A novel Anaerobic Filter Membrane Bioreactor: Prototype start-up and filtration assays

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Abstract Anaerobic digestion allows efficient treatment of high loaded wastewater and membrane technology allows obtaining high quality effluents with complete biomass retention. However, high biomass concentration interferes with membrane fouling. In the present work a new bioreactor that integrates an attached biomass anaerobic culture on a fixed bed and a submerged membrane has been started-up. The recirculation between the digestion and filtration chambers is coupled to the gas-lift effect of the bubbling employed for the scouring of the membranes avoiding the use or electromechanical pumps that damage the suspended biomass. The support material retains the biomass in the digestion tank despite the downwards flow, avoiding that the submerged membrane contacts with a high concentrated suspension. This novel system, called Anaerobic Filter Membrane Bioreactor (AnFMBR) was immediately started up, achieving COD removal efficiencies of 96% at OLR of 7 kg COD/m^3·d. In order to select filtration flux, specific gas demand and backwash interval, the results of fifteen short term assays, 8 hours each one, is presented for fluxes between 15.7 and 17.7 L/m^2·h, backwash interval between 10 and 30 minutes, and three levels of scouring. It was checked that reversible and irreversible fouling were directly related when dTMP/dt > 2.5 mbar/min.

Keywords: Anaerobic Membrane Bioreactors; Anaerobic Filter; Anaerobic Filter Membrane Bioreactor; gas-lift

INTRODUCTION

Industry wastewater characterizes by its high pollutant loads, whose profile is determined by the type of industrial activity. The main contaminants found in effluents produced by food or slaughterhouse factories have an organic nature and usually show high levels of BOD and SS (Ferreira *et al.* 2018). Elevated operational and management costs make aerobic biological treatment unsuitable for high-strength wastewater. The generation of energy in the form of methane gas associated to the reduction of organic load makes anaerobic digestion a viable alternative though. In addition to that, this process eliminates the need for aeration and its biomass growth rate is smaller, resulting in lower sludge generation (Ward *et al.* 2008; Hamza *et al.* 2016).

Anaerobic bioreactors have been widely used to treat industrial wastewater and there are many configurations to choose from, depending on the nature of wastewater (McCarty 2001). High-rate anaerobic bioreactors are particularly interesting. The low growth rate of anaerobic biomass makes the efficiency of anaerobic systems dependent on the capacity of the reactor to retain biomass. High-rate anaerobic bioreactors uncouple hydraulic retention time (HRT) from solids retention time (SRT), using different ways to accumulate biomass (van Lier *et al.* 2008). Anaerobic contact process (ACP) involves a secondary clarifier with return flow, similar to the activated sludge processes. The upflow anaerobic sludge blanket reactor (UASB) uses biomass granulation in such a way that its size and density are high enough to stands in the reactor forming a blanket that tolerates the water upward flow, so biological treatment and clarification steps are combined in only one reactor. Biomass can also be attached to an inert support material placed into the reactor. Bioreactors based on this principle are known as anaerobic filters (AF).

An AF is a fixed-bed biological reactor with a series of one or more filtration chambers. As wastewater flows through each chamber, particles and organic matter are trapped and degraded by active biomass retained by the inert support material. The ways biomass is retained by support material are: biofilm formation on carrier material, entrapment of suspended sludge in interstitial void space and formation of well settling sludge aggregates (Young 1991). There is not a recommended support material for each application. On the contrary, it is possible to find several options for packaging in literature. Rocks, coral or mussel shells, polypropylene or other plastic materials or non-woven fabric media have produced good results.

AF can be operated in upflow and downflow configurations. In upflow configuration, wastewater is pumped to the bottom of the reactor and flows upwards passing through the interstices of the support material were the biomass is retained. The treated effluent is recovered at the upper part of the tank. On the other hand, in downflow configuration, wastewater is directly pumped to the top of the tank and over the support material; then it is treated on its way down to the button of the tank (Young & Dahab 1983). This technology has been successfully used to treat different high strength wastewaters such as pig slurry (Wilkie & Colleran 1986), distillery waste (Silverio *et al.* 1986), abattoir (Gannoun *et al.* 2009), meat and dairy industries (León-Becerril *et al.* 2016; Kispergher *et al.* 2017) and vinasse (Cabrera-Díaz *et al.* 2016). COD removal efficiencies reached values between 80 – 90% in most cases.

