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Separation Science and Technology

Publication details, including instructions for authors and subscription information:

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To cite this Article Jeison, D. , van Betuw, W. and van Lier, J. B.(2008) 'Feasibility of Anaerobic Membrane Bioreactors for the Treatment of Wastewaters with Particulate Organic Matter', Separation Science and Technology, 43: 13, 3417 — 3431

To link to this Article: DOI: 10.1080/01496390802221659

URL: <http://dx.doi.org/10.1080/01496390802221659>

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Feasibility of Anaerobic Membrane Bioreactors for the Treatment of Wastewaters with Particulate Organic Matter

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Abstract: The application of anaerobic submerged membrane bioreactors was studied for the treatment of wastewaters containing suspended solids. A mesophilic and a thermophilic reactors were operated with a synthetic wastewater. The thermophilic reactor achieved higher volumetric loading rates than the mesophilic reactor, reaching 14 g COD/L · d (0.47 g COD/g VSS · d). The mesophilic reactor showed signs of overload, when reaching a volumetric loading rate of 10 g COD/L · d (0.32 g COD/g VSS · d). Cake formation was identified as the main factor governing applicable flux. Low levels of irreversible fouling were observed in both reactors. Low fluxes were attained and gas sparging was ineffective in increasing the critical flux.

Keywords: Anaerobic, cake, fouling, MBR, membrane, suspended solids

INTRODUCTION

Anaerobic digestion is nowadays one of the most cost-effective alternatives for organic matter removal from wastewaters. Low biomass yield represents one of the important advantages of anaerobic biotechnology, since it translates into the generation of low amounts of waste sludge, up to ten times less than during aerobic treatment. However, during the first developments of anaerobic processes this feature represented a major

Received 6 December 2007; accepted 28 April 2008.

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drawback when trying to increase the biomass concentration in anaerobic reactors (1). The recognition of the anaerobic sludge granulation concept significantly contributed to the development and application of anaerobic high rate treatment processes, since it enables reactor operation at high sludge concentrations, and therefore high loading rates. Since the installation of the first full-scale upflow anaerobic sludge blanket (UASB) reactor, three decades ago (2), the anaerobic process has been successfully used for the treatment of many kinds of industrial wastewaters as well as sewage. Nowadays, it can be considered an established technology that offers the possibility of an efficient treatment with low capital and operational costs (3). However, the application of granular sludge bed reactors for the treatment of wastewaters with a high content of suspended solids has limitations. Particulate material can affect the sludge bed development in different ways, like blocking liquid distribution systems and favouring growth over the particle surface rather than in granular biomass (4). Besides, depending on the imposed hydraulic retention time, part of the incoming suspended solids may not be adequately retained, leaving the reactor without treatment.

Anaerobic membrane bioreactors (AnMBR) have generated an increasing interest during the last decade. They represent a breakthrough technology for wastewater treatment. However, their application has been restricted due to high associated costs. Nevertheless, they are considered an interesting alternative for those situations where biomass retention by granule formation may not be effective and/or when total solids retention is of interest, like during the treatment of wastewaters with a high content of suspended solids. In AnMBRs, biomass and particulate organic matter are physically retained inside the reactor, providing optimal conditions for organic matter degradation. The application of AnMBRs for the treatment of high suspended solids wastewaters have been studied using different substrates, such as slaughterhouse effluent (5), primary or secondary sludge (6,7), cellulose as synthetic substrate (8), and manure (9), with promising results. However, data is still limited regarding treatment and membrane performance, so further research is needed.

Biomass retention under thermophilic conditions has also proven difficult. Granule formation seems to be more problematic under thermophilic conditions than under mesophilic temperatures (10). Thermophilic microorganisms are characterized by higher substrate utilization rates, when compared with mesophilic bacteria, which means higher loading potentials (11). The efficient biomass retention provided by AnMBR technology would allow taking full advantage of the higher loading capacities of thermophilic treatment, which would enhance the treatment of wastewaters with a high content of particulate organic matter.

