Pretreatment for seawater desalination by flocculation: performance of modified poly ferric silicate (PFSi-δ) and ferric chloride

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Abstract

Submerged micro-filtration system coupled with in-line flocculation as pretreatment for seawater reverse osmosis (SWRO) has the potential to remove organic compounds and mitigate fouling. The effects and characteristics of flocculation on the performance of micro-filtration (MF) were investigated using two flocculants namely modified poly ferric silicate (PFSi-δ) and ferric chloride ($FeCl_3$). The experimental results were analysed in terms of organic removal efficiency and critical flux. Both PFSi-δ and $FeCl_3$ were found to be applicable to pretreatment because of their capacity of remove organic compounds. The results show that PFSi-δ was better than $FeCl_3$ in terms of removing turbidity and dissolved organic carbon (DOC), particular in removing hydrophilic compounds. The highest DOC removals of 68% and 57% were obtained when PFSi-δ at a dose of 1.2 mgFe^{3+}/L and $FeCl_3$ at 3 mgFe^{3+}/L were used respectively. It is observed that PFSi-δ (1.2 mgFe^{3+}/L) and $FeCl_3$ (3 mgFe^{3+}/L) led to an increase of critical flux from 20 L/m^2.h to 35 L/m^2.h and 55 L/m^2.h respectively.

Keywords: ferric chloride; flocculation; modified poly ferric silicate; pretreatment; seawater

1. Introduction

The supply and sanitation of water will face enormous challenges over the coming decades where the water industry will need to meet the fast growing global requirements. Seawater desalination has become a promising technology to meet the ever increasing water demands. Compared to other desalination technologies such as thermal distillation, seawater reverse osmosis (SWRO) desalination was chosen as the most appropriate technique since RO has demonstrated cost-effectiveness progress for production of drinking water [1]. However, one of major challenges for SWRO system is the membrane fouling. To reduce the fouling consisting of organic, colloidal and bio-fouling in SWRO, pretreatment for raw seawater is required together with the application of proper operating conditions that controls and limits fouling since SWRO strongly depends on the variation in raw seawater [2].

Development in pretreatment technologies is led by micro-filtration and ultra-filtration processes using low-pressure membranes and it has offered significant benefits including higher flux and permeates quality, and economical operating conditions for RO plant [3]. Different membrane configurations have been evaluated by many studies on pretreatment of seawater. It has recently focused on the submerged hollow fibre membrane system to produce higher permeate quantity with lower energy cost [4].

Membrane filtration coupled with physico-chemical processes, such as flocculation, adsorption and ion-exchange, can improve the quality of feed water to RO process. In particular, membrane system with the aid of coagulation in one reactor, which is called in-line flocculation combined with submerged membrane system (IFSMS), is an effective way to reduce installation costs. Flocculation also can remove organic matter in seawater which cause membrane fouling and improve membrane performance by modifying the deposit on the membrane [5-8]. Lee et al. (2009) [9] suggested that the hybrid filtration with in-line flocculation of the influent seawater improved the DOC removal and could effectively reduce the fouling potential of membranes. In-line flocculation with dual media filtration also led to better removal of seawater organic matter [10].

Due to the potential biologic toxicity produced by residual Al in the effluent, Fe-polysilicate coagulant has been attractive in the field of water treatment. A new kind of iron-based inorganic polymer, poly-ferric-silicate (PFSi) coagulant, which is a complex compound of positively charged Fe-coagulant and negatively charged polysilicic acid flocculant, is being studied lately [11-13].

The aim of this study is to evaluate the performance of modified PFSi (PFSi-δ) as a new flocculant in pretreatment for seawater desalination. The results will be compared with those of ferric chloride ($FeCl_3$) used as conventional flocculant for seawater pretreatment. The characteristics of the flocs formed by two flocculants were also examined.

2. Materials and methods

2.1 Materials

2.1.1 Seawater

Seawater from Chowder Bay, Sydney, Australia was used in this study. Seawater was pumped from 1 m below the sea surface level and filtered using a centrifuge filtration system to remove the large particles. The pH value, salinity, total suspended solid (TSS) and turbidity of the seawater used in this study were 8, 35.5~40 g/L, 2~13 mg/L and 0.41~1.7 NTU respectively. Dissolved organic carbon (DOC) which is an indicator of organic matter was 1.29~1.45 mg/L.