As in other anaerobic technologies, further treatment steps could be necessary to stabilize the AF effluent, especially in the treatment of high-strength wastewater. An option to improve the effluent quality could be to combine AF technology with membrane bioreactors (MBR), creating a hybrid system (Hamza et al 2016). The use of membranes not only improves the biological effluent quality, but also allows to overcome some bioreactors weakness points. Membranes retain biomass that would be missed from the system due to unfavourable characteristics of the wastewater as slowly biodegradable solids or oil and grease in suspended growth reactors, or simply they can help biomass to stay in the system in order to form biofilms on the inert support media, mainly in start-up periods (Judd 2011). This last benefit refers to one of the handicaps for AF technology: biomass attachment to support material is a slow process that requires long start-up periods (Tilley *et al.* 2014).

When combining AF and AnMBR, the main obstacles of both technologies can be overcome. Long start-up periods and clogging issues of AF reactors and membrane fouling as the main drawback of AnMBR technology. Lower suspended biomass concentration, proper filtration-backwashing cycles, scouring and chemical cleaning (Judd 2011), can deal with membrane fouling, whilst clogging is controlled by the superficial velocity and support media configuration (Young & Dahab 1983).

A novel integrated system named Anaerobic Filter Membrane Bioreactor (AnMBR) that combines a downflow anaerobic filter and a submerged ultrafiltration tank, in which the recirculation between the two section is coupled with the membrane scouring by gas-lift effect, is presented in this paper. Early results including a prototype start-up and ultrafiltration assays are shown.

MATERIALS AND METHODS

Experimental setup. The AnFMBR consisted of a vertical vessel with a volume of 0.18 m^3 divided into two chambers: downflow anaerobic filter; and an upflow filtration tank where a submerged membrane was placed (Figure 1). A floating plastic material (Biofill-C, Bio-fil, Spain) with a specific surface of 460 m^2/m^3, was used as support media for biomass immobilization. The filtration unit was equipped with a PVDF hollow fibre membrane with a pore size of 0.04 µm and a filtration area of 0.93 m^2. Biological chamber and the concentric filtration tank were connected at the bottom, through holes that prevent the passage of support media to filtration tank, and by pipes located at the upper part of the filtration tank that overflow over the biological chamber.

Wastewater was fed by a peristaltic pump (Watson Marlow 520U) over the upper part of the biological zone, with a homogenous distribution over support media. A reversible wear pump (Micropump Eagle Drive GJ-N21) was used for filtration and backwash. The filtration pump is connected to a permeate tank that collects membrane effluent for backwashing.

Biogas, collected from the upper part of the vessel, was recirculated through the membrane for scouring. Two diaphragm compressors of different flow rate (Secoh SV50 and KNF laboport) were alternatively used to adjust specific gas demand (SGD). Gas sparging provokes recirculation between the two chambers by gas-lift effect. An additional compressor (Secoh SV50) was intermittently used to intensify gas-lift avoiding any other pumping devices for the mixed liquor circulation.

Temperature of biological process was kept between 35±0.5 ºC by means of an electric blanket heating. Electronic pressure sensors (PN 2569, IFM) monitored transmembrane pressure and reactor level. Temperature (TR2432, IFM), pH (Liquiline CM14, Endress + Hausser) and biogas production (FCI ST75) were continuously monitored, and the biogas flow rate of scouring was measured using a rotameter (PS Series, Tecfluid). The entire system was controlled by an Arduino based PLC (M-Duino 38R, Industrial Shields) connected to a PC for remote control and real time monitoring.

Inoculation and start-up. The bioreactor was inoculated with 90 L of dispersed anaerobic sludge from a food industry biowastes digester (Ecoalia, Burgos, Spain) with a total and volatile solids concentration of 56 and 42 g/L, respectively. The system was started-up and operated during 6 weeks at the laboratory, fed with synthetic wastewater before moving to an industrial slaughterhouse (Campofrio Food Group, Burgos, Spain), where raw wastewater was fed. Solids retention time was controlled by daily wastage of 2 L of sludge from the filtration chamber.

