Impact of organic flux enhancer on pilot anaerobic membrane bioreactor (AnMBR).

Additional thesis

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ABSTRACT

In this work, the effects of the addition of a cationic polymer ADIFLOC KD 451 (ADIPAP, France) in the performance of a decentralized Anaerobic Membrane Bioreactor (AnMBR) pilot plant treating 1.5 m^3 blackwater per day was studied. To this end, on-line sludge filterability characterizations were performed following the Delft Filtration Characterization (DFC) method. Likewise, a Supervisory Control and Data Acquisition (SCADA) system was used to measure and record the variations in transmembrane pressure (TMP). Polymer addition resulted in the modification of the biomass properties such as increased particle size measured as d50 from 19.49µm to 32.85µm and lower colloidal particles concentration. The combined effect of these changes influenced the fouling cake layer development rate. The preceding resulted in lower TMP in the pilot plant which indicates that higher operational fluxes can be achieved due to flux enhancer addition.

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List of symbols

m	Mass (g)
t	Time (s)
ρ	Density (Kg m^{-3})
T	Temperature (°C)
υ	Cross flow velocity (m s^{-1})
Q	Sludge flow (m^{3} h^{-1})
A_{cs}	Area membrane cross section (m^{2})
P_f	Feed pressure (bar)
P_c	Concentrate pressure (bar)
P_p	Permeate pressure (bar)
ΔR	Additional resistance of the fouling layer (m^{-1})
R_t	Total resistance (m^{-1})
TMP	Transmembrane pressure (bar)
η	Permeate viscosity (Pa s^{-1})
J	Flux (L m^{-2} h^{-1})
R_m	Membrane resistance (m^{-1})
ΔR_{20}	Estimated additional resistance when V=20Lm^{-2} ($1x10^{12}\,m^{-1}$)
V	Volume of permeate per m^{2} of membrane (Lm^{-2})

List of abbreviations

AnMBRs	Anaerobic membrane bioreactors
BFM	Berlin filtration method
CFV	Cross flow velocity
COD	Chemical oxygen demand
DFC	Delft filtration characterization
EPSs	Extracellular polymeric substances
GAC	Granular activated carbon
HRT	Hydraulic retention time
MBRs	Membrane bioreactors
MFI	Modified fouling index
MLSS	Mixed liquor suspended solids
OLR	Organic loading rate
PAC	Powdered activated carbon
PACl	Polyaluminum chloride
PAM	Polyacrylamide
PSD	Particle size distribution
SCADA	Supervisory control and data acquisition
SMPs	Soluble microbial products
SRT	Sludge retention time
SVI	Sludge volume index
TMP	Transmembrane pressure
TS	Total solids
TSS	Total suspended solids
VFM	VITO fouling measurement
VS	Volatile solids
VSS	Volatile suspended solids

Impact of organic flux enhancer on pilot anaerobic membrane bioreactor (AnMBR) fouling rate.

1. Introduction

In recent years, water resources depletion has led to the exploration of unconventional wastewater treatment schemes (Opher & Friedler, 2016). Ordinarily, water supply and treatment systems are based on a centralized approach however as the urban population is growing many agree that the conventional approach for wastewater treatment must be shifted from a centralized to a decentralized approach (Capodaglio, 2017; Larsen *et al.,* 2013). In the same way, source separation presents several advantages, with one of the most important being resource recovery as it involves the segregation of wastewater discharges into different streams such as blackwater and greywater. These streams have diverse characteristics in terms of pollutants and hazardous organisms hence their treatment schemes differ (Opher & Friedler, 2016). Blackwater streams are comprised of flush water, feces, urine and toilet paper, which result in a high organic content that needs to be removed to a great extent in order to produce water suitable for discharge and/or reuse. In order to achieve organics removal, different purification processes can be applied; it has been found that blackwater obtained from conventional toilets is suitable for anaerobic digestion systems. For instance, Gao *et al.,* (2018) carried out a biomethane potential assay with a retention time of 46 days under mesophilic conditions that resulted in 48% of methane production in relation to the feed chemical oxygen demand (COD). These represent an advantage from the energy recovery point of view (Bartacek *et al.,* 2017).

Over the past years the diffusion of membrane bioreactor technologies has been increasing. It has been demonstrated that these systems offer several advantages over conventional wastewater treatment systems such as a higher effluent quality, increased disinfection capability, and reduced footprint (Lin *et al.*, 2014). Membrane systems can be operated under aerobic or anaerobic conditions; the latter has the main advantage of producing energy in the form of biogas and a lower sludge yield. Anaerobic membrane bioreactors (AnMBRs) lead to reduction in the overall energy consumption of the waste water treatment plants as no aeration is required in these systems compared to conventional activated sludge systems (Robles *et al.,* 2013). Furthermore, AnMBRs can be operated under extreme conditions such as high salinity, high-suspended solids content and poor biomass granulation (Dvořák *et al.*, 2015). Additionally, AnMBR effluent has a high quality, containing macronutrients such as ammonia and orthophosphate that make it suitable for direct use in irrigation/fertilization systems: this is especially important for water depleted regions (Ellouze *et al.,* 2009). Moreover, AnMBRs effluent is relatively pathogen free with the microorganisms being retained in the membrane (Ozgun *et al.*, 2013).

However, widespread application of AnMBR technology faces several constraints such as low flux and high capital and operational costs that are related with membrane fouling (Zhang *et al.*, 2010). Membrane fouling can be classified into reversible, irreversible and irrecoverable fouling according to the type of cleaning needed to remove it. Reversible fouling can be removed by physical techniques such as relaxation and/or back washing as it is formed mainly by loosely bound particles; irreversible foulant particles are strongly adhered to the membrane and can only be removed by applying chemicals (Huyskens *et al.*,2008); and irrecoverable fouling cannot be removed by either physical or chemical cleaning. According to Bagheri & Ahmad (2018), three main factors are related with membrane fouling: membrane characteristics, reactor operating conditions and biomass characteristics. There are several approaches for the reduction of membrane fouling rate such as increasing fluid velocity, prolonged relaxation periods, physical cleaning by backwash, and addition of flux enhancers, among others. By altering the biomass properties, an increase on operational membrane flux could be achieved (Diaz *et al.*, 2014). In this sense, the addition of flux enhancers can be seen as a reliable technique. Flux enhancers are adsorbents, coagulants and flocculants that help increase the sludge filterability by different mechanisms such as adsorption, coagulation and flocculation of soluble microbial products (SMPs) (Drews, 2010).

Nguyen *et al.*, (2010) tested the individual and combined effects of the addition of inorganic and organic flocculants ($FeCl_3$ with MPE50) to an AnMBR. Both flocculants impacted the molecular weight distribution of the soluble microbial products and the particle size distribution of the sludge flocs; the fouling rate was assessed by observing the changes in transmembrane pressure (TMP) which was reduced from 5 $kPad^{-1}$ to 1.3 and 3.3 $kPad^{-1}$ with $FeCl_3$ and MPE50, respectively. In the same way, the combined addition of the flocculants resulted in a fouling rate reduction of 58% to 83% when compared to the individual addition. Similarly, Zhang et al, (2014) found that the addition of MPE50 leads to a reduction in the membrane fouling rate by changing the sludge properties such as, reduction of soluble microbial particles concentration, larger particle size, increased zeta potential and enhanced hydrophobicity of the flocs. The sludge filterability at different MPE50 concentrations was measured by applying the modified fouling index (MFI) protocol; the membrane fouling rate was observed according to the changes in the TMP. The addition of MPE50 resulted in a significant reduction of the TMP change from 12.17 $kPad^{-1}$ to 2.34 $kPad^{-1}$.

Sludge filterability has been found to be correlated with the membrane´s fouling rate. Therefore, in order to test the effects of the addition of flux enhancers in AnMBR systems a sludge filterability characterization method can be conducted. The characterization must be carried out before and after flux enhancer addition; the latter can be done during an established period of time to observe not only the immediate effect of the flux enhancer addition but also the changes in time due to flux enhancer depletion. Furthermore, it is important to conduct assays to determine the optimal concentration of flux enhancer before this is added to the system under study. On this basis, the Delft Filtration Characterization (DFC) method which makes use of a DFC installation can be considered as a valuable method since it allows the determination and comparison of sludge filterability under different conditions. The DFC installation, records the changes in membrane fouling under fixed operating conditions (*e.g.* cross-flow velocities, permeate production) and under fixed membrane characteristics. Therefore, the additional resistance given by the sludge can be measured which results in the characterization of the sludge filterability. The DFC method gives as a main output ΔR_{20} values (*i.e.* is the cake layer resistance after 20 L of permeate per m^2 of membrane surface have been extracted).

In this work, the effect of flux enhancer addition to a pilot AnMBR on sludge characteristics and reactor performance was studied. The system under study is comprised of a decentralized AnMBR unit treating 1.5 m^3 of blackwater per day obtained from Porto do Molle business center located in Nigran, Spain. The flux enhancer used was ADIFLOC KD 451, which is a cationic polymer obtained from ADIPAP Company (ADIPAP, France). The sludge characteristics studied were as follows: filterability, solid concentration, colloidal and soluble particles concentration and particle size distribution. The reactor performance was assessed in terms of membrane filtration performance and permeate quality.