2.1.2 Flocculants

Ferric chloride ($FeCl_3$) was selected as a conventional flocculant and used as a stock solution (Fe^{3+} = 1,000mg/L) due to its high effectiveness in DOC removal and low molecular weight organic matter through a complexation mechanism [8]. $FeCl_3$ was used in 5 different doses from 0.5mg Fe^{3+}/L to 4.0mg Fe^{3+}/L.

The procedure of the preparation of modified poly ferric silicate (PFSi-δ) consisted of three processes: (i) preparation of polysalicic acid (PSiA) (ii) preparation of poly ferric silicate (PFSi) and (iii) modification of PFSi (PFSi-δ).

Firstly, PSiA was prepared by acidification of sodium silicate (Na_2SiO_3) containing 26.6% SiO_2. Diluted hydrochloric acid of 20% was poured slowly into Na_2SiO_3 solution while the mixture was stirred with magnetic stirring apparatus at room temperature (25°C). In order to obtain good polymerisation of silicate, diluted hydrochloric acid was added until the pH decrease to 1.8.

To obtain PFSi, 3.1M solution of $FeCl_3$ was then mixed rapidly with the PSiA solution at 40-60^0C. In this study, the ratio of Fe to Si was kept at 1. The PFSi was produced in brown-yellow gel-type solution.

Finally, PFSi was modified to become PFSi-δ by dilution with deionised water 40 times in order to decrease the iron content in final solution.

2.2 Experimental

2.2.1 Flocculation test

PFSi-δ and $FeCl_3$ were used as flocculants. Standard jar tests were carried out to determine the optimum dosage and to investigate the characteristics of flocs and DOC removal capacities of both kinds of flocculants. The experiments were performed in 1 L beakers by adding a predetermined amount of flocculants. The pH was considered at natural condition to prove the pH range to make good flocculation after addition of flocculants. The seawater after adding flocculants was then stirred rapidly for 2 minutes at 120 rpm, followed by 20 minutes of slow mixing at 30 rpm, and 30 minutes of settling. To examine the effect of flocculants, samples were collected immediately after rapid mixing and after settlement. The supernatant was taken and analysed for DOC to determine the ability of flocculants to remove organic matter. The flocs also were taken and measured for the sizes, charges, the amounts as well as the organic contents.

2.2.2 In-line flocculation coupled with hollow fibre micro-filtration

The schematic diagram of the in-line flocculation coupled with hollow fibre MF experiments used in this study is represented in Fig. 1. In-line flocculation by PFSi-δ or $FeCl_3$ and MF experiments were carried out using hollow fibre MF in a column-type reactor. The effective volume of the reactor was 6 L, the total membrane area was 0.1 m^2 and air was injected from the bottom of reactor at a predetermined aeration rate of 1 L/min. Permeate was pumped out using a peristaltic pump at constant flux. After each experiment, membrane cleaning was carried out with chemicals. The membrane was placed into 0.2% NaOH and 0.2% NaOCl solution using a horizontal shaker at 60 rpm for 3h.

Fig. 1. Schematic diagram of in-line flocculation coupled with hollow fibre micro-filtration

After flocculation, the flocs formed were analysed to measure the particle size and charges by the Malvern Zetasizer 3000. It was utilised to measure the zeta potential of particles in the bulk solution after initial rapid mixing in a short time and the supernatant after settlement and the particle size as z-average. It took 2 minutes of gentle stabilisation and the data were recorded automatically.

After initial rapid mixing, flocs were sampled directly and analysed immediately, and after settlement, fine particles remaining in supernatant were sampled carefully. The flocs samples were analyzed without dilution or other treatments.

2.3 Analyses

2.3.1 Characterisation of flocculation flocs

After flocculation, the flocs formed were analysed to measure the particle size and charges by the Malvern Zetasizer 3000. It was utilised to measure the zeta potential of particles in the bulk solution after initial rapid mixing in a short time and the supernatant after settlement and the particle size as z-average. It took 2 minutes of gentle stabilisation and the data were recorded automatically.

After initial rapid mixing, flocs were sampled directly and analysed immediately, and after settlement, fine particles remaining in supernatant were sampled carefully. The flocs samples

were analyzed without dilution or other treatments. Mixed liquid suspended solids (MLSS) and volatile suspended solids (VSS) of flocs samples were analysed in the laboratory and all analytical procedures complied with the standard methods of American Public Health Association; American Water Works Association and Water Environment Federation [14].

