MBR fouling control and permeate quality enhancement by polyaluminium chloride dosage: a case study

A. Teli, M. Antonelli, L. Bonomo and F. Malpei

ABSTRACT

Scope of this study was to evaluate the effectiveness of a metal salt (polyaluminium chloride, PACl) dosage into a pilot-scale MBR (membrane bioreactor) in terms of fouling control and permeate quality enhancement, especially with reference to specific textile macro-pollutants (dyes and surfactants). The pilot plant was fed with a mixed domestic-textile wastewater (textile wastewater accounted for 65% of total flow and for 70% of total chemical oxygen demand, COD, load) and operated for 7.5 months without flux enhancers (step 1) and 3 months with the addition of PACl (step 2). The optimum dose was defined performing a jar-test campaign between step 1 and step 2 (12.5 mg gMLSS^{-1} that corresponds to 0.4 g d^{-1}). The addition of PACl resulted in a significant decrease of the filtration resistance due to cake layer formation (R_c, −65.4%) and of the irreversible fouling rate, evaluated as the average variation per unit time of the filtration resistance due to foulants adsorption on membrane pore wall (FR, −45.3%). As for permeate quality, removal rates related to total phosphorus and textile macro-parameters such as colour and anionic surfactants, increased by +64, +16 and +7%, respectively. No significant effect was observed on COD, non-ionic surfactants and nitrogen compounds removal.

Key words | flux enhancers, fouling, membrane bioreactor, polyaluminium chloride

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INTRODUCTION

Membrane bioreactors (MBRs) have proven to be an effective treatment for domestic as well as industrial wastewaters (Yang *et al*. 2006). However, membrane fouling limits the application, reducing filtration performance and leading to higher operating costs due to fouling mitigation efforts. Apart from membrane characteristics (e.g. materials, pore size and hydrophobicity) that are expected to play a minor role during extended filtration periods (Le-Clech *et al*. 2006), fouling is directly determined by sludge characteristics (e.g. soluble microbial products, SMP; extracellular polymeric substances, EPS; mixed liquor suspended solids, MLSS; flocs size and hydrophobicity) and hydrodynamic conditions (Meng *et al*. 2009). However, operating conditions (e.g. sludge retention time, SRT; temperature; dissolved oxygen, DO) and feedwater characteristics (e.g. nature of feed; divalent cations content) have indirect effects by modifying biomass characteristics (Meng *et al*. 2009). As a consequence, fouling is a complex phenomenon and its control and reduction is a key issue for MBR processes. Several design and operating strategies can be implemented to achieve this scope such as membrane scouring, relaxation (RX), backwashing, optimization of aeration or sustainable flux operation (Le-Clech *et al*. 2006; Drews 2010). In addition, flux enhancers such as powdered activated carbon (PAC), cationic polymers or metal salts have the potential to reduce fouling by different mechanisms of action, for instance: charge neutralization/adsorption of organic foulants such as SMP, bridging particles into larger flocs improving bio-flocculation and sludge filterability and, as for PAC, increase in shear resistance due to particles inclusion in flocs (e.g. Wu *et al*. 2006; Iversen *et al*. 2009a; Remy *et al*. 2010). Additionally, flux enhancers improve permeate quality, as pollutants can be flocculated or adsorbed and thus retained by the membrane. Therefore, this application can be of particular interest in the treatment of industrial wastewater containing recalcitrant or slowly biodegradable organics which cannot be retained or completely biodegraded. An example of this is the treatment of textile wastewater for which MBRs have been studied by several authors (e.g. Malpei *et al*. 2003; Lubello *et al*. 2007)

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showing better removal efficiencies of chemical oxygen demand (COD), colour and surfactants with respect to conventional activated sludge processes. This study is aimed to evaluate the effectiveness of a metal salt dosage into a pilot-scale MBR fed with a mixed textile and domestic wastewater (textile wastewater accounted for 65% of total flow and for 70% of total COD load) in terms of fouling control and permeate quality enhancement. Polyaluminium chloride (PACl) was chosen on the basis of a previous experience on the same wastewater (Bonomo *et al*. 1995).

