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Start-up of decentralised MBRs Part II: the use of additives as initial inoculum

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ABSTRACT

This paper corresponds to the second part of a study aiming to establish the best conditions to start-up decentralised membrane bioreactors. The first part focused on the impact of different operational parameters on the start-up, whereas this second part aims to find a substitute for activated sludge to serve as initial inoculum. Both low powdered activated carbon addition and Alumin 7 (alkaline coagulant) demonstrated a low performance in terms of filterability and operation. In turn, ferrous chloride ($FeCl_2$), due to its ability to coagulate soluble and colloidal matter, was able to create a cake layer composed of large coagulated particles acting as a prefilter. Additionally, the combination of wastewater plus $FeCl_2$ allowing sufficient contact time before the filtration starts has demonstrated to be the best way to start-up decentralised membrane bioreactor using this additive. Eventhough some drawbacks are associated with its high acidity, i.e. low pH, high conductivity and low NH_4^+–N removal, the excellent filterabilities observed and the possibility to create a cake layer from "zero-biomass" convert this additive as a possible substitute for activated sludge. This is supported by particle size distribution measurements suggesting that the negative effects of fine particles are outweighed by the possibility of creating a cake layer that impedes pore blocking.

Keywords: Start-up; Decentralised MBR; Additives

1. Introduction

Small wastewater treatment plants (SWwTP) using membrane technology or decentralised membrane bioreactors (MBR-SWwTP) are considered nowadays as one of the best technologies available in the market to treat wastewater in decentralised areas, in spite of the problems related to this technology, i.e. fouling of the membrane and high energy cost. Unlike its competitors, one important advantage offered by this technology is the possibility to reuse the permeate obtained after filtration for water reclamation in one single step. As a consequence, owners of MBR-SWwTP can benefit from a constant, cheap and safe effluent of regenerated water.

The start-up phase of such devices is crucial and it will condition the performance, removal efficiency and plant operation on the long term. Trials without initial inoculum [1]—with negative results—as well as tests to check the impact of different parameters on the first days of operation [2] have been carried out. The idea of employing additives to substitute

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activated sludge as initial inoculum is at least attractive from a logistic point of view. A large number of MBR-SWwTP are located in remote areas where activated sludge sources are simply not available. Additionally, if activated sludge is available, the use of additives could simplify the start-up since fewer amounts are required.

From a practical point of view, quickly enhancing the pollutant treatment performance is the main purpose of the start-up. For this reason, additives should be carefully chosen since some of them may depress the biological activity. Addition of adsorbents or coagulants can decrease the level of solutes and colloids or enhance the flocculation ability and thus the filterability. The addition of powdered activated carbon (PAC) for instance is a common practice for fouling control; however, it has not been implemented to full-scale plants. Akram and Stuckey [3] found a combined effect of adsorption of fine colloids and solutes, and the formation of a thin cake layer resulting in significant flux improvement from 2 to 9 L $m^{-2} h^{-1}$ in the presence of 1.67 g PAC L^{-1}. However, the addition of 3.4 g PAC L^{-1} reduced the flux to 5 L $m^{-2} h^{-1}$. It seems that PAC addition beyond the optimal value is detrimental to the operation. Besides, the improved performance as a result of the bioflocs formed by PAC addition requires regular replacement of aged biologically activated carbon with fresh PAC. Attempts have also been made to use aluminum sulphate, ferric chloride, polymeric ferric sulphate (PFS) and chitosan as coagulants. Zhang et al. [4] reported the addition of ferric chloride at the optimal concentration to reduce both soluble microbial products with molecular weight < 10 kDa in the supernatant and the fraction of small particles in the range of 1–10 μm. Lee et al. [5] found that soluble foulants in the bulk phase of the MBR were entrapped in sludge flocs within the flocculation process after the addition of a cationic polymer. They observed an enhanced filtration performance as a result of a more porous biofilm. Among the coagulants previously cited, Ji et al. [6] found PFS as the best coagulant improving sludge filterability. Wu et al. [7] compared eight inorganic coagulants. Among those, PFS was found to be the most effective to enhance membrane filterability of mixed liquor in MBR with the optimal dose of 1.05 mM Fe. Two main mechanisms were identified responsible for fouling retarding: removal of supernatant organics and enlargement of sludge floc size. Analogously, Lee et al. [8] and Zhang et al. [9] used aluminum sulphate and ferric chloride to control fouling in MBR, respectively. In this research, ferrous chloride ($FeCl_2$) has been used since it has already proved to be a good coagulant in conventional activated sludge processes.

The election of the procedure to add the additives into the reactor is also important to obtain the best results. Wu et al. [7] found that PFS addition before filtration made better effect on depressing the increase of the transmembrane pressure (TMP) than its addition during the operation. This can be explained as follows: at the beginning of the start-up, in the very first seconds of the suction phase—before superficial deposition—some contribution to irreversible fouling, mainly caused by pore blocking occurs. Thus, the period of TMP increase can be divided in two stages [10]: initially, colloidal and soluble fractions govern the fouling process blocking the pores; in a second stage, with a high number of pores, partial or completely blocked, the rapid fouling is driven by suspended flocs.

In spite of the large number of publications testing additives to enhance membrane filterability, none has focused their efforts on the search of a substitute for activated sludge in the start-up phase of MBR-SWwTP. This paper is the second part of a study aiming to establish the best conditions to start-up MBR-SWwTP. In the first part of this study, Gil et al. [2] focused on the impact of different operational parameters on the start-up phase. Among their findings, activated sludge from a SWwTP showed a better performance than activated sludge from a full-scale plant probably due to a better acclimatisation of the former to domestic influents. Wastewater showed a rapid increase of the TMP and was excluded as a possible initial inoculum. A possible optimum value for the mixed liquor suspended solids (MLSS) concentration was suggested (1 g L^{-1}), whereas in terms of aeration, the higher the specific aeration demand per membrane, the better the performance observed. Gil et al. concluded their study advising to avoid low temperatures and high hydraulic retention times (HRTs) during the start-up phase to extend the operation and achieve a fast transition between the start-up and the steady-state phases.

Unlike the rest of SWwTP, MBR-SWwTP will overflow due to a fast colmatation of the membrane. Hence, an optimal start-up phase is a "must" for a reliable, safe and sustainable operation. The goal of this study was to test three additives (PAC, Alumin 7 and $FeCl_2$) in order to find a possible substitute for activated sludge during the start-up phase in MBR-SWwTP.

2. Materials and methods

2.1. Experimental setup

A setup composed of three identical MBR-SWwTP has been designed as shown in Fig. 1. Eight flat sheet membranes (Kubota type H-203, pore size = 0.4 μm, Japan) were introduced in each tank (1 m^3) offering an effective filtration area of 0.88 m^2/tank. Scouring air was supplied through axial perforated tubes (3 mm), whereas aeration for the microorganisms was supplied through 12″ fine bubble diffusers. After a settling tank (HRT > 2 h), wastewater was pumped into the three tanks following the timetable indicated in the standard hEN 12566-3 [11]. Both inflow and outflow were controlled by level sensors whereas the TMP and the flow were monitored thanks to pressure gauges C01 (STW, Germany) and flowmeters Promag 30 (E + H, Germany) respectively. The temperature was adjusted with a heater in each tank plus a thermopar in the first one. All experiments were conducted with dissolved oxygen (DO) in excess (> 4 mg/L) and HRT ~ 65 h. No sludge was withdrawn except insignificant volumes for analysis. Experiments were terminated when the TMP reached 200 mbar. Cleanings in line (CIL) with sodium hypochlorite (NaClO, 0.5%) during 2.5 h were performed between experiments. To fully recover the permeability, additional cleanings with citric acid (pH ~ 3, *T* ~ 65°C, 1 h) were carried out when inorganic additives were used.

