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Comparison of alternative additives used for the mitigation of membrane fouling in membrane bioreactors

S. Malamis*, A. Andreadakis, D. Mamais, C. Noutsopoulos

Sanitary Engineering Laboratory, School of Civil Engineering, National Technical University of Athens,
5 Iroon Polytechniou St., Zographou Campus, Athens 15780, Greece
Tel. +30 210 772 2897; Fax: + 30 210 772 28 99; email: malamis.simos@gmail.com

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ABSTRACT

This work examined the impact of three natural minerals (zeolite, bentonite and perlite), three coagulants [ferric chloride, alum and polymeric aluminium chloride (PACl)] and a cationic polymer (MPE50) on the reduction in membrane fouling in membrane bioreactors (MBRs). The experiments were conducted using an ultrafiltration (UF) membrane module that was submerged in a reactor. Mixed liquor taken from an MBR was spiked with specific additive concentrations and was filtered through the UF membranes. Membrane fouling was assessed by determining the increase in the normalized membrane permeability L_{60}/L_0 when the additive was added compared to the L_{60}/L_0 obtained when sludge was filtered without any additive (i.e. control experiment). The use of additives resulted in a net increase in membrane permeability compared to the control experiment. Fouling mitigation followed the order $\text{MPE50} > \text{PACl} > \text{FeCl}_3 > \text{Al}_2(\text{SO}_4)_3 > \text{zeolite} > \text{bentonite}$, with perlite increasing fouling. The addition of 400 mg/L MPE50 resulted in an increase in L_{60}/L_0 by 229%, while the addition of 200 mg/L PACl resulted in a respective increase by 158%. The economic evaluation showed that a more feasible solution was the use of 200 mg/L PACl, which incurs a cost of 0.601 €cents/m^3 treated wastewater. Additive use also resulted in a significant decrease in the colloidal substances in sludge which was in accordance with the fouling reduction.

Keywords: Membrane bioreactors; Fouling mitigation; Coagulants; Minerals; Cationic polymer

1. Introduction

Membrane bioreactors (MBRs) are successfully employed for the biological treatment of municipal wastewater. Their advantages include high effluent quality, limited footprint, ability to operate at high solids retention times (SRT) and high mixed liquor suspended solids (MLSS) with no operational problems. Furthermore, problems associated with poor sludge settling are not encountered [1]. Membrane fouling is the main operational problem of MBR systems, which limits their wider adoption [2–3]. Several measures are usually undertaken to limit fouling, including coarse bubble aeration, cyclic permeate backwash, manipulation of the operating conditions and chemical cleaning of the membranes [4]. The use of conditioners to reduce fouling is usually not employed in full-scale MBRs and may be a viable alternative. These conditioners alter the properties of sludge, enhancing its filterability.

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*Corresponding author.