METHODS

Pilot-scale MBR

The pilot-scale MBR was designed according to the modified Ludzack-Ettinger process for biological nitrogen removal, with a separate membrane compartment equipped with two laboratory-scale Siemens Water Technologies (MemJet®) membrane modules (surface area 0.5 m^2 and pore size 0.04 μm). The pilot plant was placed after the pre-treatments (coarse screen, 1 × 5 mm screen, grit chamber) of a full-scale Wastewater Treatment Plant (WWTP) near Como (Italy), and after a pilot stainless steel mesh (0.49 mm), to remove residual textile fine fibres in the wastewater. The reactor was seeded with the activated sludge from the full-scale WWTP in order to maintain a biomass concentration of 5.9 ± 0.50, 4.4 ± 0.66 and 7.6 ± 0.63 g MLSS L^{-1} in the aerobic, anoxic and membrane tanks, respectively. The percentage of mixed liquor volatile suspended solids (MLVSS/MLSS) was 80 ± 2.0%. Operating parameters were: permeate flux (J), 10 L m^2 h^{-1} (LMH); membrane air flow rate, 420 L_N h^{-1} m^{-2}; RX, 8:1 (i.e. 8 min filtration, 1 min RX); HRT (hydraulic retention time), 14.5 h; SRT, 25 ± 1.0 d; F/M (food to microorganism ratio), 0.10 ± 0.020 kg COD kgMLSS^{-1} d^{-1}. Other operating parameters are summarized in Table 1.

Design of the experiments

The pilot plant operated for 7.5 months without flux enhancers (step 1) and then for 3 months with the addition of TILLPAC18, an 18% PACl produced by TILLMANNS S.p.A. (step 2). The optimal PACl dose was selected performing a jar-test campaign, after 75 days (3·SRT) from the beginning of sludge wasting (day 133), assuring the complete adaptation of the biomass to MBR conditions. At the beginning of each step, membrane cleaning was performed according to the manufacturer procedures (physical clean; oxidative soaking, sodium hypochlorite at 1.500 mgCl_2 L^{-1}, ≥3 h; basic soaking, NaOH at 20.000 ppm, ≥3 h; acid soaking, citric acid at 0.5% and H_2SO_4 until pH = 2, ≥3 h).

From day 53 onwards a high concentrated solution of sodium acetate was fed into the anoxic tank with a loading

Table 1 | Main characteristics related to the influent wastewater and to monitoring parameters (ORP, oxidation-reduction potential)

	Step 1	Step 2
Influent wastewater		
COD (mg L^{-1})	284.6 ± 83.52	173.8 ± 57.27
TKN[a] (mg L^{-1})	41.1 ± 11.47	29.2 ± 8.87
Total phosphorus (mg L^{-1})	3.2 ± 0.64	1.2 ± 0.52
Colour (cm^{-1})	0.204 ± 0.1168	0.166 ± 0.0433
Non-ionic surfactants (mg L^{-1})	7.4 ± 2.47	3.9 ± 1.53
Anionic surfactants (mg L^{-1})	3.6 ± 0.97	2.6 ± 1.02
Operating parameters		
Aerobic tank		
pH	7.4 ± 0.34	7.5 ± 0.16
DO (mg L^{-1})	2.4 ± 2.32	1.3 ± 1.09
T (°C)	21.5 ± 4.15	13.1 ± 2.11
Anoxic tank		
pH	7.7 ± 0.23	7.9 ± 0.15
ORP (mV)	−349 ± 183	−416 ± 146.9

[a]TKN represented the major macro-pollutant for nitrogen during step 1 and 2, with an average percentage of ammonium ranging between 68 and 70% (NH_4^+/TKN). Nitrate and nitrite concentrations were much lower than TKN with values always <1.0 mgN L^{-1} (nitrate) and <0.5 mgN L^{-1} (nitrite).

of 35 g COD d^{-1} (44 ± 10% of the total F/M) to provide a readily biodegradable COD source to support denitrification.