Since it is directly related to the evolution of membrane fouling, TMP curves presented in phase I (first MBR reaching the TMP threshold) and phase II (second MBR reaching the TMP threshold) plus accompanying filterabilities have been selected to compare the different scenarios to start-up MBR-SWwTP.

Table 1 shows the operating conditions for the three MBRs during the different experiments. In experiment 1, a comparison test between activated sludge (MBR1), PAC (MBR2) and $FeCl_2$ (MBR3) was carried out. In experiment 2, activated sludge (MBR1), Alumin 7 (MBR2) and $FeCl_2$ (MBR3) were compared. At the end of experiments 1 and 2, $FeCl_2$ demonstrated to be a possible substitute for activated sludge. Therefore, the addition of $FeCl_2$ was optimised in experiment 3, by adding $FeCl_2$ + tap water (MBR1), $FeCl_2$ + wastewater (MBR2) and $FeCl_2$ + wastewater + contact time (MBR3).

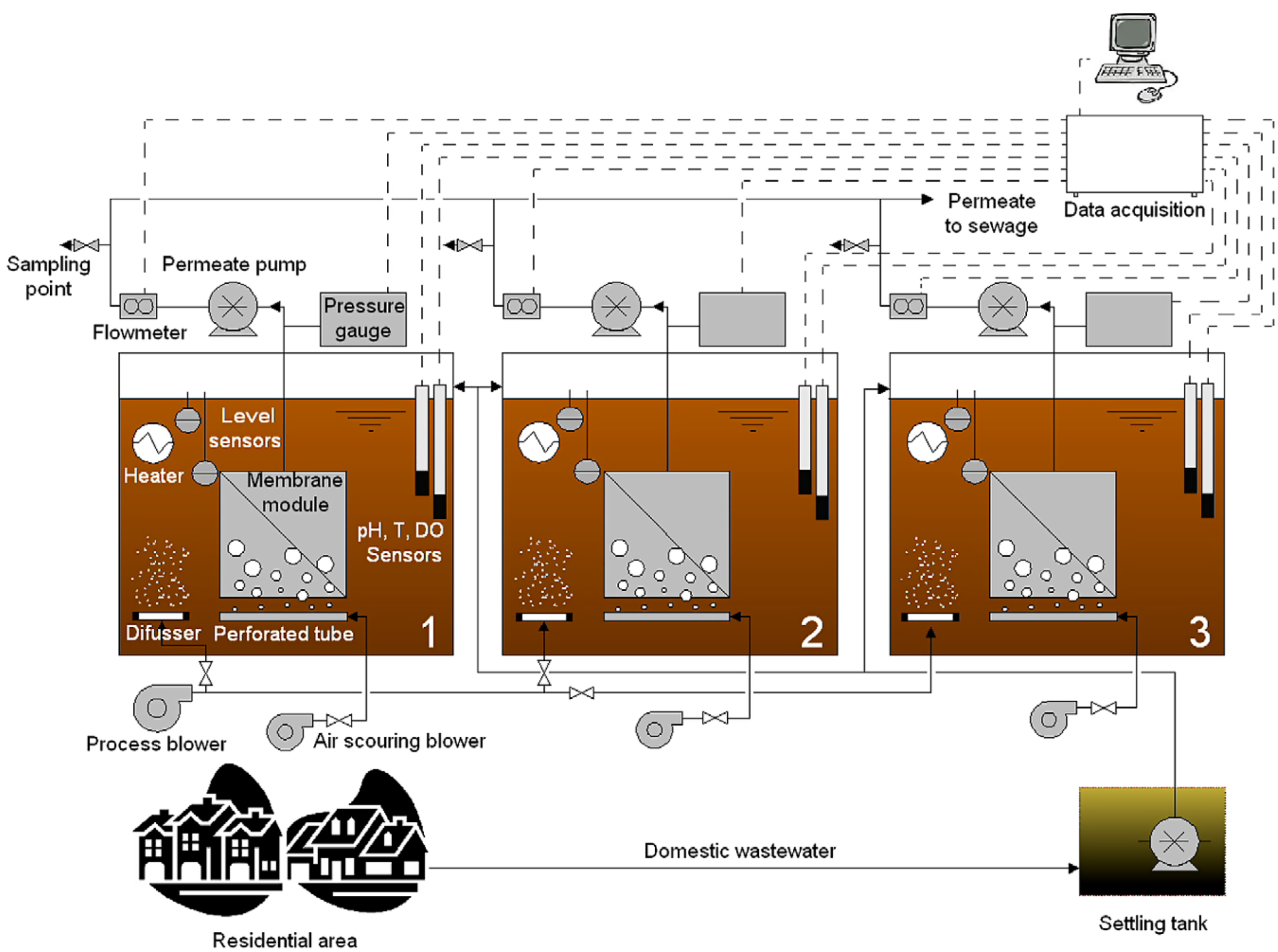

Fig. 1. Setup of the 3 identical MBR-SWwTP.

Table 1
Operating conditions for the three MBRs

Parameter	Units	Experiment 1	Experiment 2	Experiment 3	MBR
Additives	–	Activated sludge	Activated sludge	$FeCl_2$	1
		PAC	Alumin 7	$FeCl_2$ + Ww[a]	2
		$FeCl_2$	$FeCl_2$	$FeCl_2$ + Ww + Contact time	3
Temperature	°C	15.4 ± 0.4	15.3 ± 0.3	16.0 ± 0.7	
		15.2 ± 1.0	15.1 ± 1.0	15.3 ± 0.5	
		15.5 ± 0.7	15.3 ± 0.4	15.4 ± 0.5	
pH	–	6.9 ± 0.4	6.9 ± 0.5	5.5 ± 0.8	
		7.3 ± 0.1	8.8 ± 0.7	5.6 ± 0.9	
		5.1 ± 0.6	5.3 ± 0.6	5.5 ± 0.7	
DO	mg L^{-1}	6.7 ± 2.0	6.3 ± 2.2	8.8 ± 0.8	
		8.4 ± 1.5	8.5 ± 2.1	9.9 ± 1.2	
		8.5 ± 0.6	8.4 ± 0.8	8.2 ± 1.0	
Flux	L m^{-2} h^{-1}	14.8 ± 0.3	15.4 ± 0.5	14.7 ± 0.0	
		14.9 ± 0.4	15.0 ± 0.8	14.7 ± 1.2	
		14.9 ± 0.4	14.9 ± 0.3	15.1 ± 0.6	
MLSS[b]	g L^{-1}	1.3 ± 0.1	1.2 ± 0.1	–	
		–	–	–	
		–	–	–	
OLR[c]	kgCOD/m^{-3} day^{-1}	0.14 ± 0.1	0.15 ± 0.0	0.47	.
		0.10	0.14	0.47 ± 0.01	.
		0.14 ± 0.1	0.14 ± 0.0	0.47 ± 0.01	.
F/M[d]	kgCOD[e] $kgVSS^{-1}$ day^{-1}	0.15 ± 0.1	0.16 ± 0.0	1.9	1
		0.23	1.50	0.92 ± 0.2	2
		1.8 ± 0.0	0.82 ± 0.3	1.1 ± 0.5	3

[a]Wastewater.
[b]To achieve low MLSS concentrations, activated sludge from Aachen-Soers WwTP at 5–6 g L^{-1} was diluted with tap water in experiments 1 and 2.
[c]Organic loading rate.
[d]Food to microorganism ratio.
[e]VSS: Volatile suspended solids.