Three types of additives have been employed to reduce fouling in MBR systems: (a) coagulants, (b) adsorbents and (c) cationic polymers. The effect of ferric chloride ($FeCl_3$), alum [$Al_2(SO_4)_3$], polymeric aluminium chloride (PACl) and polymeric ferric sulphate (PFS) on membrane fouling has been examined [5–9]. Effective flocculation of activated sludge with the addition of coagulants results in the reduction in small flocs which are scattered inside the biological reactor. These flocs deteriorate the system's filtration performance, while larger flocs can be more easily removed when attached to the membrane surface through coarse bubble aeration. Coagulants introduce positive charges, neutralizing the negative charges of biomass, thus enhancing flocculation [5]. Zhang et al. [9] found that the addition of 67 mg/L Fe(III) increased sludge filterability and reduced the concentration of soluble microbial products (SMP). Fan et al. [6] concluded that the use of ferric chloride, alum and a cationic polymer significantly reduced transmembrane pressure (TMP). Holbrook et al. [10] found that the use of alum reduced the carbohydrate SMP concentration and thus mitigated fouling. Wu et al. [5] investigated the effect of two monomeric [$FeCl_3$ and $Al_2(SO_4)_3$] and three polymeric coagulants [PACl, PFS and polymeric aluminium ferric chloride]. The authors observed that the polymeric coagulants resulted in higher fouling reduction. Zeolite and activated carbon are adsorbents which have been employed for the reduction in fouling in MBRs [11,12]. Ng et al. [13] found that the addition of 1 g/L powdered activated carbon (PAC) minimized the specific resistance of the cake layer formed on the membrane surface, while the addition of 5 g/L PAC reduced irreversible fouling. Lesage et al. [14] observed that the addition of PAC accomplished significant fouling reduction. Li et al. [15] operated two parallel MBR systems and observed that the addition of 1.2 g/L PAC into one of the two reactors reduced fouling by 44% compared to the other MBR. Ng et al. [16] did not observe fouling mitigation with the addition of 5 g/L PAC in an MBR with no sludge wasting; this was attributed to the saturation of PAC. Cationic polymers have also been employed for the reduction in membrane fouling. Koseoglu et al. [17] examined the performance of three different cationic polymers (MPE50, MLP30 and KD452) and two coagulants ($FeCl_3$ and PACl) with respect to fouling reduction. The authors found that cationic polymers could achieve higher critical flux than the coagulants, with MPE50 resulting in the highest critical flux. Yoon and Collins [18] examined the impact of MPE50 in a full-scale MBR system having a flow capacity of 2,500 m^3/d and found that the addition of 400 mg/L MPE50 into the mixed liquor, resulted in attaining a permeate flux that was 35% greater than the critical flux for 1 day, while this was possible only for 2–3 h when MPE50 was not added.

In the literature, most works have focused on the effect of limited number of additives. The direct comparison among different research works of the most effective additive is not possible since each work was conducted under different experimental conditions. The aim of this work was to investigate and compare the effect of three natural minerals, three coagulants and a cationic polymer on the mitigation of membrane fouling and on organic colloidal matter reduction under the same experimental conditions. The comparative assessment of these additives is important under controlled experimental conditions in order to be able to conclude which one results in the highest fouling reduction.

2. Materials and methods

2.1. Additives employed

Zeolite, bentonite and perlite are low-cost minerals having high adsorption capacity. Natural zeolite and bentonite were supplied by S&B Industrial Minerals S.A., while perlite was supplied by Mathios Refractories S.A. The minerals were washed with deionized water and dried at 80°C for at least 24 h. Then, they were stored in desiccators until their use. The minerals were used in powder form (<0.18 mm), in their natural state without any chemical or other thermal pre-treatment. Ferric chloride and alum were supplied by Merck and were of analytical grade. The cationic polymer MPE50 was supplied by Nalco Hellas S.A., and high alkalinity PACl was supplied by Feri-Tri S.A. ($Al_n(OH)_mCl_{(3n-m)}$). The stock solutions of the chemical additives were prepared in deionized water just before the filtration experiments.

2.2. Experimental set-up

The filtration experiments were conducted in a cylindrical, plexiglas reactor having a total volume of 6.5 L. The ultrafiltration (UF) membrane module (ZeeWeed 1) was supplied by GE Water and Process Technologies and consisted of hollow fibres made of polyvinylidene fluoride, having a nominal pore size of 0.04 μm and a surface area of 0.047 m^2. The UF module was immersed inside the reactor. Mixed liquor was collected from the aerobic reactor of an MBR treating municipal wastewater, operating under steady-state conditions (SRT = 15 days) and was transferred to the laboratory where it was immediately used for the filtration experiments. The mixed liquor characteristics are given in Table 1a.