The present research studies the application of membrane assisted biomass retention to the anaerobic treatment of wastewaters containing a high proportion of particulate organics. Two anaerobic submerged membrane bioreactors (AnSMBR) were operated, under mesophilic and thermophilic conditions. The performance of these reactors is evaluated and compared in terms of their loading potential and membrane filtration performance.

MATERIALS AND METHODS

Reactors Setup

Two identical AnSMBRs of 3.8 L of useful volume were used to conduct this study. Reactors were operated under thermophilic (55°C) and mesophilic (30°C) conditions. Both reactors were fitted with identical tubular polysulphone micro-filtration membranes (Triqua, The Netherlands). The membrane modules were composed of 4 tubes of 36.7 and 0.9 cm of length and diameter, respectively, while total membrane area was 0.042 m². Biogas recirculation was applied to provide mixing as well as shear over the membranes surface. The AnSMBRs were operated as gas-lift reactors, with the membranes placed inside the riser. Gas superficial velocities (V_G) have been evaluated considering the free area of the riser. Biogas was recirculated by means of compressors (KNF N810FT.18, Germany). Gas flow rates were measured using variable area rotameters (Brooks Instrument R2-15-A, The Netherlands). Permeate was collected by means of peristaltic pumps (Watson Marlow 323U, UK), that provided the required trans-membrane pressure (TMP). TMPs were measured by pressure sensors (AE Sensors ATM, The Netherlands) located in the permeate lines. Figure 1 presents a scheme of each reactor setup.

Reactors Operation

The reactors were fed with a synthetic wastewater composed of a volatile fatty acids (VFA) mixture and ground up potato peels as a source of suspended solids. The acetate:propionate:butyrate ratio of the VFA solution was 1:1:1, expressed as chemical oxygen demand (COD). A concentrated VFA solution of 200 g COD/L was fed continuously to the reactors, which was diluted in-line with tap water. Potato peels were fed manually, on a daily basis. Suspended solids particle size was in the range 0.1 to 3 mm. The feeding of potato peels and VFA was performed so both contributed to the same extent to the applied organic loading rate (OLR).

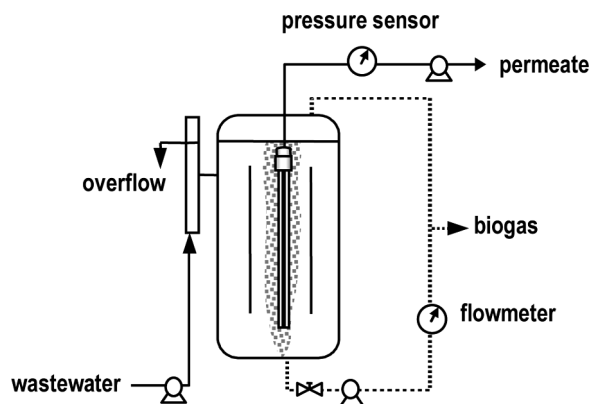


Figure 1. Schematic representation of the AnSMBR setup.

The average inlet COD concentration was 10 g/L. When necessary, an oil based antifoaming (Foamtrol AF4030, BetzDearborn NV) was added in order to control excessive foam formation. The AnSMBRs were operated with cycles consisting of 500 seconds of filtration, followed by 15–30 seconds of back-flush. Back-flush was applied by reversing the flow direction, keeping the flux at the same value as during filtration. As a safety measurement, whenever TMP reached 0.2 bar, filtration cycle was interrupted and a back-flush was performed. Reactors were inoculated with mesophilic and thermophilic suspended anaerobic sludge coming from previous researches (12,13). Initial volatile suspended solids (VSS) concentrations were 35 and 13 g/L for the mesophilic and thermophilic AnSMBR respectively. Reactors were operated at a V_G in the range 70–75 m/h.

Analyses

Total suspended solids (TSS) and VSS were determined according to Standard Methods (14). COD was determined using Lange COD cuvette tests (Hach Lange, Germany). The soluble COD was measured after sample filtration through a membrane filter of 0.45 μm pore size (Aqua 30/0.45CA, Whatman, Germany). VFA were determined in an Hewlett-Packard gas chromatograph (model 5890 series II) with a flame ionization detector (FID), equipped with a column AT Aquawax-DA (Alltech). Proteins were determined by the Lowry method, Onishi and Barr modification (Sigma-Aldrich TP0200 total protein kit). Carbohydrates were analyzed using a modified anthrone method (15).