We hypothesized that flux enhancer addition enhances the anaerobic sludge filterability by modifying the sludge characteristics such as particle size and soluble and colloidal particles concentration. Therefore, a lower fouling rate and higher operational flux could be achieved. In order to test this hypothesis, first, a screening test was performed on site by using a DFC installation unit in order to evaluate the sludge filterability at different operating conditions such as cross-flow velocities and flux typically expressed as liter of permeate produced per m^{-2} membrane per h (LMH) . After this, the impact of flux enhances on the sludge filterability was evaluated at different polymer concentrations in order to determine the optimal concentration to be added in the AnMBR pilot system. In both of the above stages the ΔR_{20} values were measured and considered as the parameter for sludge filterability comparison under each condition, ΔR_{20} is inversely related with filterability. A concentration of 50 mg of polymer per L sludge was obtained as optimal for addition into the pilot plant. Before and after the addition of the flux enhancer in the AnMBR system, the changes in the TMP were monitored and recorded by a Supervisory Control and Data Acquisition (SCADA) control system. Additionally, a throughout characterization of the blackwater, permeate and sludge in was carried out.

2. Literature review

Conventional water treatment approaches need to be adapted to cope with the ever increasing water demand that urban population growth carries. In the past years, several water shortage events have been seen all around the world. This puts pressure on governments and institutions to provide distribution and treatment schemes that guarantee a constant high quality water supply. According to the US Environmental Protection Agency (US EPA, 2005), a decentralized wastewater management approach (*i.e.* small scale wastewater transport, handling, treatment, disposal and/or reuse) could help to achieve public health and water quality requirements in a cost effective and long term manner. Among the main advantages of the decentralized approach we can find cost reduction, local economic strength and community wellbeing (Biggs *et al.*, 2008). Likewise, these schemes can help to increase the water security locally as they have a lower sensibility to extreme events such as natural disasters and operational errors. Decentralized systems can be applied at different small scales that range from single households or buildings to districts thus, they require lower capital and operational costs since they have shorter distribution systems (Capodaglio, 2017). Furthermore, decentralized systems are compact and can be operated under flexible conditions. In order to increase the benefits of the decentralized approach, the wastewater can be divided into different streams according to their polluting characteristics and their resource recovery potential (Capodaglio, 2017; Opher & Friedler, 2016). In this manner, the complexity of the treatment lines can be reduced as they will be designed for a specific wastewater stream. Conventionally, five different streams can be separated in sewage: blackwater, brown water, yellow water, greywater and rainwater. Of these, blackwater has the highest organic, nutrient and pathogen content (Larsen *et al.*, 2013).

Unfortunately, not only water demand is increasing worldwide but also energy consumption. Hence, it is advisable to explore the feasibility of water treatment schemes that allow energy recovery from wastewater streams. For instance, blackwater composition make it suitable for anaerobic digestion treatment; over the past years, the diffusion of AnMBR has been increasing as the production of biogas and further use as an energy resource makes this system highly sustainable. Furthermore, AnMBRs can retain biomass for longer periods of time than standard anaerobic digestion schemes, which helps to achieve a higher treatment efficiency; this is of importance since, conventional toilets yield diluted blackwater streams that require a high biomass retention time (Larsen *et al.*, 2013; Le-Clech, 2010). Furthermore, in AnMBR systems it is possible to decouple hydraulic residence time (HRT) from sludge residence time (SRT); it has been suggested (Dereli *et al.*, 2014) that an infinite SRT, this is operating without sludge discharge, is possible.

Membranes are fabricated with finely porous materials with pore size ranging from 0.001 to 0.01µm for nanofiltration, from 0.01 to 0.1µm for ultrafiltration and, from 0.1 to 1µm for microfiltration applications. The pollutant constituents of sludge will be retained by the membrane hence, pore size selection goes in hand with sludge characteristics and the required effluent quality to be achieved. In the same way, AnMBRs systems can have different configurations, for instance, the membranes can be submerged (either on the reactor or in a separate module) or operate as external cross-flow systems. Geilvoet (2010) suggested that the submerged configuration by placing an external membrane tank allows a better process control and enhanced permeate quality since the sludge has already been biologically treated in the anaerobic reactor before being fed into the membrane tank. Different membrane types can be used according to the configuration; flat sheet and hollow fiber membranes are desired for submerged AnMBRs whereas tubular membranes are used for external cross-flow configurations. The external configuration presents some advantages over the immersed configuration such as better fouling control, easier membrane replacement and higher fluxes. However, as a high flow needs to be pumped, the energy requirement for these systems is higher (Ozgun *et al.*, 2013). In order to achieve water separation from the pollutant particles it is necessary to apply a driving force that it is usually achieved by imposing a pressure gradient between the feed side and the treated water (*i.e.* permeate) side of the membrane, this is commonly referred as the TMP (Geilvoet, 2010).

The TMP and permeate flow (*i.e.* flux) are two of the main parameters to be monitored and controlled in AnMBR plants. It is a common practice to maintain one of these two variables constant; in most of the cases AnMBRs are operated at a constant flux and fluctuating TMP that will change along with the fouling state of the membranes. In order to produce permeate the TMP needs to overcome the resistance of the system to filtration, this is given by the membrane intrinsic resistance (*i.e.* the resistance experienced by a clean membrane when demineralized water is filtrated), concentration polarization and the fouling resistance (*i.e.* the resistance given by soluble particles and colloids deposition in the membrane´s surface and pores). Concentration polarization is negligible in membrane bioreactors when compared to the other fouling mechanisms. In membrane bioreactors operating at constant flux, when fouling is starting to occur a short term rise in TMP will occur; as fouling starts to develop the TMP will continue to increase either linearly or exponentially until a sudden change in TMP is observed (Zhang *et al.*, 2006). In order to prevent severe fouling and guarantee a continuous stable operation it is necessary that the system operates below the critical flux (*i.e.* the flux above which deposition of particles and colloids occurs on the membrane surface) (Jeison *et al.*, 2006; Meng *et al.*, 2009). For instance, an AnMBR system treating municipal water reached unstable operation when its flux was increased by 20%; most likely as a result of a higher fouling rate Martinez-Sosa *et al.*, (2011). The determination of the critical flux can be done by different flux-step methods for instance, the classical protocol in which the flux is increased gradually up to predetermined upper value and then it is gradually decreased down to the initial value. Likewise, a filtration/relaxation protocol can be applied, this methodology differs from the classical protocol as a relaxation time is given between changes in the flux. Similarly, a pre-step protocol in which a small low flux filtration step is included before each change in the operational flux can be carried out (de la Torre *et al.*, 2009).

2.1 Membrane fouling

It is important to know the different fouling mechanisms that can be present in AnMBRs so that a mitigation strategy can be developed and applied accordingly. Membrane fouling is caused by different mechanisms that are likely to occur simultaneously. As it can be observed from Fig.1, three main factors are related with membrane fouling: operating conditions, biomass characteristics and membrane characteristics.

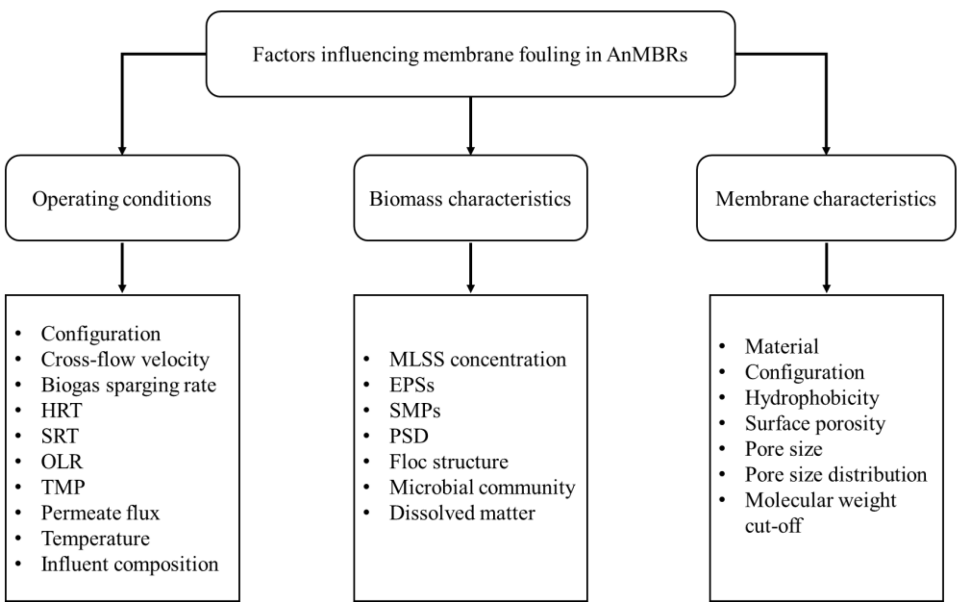

Fig. 1 Factors influencing membrane fouling in AnMBRs, adapted from Bagheri & Ahmad (2018). EPSs: extracellular polymeric substances; HRT: hydraulic retention time; OLR: organic loading rate; PSD: particle size distribution; SMPs: soluble microbial particles; SRT: sludge retention time; TMP: transmembrane pressure.