Wastewater characteristics Synthetic wastewater were prepared by diluting in water dry pet food, which main ingredients are poultry meat and animal grease, to a certain extent seen as wastewater. Because of laboratory storage capacity 50 L were prepared daily. COD concentration was adjusted between 6.4 and 22 g/L according to organic loading rate target, between 2 and 7 kg $COD/m^3 \cdot d$. Once in the slaughterhouse wastewater concentrations were in the range of 2530 to 5210 mg/L of COD, 1150 to 2030 mg/L of TOC, 286 to 403 mg/L of TN and 830 to 960 mg/L of oil and grease.

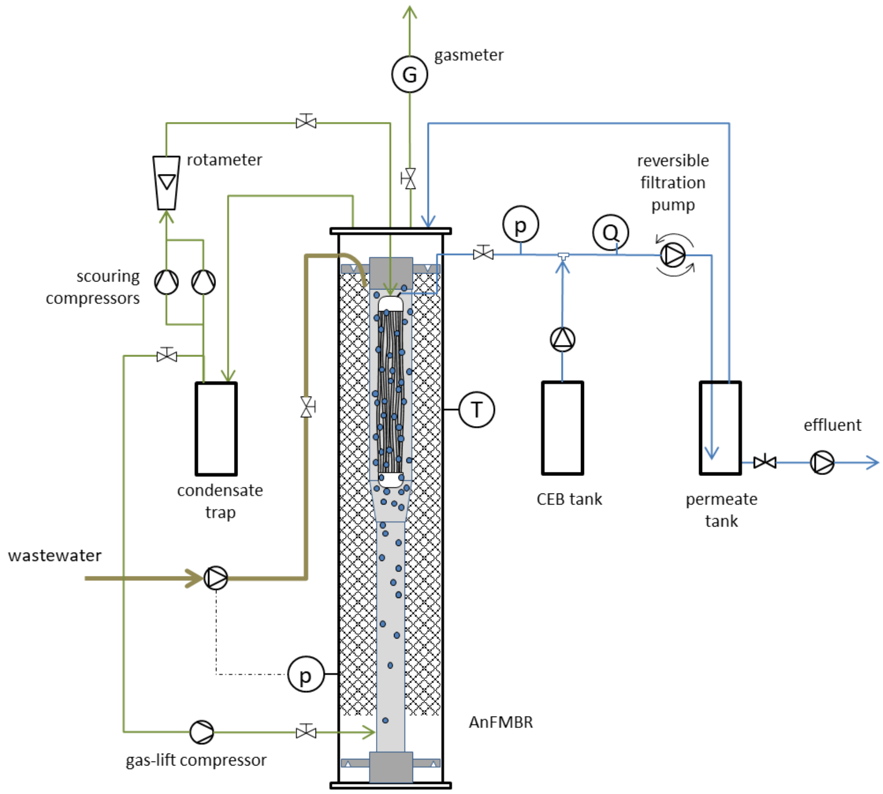

Figure 1. Schematic diagram of the AnFMBR prototype

Physical and chemical analysis. Samples of permeate and mixed liquor from filtration tank were taken daily. Volatile Solids (VS), Total Suspended solids (TSS), Volatile Suspended Solids (VSS), Chemical Oxygen Demand (COD), total alkalinity were analysed according to Standard Methods for the Examination of Water and Wastewater (APHA 2001). Total Organic Carbon (TOC), Inorganic Carbon (IC), Total Carbon (TC) and Total Nitrogen (TN) using a TOC/TN analyser (Shimadzu). Biogas composition was determined using a Multitec 545 mobile gasometer device with H_2S, O_2, CO_2 and CH_4 sensors.

Filtration assays. The filtration flux, backwash interval and specific gas demand were varied. Specific gas demand was adjusted by combining two compressors of different flow which individual SGD were 1.1 and 1.5 $Nm^3/m^2{\cdot}h$. The compressors operated intermittently in 30 seconds intervals, distributed in 10/20, 15/15 and 20/10 seconds, so average SGD was 1.23, 1.30 and 1.37 $Nm^3/m^2{\cdot}h$.

Filtration flux was adjusted at 15.8±0.1, 16.7±0.2 and 17.7±0.2 $L/m^2{\cdot}h$ being backwash flux 150% of filtration flux. Net flux was determined by subtracting from the volume filtered the volume used in the backwash and dividing by the total duration of the cycle. The cycles are composed of four stages: 30 s for relaxation between filtration and backwash, 30 s of backwash, 30 s for relaxation between backwash and filtration, and a variable time of filtration, until the total duration cycle or backwash interval, 10, 20 and 30 min.