The specific methanogenic activity (SMA) was determined in duplicates by pressure increase in experiments performed in 117 mL serum bottles, with 50 mL of media. Biomass concentration was 1 g VSS/L. A neutralized VFA mixture of acetate, propionate, and butyrate at a COD ratio of 1:1:1, was used as substrate, at an initial concentration of 1.5 g COD/L. The SMA was evaluated as the maximum specific methane production rate. The basal medium consisted of (mg/L): NH_4Cl (170), $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (8), KH_2PO_4 (37), $\text{MgSO}_4 \cdot 4\text{H}_2\text{O}$ (9), yeast extract (200), $\text{FeCl}_3 \cdot 4\text{H}_2\text{O}$ (2), $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (2), $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ (0.5), $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (0.03), ZnCl_2 (0.05), H_3BO_3 (0.05), $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ (0.09), $\text{Na}_2\text{SeO}_3 \cdot 5\text{H}_2\text{O}$ (0.10), $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (0.05), EDTA (1).

The sludge stability was determined, in duplicates, by the specific amount of methane produced under unfed conditions over a period of 100 hours. For this purpose, samples of 50 mL of sludge were placed in 250 mL bottles, and the headspace was flushed with a mixture of 80% nitrogen and 20% carbon dioxide, to provide anaerobic conditions. Methane production was followed in time measuring the pressure and composition of the gas in the bottles headspace. The amount of methane, expressed as COD, produced after 100 hours of incubation is reported. This determination represents an indication of the amount of unconverted substrates still remaining in the sludge.

SMA and sludge stability analysis were performed at the same temperature of the corresponding reactors operation: 30°C for the samples of mesophilic sludge, and 55°C for the samples of the thermophilic sludge.

Partial Filtration Resistances and Critical Flux

The flux through the membrane (J) is a function of the TMP, the permeate viscosity (η) and the total resistance (R_T):

$$J = \frac{\text{TMP}}{R_T \cdot \eta}$$

The total filtration resistance can be divided in several partial resistances:

$$R_T = R_M + R_I + R_R$$

where R_M represents the resistance of the new membrane, and R_I and R_R represents the resistances due to irreversible and reversible phenomena, respectively. The term reversible is used here to account for those phenomena that can be reverted by the applied back-flush cycles.

Membrane modules were extracted from the reactors several times during the operation, to perform resistance determinations. Resistance

was measured recording the TMP at increasing flux steps, in demineralised water. Flux steps were 5, 15, 20, and 25 L/m² · h, and were maintained for 5 minutes. The membrane was simply rinsed with clean water prior to resistance determination. The latter procedure only removed the loosely attached cake from the membrane surface, i.e. that producing the resistance R_R . Then, the value measured during resistance determinations corresponds to the sum of R_M and R_I . Since R_M is known, R_I can be then evaluated.

The critical flux was determined applying a flux step method (16). It consisted of successive filtration steps of 10 minutes, with flux increments of 1 L/m² · h. One minute of back-flush was included between each cycle. Back-flush was applied by reversing the flow direction, keeping the flux at the same value as during filtration. Critical flux measurement automatically stopped when TMP reached 0.2 bar. The critical flux was assumed to be exceeded when the slope of the TMP against time failed a hypothesis test, with null hypothesis $dTMP/dt = 0$ (17). Significance level was 95%.