2.3.2 Dissolved Organic Carbon (DOC)

DOC was measured after filtering the samples through a 0.45 μm filter. The organic fractions were measured by DOC-LABOR Liquid Chromatography - Organic Carbon Detector (LCOCD). This system uses size-exclusion chromatography to separate classes of dissolved organic materials (organic acids, bases and neutral species) before measuring by a catalysed UV oxidation. LC-OCD was measured to identify the different classes of organic compounds present in seawater that cause organic fouling. It gives the both qualitative and quantitative detailed information of the organic matter present in seawater before and after treatment.

3. Results and discussion

3.1 Flocculation test

3.1.1 Flocculation performance of Ferric chloride ($FeCl_3$)

It can be seen from Table 1 that the final pH of seawater after flocculation slightly decreased with an increase of $FeCl_3$ dosage from 0.5 mg/L to 4.0 mg/L (in terms of Fe^{+3}). However, the pH values of all samples were still in the range of the coagulation zone of 7-8. The observation from the jar test also shows that there were no visible flocs when the concentration of FeCl3 was below 1 mg/L. However, at the FeCl3 concentration lower than 1 mg/L, about 50% of organic matter had been removed. The reason for this phenomenon was that at low concentration of FeCl3, the flocs size could be small enough to be detectable by human eyes, but could be high enough to be separated by micro-filtration. The growth of flocs size was observed as ferric concentration increased and flocs settled down completely at ferric concentration of 3.0 mg/L.

Table 1. The changes of turbidity, pH, DOC fractions and removal efficiencies of seawater by flocculation with $FeCl_3$

The addition of ferric chloride may generate ferric hydroxide and ferric oxide which caused to the increase the turbidity after coagulation [15]. The increase of turbidity was presented in all experimental $FeCl_3$ doses. Thus, optimum dose of $FeCl_3$ cannot be determined in terms of turbidity. Chinu et al. (2010) [9] showed similar trend of turbidity of initial seawater varied from 0.5 to 0.7NTU and turbidity of the feed water increased to 1.31 NTU after the addition of $FeCl_3$.

The analyses of organic fractions of seawater by LC-OCD show that the DOC of seawater was 1.29 mg/L in which 33% of total DOC was hydrophobic. The majority of organic in hydrophilic part was high molecular weight (MW) fractions in which humic substances (MW about 1000 Da) and bio-polymers (MW > 20,000 Da) were 34% and 10% of total organic matter respectively. The neutrals with the lowest MW (below 350 Da) only accounted for 7%. It can be seen from Table 1 that more than 57% of DOC was removed at ferric concentration of 3.0 mg/L. At lower doses, the DOC removal efficiencies were below 50%. When the concentration of ferric was increased to 4.0 mg/L, the DOC removal efficiency started decreasing due to the re-stabilisation of the colloidal particles.

Detailed analyses of organic fractions after flocculation show that flocculation by $FeCl_3$ could remove a majority of hydrophobic fraction. However flocculation was not effective in removing hydrophilic, except neutrals part with low molecular weight and bio-polymer with high molecular weight (Table 1). Chinu et al. [7] and Shon et al. [16] observed that flocculation with 1 mg/L $FeCl_3$ removed the majority of large MW (1510 - 1180 Daltons) representing polysaccharides, proteins, and fulvic acid. However, flocculation could not remove the small range MW (530 - 130 Daltons).

3.1.2 Flocculation performance of PFSi-δ

The results of PFSi-δ performance on seawater flocculation are presented in Table 2. It is interesting to observe that pH values were stable and not depend on the experimental concentrations of PFSi-δ. As expected, a higher dose of PFSi-δ led to higher turbidity and DOC removal efficiency. After flocculation, pH was increased to about 8.2, a suitable range for flocculation, regardless of concentration. At dosage of 1.2 mg Fe^{3+}/L, more than 60% turbidity removal efficiency was exhibited.

Table 2. The change of turbidity, pH, DOC fractions and removal efficiencies of seawater by flocculation with PFSi-δ

DOC removal efficiency of different fractionations of organic matter by flocculation analysed by LC-OCD is presented in Table 2. The hydrophobic compounds and hydrophilic compounds in the seawater before flocculation by PFSi-δ were 41% and 59% respectively.