2.2. Characteristics of the activated sludge and composition of the wastewater

Activated sludge (MLSS 5–6 g L^{-1}, solid retention time –SRT– ~ 14 d) was taken from the Aachen-Soers wastewater treatment plant (WwTP) in Germany. Domestic wastewater bypassed from a conduction coming from a residential area in the city of Aachen was used as the influent. Table 2 shows the characteristics of the inflow.

2.3. Critical flux determination

In order to select the flux to perform the experiments, the critical flux (J_c) was determined using the stepwise method [10,12] by averaging two values of

Table 2
Influent quality

Experiment	COD[a] (mg L^{-1})	NH_4^+–N (mg L^{-1})	TP[b] (mg L^{-1})	T (°C)	pH	Turbidity (NTU[c])	Conductivity (μS cm^{-1})	SS (mg L^{-1})
1	397 ± 115	31 ± 3	5.8 ± 1.2	10 ± 0.4	7.3 ± 0.3	92 ± 47	881 ± 55	70 ± 60
2	392 ± 4	33 ± 3	6.3 ± 0.1	12 ± 0.1	7.4 ± 0.1	67 ± 2	962 ± 13	40 ± 20
3	401 ± 35	34 ± 4	5.9 ± 1.2	13 ± 1.0	7.2 ± 0.1	81 ± 16	947 ± 16	90 ± 10

[a]Chemical oxygen demand.
[b]Total phosphorus.
[c]Nephelometric turbidity unit.

the permeate flux: the maximum flux at which the TMP increases perceptibly over one step length and the minimum flux at which TMP does not change. Before determining J_c, a CIL was performed according to Section 2.1.

2.4. Analytical methods

MLSS, mixed liquor volatile suspended solids (MLVSS) and specific oxygen uptake rate (SOUR) were determined according to standard methods. The mixed liquor was fractionated in two fractions: supernatant and soluble. The supernatant was obtained after filtration of the mixed liquor with a 4–12 μm filter paper Whatman 589/2 (GE Healthcare, USA) in order to remove solids and cells. Subsequently, the supernatant was filtered through a 0.45-μm-pore-diam Acetate Cameo 30 A syringe filter (GE Water & Process Technologies, USA) thus removing a part of the colloidal fraction. The filtrate obtained corresponds mainly to the soluble fraction. Measurements of soluble chemical oxygen demand (SCOD) and dissolved organic carbon (DOC) were determined in the soluble fraction photometrically with standard test kits (Test Cells Merck KGaA-Photometric method, Germany) and two thermoreactors CR 3000 and CR 3200 (WTW, Germany). Particle size distribution (PSD) was measured in the supernatant with a Zetasizer Nano ZS (Malvern Instrument Ltd., UK). Supernatant samples were diluted (1/100) previously to PSD analysis.

Regarding the influent and the permeate, pH, conductivity and temperature were measured with a pH meter-conductimeter Inolab Multilevel 3 (WTW, Germany), whereas turbidity was measured with a turbidimeter 2100 AN (Hach, Germany). Composite samples (three times a day) were taken to determine photometrically the removal efficiency of chemical oxygen demand (COD), NH_4^+–N and total phosphorus (TP). Finally, to quantify the retention of pathogens, *Escherichia coli* and coliforms were quantified as colony-forming units (cfu) in the permeate utilising Compact Dry EC medium plates (HyServe & Co. KG, Germany).

2.5. The Delft Filtration Characterisation Method

To quantify the impact of the different additives tested, on the filterability of the mixed liquor, the Delft Filtration Characterisation Method (DFCm) has been used. The DFCm consists of a standardised small-scale membrane Filtration Characterisation unit (FCu) operated on the basis of a standardised measuring protocol. The DFCm facilitates the measuring and characterisation of different samples of activated sludge under the same conditions allowing unequivocal comparison of the filterability quality of activated sludge samples operating under different circumstances or from different installations. The DFCm has been extensively used throughout numerous (pilot and full-scale) MBR installations around Europe. This method has been well described and reported by different DFCm operators [13–15] and researchers [16,17]. During each experiment, an activated sludge sample is circulated at a crossflow velocity of 1 m s^{-1} through a single tubular, inside-out X-flow membrane (Norit, The Netherlands), with a diameter of 8 mm and a nominal pore size of 0.03 μm. Activated sludge circulation and permeate extraction are achieved by two peristaltic pumps. Extraction of permeate during the standardised experiment is achieved at a constant flux of 80 L m^{-2} h^{-1}. Three pressure sensors installed at the feed, concentrate and permeate sides of the membrane allow the calculation of the resistance according to Darcy's law. For easy comparison between tests, the so-called ΔR_{20} value is used. This value refers to the resistance increase after a specific permeate production of 20 L m^{-2}. Filterability is qualified in accordance with the scale adapted from Geilvoet [15] (Table 3) which is based on hundreds of measurements in pilot and full-scale MBRs [13–15,18–23].

Table 3
ΔR_{20} and corresponding filterability qualification

ΔR_{20} [*10^{12} m^{-1}]	Qualification
< 0.1	Good
0.1–1.0	Moderate
> 1.0	Poor

2.6. Additives as substitute for the initial inoculum

PAC distributed by Carl Roth GmbH (Germany), $FeCl_2$ provided by Aachen-Soers WwTP and Alumin 7 provided by Aachen-Horbach WwTP have been used as additives. Table 4 shows the characteristics of these compounds.

3. Results and discussion

3.1. Determination of the working flux

The determination of both the critical and the working flux is described in detail by Gil et al. [2] in the first part of this study. J_c ranges of 18–19 L m^{-2} h^{-1} for MBR1 and MBR2, and 16–17 L m^{-2} h^{-1} for MBR3 were found by means of the flux step method. Consequently, a value of 15 L m^{-2} h^{-1} was chosen to conduct the experiments.

Table 4
Characteristics and composition of the additives used as initial inoculum

Characteristics	PAC	$FeCl_2$	Alumin 7
Composition			
		Fe^{2+} 8.7%	
	Ca < 0.02%	Ca 2.8%	Al_2O_3 13.2%
	Fe < 0.015%	Mn 1.4%	NaOH 24.1%
	PO_4 < 3.5%	Mg 0.6%	Na_2O 18.7%
	H_2O < 15%	HCl 0.3%	[$NaOH{\cdot}Al(OH)_3$] 44.4%
		Insol. < 0.001%	
Content	–	87 gFe kg^{-1}	98 gAl L^{-1}
Appearance	Black powder	Dark-green solution	Brownish solution
Density [kg m^{-3}]	290	1365	1440
pH	2.0–3.5	< 1	> 13
Viscosity (20°C) [mPa s]	–	3	50
Particle diameter	> 75 μm 19%	–	–
	> 10 μm 10%		
CAS-Nr[a]	7440-44-0	7758-94-3	1302-42-7
Methylene blue adsorption	⩾25 g 100 g^{-1}	–	–

[a]Chemical abstract service number.

