Methane recovery efficiency in a Submerged Anaerobic Membrane Bioreactor (SAnMBR) treating sulphate-rich urban wastewater: Evaluation of methane losses with the effluent.

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Abstract

The present paper presents a submerged anaerobic membrane bioreactor (SAnMBR) as a sustainable approach for urban wastewater treatment at 33ºC and 20ºC, since greenhouse gas emissions are reduced and energy recovery is enhanced. Compared to other anaerobic systems such as UASB reactors, the membrane technology allows the use of biogas-assisted mixing which enhances the methane stripping from the liquid phase bulk. The methane saturation index obtained for the whole period (1.00 ± 0.04) evidenced that the equilibrium condition was reached and the methane loss with the effluent was reduced. The methane recovery efficiency obtained at 20ºC (53.6%) was slightly lower than at 33ºC (57.4%) due to a reduction of the treatment efficiency, as evidenced by the lower methane production and the higher waste sludge per litre of treated wastewater. For both operational temperatures, the methane recovery efficiency was strongly affected by the high sulphate concentration in the influent wastewater.

1. INTRODUCTION

Sustainable operation of wastewater treatment plants (WWTP) is nowadays a general goal. Currently, the power required for the treatment of urban wastewater in Spain is approximately 300 MW, equivalent to an average of 5.6 W/PE or a consumption of 50 kWh/(PE year) (Fernández *et al.*, 2011). Thus, operation of WWTP should be focussed on making sewage treatment self-sufficient, reducing energy requirements and global greenhouse-gas emissions (Hartley and Lant, 2006).

Anaerobic wastewater treatments present several advantages over conventional aerobic systems: (a) minimum sludge production, due to the low biomass yield of anaerobic organisms, which reduces the cost of sludge handling, stabilization and final disposal, (b) low energy demand since no aeration is required and (c) recovery of energy from the methane gas produced in the process. Most anaerobic wastewater treatment processes are typically conducted within mesophilic (25-45ºC) or thermophilic (45-60ºC) temperature ranges because most of the biochemical reactions involved in organic matter biodegradation proceed slower under psychrophilic (< 20ºC) conditions (Bandara *et al.*, 2011). However, for urban wastewaters, the low influent COD (typically less than 1 g/L) results in low methane production, and an external energetic contribution is usually required in order to heat the reactor up to mesophilic conditions (Lew *et al.*, 2009). Therefore, the most attractive option for the anaerobic

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low-strength wastewater treatment is the reactor operation without heating, i.e., at ambient temperature.

In tropical and subtropical regions, where the ambient temperatures do not drop below 15ºC, intensive anaerobic treatments have been successfully applied (Kalogo and Verstraete, 2000; Lew *et al.*, 2003). However, in moderate climates, the feasibility of anaerobic treatments in full-scale WWTP is still under research. At low temperatures, longer solids retention times (SRT) are required to achieve proper treatment performance and to prevent biomass washout than for mesophilic or thermophilic conditions, since anaerobic microorganisms growth rates decrease with temperature. In conventional completely mixed reactors, both hydraulic retention times (HRT) and SRT are the same and, thus, high working volumes are required. The combination with membrane separation processes ensures biomass retention, needed for decoupling HRT and SRT. Therefore, the application of membrane separation processes by the so-called membrane bioreactors (MBR) in anaerobic wastewater treatments represents a significant improvement of the process. In recent years, anaerobic membrane bioreactors (AnMBR) have gained attention as a suitable way to treat low-strength wastewater using different membrane module configurations. Among the different types of membranes, the hollow-fibre ones have been identified to achieve high effluent streams and quality with low filtration energy demand (Lew *et al*., 2009).

The CH_4 production in the anaerobic biodegradation of organic matter depends on treatment efficiency. However, part of this CH_4 produced is lost with the effluent and not available for energy production. The loss of dissolved methane (CH_4) with the effluent is one of the key issues which must be overcome in order to successfully apply anaerobic MBR technology to urban wastewater treatment, since it is a powerful

greenhouse gas which contributes to global warming. Methane loss becomes especially important at low operational temperature processes since CH_4 solubility in the liquid phase inversely depends on temperature. A post-treatment process will be required in order to avoid dissolved methane release to the atmosphere. A higher dissolved methane concentration in the reactor effluent leads to a decrease in the CH_4 recovery efficiency ($\eta_{CH_4}^{Rec}$), which stands for the percentage of the total CH_4 produced (biogas + lost with the effluent) that is recovered with the biogas. This parameter is another important issue of concern when CH_4 is intended to be used as energy source.