The following parameters of influent and permeate were weekly analysed on 24 h average samples: COD, nitrogen compounds (total Kjeldahl nitrogen (TKN), NH_4^+, NO_2^-, NO_3^-), total phosphorus, and colour, anionic and non-ionic surfactants, as for textile macro-pollutants. Effluent sampling was shifted of an HRT value from the influent sampling. Trans-membrane pressure (TMP) and extracellular biopolymers (proteins and carbohydrates), both freely present in mixed liquor as SMP and bound to the flocs as EPS, were monitored as well. The average characteristics of the influent wastewater are summarized in Table 1.

As for the jar-test campaign, four doses of PACl were investigated in duplicate (6.25, 12.5, 25, 50 mg PACl $gMLSS^{-1}$) and the applied procedure consisted of conditioning (i.e. PACl dosage), rapid mixing (150 rpm, 60 s), flocculation (45 rpm, 90 min) followed by bulk liquid separation. The PACl potential of fouling reduction and permeate quality enhancement was evaluated by the detection of: (1) specific resistance to filtration (SRF) and modified fouling index (MFI), both measured on flocculated sludge; (2) total suspended solids (TSS) measured on the bulk liquid after sludge centrifugation (ALC4235, 2,600 g, 10 min); (3) SMP proteins and carbohydrates (SMPp, SMPc), COD and colour, measured on the bulk liquid. Four mixed liquor samples were collected, characterized by: MLSS, 5.8 ± 0.30 g MLSS L^{-1}; SRF, 2.5 ± 0.5410^{13} m kg^{-1}; MFI, 4,800 ± 1,180 s L^{-2}; supernatant TSS, 34 ± 3.9 mg SS L^{-1}; SMPp, 21.6 ± 6.01 mg L^{-1}; SMPc, 7.6 ± 3.33 mg L^{-1}; bulk COD, 69.8 ± 18.63 mg L^{-1}; bulk colour (abs. at 426 nm), 0.046 ± 0.0206 cm^{-1}.

Experimental procedures and analytical techniques

MLSS, TSS, MLVSS, COD and TKN were measured according to Standard Methods (APHA, AWWA, WEF 2005). Inorganic nitrogen compounds were measured according to Italian standards (IRSA 2003), which are quite similar to those described in Standard Methods (APHA, AWWA, WEF 2005). Colour was measured according to AbwV (2002) as absorbance at 426 nm, 558 nm, 660 nm (optical path: 1 cm) using a spectrophotometer (model Xion 500, Dr Lange). According to previous experiences on the same wastewater (inter alia, Rozzi *et al.* 2000) the absorbance at 426 nm is considered as reference parameter for colour in data interpretation. Dr Lange kits were used for the analysis of anionic and non-ionic surfactants (LCK332, LCK333) and for total phosphorus (LCK349).

SRF and MFI were evaluated in series using two types of filter cut-off (8 μm, Wathman paper filter grade 40 for SRF and 0.45 μm, Wathman mixed acetate-cellulose ME25 for MFI) adopting the procedures of Christensen *et al.* (1993) and Schippers & Verdouw (1980), respectively. Bulk liquid separation and EPS extraction were performed according to the thermal method (Le-Clech *et al.* 2006). An aerobic mixed liquor sample (80 mL) was centrifuged (ALC4235, 2,600 g, 10 min) and filtered (Wathman ME25, 0.45 μm) to collect the bulk liquid, containing residual substrate and SMP. With regards to the extracted EPS (eEPS), the pellet was resuspended with deionized water and heated at 80 °C for 10 min. Then, the sample was cooled to ambient temperature, centrifuged (ALC4235, 2,600 g, 10 min) and filtered (Wathman ME25, 0.45 μm). Finally, eEPS and SMP solutions were characterized by the content of proteins and carbohydrates adopting the Lowry *et al.* (1951) method (TP0300, Sigma-Aldrich, Bovine Serum Albumin as standard for method calibration) and the Dubois *et al.* (1956) method (D-glucose monohydrate as standard).