Fixed concentrations of the additives were added in the mixed liquor (Table 1b). This was conducted by weighting the required mineral mass and by appropriate dilutions of the chemical concentrations. Once the required concentration was added, the mixed liquor was agitated for 1 h at 800 rpm using a Heidolph RZR 2041 mechanical stirrer. A blank experiment was also conducted each time in which additives were not added into the mixed liquor. The mixed liquor was transferred to the UF reactor and filtration took place at constant TMP of 0.3 bar for 60 min. Coarse bubble aeration (5 L/min) was supplied to the membrane module to minimize fouling, and fine bubble aeration (5 L/min) was also supplied to maintain the mixed liquor under suspension. The mixed liquor pH was maintained at 7.5 ± 0.3. The duration of each filtration experiment was 60 min since within this time the permeate flux reduction seemed to stabilize. During filtration, permeate was returned back into the reactor to maintain relatively stable the initial MLSS concentration. The filtration experiments were conducted at room temperature (i.e. 20 ± 1°C). After each filtration experiment, the membrane module was chemically cleaned by placing it in NaOCl solution (1,000 mg/L Cl_2) for 8 h and afterwards in 4,000 mg/L citric acid solution for 4 h. Through the measurement of the permeate flux and permeability, it was possible to assess both the initial fouling of the membrane caused during the first 5–15 min and the subsequent fouling.

The permeate flux was determined by measuring the mass of the permeate per unit time. Membrane permeability was determined during the 60-min filtration experiment as the ratio of the permeate flux to the TMP. The membrane permeability at the end of the filtration experiment, normalized to the clean membrane permeability, is given by L_{60}/L_0 where L_{60} [$L/(m^2\,h\,bar)$] is the membrane permeability at the completion of the 60 min filtration experiment and L_0 [$(L/(m^2\,h\,bar))$] is the clean membrane permeability. The latter was determined in water at 20 ± 1°C, at a constant TMP of 0.3 bar after the membrane was chemically cleaned in order to restore its permeability. The use of additives resulted in a different value of L_{60}/L_0 compared to that obtained when sludge was filtered without any additive (i.e. control experiment). Thus, the percent difference of L_{60}/L_0 obtained with the use of additives minus the value of L_{60}/L_0 without any additive use provides the "net" membrane permeability increase. The impact of additives on the organic colloidal matter concentration of the mixed liquor was also assessed. Colloidal chemical oxygen demand (COD) was measured as the difference between the COD of the filtrate passing through GF/C Whatman filters with pore size 1.2 μm minus the COD of the permeate passing through UF membranes with pore size 0.04 μm.

Table 1a
Initial characteristics of mixed liquor

Parameter	Average value	Variation
pH	7.17	6.82–7.44
TS (g/L)	6.02	5.62–6.51
MLSS (g/L)	5.11	4.78–5.47
MLVSS (g/L)	4.23	3.77–4.50
COD (g/gTS)	1.27	1.16–1.38
N (mg/gTS)	64	55–76

2.3. Analytical methods

COD in liquid samples was determined using the Spectroquant Merck test kits and the Nova 60 Spectroquant photometer (Merck). TS, MLSS, mixed liquor volatile suspended solids (MLVSS) COD, N and pH in activated sludge samples were determined using standard methods [19].

3. Results and discussion

3.1. Minerals

The minerals that were employed (zeolite, bentonite and perlite) are characterized by significant adsorption capacity. The colloidal and soluble organics of the mixed liquor can be adsorbed on the mineral sorption sites, thus reducing the amount of free organics that may foul the membrane. The gradual increase in

Table 1b
Additives and concentrations employed

Additives	Concentration (mg/L)
Zeolite	2,500, 5,000, 10,000, 15,000
Bentonite	2,500, 5,000, 10,000, 15,000
Perlite	5,000, 10,000
$Al_2(SO_4)_3$	100, 200, 300, 400 (15.8, 31.5, 47.3, 63.1)[a]
$FeCl_3$	100, 200, 300, 400 (34.4, 68.9, 103.3, 137.7)[b]
PACl	20, 50, 100, 200, 300, 400 (5.1, 12.8, 25.5, 51.1, 76.6, 102.1)[a]
MPE50	50, 100, 200, 300, 400, 500

[a] Concentration given as Al(III).

[b] Concentration given as Fe(III).