Compared with $FeCl_3$, PFSi-δ could remove turbidity of seawater effectively. In terms of DOC, the removal efficiency of PFSi-δ with lower ferric contents was nearly 20% higher than that of $FeCl_3$.

In a similar manner to flocculation by $FeCl_3$, the results also show that PFSi-δ could remove a majority of hydrophobic compounds. Even at a low dose of 1.2 mg Fe^{3+}/L, PFSi-δ could remove more than 88% of hydrophobic compounds. PFSi-δ was found to have better hydrophilic removal in comparison with $FeCl_3$. It removed up to 55% of hydrophilic at dose of 1.2 mg Fe^{+3}/L, nearly 2 times more than that of $FeCl_3$ whereas the dose of PFSi-δ in terms of ferric was nearly a half.

The removal efficiencies of bio-polymer (MW >> 20,000 Da) and low molecular weight of neutrals (MW < 350 Da) were also high with 92% and 89% respectively. However, the removal efficiency of building blocks (MW 300~500) by PFSi-δ was only below 32%. It is interesting to find out that even at lower doses, PFSi-δ could remove 23-32% more hydrophilic fractions except neutrals compared to $FeCl_3$ (Tables 1 and 2).

The change of zeta potential of particles was measured after flocculation with PFSi-δ. With the increase of flocculant dosage, the charge of particles also increased from negative to positive. The concentrations of 0.8 mg Fe^{3+}/L and 1.2 mg Fe^{3+}/L showed a positive charge after rapid mixing. After the settling, the negative charge of particles in the supernatant reduced from -7 mV to less than -3 mV and -2 mV respectively. After flocculation, if particles remain negatively charged, it needed to be increased to a positive charge to enhance coagulation behaviour [17].

Table 3. The charges of flocculants and particles in supernatant after flocculation, and physical characteristics of flocculated suspended solids

3.1.3 Comparison of the characteristics of flocs

The physical and chemical characteristics of flocs of both flocculants at pre-selected concentration (0.5 mg Fe^{3+}/L as lower dose and 3.0 mg Fe^{3+}/L as higher dose) are summarised in Table 3. The charge of raw seawater induced by organic matter was -1.86 mV. It can be seen that the charges of particles in seawater were neutralised after flocculation. As PFSi-δ concentration increased to 3.0 mg Fe^{3+}/L, the negative charged particles were reduced markedly. With respect to the average particle size, $FeCl_3$ flocs were larger than PFSi-δ flocs at the same ferric contents. The amount of flocs was similar at the flocculant dosage of 0.5 mg Fe^{3+}/L.

3.2 In line flocculation coupled with submerged micro-filtration system (IFSMS)

3.2.1 Critical flux

In this experiment, critical flux was measured quantitatively by a "flux stepping" method. The membrane reactor was operated at a fixed flux for around 60 minutes and the TMP was monitored simultaneously. The flux was then increased and operated at a constant flux for another 60 minutes and so on. As the flux was increased gradually, the critical flux condition was detected where TMP no longer remained steady but increased with time. The maximum flux which showed no increase in TMP was taken as the critical flux.

On large pore MF membrane, irreversible fouling was dominant due to the deposition of low MW weight NOM on the internal structure of the membrane and the fouling components appear to be colloidal and hydrophilic neutral fraction of NOM. It was concluded that medium to low MW component of NOM (300 - 1,000 Da) is responsible for the initiation of fouling [18]. At short period experiment, therefore, these factors can affect critical and limiting flux on MF.

The critical flux for conventional micro-filtration system with seawater collected from Chowder Bay was only 20 $L/m^2 \cdot h$. The coupling of in-line flocculation with hollow fibre submerged MF membrane reactor led to an increase of critical flux (Fig. 2). The critical flux increased to 30 $L/m^2 \cdot h$ (Fig. 2a) when 1.2 mg/L of Fe^{3+} were used as PFSi-δ flocculant and 55 $L/m^2 \cdot h$ (Fig. 2b) when 3.0 mg/L of Fe^{3+} were used as $FeCl_3$ flocculant. As can be seen from Table 3, with same ferric content, PFSi-δ generated more amounts of flocs and their size was also smaller than flocs obtained from ferric chloride. These may be the cause of lower critical flux by flocculation with PFSi-δ.

