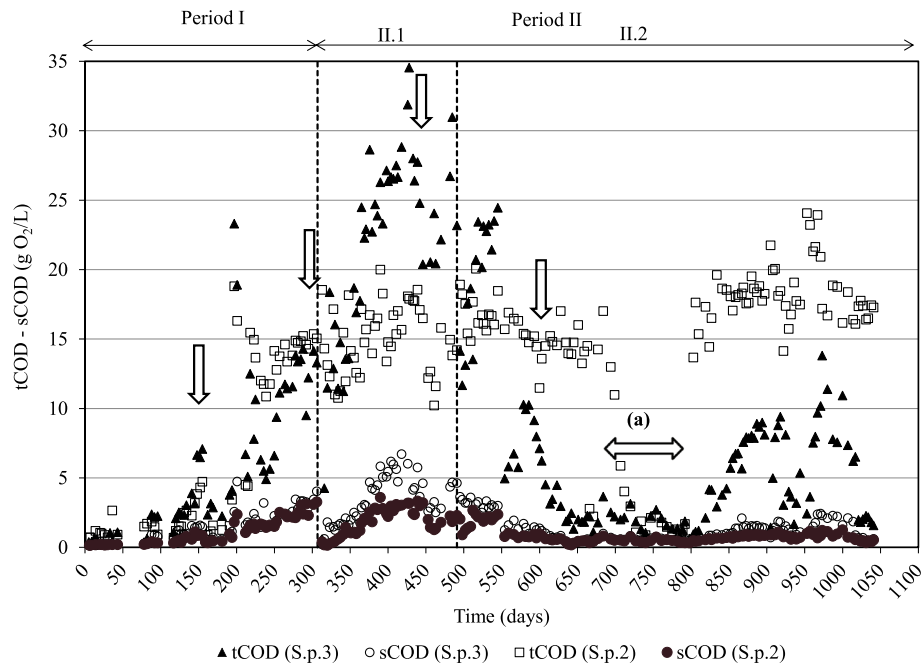


Fig. 4. Evolution of tCOD, sCOD in the UASB (S.p.2) and filtration section (S.p.3) during the entire period of operation (arrows indicate the purges carried out; (a) – continuous purge).

($g VS_{wasted}/g tCOD_{removed}$), WR is the wasting ratio (%) and Q_{fed} the flow rate of wastewater fed to the UASB reactor (L/d).

As shown in Table 5, the purges were carried out with different VS concentrations in the filtration section, and different volumes. Under these conditions, the biosolid production was between $0.083 g VS/g tCOD_{removed}$, and $0.05 g VS/g tCOD_{removed}$, with a wasting ratio between 1.1% and 0.3% respectively. The lower biosolid production from the purge on day 609 was due to the lower concentration of VS in the filtration section, caused by the recirculation from the filtration section to the bottom of the biological section. During the continuous purge, even when operating with a wasting ratio of 3%, the biosolid production was around $0.03 g VS/g tCOD_{removed}$ due to the low solid concentration in the filtration section, lower than $1 g VS/L$.

The results obtained in the present work were similar to those reported in the literature. Shin et al. (2014) in a pilot scale anaerobic fluidized membrane bioreactor (AFMBR) working without temperature control (9–30 °C), obtained an average biosolid production of $0.051 g VSS/g COD_{removed}$ with a wasting ratio of 1%. Bae et al. (2014), using synthetic wastewater at 25 °C in an AFMBR, obtained a sludge production of $0.003 g VSS/g COD_{removed}$ with a wasting ratio between 0.8–0.5%. Nevertheless, Pretel et al. (2014), working with an AnMBR, treating sulfate-rich urban wastewater, obtained low/moderate sludge production of 0.16, 0.43 and

$0.55 kg TSS/kg COD_{removed}$ at 35, 22, and 17 °C, respectively. Wei et al. (2014) reported a biomass yield between 0.015 and $0.026 g MLVSS/g COD$ working at mesophilic conditions with synthetic municipal wastewater.