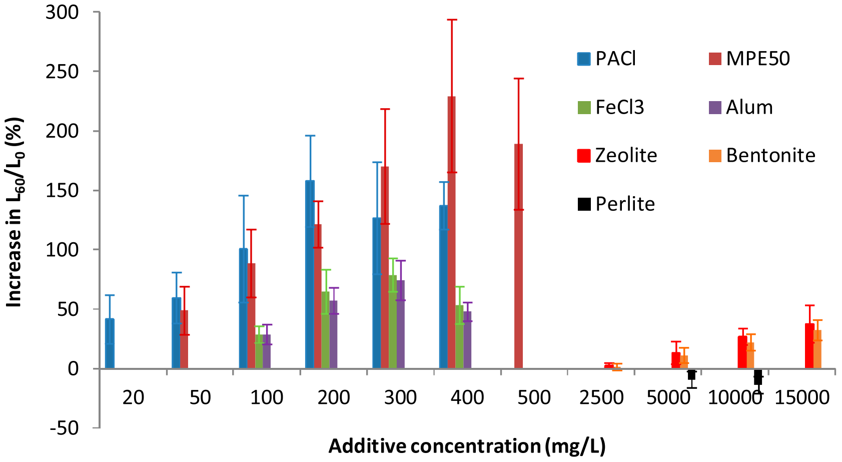

Fig. 1. Membrane permeability increase due to the addition of conditioners into the mixed liquor.

zeolite's concentration resulted in a gradual decrease in fouling since more sites were available for adsorption of colloidal matter, extracellular polymeric substances and other organics of the mixed liquor (Fig. 1). The highest permeability increase was 38% and was obtained for the zeolite concentration of 15 g/L. Bentonite addition was also beneficial with respect to fouling reduction, since the addition of 15 g/L bentonite increased the permeability compared to the control experiment by 33%. Bentonite concentrations lower than 5 g/L had a small effect on fouling mitigation. Zeolite has a more open structure than bentonite which facilitated the adsorption of organic substances at its surface and interior.

Perlite addition in the mixed liquor adversely impacted on membrane fouling. The fouling propensity of perlite was not initially expected since perlite is considered to enhance the filtration performance of several liquid media. However, the visual inspection of the membrane fibres revealed significant clogging by perlite/sludge aggregates between the membrane fibres which probably resulted in a decrease in permeate flux. Therefore, the use of perlite in a suspended form is not recommended.

3.2. Coagulants

Activated sludge flocs are negatively charged and this charge does not allow any further flocculation. Coagulant addition introduces positives charges, neutralizing the negative charge and thus favouring flocculation [5]. The addition of $Al_2(SO_4)_3$ up to 300 mg/L [47.3 mg/L Al(III)] resulted in the increase in membrane permeability on average up to 75%

Table 2
Effect of additive concentration on the COD concentration of colloidal matter

Additive concentration (mg/L)	Colloidal matter COD (mg/L)					
	Zeolite	Bentonite	$Al_2(SO_4)_3$	$FeCl_3$	PACl	MPE50
0	97	103	74	87	98	89
20	–	–	–	–	55	–
50	–	–	–	–	46	75
100	–	–	51	72	33	58
200	–	–	43	55	30	47
300	–	–	39	43	51	41
400	–	–	47	62	53	36
500	–	–	–	–	–	44
2,500	93	97	–	–	–	–
5,000	80	91	–	–	–	–
10,000	72	80	–	–	–	–
15,000	68	81	–	–	–	–

(Fig. 1). However, the further increase in $Al_2(SO_4)_3$ concentration to 400 mg/L resulted in lower fouling reduction. Consequently, the optimum coagulant concentration was found to be 300 mg/L $Al_2(SO_4)_3$. The high coagulant concentration caused biomass to become positively charged, and thus, partial deflocculation was favoured leading to an increase in colloidal matter (Table 2). This observation agrees with the findings of Song et al. [8] who found that the increase in Al(III) from 23.7 to 39.5 mg/L resulted in an increase in the specific cake layer resistance from 6.68×1.0^{18} to $2.23 \times 1.0^{19}\,m^{-1}$ in filtration experiments.