Fig. 2. Variation of TMP values (filtration flux 20 $L/m^2.h$)

3.2.2 Organic removal

It was observed that by coupling in-line flocculation with MF, up to 58% of hydrophilic compounds could be removed at a dose of $FeCl_3$ 3.0 mg/L (in terms of Fe^{3+}) (Table 4a). This value was more than 20% than that of flocculation alone. As a result, the DOC removal of in-line flocculation coupled with a submerged membrane system was also 14% higher than that of flocculation alone.

The results from MF filtration coupled with in-line flocculation with PFSi-δ show that 78% of DOC could be removed after 6.0 hrs (Table 4b). This value was about 10% higher than that of flocculation alone. In particular, bio-polymer and neutrals were removed completely by IFSMS. The results also showed that higher DOC removal efficiency was obtained by PFSi-δ than that by $FeCl_3$.

Table 4. Fraction of organic matter after in-line flocculation coupled with MF filtration (filtration flux 20 $L/m^2 \cdot h$)

4. Conclusions

The performances of PFSi-δ and $FeCl_3$ flocculants were conducted in both batch test and in-line flocculation coupled with submerged microfiltration system (IFSMS). Both PFSi-δ and $FeCl_3$ were effective in removing dissolved organic compounds in seawater. PFSi-δ at a lower dose shows higher removal efficiencies of turbidity and DOC in comparison with $FeCl_3$. In particular, hydrophilic compounds were removed better by PFSi-δ. About 55% of hydrophilic compounds in seawater were found to be removed by PFSi-δ 1.2 mg Fe^{3+}/L. However, the amount of flocs with PFSi-δ was twice than that with $FeCl_3$ and the size of the flocs by PFSi-δ were slightly smaller than that by $FeCl_3$. This may be attributed to the formation of fouling on the MF membrane and a lower critical flux of PFSi-δ than that of $FeCl_3$.

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Table No.

Table 1. The changes of turbidity, pH, DOC fractions and removal efficiencies of seawater by flocculation with $FeCl_3$

Table 2. The change of turbidity, pH, DOC fractions and removal efficiencies of seawater by flocculation with PFSi-δ

Table 3. The charges of flocculants and particles in supernatant after flocculation, and physical characteristics of flocculated suspended solids

Table 4. Fraction of organic matter after in-line flocculation coupled with MF filtration (filtration flux 20 $L/m^2.h$)

Table 1. The changes of turbidity, pH, DOC fractions and removal efficiencies of seawater by flocculation with $FeCl_3$

		Seawater		$FeCl_3$									
as Fe^{3+} (mg/L)		0		0.5		1.0		2.0		3.0		4.0	
Turbidity		0.41		1.26		1.83		1.33		1.43		1.65	
pH	Initial	7.98		-		-		-		-		-	
	Final	-		7.53		7.41		7.25		7.22		7.16	
		mg/L	%(a)	mg/L	%(b)	mg/L	%(b)	mg/L	%(b)	mg/L	%(b)	mg/L	%(b)
DOC		1.29	100	0.65	50	0.66	49	0.69	47	0.56	57	0.69	47
Hydrophobic		0.43	33	0.03	93	0.01	98	0.11	74	0.02	95	0.16	63
Hydrophilic		0.86	67	0.62	28	0.65	24	0.58	33	0.54	37	0.53	38
Biopolymer		0.13	10	0.08	38	0.07	46	0.06	54	0.05	69	0.04	69
Humic		0.44	34	0.37	16	0.39	11	0.37	16	0.36	18	0.34	23
Building blocks		0.20	16	0.17	15	0.15	20	0.13	30	0.14	30	0.13	30
Neutrals		0.09	7	0.00	100	0.03	67	0.01	89	0.00	100	0.01	89

%(a): composition of the different organic fractions in seawater.

%(b): removal efficiencies of the different organic fractions in seawater after flocculation.