RESULTS AND DISCUSSION

Figure 2 presents the OLR applied to each reactor. Initial volumetric OLRs were different, due to different initial biomass concentrations. However, initial specific OLRs were similar, i.e. about 0.2 g COD/g VSS · d. The OLR of the mesophilic AnSMBR was increased to 10 g COD/L · d on day 26. Sludge wastes were frequently performed between days 15 and 52 to keep TSS concentrations below 40 g/L, as can be seen in Fig. 3. Effluent COD and reactor soluble COD concentrations remained low up to day 52, when a sharp increase was observed (Fig. 2). On day 55 permeate COD exceeded 2 g/L, and reactor soluble COD reached 5 g/L. VFA analysis showed that the COD increase was due to accumulation of acetate and propionate acids, in similar proportions. Reactor feeding was stopped on day 55, in order to allow reactor recovery. During mesophilic AnSMBR operation at an OLR of 10 g COD/L · d, 2.3 L of sludge were wasted from the reactor, which equals an overall sludge waste of 77.6 g VSS. If the period of operation at an OLR of 10 g COD/L · d is considered, an apparent yield of 0.080 g VSS/g COD can be evaluated, which is the result of biomass growth and the accumulation of non-degraded substrates. When measuring sludge stability on day 54 a value of 0.30 g COD/g TSS was obtained. The sludge sample for the stability measurement was not washed, so the degradation of the soluble organic matter present in the sludge also contributed to the value of the stability. The high apparent yield and the low sludge stability

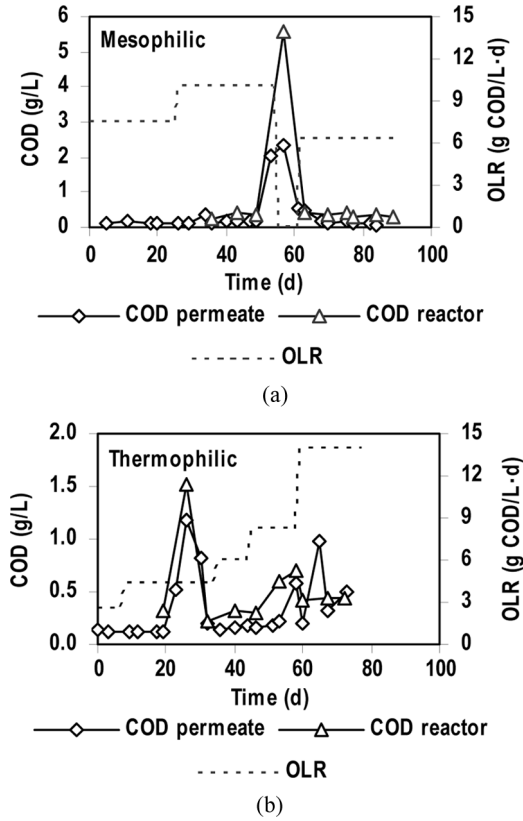


Figure 2. Applied OLR, permeate COD and soluble reactor COD during the operation of the mesophilic and the thermophilic AnSMBRs.

are indications of reactor overloading; even though the permeate COD concentration remained low until day 52. This is due to the fact that the membrane was apparently able to retain the unconverted particulate organics inside the reactor. The high level of sludge waste performed during the first 55 days of operation most likely resulted in the withdrawal of active methanogenic bacteria. Indeed, SMA decreased from 1.3 to 0.48 g COD/g VSS · d between start-up and day 55.

On day 62 the feeding of the mesophilic AnSMBR was resumed, but with an OLR of 6.3 g COD/L · d, corresponding to 0.32 g COD/g VSS · d. Between days 64 and 77 the observed yield was 0.040 gVSS/g COD, 50% lower than the one observed at the OLR of 10 g COD/L · d. During the last 10 days of operation (days 80–90), the reactor was fed solely with VFA, keeping the same OLR (6.3 g COD/L · d). During this

period, the VSS concentration indeed decreased, most likely due to degradation of the accumulated unconverted particulate substrate. The sludge stability at the end of the operation was 0.12 g COD/g TSS.

The behavior of the mesophilic AnSMBR shows that even though membrane filtration provides a solids free effluent, it is only a physical separation device. Organic matter degradation rates are dependent on the concentration of active microorganisms and a reactor overload obviously results in an accumulation of unconverted solids inside the reactor. Such accumulation subsequently dilutes the active biomass, and reduces the stability of the entire treatment system.