The addition of ferric chloride had similar effects as alum. Specifically, the increase in $FeCl_3$ from 100 [34.4 mg/L Fe(III)] to 300 mg/L [103.3 mg/L Fe(III)] resulted in an increase in membrane permeability on average up to 79%. Higher $FeCl_3$ concentrations (>300 mg/L) resulted in lower performance with respect to fouling reduction as was the case for alum. The optimum dosage was 103.3 mg/L Fe(III) which is higher than 67 mg/L Fe(III) obtained by Zhang et al. [9], but agrees with the results of Fan et al. [6]. Comparing the performance of $FeCl_3$ and $Al_2(SO_4)_3$, it is deduced that higher Fe(III) concentrations are required compared to Al(III) concentrations to achieve similar permeability reduction.

The use of PACl significantly enhanced MBR performance. The increase in PACl concentration from 20 to 200 mg/L increased fouling reduction on average up to 158%, while the further increase in PACl was not as effective in fouling mitigation. The two monomeric and one polymeric coagulants exhibited similar behaviour since fouling reduction was enhanced with increasing coagulant concentration up to a certain concentration, while the further addition of coagulant was not as effective. The polymeric coagulant was more effective than the monomeric ones since it resulted in a higher increase in membrane permeability. PACl could provide more positive charges and larger chain molecules, thus enhancing sludge filterability [6]. Also, the Al(III) concentration required to achieve the highest fouling reduction was comparable for the polymeric coagulant compared to alum. PACl addition also resulted in a high reduction in organic colloidal matter in sludge (Table 2), which can explain the respective decrease in membrane fouling. For example, the addition of 200 mg/L PACl reduced colloidal matter COD from 98 to 30 mg/L. Previous work has shown that colloidal matter is a major contributor to membrane fouling [20].

A problem associated with coagulant addition is that it can reduce the mixed liquor pH to dangerously low values and may cause biomass inhibition. The concentrations of 300 mg/L $FeCl_3$ and 300 mg/L $Al_2(SO_4)_3$ lowered the biomass pH to unacceptably low values (6.03 and 4.35, respectively) and, despite being the optimum concentrations, cannot be employed in an MBR system. Instead the concentrations of 200 mg/L are recommended for use. On the other hand, the use of high alkalinity PACl did not cause such problems.

3.3. Cationic polymer

The use of cationic polymer MPE50 resulted in a significant decrease in membrane fouling. In Fig. 2, a typical filtration experiment is presented with the variation of normalized permeability (L_t/L_0) with time where L_t is the membrane permeability at time t. The blue line shows the reduction in membrane permeability in sludge where the cationic polymer was not added and thus significant fouling occurred. The addition of MPE50 resulted in a remarkable increase in membrane permeability compared to the control experiment. During the first minutes of sludge filtration, the permeate flux obtained was even higher than the flux obtained in clean water. The addition of 400 mg/L resulted in permeability increase of 229% at the end of the filtration experiment compared to the permeability obtained with activated sludge without any additive; this was the highest reduction in fouling obtained among all the examined additives. However, the optimum concentration of cationic polymer required was twice than that of the polymeric coagulant.

3.4. Organic colloidal matter

Table 2 shows the COD of colloidal matter in the mixed liquor for different additive concentrations. Limited reduction in the COD of colloidal matter was observed when minerals where added due to low

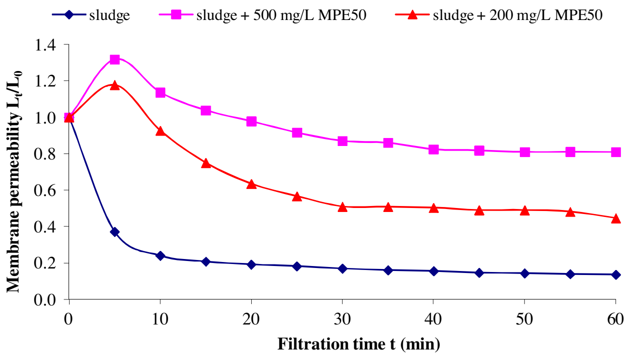

Fig. 2. Variation of membrane permeability with filtration time for 200 mg/L and 500 mg/L MPE50.