Table 2. The change of turbidity, pH, DOC fractions and removal efficiencies of seawater by flocculation with PFSi-δ

	Seawater		PFSi					
as Fe^{3+} (mg/L)	0		0.4		0.8		1.2	
Turbidity	1.70		0.74		0.69		0.67	
pH Initial	8.0		-		-		-	
Final	-		8.2		8.2		8.2	
	mg/L	%$^{(a)}$	mg/L	%$^{(b)}$	mg/L	%$^{(b)}$	mg/L	%$^{(b)}$
DOC	1.45	100	0.68	53	0.57	61	0.46	68
Hydrophobic	0.59	41	0.19	68	0.12	80	0.07	88
Hydrophilic	0.86	59	0.49	43	0.45	48	0.39	55
Biopolymer	0.13	9	0.01	92	0.01	92	0.01	92
Humic	0.45	31	0.31	31	0.31	31	0.23	49
Building blocks	0.19	13	0.15	21	0.12	37	0.13	32
Neutrals	0.09	6	0.01	89	0.01	89	0.01	89

%$^{(a)}$: composition of the different organic fractions in seawater.
%$^{(b)}$: removal efficiencies of the different organic fractions in seawater after flocculation.

Table 3. The charges of flocculants and particles in supernatant after flocculation, and physical characteristics flocculated suspended solids

		$FeCl_3$		PFSi-δ	
Concentration	mg (Fe^{3+})/L	0.5	3.0	0.5	3.0
Zeta potential (mV)	Flocculants[(a)]	3.48	20.0	7.59	13.9
	Raw seawater	-1.86	-1.86	-1.86	-1.86
	Particles (in supernatant)[(b)]	-4.50	-1.90	-6.20	-0.40
Particle size (Z-average)	(nm)	1878	2468	1757	2245
MLSS	(mg/L)	9.6	20	10	38
VSS	(mg/L)	4.8	2.0	4.4	10.8
VSS/MLSS	(%)	50	10	44	28

[(a)]: Flocculants itself at a given concentration

[(b)]: Particles in supernatant after flocculation and settling

Table 4. Fraction of organic matter after in-line flocculation coupled with MF filtration (filtration flux 20 L/m^2.h)

(a) $FeCl_3$ (3.0 mg Fe^{3+}/L)

Feed water	DOC	Hydro-phobic	Hydro-philic	Bio-polymer	Humic	Building blocks	Neutrals
Concentration (mg/L)							
Seawater	1.29	0.46	0.83	0.13	0.44	0.20	0.09
Effluent after 2 hrs	0.39	0.02	0.37	0.05	0.20	0.12	0.00
Effluent after 24hrs	0.38	0.02	0.36	0.02	0.20	0.11	0.03
Removal efficiency (%)							
Effluent after 2 hrs	70	95	55	62	55	40	100
Effluent after 24hrs	71	95	58	85	66	20	67

(b) PFSi-δ (1.2 mg Fe^{3+}/L)

Feed water	DOC	Hydro-phobic	Hydro-philic	Bio-polymer	Humic	Building blocks	Neutrals
Concentration (mg/L)							
Seawater	1.45	0.59	0.86	0.13	0.45	0.19	0.09
Effluent after 1 hrs	0.41	0.04	0.37	0.00	0.22	0.15	0.00
Effluent after 2 hrs	0.41	0.05	0.36	0.00	0.17	0.20	0.00
Effluent after 6 hrs	0.34	0.02	0.32	0.00	0.20	0.11	0.00
Removal efficiency (%)							
Effluent after 1 hrs	72	93	57	100	51	21	100
Effluent after 2 hrs	72	92	58	100	62	10	100
Effluent after 6 hrs	78	97	63	100	56	42	100

Figure No.

Fig. 1. Schematic diagram of in-line flocculation coupled with hollow fibre microfiltration

Fig. 2. Variation of TMP values (filtration flux 20 $L/m^2.h$)