Table 1. Operating conditions for filtration cycle optimization

J ($L/m^2 \cdot h$)	Backwash interval (min)	SGD ($Nm^3/m^2 \cdot h$)
15.8±0.1	10	1.30
	20	1.23
		1.37
	30	1.30
16.7±0.2	10	1.23
		1.37
	20	1.30*
	30	1.37
		1.23
17.7±0.2	10	1.30
	20	1.23
		1.37
	30	1.30

(*) 3 replicates were carried out

Table 1 shows the combination of the 3 parameters employed in the fifteen assays that always included a middle condition, excluding the assays in which the 3 parameters take extreme values. Unlike short-term flux-steps experiments performed in the classical critical flux assessment, the operating conditions tested in this work were performed with short-term assays of 8 h, 7 h at the selected operating conditions and 1 h for critical flux and compressibility determination.

RESULTS AND DISCUSSION

Biological behaviour of the AnFMBR

Inoculation and lab start-up. One of the best advantages of the novel technology was the short start-up time, even from an uncolonized support media, since it was not indispensable the initial biomass adhesion. Immediately after inoculation an OLR of 2 kg $COD/m^3 \cdot d$ was applied and 7 kg $COD/m^3 \cdot d$ was reached at week 2. The removal efficiency reached up 96% but a significant increase in effluent COD, from 532 to 805 mg/L justified a conservative decrease of OLR to 4 kg $COD/m^3 \cdot d$ to avoid biological instabilities during the filtration assays. On that conditions the removal efficiency was kept between 93 and 97% with COD concentration in the effluent between 158 and 373 mg/L. Analysis of biogas composition showed a maximum methane concentration of 73% and biogas production was about 6.1 kg $COD_{CH4}/m^3 \cdot d$ for the highest organic load. The only negative feature of the effluent was a yellow tone possibly due to humic matter realised from the biomass because the shear stress provoked by scouring (Fillow *et al.* 2012; Ding *et al.* 2016).

At the inoculation stage the support media had initially a filter effect. After 24 h of operation with wastewater feeding and recirculation, and before the experimental failure described below, the suspended solids in the mixed liquor of the filtration tank was kept between 5480 and 10920 mg/L, what means a biomass retention in the support material zone between 62% and 80% of the inoculum. A failure of the level control on day 19 led the pass of pieces of the floating support media to the filtration zone. It was necessary a partial drain and replacement of the filter that led to an increase in the suspended solids concentration in the mixed liquor up to 21330 mg/L that slightly decreased in the following weeks.

Slaughterhouse acclimation. Since organic matter concentration in the slaughterhouse wastewater were notably lower than the synthetic ones lower OLR was used in the acclimation. The applied OLR was increased in the first week from 1.6 to 2.9 kg COD/m^3·d, and 4.7 kg COD/m^3·d the next, value that was maintained during the first month with organic matter removal efficiency up to 89%, total alkalinity between 2760 – 3130 mg $CaCO_3$/L normal values for anaerobic processes and an alkalinity ratio lower than 0.27, showing absolutely stable behaviour.

Short-term filtration assays

Filtration flux was maintained between 12 and 22 L/m^2·h. The highest fluxes 18 – 22 L/m^2·h were employed in the startup and the lowest filtration fluxes 12 – 14 L/m^2·h were punctually employed after operational failures or as a conservative flux previous to filtration assays. The habitual filtration flux was in the range of 15 – 18 L/m^2·h higher than the membrane flux applied in most AnMBR studies, lower than 12 L/m^2·h (Lin *et al.* 2013; Ozgun *et al.* 2013). Short-term filtration assays are presented below.

Filtration flux, J. Figure 2 shows the influence of filtration flux over irreversible membrane fouling. The AnFMBR was operated under the same SGD and backwash interval, 1.30 Nm^3/m^2·h and 10 min, respectively, and the filtration flux was fixed at 15.7 and 17.9 L/m^2·h.