Some studies (Souza *et al.*, 2010; Hartley and Lant, 2006) have reported methane super-saturation at the effluent of many anaerobic treatment systems, due to a deficient mixing which leads to low mass-transfer coefficients. Thus, the loss of CH_4 in the effluent could be higher than the one expected by the theoretical approaches and it needs to be determined for each reactor configuration and operation.

In the present study, a submerged anaerobic membrane bioreactor (SAnMBR) treating sulphate-rich municipal wastewater has been operated at 33ºC and 20ºC. Dissolved methane concentrations have been quantified in the effluent to assess its saturation degree. Moreover, the influence of several parameters on methane recovery efficiency has been evaluated.

2. MATERIALS AND METHODS

2.1. Pilot plant description

Figure 1a shows the semi-industrial SAnMBR pilot plant that was used in this study. It basically consists of an anaerobic reactor of 1.3 m^3 total volume (0.4 m^3 head-space volume) connected to two membrane tanks of 0.8 m^3 total volume each (0.2 m^3 head-space volume), resulting in a total reaction volume of 2.1 m^3. Each membrane tank includes a commercial hollow-fiber ultrafiltration membrane module (PURON®, Koch Membrane Systems, 0.05 μm pore size). A rotofilter of 0.5 mm screen size has been installed as pre-treatment system. In order to control the temperature, the anaerobic reactor is jacketed and connected to a water heating/cooling system.

Figure 1b shows the flow diagram of the plant. The pilot plant is fed with the effluent of the Carraixet WWTP pre-treatment (screening, degritter and grease removal). In order to improve the stirring conditions of the anaerobic reactor, and to favour the stripping of the produced gasses from the liquid phase, a fraction of the produced biogas is recycled through the bottom of this reactor. The sludge is continuously recycled throughout the external membrane tanks (MT) where the effluent is obtained by vacuum filtration. In order to minimise the cake layer formation, another fraction of the produced biogas is also recycled to the membrane tanks from the bottom of the membrane modules. Finally, in order to control the solids retention time (SRT) in the system, a fraction of the sludge is intermittently extracted from the anaerobic reactor all along the day. The membrane performance is established by a proper scheduling of the individual membrane operational stages. Further details of the pilot plant description and operational conditions can be found in Giménez *et al.*, 2011.

2.2. Experimental procedure

In the present study, two different experimental periods were evaluated according to the working temperature: Period I (T=33ºC) and Period II (T=20ºC). The sludge retention time (SRT) was fixed at 70 days. It is important to highlight that, even though the organic load (OL) was higher in Period I than in Period II, the organic load available for methanogenic *archaea* (OL^{MA}) was slightly higher in Period II (See Table 1). This higher OL^{MA} can be attributed to a higher COD/SO_4-S ratio in the second period. The OL^{MA} can be calculated by subtracting the Chemical Oxygen Demand (COD) consumed by sulphate reducing bacteria (SRB) to the total COD consumed in the process, considering that 2 kg of substrate as COD is consumed by SRB in order to reduce 1 kg of sulphate (SO_4-S).

2.3. Analytical methods.

Samples from influent and effluent streams and anaerobic sludge from the reactor were collected once a day. Biogas from the headspace of the reactor was sampled at least once a week. Total Suspended Solids (TSS), Volatile Suspended Solids (VSS), Volatile Fatty Acids (VFA), alkalinity (Alk), and SO_4-S were analysed every day. Total and soluble COD (TCOD and SCOD, respectively), and biogas composition (CH_4, CO_2, and H_2S) were determined once a week. Solids, COD, and sulphate determinations were performed according to Standard Methods (APHA, 2005). Carbonate alkalinity and VFA concentrations were determined by titration according to the method proposed by WRC (1992).