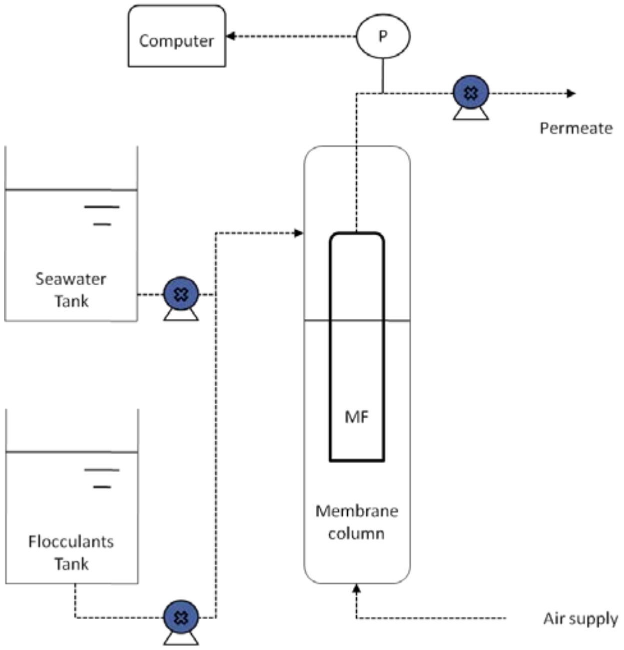
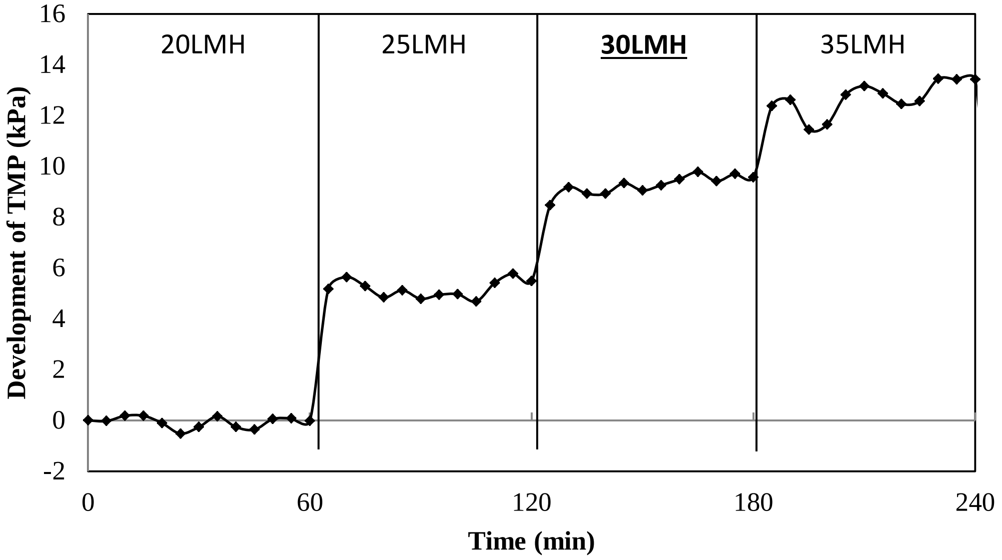
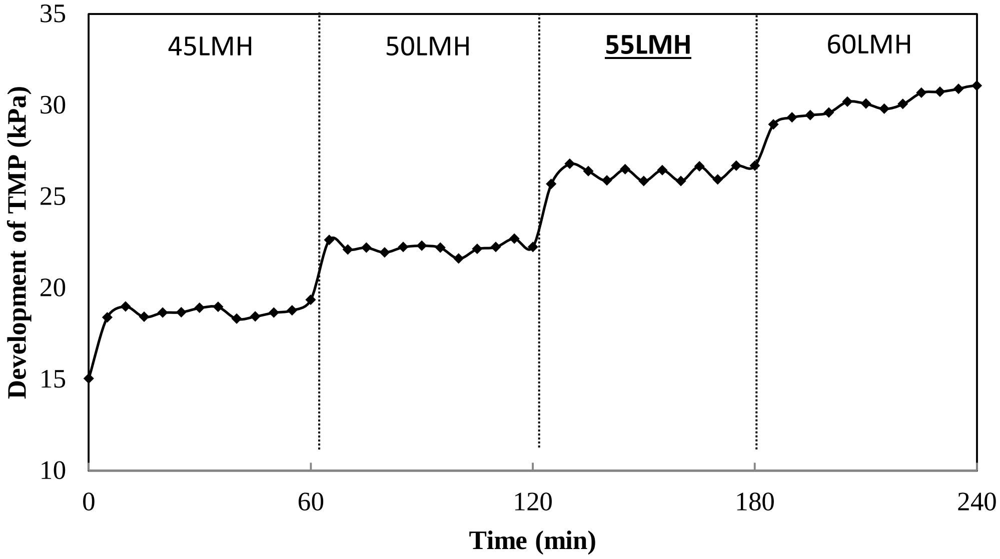


Fig. 1. Schematic diagram of in-line flocculation coupled with hollow fibre microfiltration



(a) PFSi-δ (1.2 mg Fe^{3+}/L)



(b) $FeCl_3$ (3.0 mg Fe^{3+}/L)

Fig. 2. Variation of TMP values (filtration flux 20 L/m^2.h)