Membrane fouling can be categorized according to the location where the particles are being deposited (*i.e.* internal and external fouling) as well as according to the ability of chemical and physical cleaning methods to remove this deposition (*i.e.* reversible, irreversible and irrecoverable fouling).

Organic substances present in the feed wastewater and those produced by the microorganisms inside the reactor may tend to accumulate in the membrane surface and pores contributing to fouling (Le-Clech *et al.*, 2006). Internal fouling is caused when the effective pore size of the membranes is reduced by the adsorption of colloids or soluble particles that are not able to pass to the permeate side as they have a size similar to that of the membrane pores. When particles with a size larger than the membrane pores start to accumulate in the membrane surface the formation of a cake layer occurs (Fazana *et al.*, 2017). Fouling by means of cake layer formation is generated by the accumulation and adsorption of SMPs and/or extracellular polymeric substances (EPSs) which are attached to the suspended solids (Gao *et al.,* 2013). Some operational parameters influence the concentration and composition of SMPs and EPSs, for instance SRT, organic loading rate (OLR), temperature, pH and shear rate (Ozgun *et al.*, 2013). The size and thickness of the cake layer is dependent on the membrane operation time. Likewise, the concentration of mixed liquor suspended solids (MLSS) influences the formation of the cake layer. This is especially critical in AnMBR systems as they are operated at high biomass concentrations; the higher the sludge retention time the higher the concentration of MLSS. It has been stated that a concentration below 10 gMLSS L^{-1} must be maintained in order to guarantee an optimum operation in aerobic

MBRs; however, this can vary according to the operating flux and the shear stress induced by biogas sparging (Ferreira *et al.*,2010).

In AnMBR systems fouling control is typically done by means of biogas sparging and high cross flow velocities in submerged and external cross flow operations, respectively (Smith *et al.*, 2012). Biogas sparging is applied to increase the shear force on the membrane; the changes in the membrane filterability will be a function of the frequency and duration of the biogas sparging (Vyrides & Stuckey, 2009). In the side stream configuration, the formation of a cake layer in the membrane is reduced by the fluid cross flow velocity which is controlled by a recirculation pump. AnMBRs systems are typically operated at cross flow velocities ranging from 2 to 4 m s^{-1} which allow both higher efficiency and lower energy consumption. The strategies mentioned above need to be kept at conservative values in order to reduce energy consumption and disintegration of large particles (Bornare *et al.*, 2014).

2.2 Membrane cleaning

Both internal and external fouling will result in a decrease in the membrane permeability leading to a lower flux and increased operational costs. Thus, it is common to operate the AnMBR in an intermittent filtration mode in order to clean the membrane and maintain a constant flux. Ozgun *et al.*, (2013) found that regular maintenance by means of chemical cleaning of the membranes is needed in order to avoid the formation of residual fouling that will later be more difficult to remove by conventional cleaning methods. In practice, membrane cleaning can be done physically and/or chemically depending on the fouling nature. Physical cleaning can be done either in-situ, by backwash and/or relaxation periods, or ex-situ, by removing the membranes from the tank and applying water jets (Ozgun *et al.*, 2013). The efficiency in physical membrane cleaning gives indication of whether the fouling is reversible or irreversible; the closer the cleaning efficiency is to 100% the less irreversible fouling is present in the membrane (Martinez-Sosa *et al.,* 2011). Membrane performance can be highly increased by minimizing irreversible membrane fouling since this determines the membrane lifetime. The physical cleaning method vary accordingly to the membrane type that is being used; hollow fiber modules are generally cleaned by backflush whereas flatsheet modules are commonly operated with relaxation periods (Drews *et al.,*2010).

Chemical cleaning methods are applied when the membrane presents severe fouling. The cleaning substances are transported to the membrane interface and they penetrate in the fouling layer leading to the solubilization and loosening of the foulants creating a waste stream (Bagheri & Ahmad, 2018). Chemical cleaning can be carried out with the addition of alkali, oxidant or acid reagents depending on the foulant and membrane characteristics. For instance, alkali and oxidant solutions can be used for the removal of organic fouling, namely proteins, polysaccharides, carboxylic and phenolic groups, by means of solubilization and hydrolysis reactions. On the other hand, acid solutions are normally used for the removal of inorganic foulants, such as metal hydroxides and divalent cations, since acid reagents can oxidize specific functional groups (Zhou *et al.,* 2017). Chemical cleaning can be performed both in-situ and ex-situ according to the fouling state of the membrane and the design of the reactor. If the membrane is not highly fouled, in-situ cleaning could be performed allowing the reestablishment of the membrane permeability. On the other hand, when the membrane is severely fouled, ex-situ cleaning could be performed by transferring the membrane from the reactor to a cleaning tank containing the chemical solution (Meng *et al.,* 2017; Zhou *et al.,* 2017).

2.3 Flux enhancers

Several methods have been investigated in order to decrease membrane fouling, such as altering the membranes material, changing the process hydraulic conditions as well as the addition of flux enhancers. Flux enhancers are adsorbents, coagulants and flocculants. Adsorbents, such as PAC and GAC, increase the removal of COD in AnMBRs; removing the soluble organic compounds by adsorption results in a reduction of organic fouling leading to a higher operating flux (Hu & Stuckey, 2007). Sewage production present variation throughout the year as a result of seasonal water consumption variations. Therefore, AnMBR systems for sewage treatment could present variations in their operational flux throughout the year; this leads to an overestimation in the AnMBR required capacity as the system needs to be able to treat all the wastewater that enters the process. Flux enhancer addition can be adapted according to the flux requirements and sludge properties; hence, its application could help to cope with temporary high capacity demands leading to a reduction in the designed overestimation (Díaz *et al.*, 2014).

Furthermore, flux enhancers could affect the sludge sedimentation speed, the sludge volumetric index and the turbidity of the supernatant. If these parameters are increased then bigger, stronger and denser flocs are observed in the sediment (Siah *et al.,* 2014). The sludge floc size has been found to be positively correlated with the permeate flux as larger flocs are more likely to be carried away from the membrane surface. In the same manner, larger flocs deposition on the membrane surface leads to a lower fouling resistance as the cake layer is more porous and permeable (Ozgun *et al.*,2013). Therefore, it can be argued that particle size distribution influences, to a high extent, the membranes fouling rate. Hence, one of the main reasons why flocculants are used as flux enhancers relies on their ability to cause small particles and colloids aggregation into larger conglomerates (Díaz *et al.*, 2014). For instance, high molecular weight cationic polymers are usually employed for direct coagulation-flocculation as they help to neutralize the negative charges of the colloids and induce aggregation of the particles (Siah *et al.*, 2014). Yu *et al.* (2015) examined the effect of polyaluminum chloride (PACl) and polyacrylamide (PAM) finding that both substances lead to an enhanced sludge filterability. However, the substances presented different effects on the sludge characteristics; PACl reduced the concentration of SMPs whereas PAM led to higher particle size. In a study carried out by Dong *et al.*, (2015), it was found that $FeCl_3$ addition led to reduction of both reversible and irreversible fouling by increasing the particle size distribution due to soluble and colloidal particles agglomeration. Moreover, Díaz *et al.* (2014) found that the enhancement of the operational flux due to flocculant addition was seen up to several weeks after the flocculant addition.

2.4 Sludge filterability measurement

One of the ways to test the effects of flux enhancer on AnMBR performance at the moment of addition and over time is by performing anaerobic sludge filterability on-site characterizations. These can be done with different protocols such as the Delft filtration Characterization method (DFC method). As it was mentioned before, membrane fouling is caused by variations in the operational parameters, sludge characteristics, and membrane characteristics. If the single effect of the changes in one of these variables on membrane fouling needs to be assessed it is important to maintain the other two constant. For instance, if the sludge filterability under different sludge characteristics needs to be studied it is important to maintain both the operational parameters and membrane characteristics constant. In view of the above, the DFC method can be used since it is possible to operate the DFC installation with fixed membrane characteristics and constant operational parameters. Therefore, wastewater treatment plant operators are able to determine whether an increase in fouling rate should be attributed to the sludge filterability or to operating conditions (see Fig. 1). The DFC method gives as a main outcome ΔR_{20} (*i.e.* is the cake layer resistance after 20 L of permeate per m^2 of membrane surface have been extracted); this parameter is inversely related to the sludge filterability. For aerobic sludge ΔR_{20} values between 0.1 x 10^{12} m^{-1} and 1.0 x 10^{12} m^{-1}) indicate good and moderate filterability respectively whereas values higher than 1 x 10^{12} m^{-1} indicate poor filterability (Geilvoet, 2010). However, for anaerobic sludge the ΔR_{20} values measured are considerably higher and the relation with filterability should be redefined.