Membrane fouling analysis

The membrane filtration process can be described according to Darcy's law, considering the correction to 20 °C to account for the dependence of permeate viscosity μ_T on temperature, as defined by Rosenberger *et al.* (2006):

$$J = \frac{\mathrm{TMP}}{\mu_T \cdot R} = \frac{\mathrm{TMP}}{(\mu_{20} \cdot e^{-0.0239 \cdot (T-20)}) \cdot R} \quad (1)$$

where J is the permeation flux (m s^{-1}), TMP the trans-membrane pressure (Pa), μ_{20} the viscosity of permeate at 20 °C (Pa s), T the temperature (°C) and R the total filtration resistance (m^{-1}). According to the resistances in series model, it is assumed that R is the sum of three terms (Bae & Tak 2005):

$$R = R_m + R_c + R_f \quad (2)$$

where R_m: intrinsic membrane resistance, R_c: resistance by cake layer on the membrane surface, a sort of a porous media composed by sludge flocs and colloids giving a complex system of interconnected interparticle voids, R_f: fouling resistance caused by irreversible adsorption of foulants on membrane pore wall or surface.

The cake layer resistance R_c refers to reversible fouling that can be removed by physical means such as RX and backflushing. Contrarily, R_f represents the irreversible

fouling that can only be removed by chemical cleaning. The three resistance terms can be obtained from the total filtration resistance adopting the method of Bae & Tak (2005):

$$R_m = \frac{TMP_w}{\mu_T \cdot J_w} \quad (3)$$

$$R_f = \frac{TMP_w'}{\mu_T \cdot J_w'} - R_m \quad (4)$$

$$R_c = \frac{TMP_{AS}}{\mu_T \cdot J_{AS}} - R_m - R_f \quad (5)$$

where J_{AS} and TMP_{AS} are flux and TMP observed during the filtration of activated sludge, whereas J_w, TMP_W and J_w', TMP_W' are related to filtration tests with fresh water using new membranes ($R_m = 4.64 \times 10^{11}\ m^{-1}$) and physically/chemically cleaned membranes, respectively.

RESULTS AND DISCUSSION

Determination of the optimal PACl dose

Four different kinds of indexes were taken into consideration during the jar-test campaign, the main results of which are shown in Figure 1, where the variations for SRF, MFI and low density suspended solids (LDSS) and the removal rates for SMP, bulk COD and bulk colour with respect to raw sludge samples were reported. In particular, bulk COD and colour were taken into account as indexes of effluent quality. SRF and MFI are indexes of sludge filterability: SRF is related to the cake formation on the filter during the test, whereas MFI provides information about the effects of little particles and colloids forming a sort of gel structure on the filter. Suspended solids measured on the supernatant after sludge centrifugation represent low density suspended material (LDSS) in sludge and they can be considered as an index of sludge flocculation ability. For instance, a reduction in LDSS implies an improvement in sludge flocculation. SMP can be considered as an index of fouling potential as such molecules are believed to be one of the main causes of fouling (e.g. Le-Clech *et al.* 2006; Rosenberger *et al.* 2006).

With regards to indexes of effluent quality, PACl showed high removal rates even at low doses for bulk COD (49–60.8%). Removal of colour showed a monotonically increasing trend from above 55% reaching a maximum removal of approximately 80% at 25 mg $gMLSS^{-1}$. With regards to mixed liquor filterability and flocculation, data showed a significant effect for a dose of 12.5 mg $gMLSS^{-1}$, giving a variation of −35.4% for SRF and of −53.5% for LDSS, respectively. Differently, MFI offered an excellent result at 6.25 mg $gMLSS^{-1}$ (−73.0%). Then, for all these parameters, relative variation increased and reached a maximum value between −80 and −90% at a dose of 50 mg $gMLSS^{-1}$. Fouling indexes showed interesting removal rates even at the lowest dose tested (6.25 mg $gMLSS^{-1}$): SMPp and SMPc removal rates were 35.9 and 31.8%, respectively. SMPp removal rates remained almost constant increasing PACl