2.3.1. Biogas characterization

2L Tedlar bags (Sigma-Aldrich) were used to collect biogas samples from a sampling point connected to the top of the reactor in order to assure the representativeness of the characterization. Methane richness of the biogas was determined using a gas chromatograph equipped with a Flame Ionization Detector (GC-FID, Thermo Scientific). 0.2 ml of biogas were collected by a gas-tight syringe and injected to a 30m×0.319mm×25μm HP-MOLESIEVE column (Agilent Technologies) which was maintained at 40ºC. Helium was used as the carrier gas at a flow-rate of 40 mL·min^{-1}. Methane pure gas (99.9995%) was used as standard. MSA AUER color-indicating detector tubes were used once a week in order to determine the carbon dioxide (detection range 0.5 - 10 vol. %) and hydrogen sulfide (detection range 0.1 - 4 vol. %) content of the biogas.

2.3.2 Determination of dissolved CH$_4$ in the effluent.

The dissolved methane concentration of the effluent was estimated according to the methodology proposed by Souza *et al.*, 2010 in order to evaluate the methane saturation index. This parameter can be defined as the ratio between the measured concentration of methane at the liquid phase and the saturation concentration.

Effluent samples were carefully collected in known-volume sealed vials with PTFE septa, and stored in an incubator for at least 4 hours before measuring the head-space methane content, in order to assure the equilibrium condition. The net weight of the vial (m_V), as well as the weight of the water-filled vial (m_V^W), and the vial with the sample (m_V^S) were determined in order to calculate the total (V_T), liquid phase (V_L) and gas

phase (V_G) volumes, assuming a specific mass of 1.0 g·mL^{-1} for both the water and the sample, according to the following:

$$V_T = m_V^W - m_V \qquad (1)$$

$$V_L = m_V^S - m_V \qquad (2)$$

$$V_G = V_T - V_L \qquad (3)$$

Once the equilibrium condition was achieved, gas chromatography analysis was performed to the gas phase in order to determine the methane molar fraction (y^{CH_4}). In the equilibrium condition, a fraction of methane that was previously in the liquid phase is spread out to the gas phase. The volume of methane in the gas phase ($V_G^{CH_4}$) can be calculated as:

$$V_G^{CH_4} = V_G \cdot y^{CH_4} \qquad (4)$$

The mass of methane in the gas phase ($m_G^{CH_4}$) can be calculated from the combination of equation (4) and the equation of ideal gases:

$$m_G^{CH_4} = \frac{P \cdot V_G \cdot y^{CH_4} \cdot \bar{M}^{CH_4}}{R \cdot T} \qquad (5)$$

Where P is the total pressure (atm), $\bar{M}^{CH_4}$ is the molecular weight of methane ($\bar{M}^{CH_4} = 16$ g/mol), R is the universal constant of gases (0.082 atm·L·mol^{-1}·K^{-1}), and T is the temperature (K).

Henry's Law establishes that, for given temperature and pressure conditions, the saturation of any gas in a liquid phase depends on its concentration in the gas phase as well as on the nature of both gas and liquid, according to the following equation:

$$P \cdot y^{CH_4} = H^{CH_4}(T) * x^{CH_4} \qquad (6)$$

$H^{CH_4}(T)$ is the temperature-dependent Henry's constant for methane (atm) and x^{CH_4} is the molar fraction of methane on the liquid phase.

The temperature-dependent Henry's constant was calculated for pure water, according to the following formula (Tchobanoglous *et al.*, 2003):

$$H^{CH_4}(T) = 10^{\left(\frac{-675.74}{T(K)} + 6.88\right)} \qquad (7)$$

Therefore, Henry's Law was used to estimate the dissolved methane molar fraction at the liquid phase under the equilibrium condition, based on the gas phase methane content and assuming that the liquid phase consisted of water and methane only. Therefore, the molarity of methane (M^{CH_4}) in the effluent was calculated as:

$$M^{CH_4} = \frac{M^W \cdot x^{CH_4}}{(1 - x^{CH_4})} \qquad (8)$$

where M^W is the molarity of pure water (55.56 mol·L^{-1}). By combining equations (6) and (8), taking into account the molecular weight of methane and the total volume of the liquid phase (eq. 2), the mass of methane at the liquid phase ($m_L^{CH_4}$) can be calculated by means of the following expression:

$$m_L^{CH_4} = \frac{M^W \cdot P \cdot y^{CH_4} \cdot \overline{M}^{CH_4} \cdot V_L}{H^{CH_4}(T) - P \cdot y^{CH_4}} \tag{9}$$