3. Materials and methods

3.1 Analytical methods

The performance of the pilot AnMBR was assessed by taking samples of blackwater, permeate and anaerobic sludge. Total suspended solids (TSS) and total volatile suspended solids (VSS) were analyzed in triplicate and in accordance with Standard Methods (APHA, 1999) 2540D and 2540 E, respectively. In order to determine the chemical oxygen demand (COD) of the sludge soluble and colloidal fraction, sludge samples were centrifuged at 4000rpm for a period of 10 minutes. After this, the supernatant was filtered using a glass fiber filter (Whatman 1821-047) with a nominal pore size of 1 µm. The COD of the filtered sample (csCOD) as well as the pilot´s permeate (COD_p) were measured in triplicates using HACH Kits LCK 114. With this the estimation of the colloidal COD (i.e. $csCOD_s$-COD_p) fraction of the sludge was calculated. The samples were taken three times a week directly from the pilot plant installation. The DFC installation takes sludge directly from the pilot´s membrane tank hence, the variatio in sludge characteristics in the membrane tank were analyzed by taking one sludge sample per DFC run. The samples were taken in the sludge feed line of the DFC installation.

The particle size distribution (PSD) of the sludge was measured by a Microtrac Bluewave PSD analyzer (Malvern Instruments Ltd., UK) with a measuring range of 0.01 to 2000 µm. All measurements were done in triplicates. The d50 value (*i.e.* the size in microns that splits the distribution with half above and half below this diameter) was taken as reference to characterize the particle size distribution.

3.2 Delft filtration characterization method and installation

The DFC method was used to test the influence of the flux enhancer on anaerobic sludge filterability and determine the flux enhancer concentration to be added to the pilot plant. The DFC method consists of five main stages. Step 1, a mechanical cleaning is performed by flushing water at a cross-flow velocity (CFV) above 3 m s^{-1} through the feed side of the membrane for 5 minutes. Step 2, the membrane resistance is measured by filtrating demineralized water whilst measuring and recording the TMP changes until 3 liters of permeate per m^2 of membrane are produced. The total resistance for water filtration must be below 0.5×10^{12} m^{-1}; if this is not achieved, chemical cleaning of the membrane is needed. In the third step, the filterability of the sludge is measured by circulating sludge through the feed side until 20 liters of permeate per m^2 of membrane are produced or a TMP of 0.75 bar is reached in order to guarantee correct operation of the membrane. It is important to point out that steps 2 and 3 must be carried out at the same CFV and flux values. After this, step 1 and 2 are repeated to reduce and evaluate the fouling state of the membrane before the next sludge filtration step. Lastly, after the different filtration runs are performed, manual chemical cleaning of the membrane is performed by feeding 250 mL of sodium hypochlorite solution through the permeate side of the membrane. The reader is referred to Geilvoet (2010) for further insight in this protocol. At the pilot plant location there is no demineralized water available; therefore, tap water was used during steps 1, and 2.

The DFC installation has a tubular X-flow ultrafiltration membrane provided by Pentair with a nominal pore size of 0.03 µm, an internal diameter of 8 mm and a length of 95 cm which gives a membrane area of 0.024 m^2. Fig. 2 shows the general layout of the DFC installation. The anaerobic sludge is collected online from the pilot plant and contained in a hermetic container (anaerobic sludge vessel). The sludge is continuously feed from the membrane tank of the pilot AnMBR to the DFC installation using a peristaltic pump and the concentrated sludge is returned to the pilot plant; maintaining a volume of 40L in the anaerobic vessel. The permeate is extracted also by a peristaltic pump.

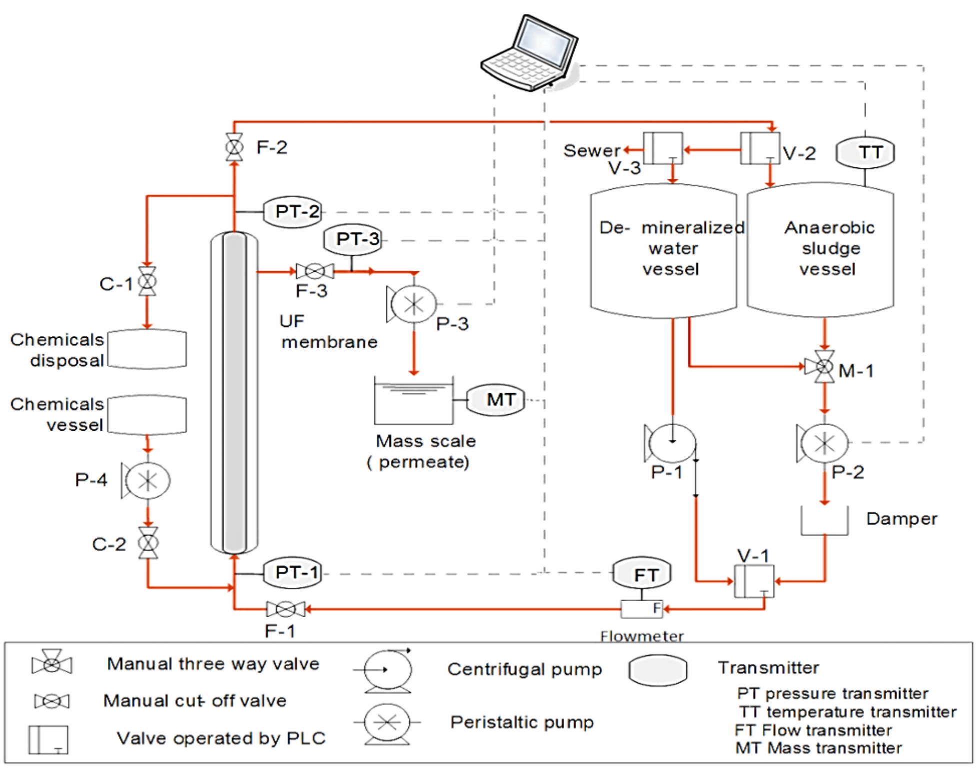

Fig. 2 Delft Filtration Characterization (DFC) installation adapted for anaerobic sludge.

The following parameters are measured and recorded during the tests: sludge temperature (°C), sludge flow through the membrane ($m^3\ h^{-1}$), feed pressure (bar), concentrate pressure (bar) and permeate pressure (bar), and permeate mass (g). The location of these sensors in the DFC installation is shown in Fig. 2 labeled as TT, FT, PT1, PT2, PT3, and MT respectively. With these the flux (LMH), CFV ($m\ s^{-1}$), and TMP (bar) are calculated. The flux (J) and CFV set points are established at the beginning of the filtration run. These parameters are calculated and controlled by the DFC installation software.

The flux is calculated according to Eq. 1:

$$J = \frac{dM}{dt}\frac{3600}{A_m \rho} \text{..........} Eq.1$$

$Where:$
$M = \text{Mass (g)}$
$t = \text{Time(s)}$
$A_m = \text{Total membrane surface } (m^2)$
$\rho = \text{Density } (Kg\ m^{-3})$

The density ρ is considered equal to water´s density and variable with temperature, Eq. 2 shows its calculation.

$$\rho = (-0.0043 * T^2) - (0.022 * T) + 1000.2 \text{..........} Eq.2$$

$Where:$
$T = \text{Temperature (°C)}$

The CFV (υ) is calculated as:

$$\upsilon = \frac{Q}{A_{cs}} \text{..........} Eq.3$$

Where:

υ = Cross flow velocity (m s^{-1})

Q = Sludge flow (m^{3} h^{-1})

A_{cs} = Area membrane cross section (m^{2})

The TMP is calculated according to Eq. 4:

$$TMP = \frac{P_f + P_c}{2} - (P_p + 0.04) \ldots\ldots Eq.4$$

Where:

P_f = Feed pressure (bar)

P_c = Concentrate pressure (bar)

P_p = Permeate pressure (bar)

3.3 ΔR_{20} estimation.

The ΔR_{20} values are obtained using the changes in TMP calculated by the DFC installation taking in consideration the variations in temperature that impact both the viscosity and density of the sludge.

After this the total resistance is calculated following Eq. 5:

$$R_t = \frac{TMP\, x 10^{8}\, x 3600}{\eta J} \ldots\ldots \text{Eq.5}$$

Where:

R_t = Total resistance (m^{-1})

TMP= Transmembrane pressure (bar)

η = Permeate viscosity (Pa s^{-1})

J= Flux (L m^{-2} h^{-1})

The permeate viscosity η is dependent on temperature and calculated as:

$$\eta = 0.001e^{(0.580-2.520\theta+0.909\theta^2-0.264\theta^3)} \text{...} Eq.6$$

$$\theta = 3.661\frac{T}{273.1+T} \text{...} Eq.7$$

$Where:$
$T = \text{Temperature (°C)}$

Below the calculation steps to obtain ΔR_{20} are shown:

$$\Delta R_{20} = \Delta R\big|_{V=20Lm^{-2}}$$

$$\Delta R = R_t - R_m \text{..} \text{Eq.8}$$

$Where:$
$\Delta R = \text{Additional resistance of the fouling layer } (m^{-1})$
$R_t = \text{Total resistance } (m^{-1})$
$R_m = \text{ Membrane resistance } (m^{-1})$
$\Delta R_{20} = \text{ Estimated additional resistance when V=20Lm}^{-2} \ (1x10^{12} m^{-1})$
$\text{V= Volume of permeate per m}^2 \text{ of membrane} \ (\text{Lm}^{-2})$

The membrane resistance R_m is the resistance measured at the beginning of the run. When the filtration run stops either for reaching maximum TMP or for producing an specific volume of 20 L m^{-2}, the calculation of the ΔR_{20} is done by applying a best fit trendline to the ΔR *vs.* specific volume values and extrapolating to an specific volume of 20 L m^{-2}.