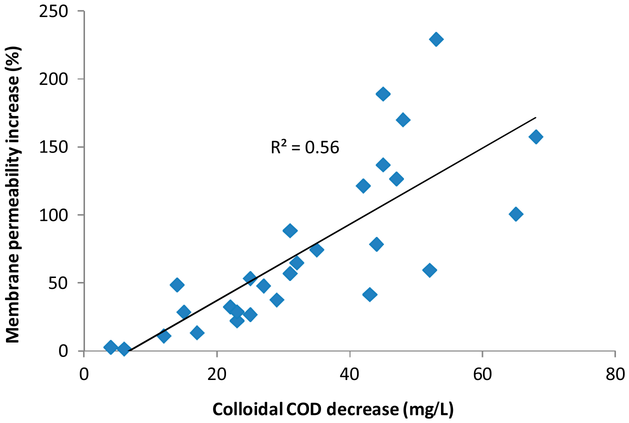

Fig. 3. Membrane permeability increase vs. the decrease in the COD of colloidal matter of the mixed liquor.

sorption of organics. The use of coagulants (particularly PACl) resulted in significant reduction in the COD of colloidal matter in sludge, while the maximum reduction was obtained with the use of the cationic polymer. The flocculation that takes place significantly reduces the concentration of colloids in the mixed liquor. Organic colloidal matter decrease was in accordance with fouling decrease, and the lowest colloidal COD was obtained for the additive dosage exhibiting the lowest membrane fouling.

In Fig. 3, the correlation between membrane permeability increase and colloidal matter COD decrease is given. These data have been gathered from several filtration experiments of the mixed liquor alone (i.e. no additives) and with the use of the additives. The COD decrease in colloidal matter is determined by considering its decrease due to the use of additives compared to its concentration in the control experiment.

Although the coefficient of determination is not very high ($R^2 = 0.56$), it shows that there exists some correlation between colloidal matter COD and membrane fouling. The three data points that are well above the graph resulted from the addition of MPE50 in activated sludge at concentrations of 300–500 mg/L. It is seen that the membrane permeability reduction was much greater than the one anticipated based on colloidal matter COD decrease. It seems that the cationic polymer MPE50 does not mitigate fouling only through the decrease in the colloidal organics in the mixed liquor. MPE50 chemistry forms polymer–biopolymer complexes, and these complexes become part of the floc structure. Also, the three data points that are well below the other points correspond to the addition of 20–100 mg/L PACl. In these cases, the reduction in fouling was lower than that anticipated based on colloidal matter COD reduction.

3.5. Economic assessment of additives

The results showed that fouling mitigation followed the order MPE50 > PACl > $FeCl_3$ > $Al_2(SO_4)_3$ > zeolite > bentonite, while perlite increased fouling. The cationic polymer MPE50 exhibited the highest reduction in membrane fouling, followed by the polymeric coagulant, the monomeric ones and finally the minerals. Minerals are not recommended for use since large quantities are required to achieve limited fouling mitigation. Monomeric coagulants have the disadvantage that the optimum dosage cannot be used as it reduces the biomass pH to very low values.

To evaluate better the effect of additives, it is also important to consider the expenses incurred due to their addition. The cost incurred by the use of additives was estimated for a medium scale MBR wastewater treatment plant having the following characteristics:

(1) $Q_{\text{influent}} = 50{,}000\ \text{m}^3/\text{d}$.
(2) SRT = 15 days.
(3) Hydraulic Retention Time (HRT) = 8 h.

It was also assumed that the entire additive is retained by the activated sludge. In the case of minerals, this follows from the fact that mineral size is much larger than the membrane nominal pore size. In the case of the aluminium-based coagulants, previous work has demonstrated that the chemical concentration in the treated effluent is negligible [21]. The same was assumed for MPE50 and ferric chloride. As a result, from the mass balances, the following equation holds [22]:

$$[\text{Additive}]_{\text{aerobic reactor}} = \text{SRT}/\text{HRT} \times [\text{Additive}]_{\text{wastewater}} \qquad (1)$$

where $[\text{Additive}]_{\text{aerobic reactor}}$ is the additive concentration in the aerobic reactor and $[\text{Additive}]_{\text{wastewater}}$ is the additive concentration in the influent wastewater.