The original dissolved methane concentration of the effluent ($[CH_4]_0^{Effluent}$) is calculated, then, adding both the dissolved methane and the methane in the gas phase by means of the following equation:

$$[CH_4]_0^{Effluent} = \left(\frac{V_G}{V_L R \cdot T} + \frac{M^W}{H^{CH_4}(T) - P \cdot y^{CH_4}} \right) \cdot P \cdot \overline{M}^{CH_4} \cdot y^{CH_4} \tag{10}$$

If any difference exists between the process working temperature and the temperature of the incubator, changes in partial pressure should also be considered mainly due to the expansion of the head-space gases.

3. RESULTS AND DISCUSSION

3.1. Wastewater and biogas characterisation.

Pre-treated domestic wastewater outgoing from the Carraixet WWTP degritter was used as the influent to the pilot plant. Table 2 shows the main characteristics of the influent for two different periods: Period I (T=33ºC) and Period II (T=20ºC). In both periods, the most noteworthy features of the influent stream are the high and almost constant sulphate concentration, and the strong organic load variability as evidenced by the wide variation range in parameters like TCOD, SCOD, VFA and COD/SO_4-S ratio.

The biogas composition in both experimental periods is shown in Table 3. N_2 fraction in the biogas was estimated assuming that biogas mainly consists of CH_4, CO_2, H_2S and N_2.

Unlike conventional anaerobic digestion of sludge, significant variations in biogas composition and high N_2 concentrations were obtained for both experimental periods as observable in Table 3. Several factors have been identified as influencing the biogas composition. Most of them are related to the influent physico-chemical characteristics such as the COD concentration, the COD/SO_4-S ratio and the wastewater temperature. The COD concentration and the COD/SO_4-S ratio contribute to modify the biogas composition by means of modifying the CH_4, H_2S and CO_2 production. The influent temperature and, in particular, the temperature gradient between the influent and the reactor determine the amount of N_2 in the biogas, since, once in the reactor, the dissolved N_2 in the influent is stripped out to the gaseous phase, contributing to dilute the biogas. For anaerobic treatment of low-strength wastewaters, as urban wastewaters, high N_2 concentrations are achieved, since less CH_4 is produced per litre of treated wastewater.

Another factor influencing the biogas production and composition is the operational temperature. At psychrophilic temperatures the activity of methanogenic *archea* is lower than at mesophilic conditions, which leads to a decrease in the methane production. Moreover, temperature inversely affects the solubility of the gaseous compounds, which results in a different distribution of the gases between both the liquid and gas phases. This explains why CH_4 concentrations in the biogas obtained in Period II (T=20ºC) were lower than in Period I (T=33ºC), despite the higher OL^{MA} obtained in Period II.

3.2. Assessment of methane saturation index in the effluent at 33ºC and 20ºC

In order to assess whether or not super-saturation takes place, the methane saturation index was evaluated. Dissolved methane was measured to 25 effluent samples by means of the methodology described in section *2.3.2*. The values obtained were compared to the saturation methane concentration given by Henry's Law for the methane content of the biogas at the working conditions (P=1 atm; T=33ºC in Period I and T=20ºC in Period II). As can be seen in Table 4, the dissolved methane concentration values were very close to the saturation concentration ones within a wide range of biogas compositions.

The results obtained for the methane saturation index during the whole period prove that the equilibrium condition between both the liquid and gas phases was reached in the anaerobic reactor for both operational temperatures. The use of membranes as a physical barrier to retain the biomass in the system requires a physical cleaning membrane by means of gas sparging. The produced biogas was used for both membrane cleaning and reactor mixing. This biogas-assisted mixing enhanced the methane stripping from the liquid phase bulk until the equilibrium was reached.