3.4 Screening test and daily ΔR_{20} values variability tests.

The DFC method was applied in three different general assays: screening tests, daily ΔR_{20} values variability tests and, flux enhancer dosage step tests. The latest is further described in Section 3.6. A screening test was performed in order to determine the best operational conditions for the DFC installation membrane, Table 1 shows the different conditions tested in the screening test.

Table 1 DFC method screening test conditions.

Run	**Flux (LMH)**	**Cross flow velocity ($m\ s^{-1}$)**
1	20	1.0
2	40	1.0
3	60	1.0
4	20	1.5
5	40	1.5
6	60	1.5
7	20	2.0
8	40	2.0
9	60	2.0

The ΔR_{20} values daily variability tests were performed at the conditions selected during the screening test during a 2 months' period. The measurements where done in duplicates 5 times a week.

3.5 Flux enhancer.

The cationic polymer ADIFLOC KD 451 (ADIPAP, France) was used in this study as flux enhancer. The polymer was selected based on its capacity to significantly enhance the sludge filterability of municipal and industrial sludge samples when applied at very low concentrations, while no effect on pH was observed (Odriozola *et al.*, 2018). The polymer was dissolved in distilled water to reach a baseline concentration of 10 g L^{-1}.

3.6 Flux enhancer dosage step tests.

The flux enhancer dosage step tests were performed in order to evaluate the effect of its addition in the sludge filterability. The test was performed two times with one week of operation in between and using 40L gab samples from the pilot AnMBR. Table 2 and 3 show the different concentrations tested for dosage steps 1 and 2 respectively.

Table 2 Dosage step 1: Concentrations of flux enhancer in anaerobic sludge

Run	**Concentration (g L^{-1})**
1	0
2	0.025
3	0.05
4	0.1
5	0.15
6	0.2
7	0.25
8	0.3

Table 3 Dosage step 2: Concentrations of flux enhancer in anaerobic sludge

Run	**Concentration ($g\ L^{-1}$)**
1	0
2	0.01
3	0.02
4	0.03
5	0.04
6	0.05
7	0.06
8	0.08
9	0.1

After performing the dosage steps, the optimal concentration to be added to the pilot plant was determined considering the impact of the flux enhancer in the sludge filterability ΔR_{20}.

3.7 Pilot plant

The decentralized pilot AnMBR system was fed with blackwater collected in a segregated pipe in the main office building (approx. 100 people working) at the Porto do Molle business center located in Nigrán, Spain. In Fig. 3, a schematic representation of the treatment facility is shown. The pilot plant is an underground facility; blackwater is collected from the building and directed to a reservoir tank. After this, the blackwater is pumped to a buffer tank. The main components of the pilot plant are an anaerobic reactor with an operational volume of 1.7 m^3 and a membrane tank with an operational volume of 1 m^3 equipped with an ultrafiltration flat-sheet membrane type MF101 provided by Martin Membrane Systems with a nominal pore size of 0.035 µm and effective surface area of 6.25 m^2. The membrane is continuously sparged with biogas; in order to decrease fouling a biogas flow of 1.12 N m^3 h^{-1} per m^2 of membrane surface was recommended by the membrane module supplier. A similar approach using biogas slug flow to minimize energy consumption was proposed by Lindeboom et al. (2011) in order to reduce the CFV while controlling the cake-layer and thereby the flux. The system operates at constant flux; the permeate is extracted by a peristaltic pump with fixed speed. Another peristaltic pump is used to provide sludge recirculation between the membrane tank and the anaerobic reactor. The system operates in a discontinuous filtration mode consisting of repeating cycles of 5 min filtration and 1.5 min relaxation.

The pilot plant is equipped with a supervisory control and data acquisition (SCADA) system with which, the operation data logging and the plant control are carried out. The online sensors measured the pH, temperature, and oxidation-reduction potential in the anaerobic reactor. Likewise, the TMP and gas pressure were measured by liquid pressure transmitters. Flow rate transmitters measure the permeate flow. The levels of the buffer tank, membrane tank and digester are measured and controlled to ensure correct operation.

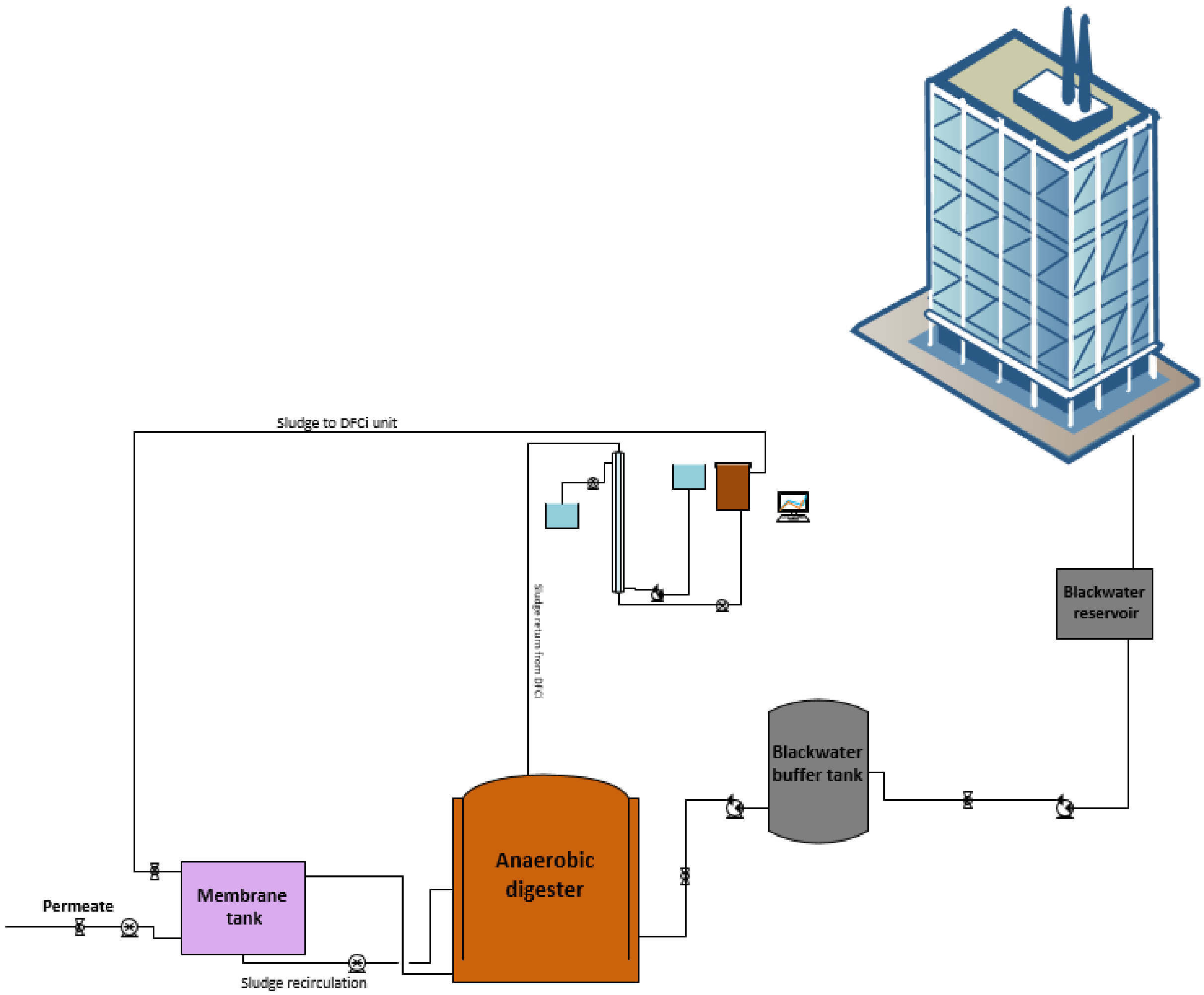

Fig. 3 AnMBR pilot plant general layout

3.8 Flux enhancer addition to pilot plant.

The flux enhancer was added one time to the pilot AnMBR. The polymer was added to reach a concentration of 50 mg per L sludge.

4. Results and discussion.

The impact of flux enhancer addition on the pilot AnMBR plant was measured by analyzing the changes in the biological and operational performance parameters before and after polymer addition which hereinafter will be referred to as phase 1 and phase 2, respectively.

4.1 Analytical measurements.

As we can see from Fig.4 a, blackwater entering the pilot plant presented high disparity in terms of influent COD. A high variation in influent concentrations leads to significant challenges in the operation and stability of pilot AnMBR (Dereli *et al.*, 2012). This high variation can be attributed to a daily difference in the amount of people working in the building and the poor mixing in the reservoir tank.