An increase on filtration flux resulted on both reversible and irreversible fouling rise. Irreversible fouling, imperceptible when reversible was 0.60 mbar/min at 15.7 L/m^2·h, went up to 0.45× 10^{12} m^{-1} per cubic metre filtered by square metre of membrane for the flux of 17.9 L/m^2·h, when dTMP/dt was 3.0 mbar/min.

It is important to note that the assays included in this work were conducted immediately after the start-up period and that abnormal membrane fouling at the beginning this period was produced, when the control parameters were being adjusted, and consequently the filtration fluxes used were under the filtration capacity of the membrane in usual conditions.

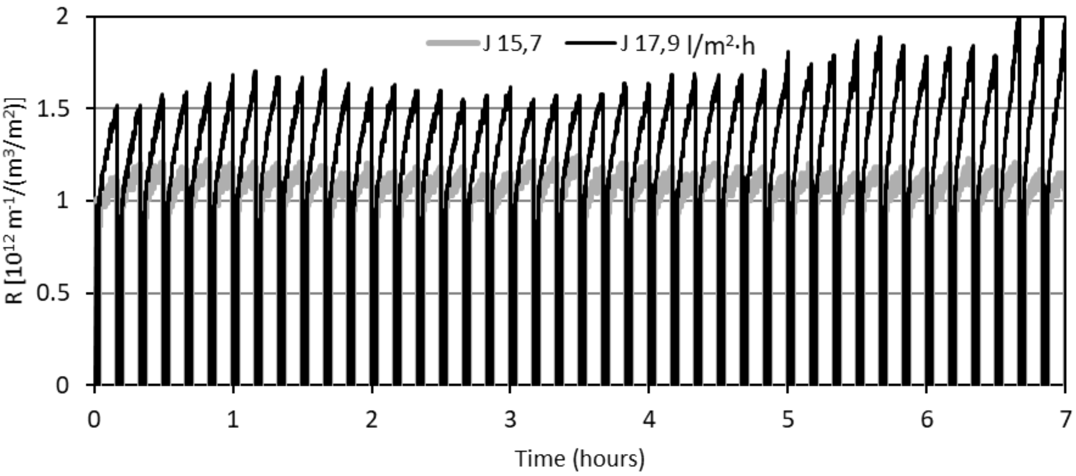

Figure 2. Evolution of filtration resistance at 15.7 and17.9 L/m^2·h for backwash interval of 10 min and SGD of 1.30 Nm^3/m^2·h

Backwash interval. To evaluate the influence of backwash interval over irreversible fouling, two backwash intervals, 10 and 30 min, were compared under a flux of 17.7±0.2 L/m^2·h and SGD of 1.30 Nm^3/m^2·h.

Huber's robust regression method was used for fouling rate calculation that reduces statistical weigh of values deviating from the global linear trend and increases the confidence interval for the calculated slope. In Figure 3 points weighed under 75% are shown between round brackets whereas those weighed under 50% are between square ones.

The increase of fouling over time was 0.129 and 0.351× 10^{12} m^{-1}/d for backwash interval of 10 and 30 min respectively, which resulted in a fouling rate 272% higher for the longer filtration cycle. However, increasing backwash frequency lowers net flux due to the reduction of the filtration time and the consumption of permeate during backwash. Notwithstanding, and even though net flux for the 10 min backwash interval, (14.1 L/m^2·h) was significantly lower than the net flux for the 30 min interval (16.5 L/m^2·h) the irreversible fouling on the net volume filtered basis was still far higher (232%) for the cycle time of 30 min than the one for the more frequent backwash, every 10 min.