Unlike many other anaerobic treatment systems (UASB, EGBR, etc…) in which methane super-saturation takes place due to a deficient mixing, the SAnMBR technology using biogas-assisted mixing avoids super-saturation and guarantees the minimum concentration of dissolved methane at the effluent, i.e. the saturation

concentration. Therefore, greenhouse gas emissions are reduced and energy recovery is enhanced.

Figure 2 shows the dissolved and saturation concentration of methane in the effluent for the different experiments carried out at 33ºC and 20ºC. As can be observed, the operational temperature in an anaerobic reactor inversely affects the solubility of the gaseous compounds, as mentioned above. According to equation (9), the solubility of methane is 23.7 mg $CH_4 \cdot L^{-1}$ at 20ºC and 18.9 mg $CH_4 \cdot L^{-1}$ at 33ºC. Therefore, although the SAnMBR technology using biogas-assisted mixing assures the minimum dissolved methane in the effluent, the methane lost at 20ºC will be higher than at 33ºC. In particular, the extra methane that is lost with the effluent by decreasing temperature from 33ºC to 20ºC will be 25% higher, according to Henry's constant at 20ºC and 33ºC.

3.3. Influence factors on CH_4 recovery efficiency

The methane recovery efficiency ($\eta_{CH_4}^{Rec}$) can be calculated by the combination of equations (5) and (9):

$$\eta_{CH_4}^{Rec} = \frac{\frac{Q^{BG}}{Q^{TREAT}}}{\frac{Q^{BG}}{Q^{TREAT}} + \frac{R \cdot T \cdot M^W}{H(T) \cdot P \cdot y^{CH4}}} \cdot 100 \qquad (11)$$

Given that $\eta_{CH_4}^{Rec}$ refers to a continuous process, V_G and V_L of equations (5) and (9) have been replaced by the biogas flow rate (Q^{BG}) and Q^{TREAT}, respectively.

This equation shows the influence of different factors on the $\eta_{CH_4}^{Rec}$ of the process. As can be observed, the methane fraction in the biogas (y^{CH4}) is not a high-influence factor since H(T) value is much higher than ($P \cdot y^{CH4}$). An increase in y^{CH4} means not only an increase in the CH4 recovery in the biogas but also an increase on CH4 lost in the effluent, according to Henry's Law. Therefore, the $\eta_{CH_4}^{Rec}$ is not significantly affected by this parameter. Other operational parameters that affect the $\eta_{CH_4}^{Rec}$ are the biogas production rate per litre of treated water (Q^{BG}/Q^{TREAT}) and the operational temperature.

Previous studies (Giménez *et al.*, 2011) have reported that the influent COD/SO4-S ratio significantly affects the Q^{BG}/Q^{TREAT} due to the competition between methanogenic *archaea* (MA) and sulphate reducing bacteria (SRB) for the available substrate. Figure 3 shows the biogas production rate per litre of treated wastewater as a function of the influent COD/SO4-S ratio. As this figure shows, the biogas produced per litre of treated wastewater increases with COD/SO4-S ratio for values above 2 since there is stoichiometrically enough substrate to completely remove the sulphate and the remaining COD is available for MA. Therefore, the methane recovery efficiency strongly depends on the COD/SO4-S ratio and needs to be taken into account for sulphate-rich wastewaters.

Figure 4 shows the theoretical influence of the biogas and treatment flow rates (Q^{BG}, Q^{TREAT}) over the $\eta_{CH_4}^{Rec}$ at three different reactor temperatures (10ºC, 20ºC and 33ºC) for a y^{CH4}=0.5 and for a constant organic load. As it can be seen in this figure, the $\eta_{CH_4}^{Rec}$ increases with the biogas flow rate and decreases as the treatment flow rate increases. This means that the $\eta_{CH_4}^{Rec}$ increases with the volume of biogas produced per litre of treated water (Q^{BG}/Q^{TREAT}). Figure 5 shows the $\eta_{CH_4}^{Rec}$ in the SAnMBR pilot plant

at 33ºC and 20ºC as a function of Q^{BG}/Q^{TREAT}, calculated from the experimental biogas flow rate and CH_4-richness, and considering that dissolved methane is in equilibrium with the gas phase CH_4 content. The theoretical curves have been obtained for 20ºC and 33ºC by means of equation (11) for y^{CH4}=0.5.