The performance of the plant in terms of COD removal was maintained at high rates (>90%) in both phase 1 and phase 2 (Fig. 4b).

Fig. 4.c shows the TSS and VSS concentrations of the sludge contained in the membrane tank were maintained around 7.4 and 6.4 g L^{-1}, respectively. The peak observed from September 4^{th} to September 7^{th} was caused due to accumulation of solids in the membrane tank as the recirculation pump was out of service.

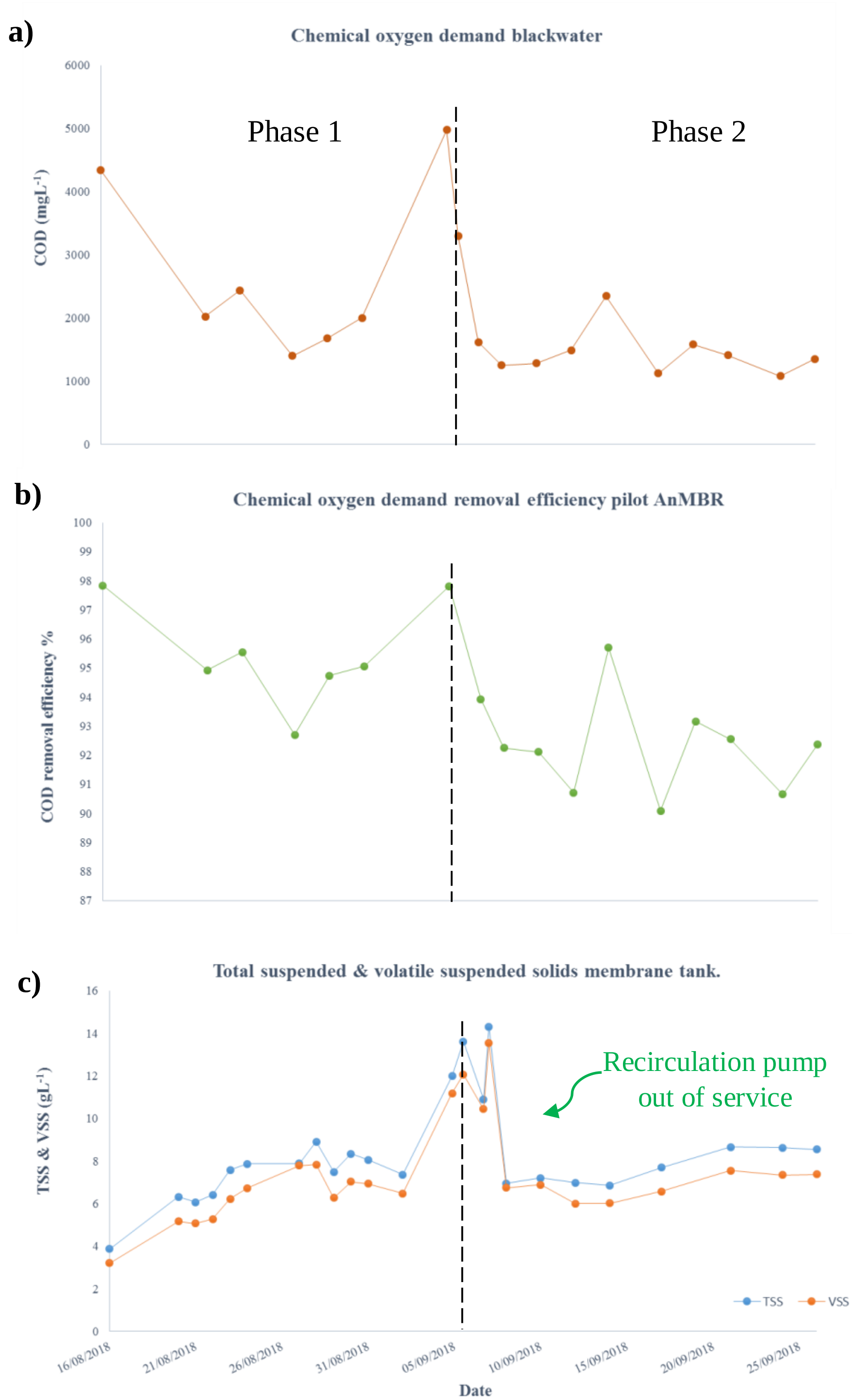

Fig. 4 Variability influent characteristics and biological treatment efficiency before and after flux enhancer addition, phase 1 and phase 2, respectively. ***a)*** *Blackwater COD (mg L^{-1}) variability;* ***b)*** *COD removal efficiency (%) variation;* ***c)*** *Total suspended solids and total volatile suspended solids (g L^{-1})variability in membrane tank.*

4.2 Screening tests.

In order to test the influence of permeate flux and CFV applied in the DFC installation on the sludge filterability and determine the optimal operational settings for filterability measurements, different DFC method runs were performed under the conditions mentioned in Table 1 DFC method screening test conditions. As mentioned in section 3.2 the duration of the sludge filtration run is limited by both the maximum allowed TMP in the DFC installation and/or a specific volume production of 20 L m^{-2}. If any of this two conditions is achieved the sludge filtration run automatically stopped.

For instance, Figure 5a shows the additional resistance of the fouling layer (ΔR) obtained when performing a filterability measurement in the DFC installation at a flux of 60 LMH and a CFV of 1.5 m s^{-1}. In this run, the maximum TMP allowed by the DFC installation was reached at a specific volume of 8 L m^{-2} hence, the run was stopped.

Therefore, in order to calculate the ΔR_{20}, a linear trendline was applied to the ΔR values *vs* specific volume graph. From Figure 5b we can observe the changes in the resistance given by the formation of a cake layer in the membrane. It was estimated that an extra resistance ΔR_{20} of 11.9 x 10^{12} m^{-1} would be generated by this foulant cake layer if a specific volume of 20 L m^{-2} would be reached. It was found that the fouling layer development presents a linear behaviour which results in a steady rate increase in the resistance. Evenblij (2006) stated that a straigth line is an indication of inert particles building a cake layer. This procedure was applied to the different runs of the screning test. Every condition of the screening test presented a steady state increase in the sludge fouling layer development and resistance.