During the operation of the thermophilic AnSMBR, the OLR was gradually increased up to 14 g COD/L·d, as can be seen in Fig. 2. COD concentration remained low during most of the operational period (Fig. 2). An increase in permeate COD and reactor soluble COD was observed between days 22 and 29, and on day 65. This was caused by an accidental increase in the feeding of VFA, due to a setup malfunction. In both cases, the reactor COD dropped to the level prior to malfunction, after few days.

The VSS concentration of the thermophilic AnSMBR showed a steady increase from 13 to 30 g VSS/L (Fig. 3). No sludge waste was performed during the continuous reactor operation. An apparent yield of 0.034 g VSS/g COD is calculated, by using the reactors VSS increase throughout the operation, and the loading rates applied. Sludge stability was measured on days 42 and 75, giving a value of 0.15 g COD/g TSS in both cases. Specific OLR by the end of reactor operation was 0.47 g COD/g VSS·d. Results confirm that high degradation rates of thermophilic microorganisms constitute a positive factor for the treatment of high suspended solids wastewaters, by enabling higher organic loading rates.

Differences between permeate COD and reactor soluble COD remained most of the time in the range of 150–200 mg/L, for both reactors. Accumulation of soluble COD inside AnMBRs bioreactors have been previously reported (18–20). Ince et al. (18) observed a soluble microbial products (SMP) accumulation that was related with the applied OLR, when operating an AnMBR with brewery wastewater. However, no clear tendency was observed during the present research with respect to SMP accumulation. During the operation of both AnSMBRs, soluble proteins and carbohydrates in the mixed liquor were generally below 120 and 40 mg/L, respectively.

Figure 4 presents the applied flux and filtration resistance during the operation of both reactors. In both cases low fluxes were applied, i.e. in the range of 3 to 6 L/m²·h. The operation of the reactors was very unstable, with sudden increases in resistance, as can be seen in Fig. 4.

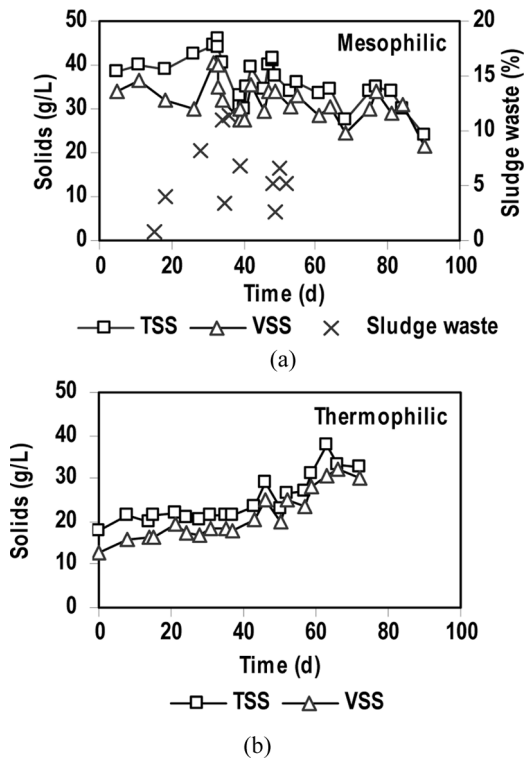


Figure 3. TSS and VSS concentrations during the operation of the mesophilic and the thermophilic AnSMBRs. In the case of mesophilic AnSMBR, the volume of wasted sludge is presented as a percentage of the useful volume of the reactor.

This was specially the case of the mesophilic AnMBR, forcing to several small adjustments in the applied flux. Low applied fluxes are in agreement with the low observed critical fluxes, presented in Fig. 5. Both reactors experienced a rapid decrease in the critical flux: from 20 to 4 L/m²·h for the mesophilic AnSMBR, and from 15 to 6 L/m²·h for the thermophilic AnSMBR. Different agents have been pointed out as fouling promoters during the operation of membrane bioreactors: biomass, colloids (21), soluble organic matter (8), inorganic precipitates (22,23), and extracellular polymers (24,25,26). However, particle deposition, and the consequent formation of a cake layer, was identified during this research as the most likely factor determining the applied flux, at both temperatures. TMP increase when surpassing critical flux showed to be almost fully reversible by the application of back-flush cycles or by simple filtration interruption. On the other hand, even though R_I increased during