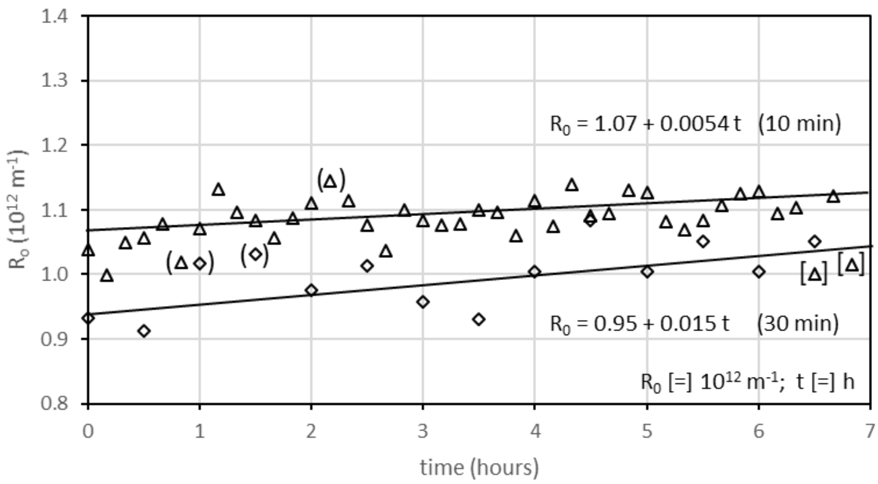

Figure 3. Effect of backwash interval on membrane resistance

Specific Gas Demand. Optimization of membrane gas scouring is a key factor in the reduction of the specific energy demand in hollow-fibre MBR's (Krzeminski, *et al.* 2017). In this study, three levels of scouring were tested in combination with filtration flux and backwash frequency. The specific gas demand used, between 1.23 and 1.37 Nm^3/m^2·h, seems higher than usual, but it is necessary to consider that the height of the experimental module was only 0.69 m, so that the SGD used corresponds to 0.42 – 0.47 Nm^3/m^2·h in an industrial module of 2 m in height. Figure 4 shows that irreversible fouling rate is very sensitive to scouring intensity. The mean irreversible fouling rate decreased from 0.72 to 0.01 × 10^{12} m^{-1}/(m^3/m^2) when SGD increased from 1.23 to 1.37 Nm^3/m^2·h. The standard deviations were logically high since the correspondent assays were carried out with different filtration flux and different backwash interval according experimental program presented in Table 1. It is also important to emphasize that some negative irreversible fouling rate were obtained after unfavourable filtration conditions, high permeate flux and/or long cycles, when more favourable conditions in flux and/or backwash frequency, are employed. That means that the part of the fouling that cannot be removed by backwashing can be removed without chemical cleaning, so that irreversibility of fouling depends on the operating conditions being possible to detach a part of non-consolidated fouling when operating conditions are moderated. Wu *et al.* (2008) found that relaxation was effective in removing irreversible fouling to some extent by removing soluble microbial products, despite it was not effective in preventing pore blocking. The possibility of reversing membrane fouling by changing filtration conditions was reported by Defrance and Jaffrin (1999), however they encountered that when critical flux was exceeded severe irreversible fouling was observed, since in our work irreversible fouling developed above the critical flux was partly detached when flux was set back to sub-critical level.

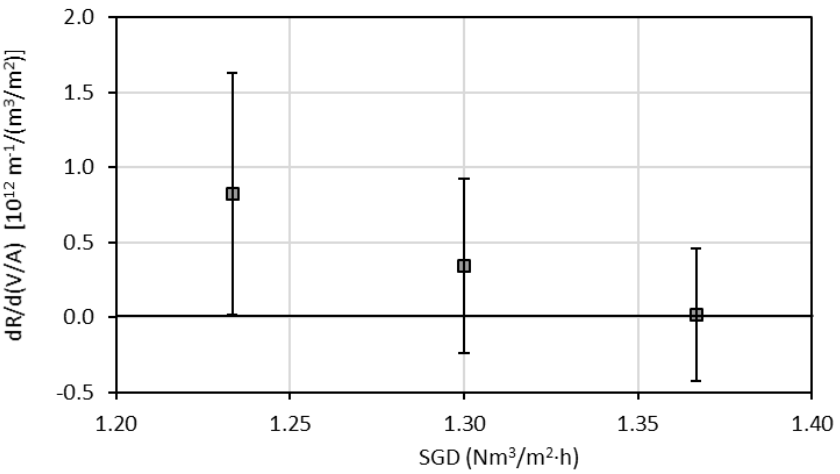

Figure 4. Irreversible fouling rate on net volume basis vs specific gas demand

Reversible vs irreversible fouling A widely accepted recommendation to reduce irreversible fouling is by keeping the filtration flux below the critical flux (Bacchin *et al.* 2006). The then so-called critical flux is taken to be the highest flux at which the TMP curve remains horizontal. For the determination of critical flux different criteria for the TMP gradient were chosen. The value recommended by Le-Clech *et al.* (2003) was 0.1 mbar/min, in the Berlin Filtration Method test cell critical flux was determined as the flux at which dTMP/dt was greater than 0.2 mbar/min, Le-Clech *et al.* (2006) assumed an arbitrary fouling rate threshold of 0.5 mbar/min, and De la Torre *et al.* (2009) and Dereli *et al.* (2014) chosen an arbitrary slope of 1 mbar/min was arbitrary to decide whether the critical flux was reached.