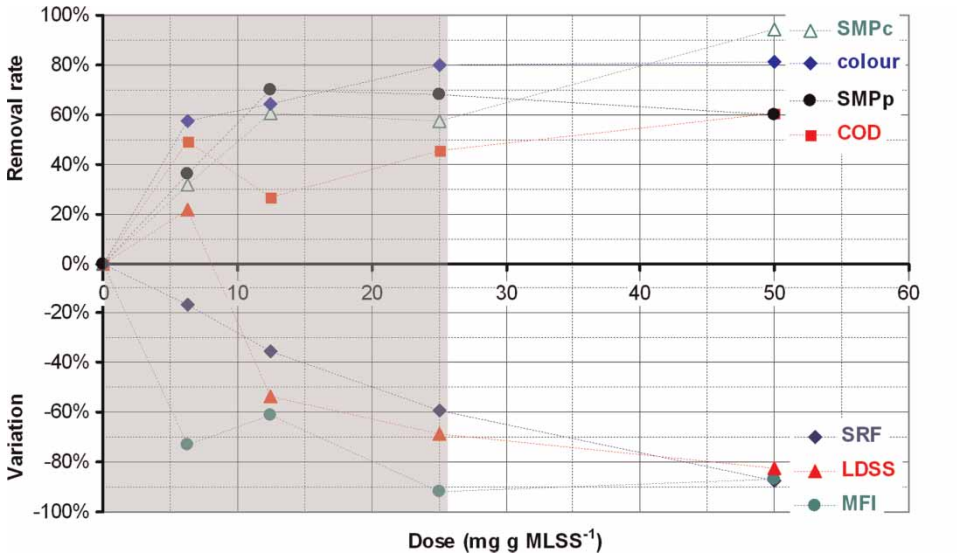

Figure 1 | Evaluation of effectiveness of PACl at different dosages (as average values). The grey area indicates doses where sludge pH was not affected by the addition of PACl.

dose, while for SMPc a rise to a maximum value of 94.2% was observed at 50 mg gMLSS^{-1}.

The optimal dose was fixed on the basis of the improvement of all the above mentioned indexes at 12.5 mg gMLSS^{-1}, corresponding to a PACl dosage of 0.4 g d^{-1} to compensate for the losses due to the excess sludge removal.

Fouling control by PACl dosing at pilot scale

Figure 2 shows the TMP_{20} (i.e. the equivalent TMP at 20 °C) evolution during step 1, in which two different periods can be noted. The first period (day 0 ÷ day 160) is characterized by the occurrence of the 'TMP jump' (Cho & Fane 2002) with a maximum TMP_{20} value of approximately 0.210 bar. Differently, highly irregular TMP_{20} variations were registered in the second period (day 160 ÷ day 225) showing a series of peaks with values between 0.100 and 0.200 bar and rapid decreases to TMP_{20} values lower than 0.050 bar. At day 160, the TMP jump was stopped due to the switching off of the process pump for some hours, caused by a failure. Membrane aeration was, however, maintained in operation resulting in a sort of extended physical cleaning. When the pump was switched on, TMP_{20} was completely recovered suggesting that the occurrence of TMP jump was related to reversible fouling. Comparing TMP_{20} data in step 1 and step 2, it can be noted that PACl offered a more stable filtration process, providing a significant fouling control. In fact, TMP_{20} values in step 2 were considerably lower than those in step 1 having also a low variability over mean values: 0.057 ± 0.032 bar and 0.036 ± 0.008 bar for step 1 and step 2, respectively.