3.2. PAC and $FeCl_2$ vs. activated sludge (experiment 1)

In first trial, PAC and $FeCl_2$ have been compared with activated sludge. Activated sludge as initial inoculum was introduced in MBR1. The MLSS concentration was diluted according to the optimal MLSS concentration (1 g L^{-1}) found by Gil et al. [2] to start-up MBR-SWwTP. Low PAC concentration (1 g L^{-1}) was introduced in MBR2 whereas $FeCl_2$ was added into MBR3 until pH ~ 3. These concentrations corresponded to 1 kg of PAC and 12 L of $FeCl_2$ respectively for our volume reactor (1 m^3). This pH value was chosen since Katz et al. [24] found ferric chloride performing well in the range 3–10, and because, based on our experiments, the greater the amount of $FeCl_2$, the better the performance obtained. PAC can be acquired in bulk at the reasonable price of 2 € kg^{-1} [25]. However, problems related with both, storage and handling of PAC make this option unfeasible for the majority of manufacturers. PAC can be also acquired from retailers at an approximate price of 20 € kg^{-1}. In this research, low PAC addition has been chosen to make its application economically viable. The limitation in the usage of $FeCl_2$ is fairly based on discharge criteria than in economical reasons (0.5 € kg^{-1} $FeCl_2$). $FeCl_2$ is quite acidic, and as a consequence, part of the inflow is solubilised resulting in a permeate with both high conductivity and SCOD, and low pH during the first days of operation. Fig. 2 shows TMP and flux curves plus accompanying filterabilities.

MBR2 demonstrated the worst performance since PAC particles itself could contribute to the total resistance. At the end of phase I, MBR2 showed a high average fouling rate, 45 mbar d^{-1} whereas MBR1 (activated sludge) indicated a slightly higher average fouling rate than MBR3 ($FeCl_2$), 18 vs. 13 mbar d^{-1}, respectively. This difference was increased at the end of phase II, with MBR1 showing an average fouling rate three times higher than MBR3 (34 vs. 10 mbar d^{-1}). $FeCl_2$ demonstrated the best performance, showing the lowest fouling rate at the end of phase II. The evolution of the TMP is also reflected by the quality of the mixed liquor. In Fig. 2b, increasing ΔR_{20} values can be observed for both, activated sludge and PAC, with poorer filterabilities for the latter. In contrast, MBR3 showed a decreasing tendency, with a moderate ΔR_{20} initial value (0.59×10^{12} m^{-1}) followed by good filterabilities in the following days (0.03×10^{12} and 0.05×10^{12} m^{-1} on days 4 and 6, respectively).

Fig. 3 shows SCOD and DOC concentrations for MBR1 (activated sludge) and MBR2 (PAC) along with time. Fig. 3 aims to compare the adsorption capacity of soluble matter between activated sludge and low PAC addition. SCOD and DOC values from MBR3 ($FeCl_2$) have not been included since due to the acidity of $FeCl_2$, an important percentage of the inflow is solubilised resulting in both higher SCOD and DOC values than the ones shown in Fig. 3. However, this disadvantage is overweighed by the aggregation of soluble and colloidal matter forming aggregates,

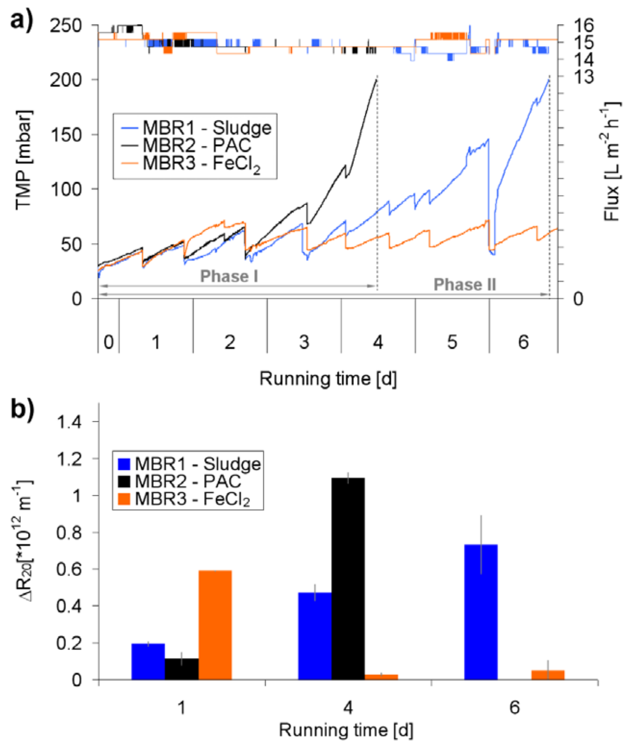

Fig. 2. (a) TMP and flux curves for experiment 1 and (b) ΔR_{20} values for experiment 1.

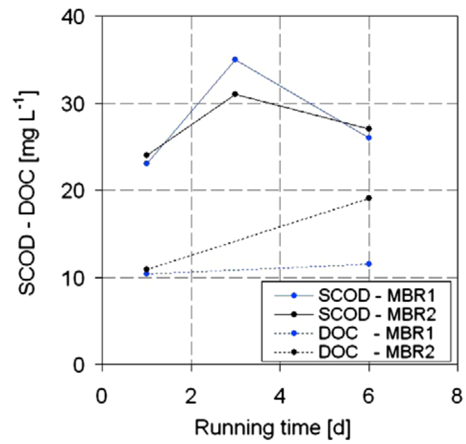

Fig. 3. SCOD and DOC concentrations for MBR1 and MBR2 in experiment 1.

which participate in the creation of a cake layer as it will be discussed in the following sections.

Either powdered or granulated activated carbon differentially adsorb the hydrophobic and part of the *amphiphilic* fractions of SCOD (75–80%). This feature, as can be seen in Fig. 3, allowed a similar adsorption–elimination of SCOD between PAC and activated sludge. Remy et al. [25] combined low PAC addition with high SRTs obtaining good results. In spite of their slightly higher working flux, (17.4 L m^{-2} h^{-1}), they noticed very low fouling rates. This can be attributed to a higher MLSS concentration (13.6 g L^{-1}). In a posterior study, Remy et al. [26] observed that SCOD and colloidal COD were removed from the permeate in the presence of low PAC addition. They concluded that the formation of stronger sludge flocs in the presence of low PAC addition seems the more likely explanation as to why low PAC concentrations help to reduce fouling. This would explain both the bad performance for the TMP curve observed in Fig. 2a for PAC, and the higher concentration of DOC detected in MBR2 (PAC) than in MBR1 (activated sludge), 19 vs. 12 mg DOC L^{-1}, respectively. Remy et al. worked with high MLSS concentrations in both studies (13.6 and 10.1 g L^{-1}) in comparison to this study. These results suggest that low PAC addition is only effective at high MLSS concentrations. Therefore, low PAC addition is not suitable to start-up MBR-SWwTP as a substitute for activated sludge. Some authors have found better results at higher PAC concentrations. Ng et al. [27] observed that to prevent irreversible fouling, 5 g L^{-1} of PAC was required. However, as commented previously, higher PAC concentrations are not considered in this study due to economical reasons.

3.3. Alumin 7 and $FeCl_2$ vs. activated sludge (experiment 2)

In the previous section, $FeCl_2$ has demonstrated a better performance than both low PAC addition and activated sludge as initial inoculum. Since $FeCl_2$ presents an acidic character, a basic coagulant was also tested as a possible substitute for activated sludge. In this section, Alumin 7 (pH > 13) has been compared with $FeCl_2$ (pH < 1) and activated sludge. The excellent performance and accompanying filterabilities demonstrated by $FeCl_2$ suggest that the amount added can be minimised. To this end, $FeCl_2$ was added till just pH = 4 corresponding to 7 L instead of the 12 L used in experiment 1. Alumin 7 was added till pH = 10 (the same variation with respect to neutral pH) corresponding to just 400 mL. Fig. 4 shows TMP and flux curves plus accompanying filterabilities.