The irreversible fouling rate, as resistance increase over net volume filtrate per unit of surface area, in 10^{12} $m^{-1}/(m^3/m^2)$, for the different combinations of operating condition was represented versus the reversible fouling rate, as slope of TMP in mbar/min (Figure 5). It was checked that above 2.5 mbar/min irreversible fouling rate was directly related to the slope of TMP. Below 2.5 mbar/min the irreversible fouling was practically null, even negative according to the previous subsection, but the opposite situation also occurred and, irreversible fouling rate up to 0.45×10^{12} $m^{-1}/(m^3/m^2)$ was reached after a relaxed filtration period for the lowest flux and maximum SGD, despite dTMP/dt was only of 0.86 mbar/min.

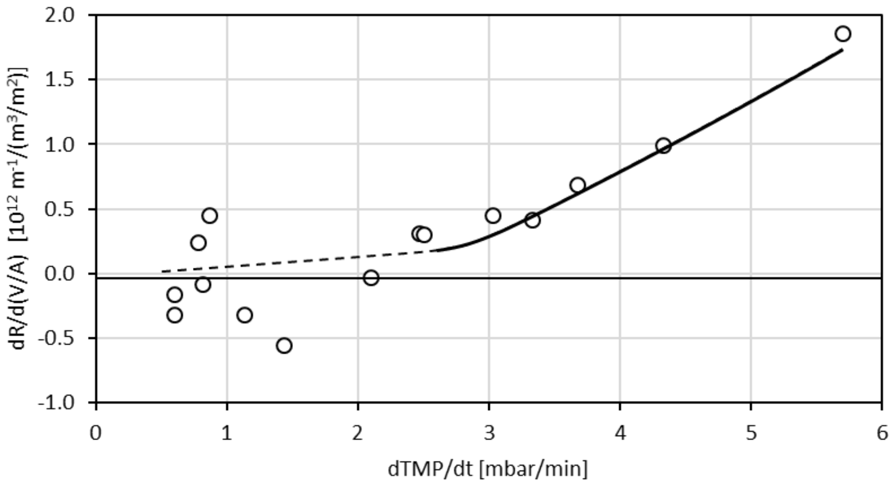

Figure 5. Irreversible fouling rate on net volume basis vs reversible fouling

Critical flux evolution. After 7 hours of filtration at the selected operating conditions backwash resistance, compressibility index and critical flux were measured. Figure 6 shows a typical TMP profile for the determination of critical flux. The backwash resistance and the compressibility index remained practically constants in $0.94 \pm 0.05 \times 10^{12}$ m^{-1} and 0.49 ± 0.06, respectively, which means that not significant increase on internal resistance neither change in fouling elasticity was detected.

The critical flux remained between 15.3 and 15.9 L/m^2·h, any effect of operating filtration flux or backwash interval was observed, but the influence of SGD on critical flux was noticed (Figure 7). It was checked that critical flux slightly increased from 15.56 ± 0.10 to 15.90 ± 0.06 L/m^2·h when SGD increased from 1.23 to 1.37 Nm^3/m^2·h. Despite the filtration fluxes employed during the fifteen assays, 15.7, 16.8 and 17.7 L/m^2·h, were equal or greater than the critical flux, the irreversible fouling rate was only 0.10×10^{12} m^{-1}/d that in increase of transmembrane pressure equals to 3.7 mbar/d.