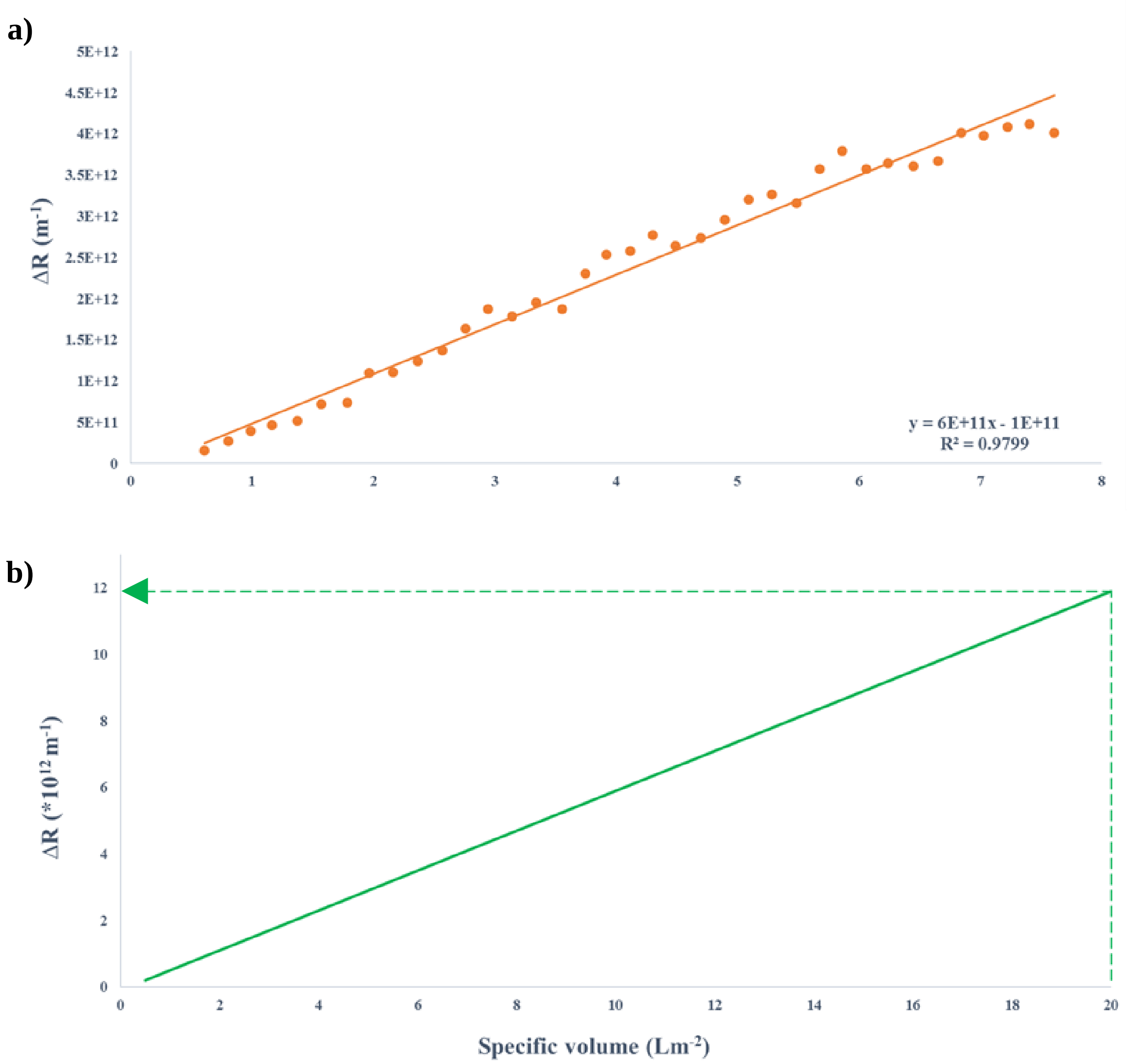

Fig. 5 Additional fouling cake layer resistance obtained by DFC method ***a)*** *ΔR values obtained operating DFC installation at a flux of 60LMH and a CFV of 1.5 ms^{-1} (run stopped at an specific volume of 8L m^{-2} due to maximum TMP reached);* ***b)*** *ΔR_{20} obtained by the extrapolation of the best fit trendline equation obtained in 5a.*

The ΔR_{20} values varied considerably among each different condition; this is sludge filterability is sensible to both the flux and CFV settings in the DFC installation. This is in line with results from Odriozola (2017) where sludge obtained from pilot AnMBR treating municipal wastewater located in Alcazar de S. Juan STP, Spain.

Table 4 shows the ΔR_{20} given by the formation of a sludge fouling layer in the membrane. Conditions with a ΔR_{20} equal to 0 indicate low ΔR values where no fouling cake layer was formed; this was the case for every run performed at a flux of 20LMH.

Table 4 Effects of flux and cross flow velocity set points on sludge filterability (ΔR_{20})

Run	**Flux (LMH)**	**Cross flow velocity (ms^{-1})**	**ΔR_{20} ($x\ 10^{12} m^{-1}$)**
1	20	1.0	0
2	40	1.0	19.7
3	60	1.0	19.9
4	20	1.5	0
5	40	1.5	1.96
6	60	1.5	11.9
7	20	2.0	0
8	40	2.0	0
9	60	2.0	3.7

As it can be observed, the ΔR_{20} at CFV of 2 m s^{-1} are low independtly of the flux set point, this is an indication of low rate fouling layer development. In this condition the system was stopped by the maximum specific volume set point. Even though this indicates good operational conditions the formation of a fouling layer is crucial to test the polymer effects on the sludge filterability which is the main aim of this work. On the other side, when operting the membrane at a CFV of 1 m s^{-1} fouling layer development rate was faster resulting in high

ΔR_{20}. This lead to shorter filtration runs as the maximum TMP was quickly reached. In contrast, when the DFC method was performed at a flux of 60LMH and CFV of 1.5 m s^{-1} a balance between both filtration run and fouling cake layer development (ΔR_{20}) which allows a longer run time and the study of the effects of the flux enhacer in fouling reduction; hence, this set of conditions was chosen to perform both the ΔR_{20} daily variability and the dosage step tests.

4.3 Flux enhancer dosage step tests.

With the purpose of determining the optimal concentration of polymer to be added to the pilot plant, dosage steps were carried out. This is of importance as the contribution of the flux enhancer to ΔR_{20} reduction can reach a maximum after which, no significant changes are observed. In fact, it was found by Wang *et al.*, (2016) that a high concentration of cationic polymers (600 mg poly-dimethyl-diallyl-ammonium chloride per L) reduces filtration efficiency due to colloidal re-stabilization. Hence, it is necessary to study its effect considering both fouling and operational costs reduction.

Figure 6 shows the impact of the flux enhancer, at the range of concentrations mentioned in Table 2 and 3, on the sludge filterability. The polymer addition increased the PSD, taken as the d50 value, and decreased the csCOD, Fig. 6 a and Fig. 6 b respectively. This resulted in a reduction of the ΔR_{20}, this is, an incresase in sludge filterability. In dosage step 1, Table 2, the ΔR_{20} value was lowered from 11.7 x 10^{12} m^{-1} with no flux enhancer addition to 0.97 x 10^{12} m^{-1} with a concentration of 0.05 g of polymer per L of sludge suggesting that at this concentration, the fouling cake layer development rate was low. In the same manner, it was observed that above this concentration no significant changes in fouling reduction are achieved even when d50 keeps increasing. A second dosage step, Table 3 , was carried out with smaller jumps in polymer concentration in an effort to test if a lower concentration of polymer could be used. It was observed that a 80% fouling reduction was achieved at 0.05 g L^{-1} in contrast with a 60% in fouling reduction at 0.04 g L^{-1}.Therefore, 0.05 g L^{-1} was established as the preferred concentration for addition to the pilot AnMBR.

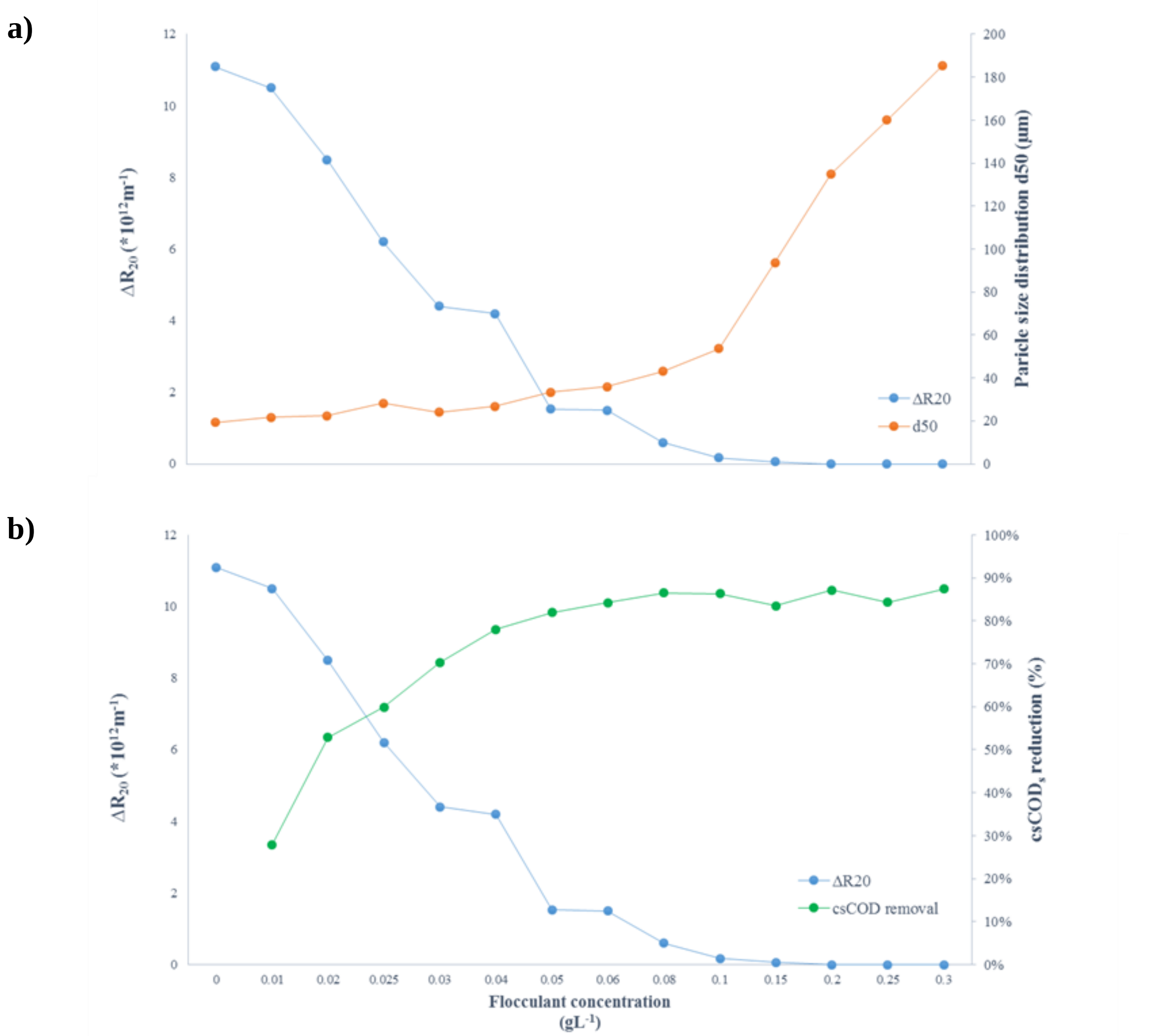

Fig. 6 Dosage step tests: Impact of flux enhancer concentration on anaerobic sludge filterability (ΔR_{20}) measured following the DFC method at a flux of 60 LMH and CFV of 1.5ms-1 due to ***a)*** *increase particle size distribution and* ***b)*** *Colloidal and soluble chemical oxygen demand (csCODs) reduction.*

4.4 ΔR_{20} daily variability tests.