MBR2 (Alumin 7) demonstrated the worst filterability showing the TMP jump on day 2. Either a basic pH or the lower amount (400 mL) introduced due to the high alkalinity of Alumin 7 exhibited both poor filterabilities and performance. At the end of phase I, MBR2 indicated an average fouling rate of 59 mbar d^{-1}, whereas MBR1 (activated sludge) and MBR3 ($FeCl_2$) showed 22 and 39 mbar d^{-1} respectively. These results exclude Alumin 7 as a possible substitute for activated sludge.

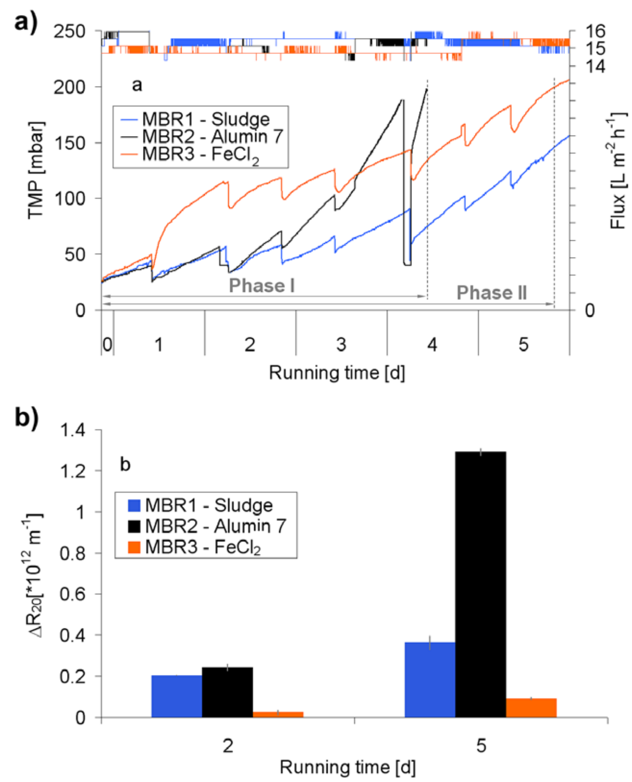

Fig. 4. (a) TMP and flux curves for experiment 2 and (b) ΔR_{20} values for experiment 2.

At the end of phase II, average fouling rates of 30 and 42 mbar d^{-1} for MBR1 and MBR3 were observed. These values suggest a priori a better performance for MBR1. However, the better filterabilities, observed for MBR3 as shown in Fig. 4b, suggest that $FeCl_2$ has the potential ability to aggregate soluble and colloidal matter which can participate in the formation of a cake layer on top of the membrane acting as a prefilter. Fig. 5 shows one of the cartridges submerged in MBR3 ($FeCl_2$) after experiment 2. The formation of a thin cake layer on top of the membrane can be observed, which can be easily removed.

The jump observed in the TMP curve corresponding to MBR3 in Fig. 4a on day 1 coincides with the first input of wastewater. Due to the lower amount of $FeCl_2$ introduced (with respect to experiment 1), the cake layer was not formed immediately resulting in a loss of performance (from day 1 on). In contrast, MBR 3 in experiment 1 ($FeCl_2$, pH = 3) registered good filterabilities. In this case, the TMP kept low values, probably due to the higher amount of $FeCl_2$ introduced and accompanying cake layer formation.

Regarding the activated sludge (MBR1), the TMP curve followed a similar pattern as the one for $FeCl_2$ (MBR3). Similar $\Delta P/\Delta t$ values for both curves from day 2 on (1.7×10^{-5} and 1.8×10^{-5} mbar d^{-1} for MBR1 and MBR3, respectively) combined with better filterabilities for MBR3 indicate the potential of $FeCl_2$ as a possible substitute for activated sludge.

Results from experiments 1 and 2 point to $FeCl_2$ as a possible substitute for activated sludge during the start-up phase of MBR-SWwTP. $FeCl_2$ is more effective to mitigate gradual fouling, which is caused mainly by supernatant organics. This phenomenon can be seen in Fig. 4a. The TMP rose on day 1 coinciding with the first input of wastewater. In this stage, the first colloidal and soluble matter react with the $FeCl_2$ and the cake layer starts to form and deposit onto the membrane. Simultaneously, due to the brief contact time prior to filtration, supernatant organics, which are not coagulated, are dragged onto the membrane blocking the pores. As a consequence, the cake layer has been deposited after initial pore blocking occurs, resulting in a rapid fouling stage (from day 2 on) governed by suspended flocs and coagulated matter. Consequently, the two TMP curves corresponding to MBR1 (activated sludge) and MBR3 ($FeCl_2$) are almost identical in shape and present similar fouling rates.

3.4. Optimization of $FeCl_2$ addition (experiment 3)

In this section, based on results from previous sections, the addition of $FeCl_2$ has been optimised with the aim to build up a cake layer before pore blocking occurs. $FeCl_2$ was added in all MBRs till pH = 4. Table 5 describes the different procedures to add

Fig. 5. Cake layer deposit composed of soluble and colloidal material coagulated by $FeCl_2$.

$FeCl_2$ into the tanks, whereas Fig. 6 shows TMP and flux curves plus accompanying filterabilities.

Initial TMP values for MBR1 were lower since the tank was filled up with tap water. However, as wastewater was gradually introduced, the TMP curve for MBR1 matched the other two TMP curves on day 3, and from this day on, the TMP increased showing an average fouling rate of 36 mbar d^{-1} at the end of phase I. In turn, MBR2 and MBR3 showed lower average fouling rates of 8.4 and 6.6 mbar d^{-1} respectively at the end of phase I. Indeed, TMP curves for MBR2 and MBR3 were identical till day 5. From this day on, MBR3 showed the best performance with a fouling rate of 9.6 mbar d^{-1}, whereas the fouling rate for MBR2 was 24 mbar d^{-1} at the end of phase II. The evolution of the TMP curves is supported by the filterability of the initial inoculum (Fig. 6b). On day 1, MBR1 (tap water) showed a better filterability (0.02×10^{12} m^{-1}) than MBR2 (wastewater) and MBR3 (wastewater + contact time), 1.09×10^{12} and 0.48×10^{12} m^{-1}, respectively. A better filterability can be appreciated in MBR3, probably due to the contact time between $FeCl_2$ and wastewater. The following days (days 4 and 7) are characterized by good filterabilities with ΔR_{20} values ranging between 0.02×10^{12} and 0.06×10^{12} m^{-1}, except for MBR1 with a moderate value of 0.16×10^{12} m^{-1} on day 7 coinciding with the reach of the TMP threshold. On day 9, the quality of the mixed liquor showed poor and moderate values for both MBR2 and MBR3 (1.40×10^{12} and 0.87×10^{12} m^{-1}, respectively), continuing this tendency on day 10, with a poor filterability (1.1×10^{12} m^{-1}) for MBR3. These results confirm our theory. MBR1, filled up with tap water, was not able to create a cake layer prior to filtration. As a consequence, initial pore blocking occurred before the cake layer was established. This led to the fast increase in the TMP even with the low ΔR_{20} values observed. In turn, MBR2 and MBR3 filled with ½ tank of tap water and completed with wastewater behaved similarly with the exception of higher ΔR_{20} values and a faster increase of the TMP for the former. Since the only difference between MBR2 and MBR3 was the contact time in the latter, these results underline the benefits of a contact time between coagulant and wastewater before filtration starts.