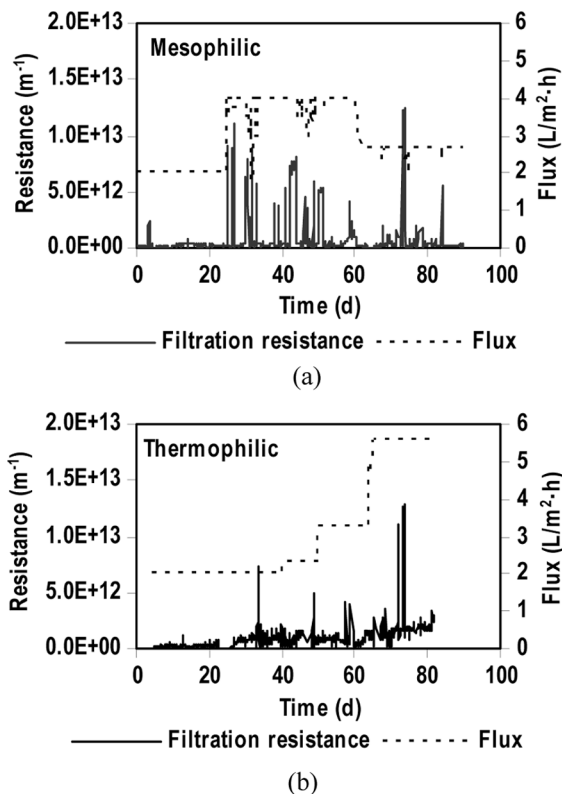


Figure 4. Filtration resistance and permeate flux during the operation of the mesophilic and the thermophilic AnSMBRs.

reactors operation, it remained below $7 \cdot 10^{11} \text{ m}^{-1}$ as can be seen in Fig. 5. Such value means that a TMP of only 0.016 bar is required as a driving pressure at a flux of $10 \text{ L/m}^2 \cdot \text{h}$, at 30°C . At 55°C , this value is 0.010 bar, due to the lower permeate viscosity. Such low level of irreversible fouling confirms that operational fluxes were indeed limited by a reversible process. This is interpreted as a clear indication that cake layer formation was the phenomena determining the critical flux, and therefore the applied flux during the operation of the reactors. This agrees with our previous results dealing with the application of AnSMBR to the treatment of soluble acidified substrates, both under mesophilic and thermophilic conditions (12,13).

Since particle deposition seems to be the decisive phenomena influencing the critical flux, an increment of surface shear should promote a higher level of back-transport, reducing particle deposition, increasing

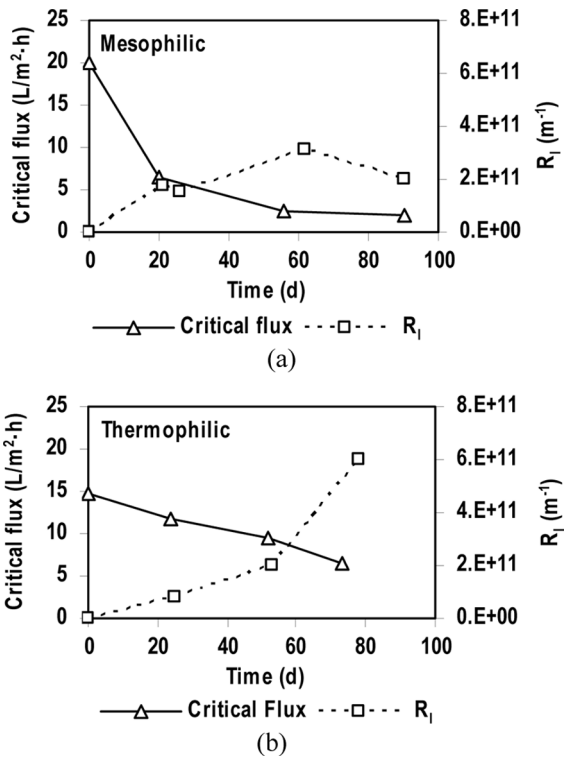


Figure 5. Critical flux at a V_G of 70 m/h and R_f , during the operation of the mesophilic and the thermophilic AnSMBRs.