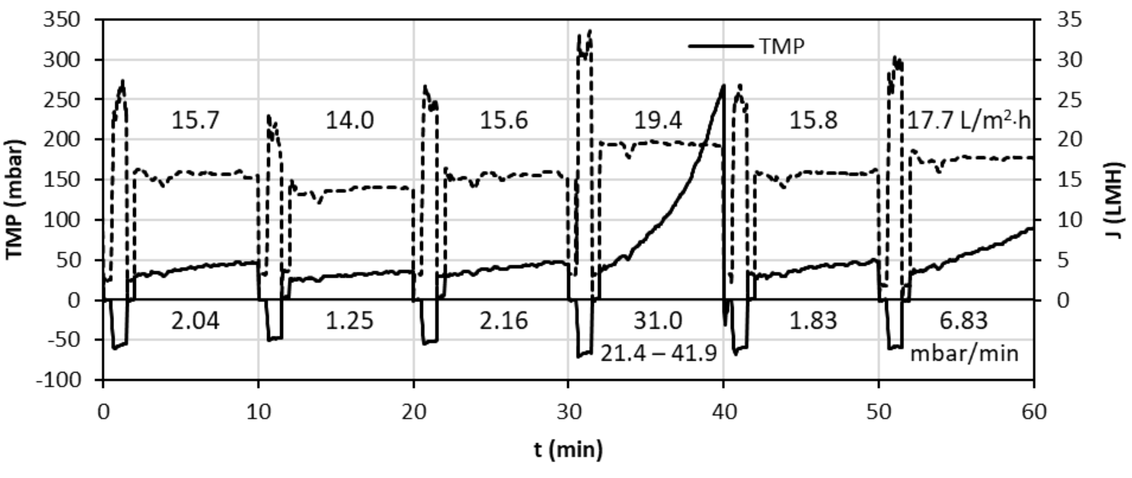

Figure 6. Flux and TMP profile during the determination of critical flux

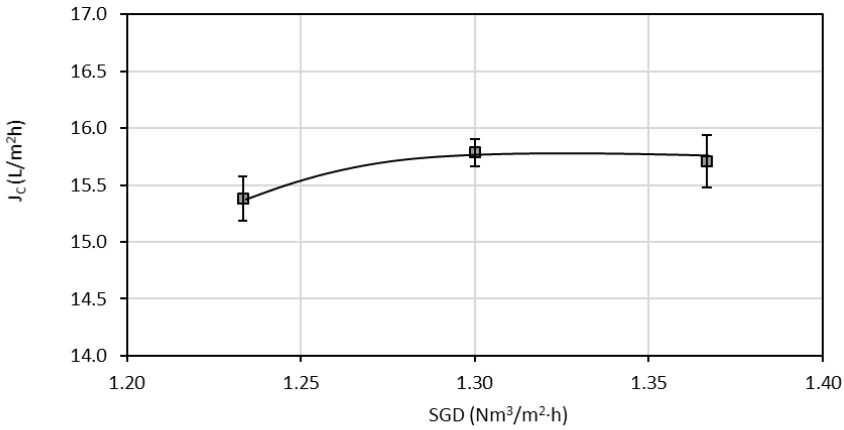

Figure 7. Effect of specific gas demand on critical flux

The critical flux for irreversibility is defined as the first permeate flux for which cake layer becomes too cohesive, pore blocking becomes noticeable and/or an irreversible gel structure due to adhesion of foulants to the membrane appears. From a practical point of view critical flux for irreversibility can be determined as the flux at which the transmembrane pressure curve starts to deviate from linearity (Bacchin *et al.* 2006). Figure 6 shows how for a flow of 17.7 L/m^2·h, despite the TMP slope was 6.8 mbar/min the TMP profile is still linear, whereas for a flux of 19.4 L/m^2·h TMP profile deviates clearly from linearity indicating a higher propension to irreversible fouling.

CONCLUSIONS

An Anaerobic Filter Membrane Bioreactor (AnFMBR) composed by a downflow anaerobic filter and a submerged membrane tank was started-up and acclimated to slaughterhouse wastewater in a short period of time. Membrane filtration allows operating a moderate to high organic loading rate, up to 7 kg/m^3·d, even from plastic support media without any previous conditioning, just after inoculation.

Gas sparging for membrane fouling control generated an upwards flow in the filtration tank and downwards flow in the anaerobic filter chamber without the need of any other pumping device. Gas-lift recirculation avoids any clogging problem in the anaerobic filter, even after an operational problem that led to an increase in suspended solids up to 21330 mg/l.

The retention of biomass on the downflow anaerobic filter chamber reduced the suspended solids concentration in the filtration tank and membrane fouling. Filtration fluxes of 15 – 18 L/m^2·h, higher than the membrane flux applied in most AnMBR, were maintained without significant irreversible fouling increase when reversible fouling rate was under 2.5 mbar/min.

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