3.5. Effect of particle size distribution

PSD was measured in the supernatant to analyse the impact of the particle size on filtration performance. Recent investigations almost reached an agreement suggesting that small particles are the major contribution to fouling. The size of the predominant contributor was located in the range 0.5–15 μm [28,29]. Van der Graaf et al. [20] found a direct relation between the filterability of the activated sludge and the concentration of sub-micron particles (colloids)

Table 5
Different procedures to start-up with wastewater and $FeCl_2$ in experiment 3

MBR	Step	Time (min)	Procedure
1	1	–	Fill in the tank with tap water (full tank = 1000 L)
			$FeCl_2$ addition adjusting pH till pH ~ 4 (6.5 L)
	2		Start filtration
2	1	–	Fill in the tank with tap water (half tank = 500 L)
			Introduce wastewater to fill the tank completely (500 L)
			$FeCl_2$ addition adjusting pH till pH ~ 4 (13.5 L)
	2		Start filtration
3[a]	1	–	Fill in the tank with tap water (half tank = 500 L)
			Introduce wastewater to cover the membranes (175 L)
			$FeCl_2$ addition adjusting pH till pH ~ 4 (7 L)
	2	120	Contact time to coagulate colloidal and soluble organics
	3	–	Input of wastewater (175 L)
			$FeCl_2$ addition adjusting pH till pH ~ 4 (4 L)
	4	120	Contact time to coagulate colloidal and soluble organics
	5	–	Input of wastewater (175 L)
			$FeCl_2$ addition adjusting pH till pH ~ 4 (3.5 L)
	6	120	Contact time to coagulate colloidal and soluble organics
	7	–	Start filtration

[a]During steps 2–7, the aeration was 1 min ON and 30 s OFF to promote mixing/favour contact between $FeCl_2$ and wastewater.

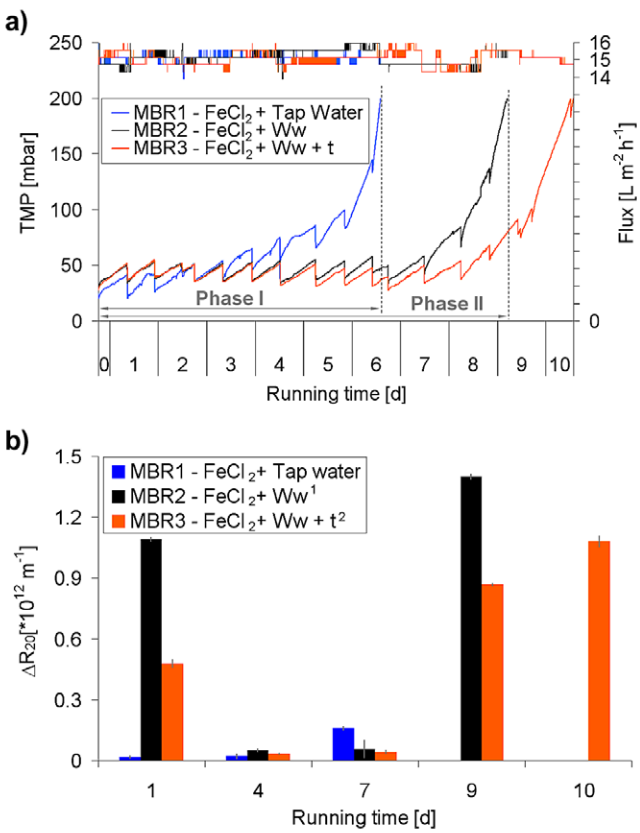

Fig. 6. (a) TMP and flux curves for experiment 3 and (b) ΔR_{20} values for experiment 3. 1. Wastewater. 2. Wastewater + contact time.

within a range of 0.1–1.0 μm in the supernatant. Since fine particles reduce the porosity and augment the specific resistance of the cake layer [2], sub-micron particles ($V_{\text{sub-micron}}$) in the range 0.1–1 μm are expected to impact negatively on the formation of the cake layer. All particles analysed in the supernatant were in the range 0.06–6 μm coinciding with part I of this study [2]. Fig. 7 summarises $V_{\text{sub-micron}}$ (expressed as %) at the beginning and at the end of the different experiments.

In experiments 1 and 2, $V_{\text{sub-micron}}$ increased slightly (68 and 65% respectively) at the end of the experiment in both MBRs initiated with activated sludge (MBR1). As commented previously, only under high MLSS concentrations, low PAC addition improves flocculation of colloidal foulants. As a result, $V_{\text{sub-micron}}$ increased slightly in MBR2 along experiment 1 (from 69 to 74%). In turn, due to its high alkalinity, just 400 mL of Alumin 7 were added. With such low addition, the coagulant effect disappeared rapidly, resulting in an increase of the $V_{\text{sub-micron}}$ (from 51 to 100%) and a raise of the TMP.

$FeCl_2$ showed different behaviours depending on the amount and the way that was added. In experiment 1, under low pH conditions (∼3), both the TMP and the filterability showed a good performance in MBR3 in spite of the increase in $V_{\text{sub-micron}}$ (from 47 to 100%), probably due to the release of particles at low pH. On the contrary, in experiment 2, a rapid increase of the TMP was observed as a consequence of the lower $FeCl_2$ addition, even with a reduction of $V_{\text{sub-micron}}$ (from 95 to 65%) and good filterabilities too. These results suggest that $V_{\text{sub-micron}}$ is proportional to the addition of $FeCl_2$. However the negative effect of high $FeCl_2$ addition ($V_{\text{sub-micron}}$ release) is outweighed by the positive effects: aggregation of large particles and deposition onto the membrane forming a cake layer.

In experiment 3, the best reduction of $V_{\text{sub-micron}}$ (from 93 to 75%) corresponded to MBR1. These fig-

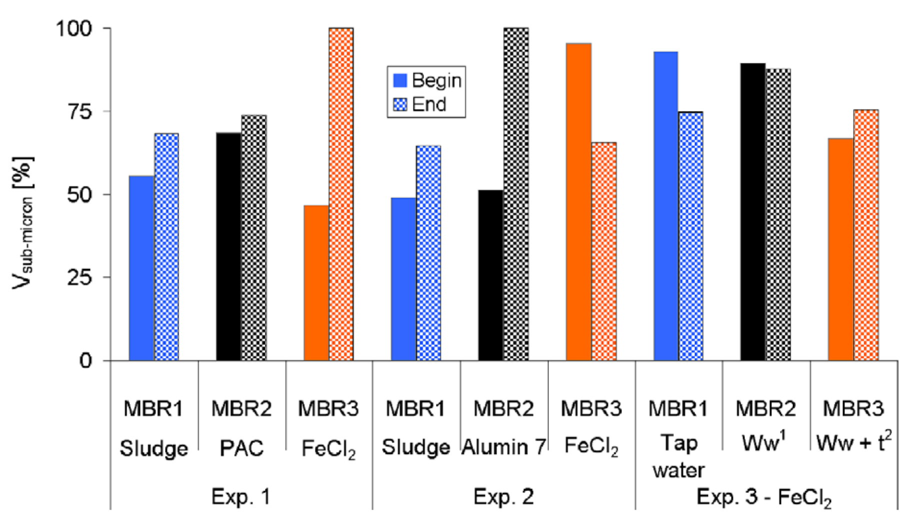

Fig. 7. $V_{\text{sub-micron}}$ expressed as % for the different experiments. 1. Wastewater. 2. Wastewater + contact time.

ures are very similar to MBR3 in experiment 2 (from 95 to 65%) since the conditions were the same ($FeCl_2$ addition till pH ~ 4 and tap water as initial inoculum). In both situations, the TMP suffered a fast increase, suggesting the late formation of the cake layer. MBR2 (from 89 to 88%) and MBR3 (from 67 to 75%) did not present big changes in $V_{\text{sub-micron}}$. Due to an improved coagulation of soluble and colloidal matter thanks to the contact time, less fine particles were present in MBR3 than in MBR2 when the filtration started. As a result, the filterability of the initial inoculum in MBR3 was enhanced, leading to a better evolution of the TMP.