the flux. Figure 6 presents the effect of V_G in the critical flux, measured several times during the operation of the reactors. The decrease in critical flux in time is evident, as previously discussed. At both temperatures, increase of V_G over a wide range of values resulted ineffective in achieving high critical fluxes. In both AnSMBRs, doubling the applied V_G produced an increase in critical flux of only 1–3 $L/m^2 \cdot h$. Apparently, gas sparging was ineffective in generating the high surface shear necessary to control particle deposition, under the conditions of the reactors operation.

Low levels of flux achieved during the present study would involve high costs when expressed per unit of permeate. Configurations that promote higher levels of surface shear, like the side-stream AnMBR may provide a higher level of flux. This, however, may come at the expense of a higher energy demand. For example, if we consider a side-stream AnMBR operating at a flux of 20 $L/m^2 \cdot h$, with a sludge viscosity of

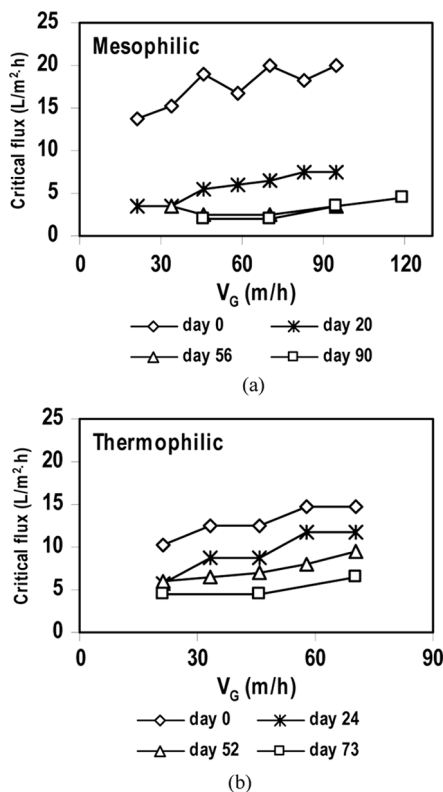


Figure 6. Effect of V_G on critical flux, during the operation of the mesophilic and the thermophilic AnSMBRs.

0.01 Pa·s and a cross-flow velocity of 1.5 m/s, energy requirement for pumping of the reactor liquor through the membrane module will be in the range of 5 kW·h per m³ of permeate (27). Nevertheless, the biogas produced during the reactor operation can be used as a source of energy, which may compensate for the increase in costs due to membrane filtration. Wastewaters containing particulate organics usually present a high COD concentration, which means an elevated methane production. At the inlet COD concentration used in this study, i.e. 10 g/L, 2.1 kg of methane will be produced per m³ of permeate, if we assume a COD removal of 95% and that 10% of the removed COD is used for cell growth production. This means 30 kWh of calorific energy per m³ of permeate, or 10 kW·h of electricity per m³ of permeate, assuming an efficiency of 35%. The latter energetic potential is twice the energy required for sludge pumping in the side-stream configuration.

CONCLUSIONS

AnMBR technology offers the possibility of total solids retention, enhancing the treatment of particulate organic matter. Furthermore, anaerobic treatment of wastewaters with a high organic fraction of suspended solids can be enhanced applying thermophilic conditions, which provides higher reaction rates and therefore higher organic loading potentials.

Low levels of irreversible fouling were observed over a 90 days period. Cake formation is the main phenomena limiting the attainable flux. However, gas sparging rate fails to provide enough surface shear to prevent cake formation, when working with tubular membranes in submerged AnMBRs, at high sludge concentrations. The use of biogas as an energy source may compensate for the costs due to membrane filtration implementation in AnMBRs.

ACKNOWLEDGMENTS

This research was financially supported by the European Union in the framework of the "Agroiwatech," project contract nr. ICA2-CT-2002-10010. Authors acknowledge the personal scholarship from the Chilean Government to David Jeison.

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