In line with section 4.1 the ΔR_{20} values were measured in both phase 1 and 2. From Figure 7 we can observe that flux enhancer addition resulted in resistance values with a lower variation. Furthermore, flux enhancer addition results in the improvement of filterability, this is, reduction in ΔR_{20} . The ΔR_{20} values were lowered from an 14.8 x 10^{12} m^{-1} to 6.9 x 10^{12} m^{-1} with flux enhancer addition. This reduction can be mainly attributed to an increase in the particle size and reduction of colloidal particles.

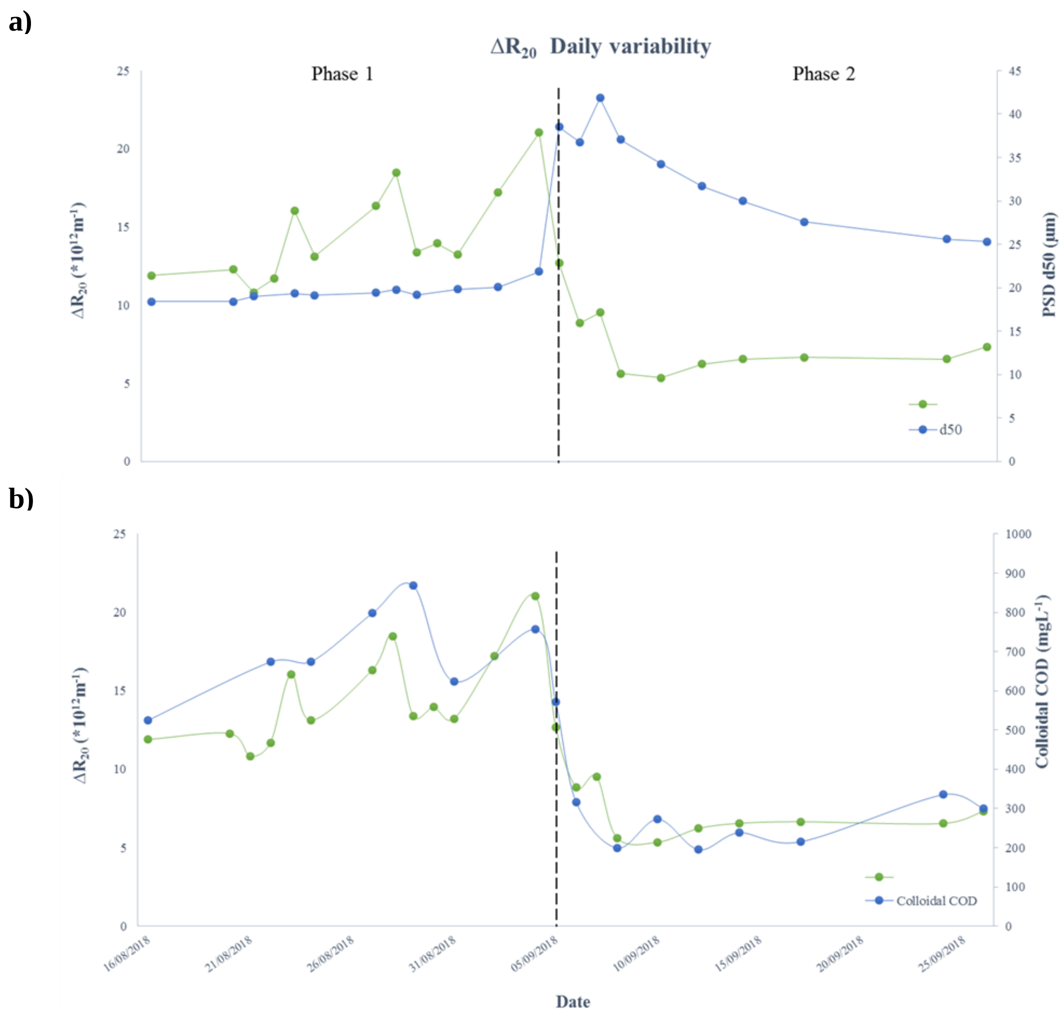

*Fig. 7 $\Delta R20$ daily variability in phase 1 and phase 2, before and after flux enhancer addition, respectively. and **a)** d50 **b)** Colloidal COD. $\Delta R20$ Values measured following the DFC method at a flux of 60LMH and CFV of 1.5ms^{-1}*

4.5 Flux enhancer effect on pilot AnMBR performance.

The effects of polymer addition on the AnMBR pilot plant performance was observed in the variations on the TMP values. The daily average TMP profile is presented in Fig. 8; we can observe that the TMP was decreased from a mean pressure of 192 mbar to a mean pressure of 142 mbar due to flux enhancer addition. Furthermore, the TMP behavior was more stable when flux enhancer was added to the pilot plant. From 25th September to 28th September, there was a peak in the TMP due to low biogas sparging. Once the biogas sparging flow was reestablished the TMP returned to normal values. In line with Diaz *et al.*, (2014) the flux enhancer effect was perceived for several days after flux enhancer addition.

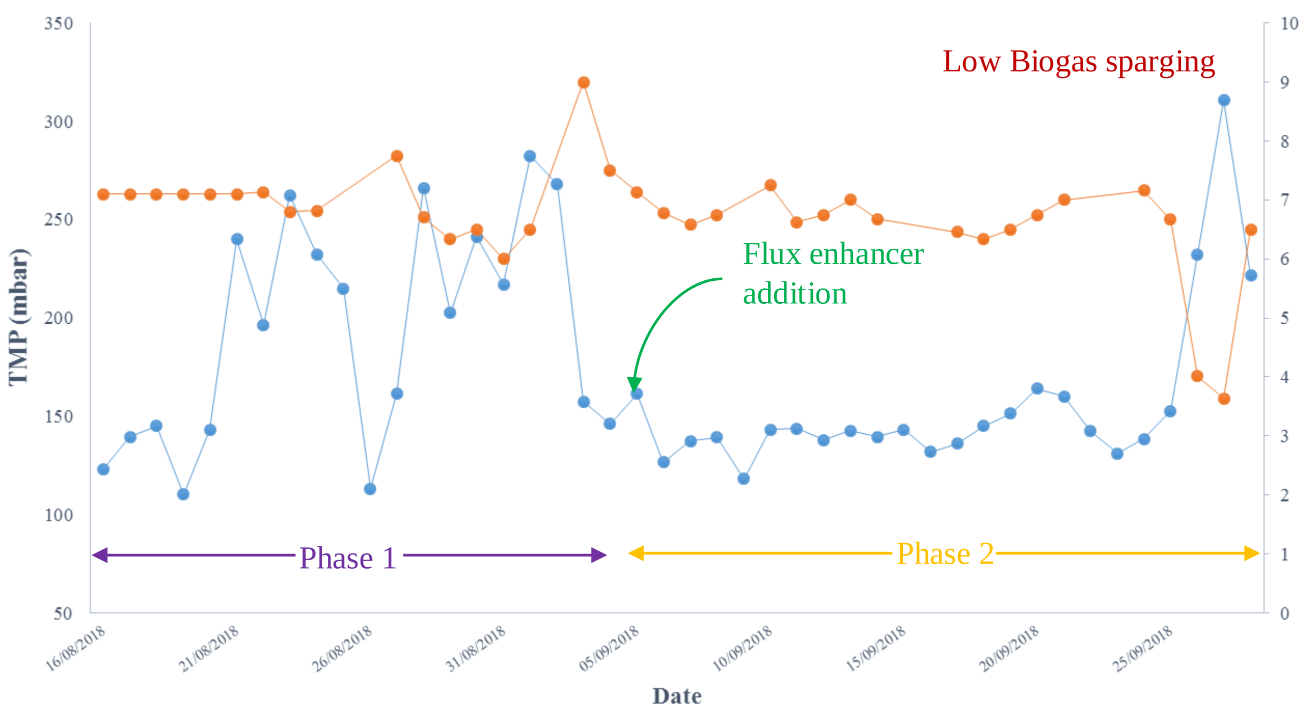

Fig. 8 Daily mean TMP in the pilot AnMBR before and after flux enhancer addition mentioned as phase 1 and phase 2, respectively.

5. Conclusion.

The addition of 0.05 g of cationic polymer ADIFLOC KD 451 per L of sludge in an AnMBR pilot plant increased sludge particle size and decreased colloidal particles concentration. This influenced the fouling cake layer development in the membrane. This resulted in lower and more stable TMP in the system, most likely due to the positive influence of the larger particle size on cake layer development and the lower colloidal particles concentration. The effect could even be observed one month after flux enhancer addition. Furthermore, the permeate COD was not affected by polymer action in the sludge particles.

These results motivate further research in order to understand the mechanisms and dynamics of flux enhancer addition on the sludge particles. With such insights, both, the concentration of the polymer and the periodicity of its addition can be optimized.

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