3.6. Microbial activity, treatment efficiency and rejection of pathogens

SOUR of the initial inoculum was determined (Table 6) at the end of the experiments to analyse the impact of the different additives on microbial activity. All SOUR values were in the range 1.7 ± 0.1–$28 \pm 1.2\,mgO_2\ gMLVSS^{-1}\,h^{-1}$. SOUR in the MBR started-up with PAC, could not be determined due to the low microbial activity observed. Activated sludge as initial inoculum was expected to present higher SOUR values in comparison to the additives tested. However, a big difference was observed between the tests carried out with activated sludge (MBR1 in experiments 1 and 2) and the tests performed with $FeCl_2$ + wastewater (MBR2 and MBR3 in experiment 3). $FeCl_2$ coagulates soluble and colloidal matter, which serves as a support for the microbiology. Additionally, the contact time between coagulant and wastewater (MBR3-experiment 3), increased the microbial activity ($28 \pm 1.2\ mgO_2\ gMLVSS^{-1}\,h^{-1}$). Using ferric chloride as coagulant, Zhang et al. [9] found a strong decrease in pH and a reduction of microbiological activity. However, their MBR was in a steady-state process with a higher MLSS concentration and a biomass well acclimatised. Starting-up from "zero-biomass", a positive effect is more probable.

The quality of the permeate can be seen in Table 7. COD, NH_4^+–N and TP concentrations were monitored to check the impact of the tested additives on the treatment efficiency. MBR-SWwTP are normally operated at a minimum HRT of 24 h. In this study, the HRT was around 65 h for all experiments. COD removal efficiency showed random values with percentages over 90% for all trials but the experiments carried out with $FeCl_2$ where the COD removal efficiency ranged from −55 till 91%. In this case, the higher the amount of $FeCl_2$ and wastewater used as initial inoculum, the worse the removal efficiency became. During the first days, low COD removal efficiencies can be expected since $FeCl_2$ solubilises part of the inflow. However, in the following days, an improvement in the COD removal efficiency was observed since $FeCl_2$ is oxidised progressively. NH_4^+–N removal was over 94% for trials carried out with activated sludge. On the contrary, the trials started-up with additives showed decreasing NH_4^+–N removal efficiencies. This difference is attributed to both, low pH and the lack of nitrifier bacteria, which are only present at high SRTs. However, a slight recovery was observed in experiment 3 in MBR3, from 2 to 12% on day 10, indicating that the capacity to nitrify starting-up with $FeCl_2$ needs acclimatisation. The system was not provided with an anaerobic zone for TP removal. Therefore, decreasing TP removal efficiencies were noticed for the trials started-up with activated sludge. PAC in turn, showed an improvement from 0 to 40% in four days of operation. In this case, the use of additives is an advantage, since TP can be eliminated via coagulation. TP removal efficiencies were over 92% for those trials using $FeCl_2$ as initial inoculum.

Suspended solids (SS) in the permeate presented an average value of $40 \pm 10\,mg\ L^{-1}$. Comparing this value with the average SS concentration of the inflow ($70 \pm 20\,mg\ L^{-1}$), the effectiveness of the settling tank as pretreatment to remove SS is confirmed.

In terms of rejection of pathogens, the membrane showed a total rejection of both *E. coli* and coliforms for all experiments, except for MBR2 (Alumin 7) at the end of experiment 2 ($154\,cfu\,mL^{-1}$), and for MBR3 ($FeCl_2$) on day 7 in experiment 3 ($4\,cfu\,mL^{-1}$). Either basic conditions or the inexistence of a prefilter acting

Table 6
SOUR[a] values analysed at the end of the experiments

Experiment	MBR1	MBR2	MBR3
1	5.3 ± 0.5 (Activated sludge)	(PAC)	1.7 ± 0.1 ($FeCl_2$)
2	10 ± 1.3 (Activated sludge)	5.0 ± 1.4 (Alumin 7)	3.8 ± 1.1 ($FeCl_2$)
3	3.9 ± 1.1 ($FeCl_2$)	19 ± 1.3 ($FeCl_2$ + Ww[b])	28 ± 1.2 ($FeCl_2$ + Ww + Contact time)

[a]Units: [$mgO_2\ gMLVSS^{-1}\ h^{-1}$].
[b]Wastewater.

Table 7
Effluent quality for the studied period

Experiment	MBR	COD (mg/L^{-1})	NH_4^+–N (mg L^{-1})	TP (mg L^{-1})	T (°C)	pH	Turbidity (NTU)	Conductivity (μS cm^{-1})
1	1	17 ± 4.3	1.0 ± 0.9	1.3 ± 1.2	15 ± 0.5	6.9 ± 0.5	0.55 ± 0.3	591 ± 132
	2	14 ± 1.4	9.0 ± 8.0	5.5 ± 2.1	15 ± 0.3	7.7 ± 0.4	0.62 ± 0.4	494 ± 185
	3	125 ± 46	12 ± 16	0.3 ± 0.1	15 ± 0.4	5.2 ± 0.7	0.44 ± 0.1	4,470 ± 3,117
2	1	15 ± 6.4	1.3 ± 0.4	1.7 ± 1.7	16 ± 0.1	6.9 ± 0.6	0.64 ± 0.3	677 ± 83
	2	34 ± 5.7	13 ± 11	0.5 ± 0.1	16 ± 0.4	8.6 ± 1.1	0.47 ± 0.0	681 ± 164
	3	92 ± 28	14 ± 19	0.4 ± 0.1	16 ± 0.4	5.8 ± 0.3	0.99 ± 0.2	3,375 ± 2,397
3	1	68 ± 31	16 ± 12	0.3 ± 0.1	16 ± 0.5	6.2 ± 1.1	1.07 ± 0.6	2,763 ± 1,839
	2	267 ± 245	18 ± 15	0.4 ± 0.1	16 ± 0.6	5.7 ± 1.1	0.72 ± 0.2	5,027 ± 4,053
	3	214 ± 240	21 ± 14	0.4 ± 0.1	16 ± 0.6	6.0 ± 1.3	0.90 ± 0.4	4,221 ± 3,997

as a barrier explain the high number of pathogens in MBR2. In the case of MBR3, the number detected was minimum, and was followed by the absence of pathogens on day 10. On the whole, the membrane behaved as an excellent barrier against pathogens under the different conditions imposed. Excluding Alumin 7, these results are in compliance with reuse normatives [30] making feasible the reuse of water since the first day of operation.

3.7. Benefits and drawbacks of $FeCl_2$ addition

Adding coagulants during the start-up may depress the biological activity in some cases, i.e. PAC and Alumin 7 (see Table 6) with respect to that of activated sludge. However, a higher biological activity was noticed when $FeCl_2$ + wastewater were added in experiment 3. The first time that $FeCl_2$ reacts with wastewater implies a strong coagulation and flocculation, which results in the formation of the first flocs that serve as support media for the microbiology. Wu et al. [31] observed that the floc size increased significantly (from 50 to 68 μm) when PFS was firstly added. The next two times they added PFS, the floc size did not increase so obviously. Similar to PFS, $FeCl_2$ has the ability to supply positive electric charges for colloidal and soluble organics. As a consequence of $FeCl_2$ addition, charge neutralization and flocculation are enhanced whereas colloids loose stability, resulting in flocs with larger size when compared to the addition of other additives. This can be checked in Fig. 8, where flocs formed at the end of the experiments by the different additives used in this study are shown.