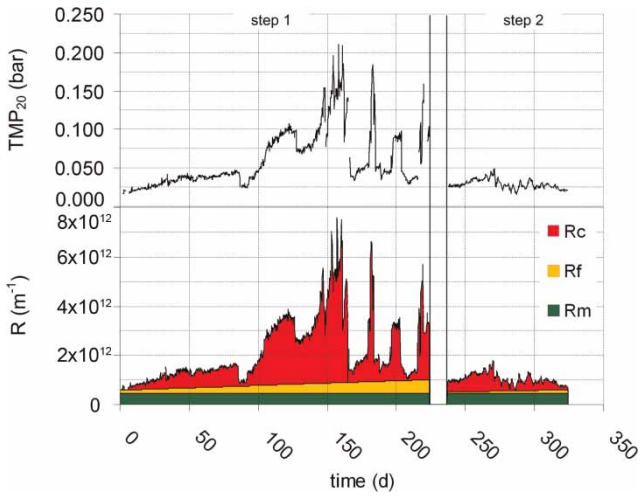

Figure 2 | Evolution along time of equivalent TMP at 20 °C (TMP_{20}) and fouling resistances (R_m, R_f, R_c).

The membrane fouling analysis was employed, calculating the resistance terms using Equations (3)–(5). Firstly, R_f was determined at the beginning and at the end of each step, after cleanings. After that, the average irreversible fouling rate (FR, m^{-1} d^{-1}) was introduced and evaluated as the variation of R_f within each step over its duration. Then, in order to calculate R_c it was assumed a linear trend of R_f with a constant fouling rate equal to FR and Equation (5) was applied for all the experimental period resulting in a complete membrane fouling analysis, as shown in Figure 2. The progressive increasing tendency of R_c values in the first 80 days, followed by a rapid rise leading to the TMP jump, can be due to changes in sludge characteristics giving a particularly adhesive cake layer.

However, the reversible nature of the cake layer was confirmed at day 160 when the extended physical cleaning permitted recovery of the TMP_{20} and R_c values. It is believed that the adhesive nature of the cake layer was also the reason for the series of rapid rises and decreases in TMP_{20} and R_c from day 160 onwards. In particular, the effectiveness of membrane scouring by aeration was probably related to the cake-layer thickness: as soon as the thickness increased, resistance values rose as well, and at the same time membrane scouring began to be more effective causing the sudden detachment of the cake with a rapid decline in TMP_{20} and R_c values. However, such a behaviour was not observed during step 2, when PACl was added. Probably, PACl altered the adhesive nature of the cake controlling R_c properly and showed a significant reduction from average resistance values of 13.84×10^{11} m^{-1} (step 1) to 4.79×10^{11} m^{-1} (step 2) corresponding to a relative variation of −65.4%. Moreover, as for irreversible fouling, PACl offered a reduction of FR, from 1.90×10^{9} to 1.04×10^{9} m^{-1} d^{-1} (−45.3%).

With regards to extracellular biopolymers (including both EPS and SMP) a significant reduction in SMP concentration would have been expected from jar-tests, but no significant variation was observed: SMPp varied from 12.1 ± 1.56 mg L^{-1} (step 1) to 12.4 ± 4.82 mg L^{-1} (step 2) and SMPc from 6.1 ± 2.43 mg L^{-1} (step 1) to 6.6 ± 1.18 mg L^{-1} (step 2). According to Iversen *et al.* (2009a) this might be due to the different conditions in the pilot plant, wastewater compounds and additional shear stress. Moreover, also the dosing mode (the spike addition in jar-tests vs the continuous dosage in the MBR) could be a relevant factor, underling the importance of studying flux enhancers under real operation conditions. Differently, variations in eEPS concentration were consistent: eEPSp varied from 41.8 ± 9.77 mg gMLVSS^{-1} (step 1) to 30.6 ± 1.3 mg gMLVSS^{-1} (step 2) and eEPSc from 3.0 ± 0.79 mg

Table 2 | Average (and standard deviation) permeate quality and removal rates in step 1 and step 2