Biochemical methane potential tests of the biosolids wasted in the current work were conducted under mesophilic conditions (35 °C) in order to determine the mesophilic anaerobic biodegradability. The BMP tests showed that the biodegradability was between 28% and 42%, with specific methane production at day 16 of between 123 and $327 mL CH_4/g VS_{fed}$.

3.8. Membrane behavior

Fig. 5 illustrates the performance of the membrane throughout the experimental period of the AnSMBR pilot plant. Biogas sparging, and the above mentioned purges, were the parameters modified to control TMP. Biogas was continuously sparged at the bottom of the hollow fibers, with different flow rates. A superficial biogas velocity of 9 m/h was enough to maintain a low increase in TMP over approximately the first 400 days of operation due to the low solid concentration and low permeate flux. When the flux was gradually increased from $8 L/m^2 h$ to $15 L/m^2 h$, together with a high solid concentration in the filtration section, a remarkable increase in TMP was originated. In order to maintain the filtration

Table 5
Purges carried out from the filtration section (S.p.3) of the AnSMBR.

Days	AnSMBR				
	156	307	448	609	684–792 ^a
V_{purged} (L)	425.32	380	160	160	1964.32
VS (g/L)	7.44	7.16	16.08	2.28	0.89
VS_{purged} (g)	3162.5	2720.8	2571.2	364.8	1748.4
Wasting flow rate (L/d)	2.73	2.52	1.13	0.99	18.19
Mass VS wasted (g VS/d)	20.27	18.02	18.24	2.27	16.19
Biosolids production (g VS/g $COD_{removed}$)	0.067	0.083	0.050	0.004	0.026
Wasting ratio (%)	0.59	1.07	0.30	0.18	3.03
Recirculation	No	No	No	Yes	Yes

^a Continuous purge from day 684 until day 792.

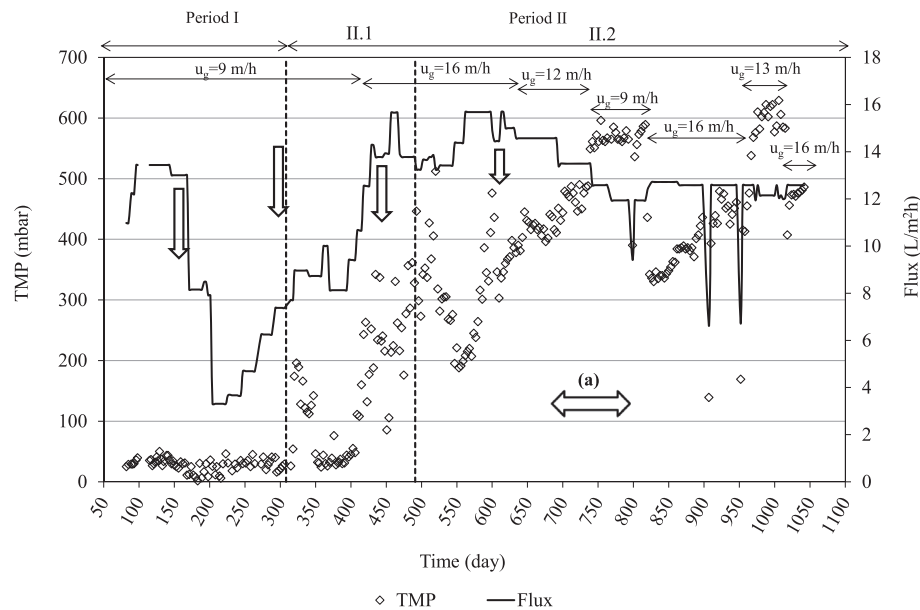


Fig. 5. Evolution of TMP and permeate flux during the entire period of operation (arrows indicate the purges carried out; (a) –continuous purge).