The accompanying formation of the cake layer by larger flocs is the key feature of the better filterability observed. Another characteristic of $FeCl_2$ is its ability to produce more excess sludge. Wu et al. [31] found a little increase on MLSS concentration (0.4 g L^{-1} on average) when a low concentration of PFS (1 mM Fe) was added. Nevertheless, to start-up MBRs, this drawback turns into a benefit since a rapid increase of the MLSS is desirable. When comparing Alumin 7 with $FeCl_2$ (experiment 2), the MLSS concentration increased in 3 days from 0.19 to 0.29 to 0.26 g L^{-1} and from 0.23 to 0.58 g L^{-1}, respectively. Additionally, in experiment 3, MLSS increased from 0.11 to 0.61 g L^{-1}, from 0.32 to 1.3 g L^{-1} and from 0.19 to 1.2 g L^{-1} in MBR1, MBR2 and MBR3, respectively.

Among the additives tested, $FeCl_2$ has demonstrated to be the best candidate to substitute activated sludge as an initial inoculum in the start-up of MBR-SWwTP. However, the addition of $FeCl_2$ presents some drawbacks that MBR manufacturers and operators must bear in mind. For starters, as a result of supplying positive electric charges, Fe_2^+ oxidises to Fe_3^+ with accompanying orange coloration. Customers could complain about esthetic problems even if the coloration can be removed with high-pressure water. Another problem is the acidity and the high conductivity associated with $FeCl_2$ addition (Fig. 9).

MBR2 and MBR3 contained approximately double the amount of $FeCl_2$ as MBR1. However, pH curves showed similar behaviours for all MBRs. Regardless of the amount of $FeCl_2$ added, the pH was similarly increased in every input of wastewater. This buffering process ends when all $FeCl_2$ is oxidised coinciding with the end of the coagulation process, restoring pH to neutral values. This happened on days 5, 7 and 8 for MBR1, MBR2 and MBR3, respectively. Conductivity in the permeate showed high values for MBR2 and MBR3 (> 9,000 μS cm^{-1}) in comparison with MBR1 (4,810 μS cm^{-1}) at the beginning of the experiment. However, as wastewater was introduced, all MBRs followed the same tendency meeting the same conductivity range on day 7 (1,200–1,600 μS cm^{-1}). Following this decreasing tendency, MBR3 finished the experiment with a con-

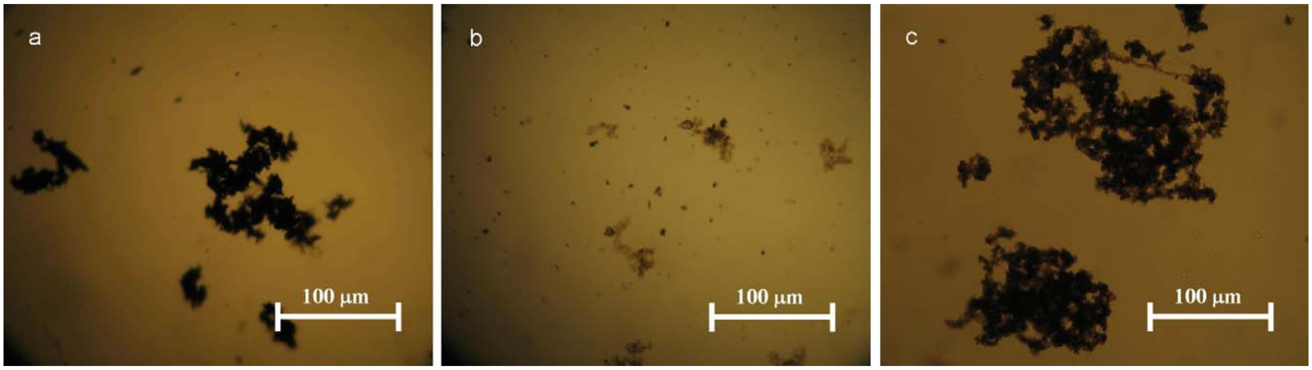

Fig. 8. Different floc size according to the additive used at the end of each experiment: (a) PAC, (b) Alumin 7 and (c) $FeCl_2$.

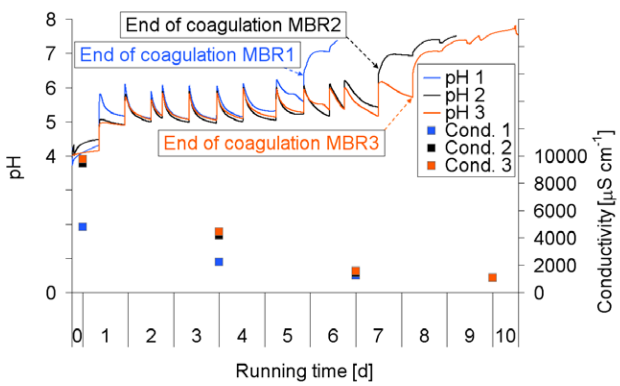

Fig. 9. pH curves and conductivity in the permeate for experiment 3.

ductivity of 1,081 µS cm^{-1} on day 10, whereas the conductivity in the inflow was 947 ± 16 µS cm^{-1}. These results suggest that, regardless of the amount of $FeCl_2$ added, the pH will follow the same pattern as wastewater is introduced. On the contrary, pH stabilisation showed to be proportional to the amount of $FeCl_2$ introduced. In terms of conductivity, independently of the amount added, the same decrease for this parameter can be expected after a few days of operation. These results suggest that the negative effects of working at low pH and high conductivity will be ameliorated in the following days to the start-up.

4. Conclusion

The test of three different additives to start-up MBR-SWwTP as possible substitutes for activated sludge as an initial inoculum has been successfully completed. Low PAC addition, which could be economically viable, resulted in a low performance. Results suggest that low PAC addition is not effective at low MLSS concentrations since the formation of stronger sludge flocs in the presence of low PAC addition at high MLSS concentrations seems the more likely explanation as to why low PAC concentrations help to reduce fouling. Therefore, low PAC addition is not suitable to start-up MBR-SWwTP as a substitute for activated sludge. Alumin 7, an alkaline coagulant, was also tested with negative results. Its high alkalinity makes its use unfeasible in the same range as $FeCl_2$. Higher addition could be employed at the expense of a pH increase, but the effects of such increase on the biomass and operation are unknown and should be studied.

In turn, $FeCl_2$ demonstrated the best results. Its potential ability to aggregate soluble and colloidal matter generates particles and former flocs from wastewater, which not only serve as support for microbiology, but also participate in the formation of a cake layer on top of the membrane acting as a prefilter. Additionally, to create the cake layer before pore blocking occurs, the use of both wastewater and sufficient contact time between coagulant and wastewater has demonstrated the best results in terms of TMP increase and filterability. High conductivities, low pH and little removal efficiency—except for TP—during the first days, together with aesthetic problems must be borne in mind when considering its application. On the other hand, a fast increase of the MLSS concentration, enhanced microbial activity, the possibility to create a cake layer that acts as a prefilter and excellent filterabilities convert this additive in a possible substitute for activated sludge. Since the coagulant effect disappears when all $FeCl_2$ has reacted with wastewater, and their negative effects disappear gradually with the input of wastewater, we encourage manufacturers and operators to reach "at least" the pH proposed in this study. Finally, the permeate has proved to be suitable for water reclamation. However, it is recommendable to wait a few days after the start-up, until the effluent stabilises to normal values.

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