Table 3 shows the cost calculations for the additive types and concentrations that were tested. The use of MPE50 results in significant expenses (1.78 €cents/m^3) owing to the significant cost of the cationic polymer. The best solution is probably the use of 200 mg PACl/L which results in a high decrease in membrane fouling and incurs a cost of 0.601 €cents/m^3.

Table 3
Economic evaluation of the different additives and concentrations employed

Additive	Additive cost (€/ton)	Concentration (mg/L)	Required amount (kg/d)	Daily cost (€/d)	Treatment cost (€cents/m^3)
Zeolite	100 (powder)	2,500	2,778	278	0.556
		5,000	5,556	556	1.111
		10,000	11,111	1,111	2.222
		15,000	16,667	1,667	3.333
Bentonite	150 (powder)	2,500	2,778	417	0.833
		5,000	5,556	833	1.667
		10,000	11,111	1,667	3.333
		15,000	16,667	2,500	5.000
$Al_2(SO_4)_3 18H_2O$	260 (8.1%[a])	15.8[a]	17.52[a]	56	0.113
		31.5[a]	35.05[a]	113	0.225
		47.3[a]	52.57[a]	169	0.338
		63.1[a]	70.09[a]	225	0.450
$FeCl_3$	160 (10% wt[b])	34.4[b]	38.22[b]	61	0.122
		68.9[b]	76.56[b]	123	0.245
		103.3[b]	114.78[b]	184	0.367
		137.7[b]	153.00[b]	245	0.490
PACl	270 (5.1% wt[a])	5.1[a]	5.7[a]	30	0.060
		12.8[a]	14.2[a]	75	0.150
		25.5[a]	28.4[a]	150	0.300
		51.1[a]	56.7[a]	300	0.601
		76.6[a]	85.1[a]	451	0.901
		102.1[a]	113.5[a]	601	1.202
MPE50	2,000	50	55.56	111	0.222
		100	111.11	222	0.444
		200	222.22	444	0.889
		300	333.33	667	1.333
		400	444.44	889	1.778
		500	555.56	1,111	2.222

[a] Concentration or mass are given in Al(III).

[b] Concentration or mass are given in Fe(III).

4. Conclusion

The results showed that the use of additives to reduce membrane fouling is a viable option. Significant fouling mitigation occurred which followed the order MPE50 > PACl > $FeCl_3$ > $Al_2(SO_4)_3$ > zeolite > bentonite, while perlite increased fouling. The cationic polymer MPE50 exhibited the highest reduction in fouling, followed by the polymeric coagulant PACl, the monomeric ones and finally the minerals. However, the economic evaluation showed that the optimal PACl concentration results in much lower treatment cost and high membrane fouling mitigation. Minerals are not recommended since large quantities are required to achieve limited fouling reduction. Additive use also resulted in significant reduction in colloidal organics in sludge. Colloidal matter COD decrease was in accordance with fouling decrease showing its importance as an indirect parameter related to fouling.

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Abbreviations

$Al_2(SO_4)_3$ — alum
COD — chemical oxygen demand
EPS — extracellular polymeric substances
$FeCl_3$ — ferric chloride
HRT — hydraulic retention time
MBR — membrane bioreactor
MLSS — mixed liquor suspended solids
MLVSS — mixed liquor volatile suspended solids
MPE50 — cationic polymer
PAC — powdered activated carbon
PACl — polymeric aluminium chloride
PAFCl — polymeric aluminium ferric chloride
PFS — polymeric ferric sulphate
PVDF — polyvinylidene fluoride
SMP — soluble microbial products
SRT — solids retention time
TMP — transmembrane pressure
TS — total solids
UF — ultrafiltration

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