pressure, the biogas superficial velocity was increased up to 16 m/h and the filtration time was simultaneously decreased from 15 to 7.5 min, and the back-flush from 1 to 0.5 min. Moreover on day 448 and on day 609 two purges were carried out, and on day 493 the mixed liquid from the filtration section was recirculated to the bottom of the UASB in order to decrease the solid concentration. The recirculation and periodical purges permitted the maintenance of a low concentration of solids in the filtration section and, therefore, a high permeate flux and a moderate increase in the TMP. During this stage the flux was maintained between 14–15 L/m² h and the TMP was kept at around 300–350 mbar. The decrease of superficial biogas velocity to 12 m/h and to 9 m/h, after 725 days of continuous operation, caused a significant increase in TMP, even when the permeate flux was gradually decreased from 15 L/m² h to 12 L/m² h. Under these conditions, the TMP increased up to 570 mbar, although the concentration of solids in the filtration section was lower than 2 g/L. The increase, once again, of the biogas velocity to 16 m/h, caused a pressure recovery from 590 mbar to 350 mbar. As can be seen in Fig. 5, the change of the superficial biogas velocity has a significant effect on the pressure filtration. When the permeate flux was decreased a great deal due to occasional operational problems, the TMP decreased, but returned quickly to the initial level after restarting. The results obtained show that the main parameters involved in the fouling of the membrane were the permeate flux, the superficial velocity of biogas and the solid concentration, which depends on the recirculation rate and periodical purges carried out. Defining the fouling rate as the daily increase of the TMP, a fouling rate of between 1–3 mbar/d was obtained with high biogas sparging (16 m/h), high permeate flux (12 L/m² h) and a solid concentration lower than 2 g VS/L. Meanwhile, the fouling rate increased up to 19 mbar/d when the permeate flux increased to 15.7 L/m² h and the solid concentration to 12–14 g VS/L. Martinez-Sosa et al. (2011), working with an AnSMBR at 20 °C and flux constant of 7 L/m² h, reported an increase on TMP of 2.61 mbar/d.

In the present work, the membrane operated for most of the time with a flow rate around 12–14 L/m² h with transmembrane pressure between 350–600 mbar. During the three years of continuous operation, the membrane was not physically or chemically cleaned.

The different membrane behavior observed in the current work and the previous work (Gouveia et al., 2015) could be due to the combination of three factors: the different membrane configuration (which allowed better solid circulation from the membrane section to the UASB reactor), the higher biosolid wasted in the current work, and the lower superficial velocity of the biogas sparging applied. In both configurations, the specific gas demand was between 0.5–1 m³/m² h, nevertheless the biogas superficial velocity was different. In the previous work, biogas velocity ranged between 30–60 m/h, and in the current work the biogas superficial velocity was between 9–16 m/h. Continuous biogas sparging is needed in the filtration section, and the superficial biogas velocity is a fundamental parameter for controlling TMP increase. Nevertheless, a high superficial velocity does not improve the fouling rate in long-term operation. Li et al. (2014), working with a submerged Anammox membrane bioreactor at 35.0 ± 0.5 °C, using synthetic wastewater, reported a flux between 7.08 to 14.16 L/m² h, with a volatile suspended solid concentration of 3 g/L and biogas sparging between 0 and 0.2 m³/h, with periodic removal for chemical cleaning. Bae et al. (2014) working with synthetic wastewater at 25 °C in an anaerobic fluidized membrane bioreactor, using a PVDF membrane with a pore size of 0.1 μm, reported a flux between 6 and 9 L/m² h. Robles et al. (2013), working with a SANMBR plant, reported that a biogas sparging of around 0.23 N m³/m² h was enough to operate for two and a half years with a flux of 8 L/m² h. Meanwhile, Fox and Stuckey (2015), working with AnMBR, fed with synthetic wastewater, reported that biogas sparging reduced fouling substantially, using a critical flux of 11.8 L/m² h and a sparging rate of 6 L/min.

4. Conclusions

The results show the reliability and stability of AnSMBR technology for the treatment of municipal wastewater at psychrophilic conditions. The membrane configuration, placed on top of the UASB reactor, together with a superficial biogas velocity of between 16–9 m/h, allows the continuous operation of the membrane for three years without physical or chemical cleaning, operating with a permeate flux between 14–12 L/m² h. The solid

concentration in the filtration section, controlled by recirculation from the filtration section to the UASB reactor and by periodical purges every 150 days, has been a fundamental parameter for the long-term stability of the technology.

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