As can be observed, the experimental data fit very well with the theoretical ones for both operational temperatures. This figure also shows the temperature effect on $\eta_{CH_4}^{Rec}$. At lower temperature the $\eta_{CH_4}^{Rec}$ decreases due to a higher loss of dissolved CH_4 in the effluent, as previously explained. However, this decrease is not significant, especially at low or high Q^{BG}/Q^{TREAT} values, when $\eta_{CH_4}^{Rec}$ tends to 0 or 100, respectively.

Table 5 shows the experimental results obtained in the pilot plant for the $\eta_{CH_4}^{Rec}$ assessment at 33ºC and 20ºC. As can be observed, the $\eta_{CH_4}^{Rec}$ obtained in Period I was slightly higher than in Period II. Despite the higher influent COD/S-SO$_4$ ratio in Period II than in Period I (6.3 vs. 3.9 g COD/gS-SO$_4$), a higher Q^{BG}/Q^{TREAT} was obtained in Period I. This can be attributed to the lower temperature maintained in Period II (20ºC) which reduced the treatment efficiency due to a lower hydrolysis of the organic particulate matter, as evidenced by the lower methane production (Q^{CH4}/Q^{TREAT}) and the higher waste sludge per litre of treated wastewater. Moreover, the increase of CH_4 and N_2 solubility at 20ºC with respect to 33ºC, contributed to reduce the Q^{BG}/Q^{TREAT} by means of increasing the amount of CH_4 lost with effluent and reducing the N_2 stripped out from the influent wastewater.

The lower $\eta_{CH_4}^{Rec}$ achieved in Period II resulted in a greater CH_4 loss with the effluent than in Period I: 46.4% of the total CH_4 generated in the reactor was lost in Period II vs. 42.6% in Period I. These values are higher than the ones obtained by Souza et al. (2010) in effluents from different UASB reactors treating domestic wastewaters at 25ºC, which reached values from 36% to 41% of the total methane generated. However, it should be highlighted that, in the present study, the presence of sulphate in the wastewater reduced the available COD for methanisation since SRB outcompete MA for the available substrate. In particular, 57% and 41% of the influent COD was consumed by SRB in Period I and Period II, respectively. Therefore, higher biogas production and $\eta_{CH_4}^{Rec}$ would be achieved if no sulphate were present. Considering that the COD consumed by SRB were available for MA, the methane recovery efficiency could reach values up to 83% and 77%, at 33ºC and 20ºC, respectively. This states the importance of the influent wastewater physico-chemical characteristics over the anaerobic treatment performance.

4. CONCLUSIONS

SAnMBR technology represents a sustainable approach for urban wastewater treatment since greenhouse gas emissions are reduced and energy recovery is enhanced. Biogas-assisted mixing avoids super-saturation and guarantees a minimum concentration of dissolved methane at the effluent. The methane recovery efficiency obtained at 20ºC (53.6%) was slightly lower than at 33ºC (57.4%) due to a reduction of the treatment efficiency and to an increase of the gases solubility. The $\eta_{CH_4}^{Rec}$ turned out to be mainly affected by the influent wastewater COD/S-SO$_4$ ratio: if no sulphate were present $\eta_{CH_4}^{Rec}$ could reach values up to 83% and 77% at 33ºC and 20ºC, respectively.

Acknowledgements

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Figure and table captions

Figure 1. General view of the pilot plant **(a)**, and process flow diagram **(b)**.

Figure 2. Dissolved and saturation concentration of methane in the effluent.

Figure 3. Biogas production per litre of treated water as a function of influent COD/SO_4-S ratio

Figure 4. Theoretical influence of biogas and treatment flow rates on the methane recovery %.

Figure 5. $\eta_{CH_4}^{Rec}$ at 33ºC (●) and at 20ºC (○).

Table 1. Operational parameters of the SAnMBR pilot plant. (Mean ± SD)

Table 2. Influent wastewater characterisation. Median (Range)

Table 3. Biogas characterisation. Median (Range)

Table 4. Methane saturation index assessment. Median (Range)

Table 5. Methane recovery efficiency. Median (Range).