	Permeate quality[a]		Removal rate (%)		
Parameter	**Step 1**	**Step 2**	**Step 1**	**Step 2**	**Δ**
COD	35.9 ± 13.32	23.6 ± 11.91	81 ± 8.3	86 ± 8.2	+5
Total nitrogen	7.2 ± 2.88	5.5 ± 1.09	81 ± 9.8	80 ± 6.2	−1
Total phosphorus	2.3 ± 0.74	0.1 ± 0.08	24 ± 38.5	88 ± 14.0	+64
Colour (abs. 426 nm)	0.039 ± 0.0095	0.026 ± 0.0071	68 ± 13.8	84 ± 6.2	+16
Non-ionic surfactants	0.35 ± 0.253	0.24 ± 0.103	94 ± 5.1	93 ± 3.2	−1
Anionic surfactants	0.80 ± 0.678	0.27 ± 0.111	79 ± 13.4	86 ± 13.1	+7

[a]All data related to permeate quality are expressed in mg L^{-1} except for colour (abs. at 426 nm, cm^{-1}).

$gMLVSS^{-1}$ (step 1) to 1.4 ± 0.59 mg $gMLVSS^{-1}$ (step 2). Jiang *et al*. (2005) suggested that a decrease in EPS levels may cause floc deterioration with a negative repercussion on cake resistance. Differently, Ahmed *et al*. (2007) observed that the specific cake resistance increased with negative effect on TMP values as EPS concentration rose. Possibly, the effects of EPS on fouling depend on the specific situation. In this study, low eEPS concentrations were observed during PACl dosage in parallel with low R_c values and this was likely the cause of the low adhesive nature of the cake layer in step 2 as discussed above.

Permeate quality enhancement by PACl dosing at pilot scale

Permeate concentration values were lower in step 2 than in step 1 due to the combined effect of the PACl and lower influent concentrations (see Table 1). As a consequence, to understand the effect of PACl, removal rates have to be considered as well (Table 2).

With regards to conventional macro-parameters, a relevant improvement of removal rates was observed for total phosphorus (+64%) with a variation of permeate concentration from 2.3 to 0.1 mg L^{-1}. This was related to precipitation of insoluble phosphorus salts and to the retention in the MBR. Limited or null improvements were observed for COD and nitrogen removal rates (+5 and −1%, respectively) however, showing that PACl did not lead to any inhibition of the biological process as reported by some authors (e.g. Iversen *et al*. 2009b). With regards to nitrogen apportionment, organic nitrogen, ammonium and nitrate accounted approximately for 30, 10 and 60% respectively, without significant differences between step 1 and step 2. Nitrite concentration was always negligible (<1%).

With regards to textile macro-parameters, a significant enhancement of colour removal (+16%) was observed and, to a lesser extent, of anionic surfactants (+7%) probably due to ionic interactions as for phosphoros. By comparison, permeate quality showed a variation in colour from 0.039 to 0.026 cm^{-1} and, as for anionic surfactants, from 0.80 to 0.27 mg L^{-1}. No improvements of removal rates related to non-ionic surfactants were detected (−1%) even if a noteworthy difference in permeate concentration resulted between step 1 and step 2 with values of 0.35 and 0.24 mg L^{-1}, respectively. Even if higher removal rates would have been expected from jar tests as previously discussed for SMP (see above), the effects of PACl on textile macro-pollutants (i.e. colour and surfactants) were significant.

CONCLUSIONS

The outcomes of the study suggest that the MBR process combined with PACl is an effective and promising technology for the treatment of textile wastewater. In fact, this study showed interesting effects of PACl on fouling control (−65.4 and −45.3% for R_c and FR, respectively) and permeate quality enhancement (+64, +16 and +7% for total phosphorus, colour and anionic surfactants, respectively), even if some improvements found during the execution of the jar-test campaign were higher. However, this underlines the importance of studying flux enhancers under real operating conditions and in long-term trials such as the present work.

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