Reduction of dissolved organic matter and trihalomethane formation potential in raw water supply reservoir by hybrid ultrafiltration process

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ABSTRACT

Hybrid microfiltration (MF)–ultrafiltration (UF) and hybrid coagulation–ultrafiltration membrane processes were investigated and compared for dissolved organic matter (DOM) reduction in the low turbidity raw water supply from Sri-Trang Reservoir, Thailand. The coagulation process was operated with 20 mg/L polyaluminum chloride (PACl) at pH 7. The MF used had a pore size of 0.1 μm and the UF was of immersible polysulfone hollow fiber membrane with a pore size of 0.008 μm. The hybrid coagulation–UF process could effectively reduce DOM in terms of dissolved organic carbon (DOC) and UV-254 by 64% whereas the hybrid MF–UF membrane process yielded lower efficiency, 58% DOC reduction and 50% UV-254, respectively. In regards to trihalomethane formation potential (THMFP) analysis, the hybrid coagulation–UF membrane process showed slightly higher efficiency in THMFP reduction than that of the hybrid MF–UF process, 87.6 and 87.1%, respectively. The resin adsorption technique using DAX-8 was also employed to fractionate DOM in all water samples into two fractions; hydrophobic (HPO) and hydrophillic (HPI) organic matter. Significantly, HPO was the major fraction of DOM in the raw water source and it was the main precursor of THMFP.

Key words | DOM, hybrid ultrafiltration process, raw water supply reservoir, THMFP, UV-254

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INTRODUCTION

Sri-Trang Reservoir is located in Hat Yai campus of Prince of Songkla University (PSU), Songkhla Province, Thailand. The water in this reservoir is used as a raw water for the PSU water treatment plant. Its characteristics are low turbidity (3–14 NTU) and low alkalinity (11–17 μS/cm) throughout the year (Labaiji *et al.* 2008; Mumean *et al.* 2008). These are the general characteristics of such kinds of raw water sources in Thailand (Kueseng *et al.* 2011). Moreover, the previous work (Kueseng *et al.* 2011) indicated that this reservoir may be contaminated by natural organic matter (NOM), especially dissolved organic matter (DOM) discharged from agricultural activities upstream in addition to the NOM originating from natural decomposition.

The NOM, especially DOM, was reported to be of concern when it is present in a raw water supply. The remaining DOM serves as a precursor that can react with chlorine, which is commonly used for disinfection in water treatment processes and form carcinogenic disinfection by-products (DBPs) such as haloacetic acids (HAAs) and trihalomethanes (THMs) (Rook 1974). Kueseng (2010) reported that tap water from the PSU water treatment plant contained THMs at higher concentrations than that stipulated in the WHO guidelines. THMs can be taken in by drinking the water and by breathing its vapour (for example, when showering). THMs are absorbed, metabolized and eliminated rapidly by mammals after oral or inhalation exposures. Following absorption, highest concentrations are accumulated in fat tissues, liver and kidneys (Marhaba & Van 1999). Therefore, an investigation of alternative processes in order to reduce THMs for the conventional PSU water treatment plant is deemed necessary.

In this study, the use of hybrid processes combining either the traditional water treatment (by coagulation) or a membrane microfiltration process (MF) and ultrafiltration process (UF) were employed. Membrane process information for water treatment in Thailand, especially for raw water supply reservoirs, is still insufficient. However, MF and UF processes are widely used nowadays in water treatment to remove DOM in a raw water supply (Zularisam *et al.* 2006) because it is more cost-effective than nanofiltration (NF) and reverse osmosis (RO) (yielding higher permeate flux in comparison) and high DOM removal efficiency (Shon *et al.* 2004). Ates *et al.* (2009) reported that the UF process could remove DOM in terms of UV-254 and DOC (dissolved organic carbon) by 74 and 61%, respectively. These removed DOM significantly indicated rejection not only by pore size exclusion, but also other separation mechanisms such as adsorption onto the membrane surface, adsorption onto particles in the cake layer or sieving as a result of physical constriction of the membrane pores due to irreversible fouling (Jacangelo *et al.* 1995; Walsh *et al.* 2008). Resin adsorption has lately been successfully utilized as a fractionation technique to fractionate the groups of DOM into hydrophobic (HPO) and hydrophilic (HPI) fractions by using a DAX-8 resin (Leenheer 1981) and thus it has been used in this study to better understand the characteristics of DOM and the formation kinetics of THMs. HPO fractions were often found to have higher THMs reactivity than the HPI (Collin *et al.* 1986). This paper emphasizes the use of these techniques to investigate the DOM and trihalomethane formation potential (THMFP) reduction of raw water supply from the Sri-Trang Reservoir. The efficiency of hybrid ultrafiltration with a pre-treatment technique, coagulation or MF process, was reported in terms of both the total DOC and DOM fractionated group removal.

MATERIALS AND METHODS

Raw water

Raw water samples from the Sri-Trang Reservoir were collected from the pumping station of the PSU water treatment plant (UTM 666327 774845) in September 2009 and preserved at 4 °C until experiments were conducted.

The raw water samples were analyzed to determine their pH, turbidity, alkalinity, conductivity, ultraviolet adsorption at 254 nm wavelength (UV-254), dissolved organic carbon (DOC), fluorescent excitation–emission matrix (FEEM) and trihalomethane formation potential (THMFP) in accordance with *Standard Methods for the Examination of Water and Wastewater* (APHA 1998).

Membrane and coagulant

An immersible polysulfone hollow fiber microfiltration membrane (MF) model MF2505Mi with a pore size of 0.1 μm and an ultrafiltration membrane (UF) model UF2505Mi with a nominal pore size of 0.008 μm operating in an outside/in and dead-end filtration mode provided by Polymem, France, were used in this study. Before the experiment, the membranes' surface was washed with deionized water and soaked in 0.1 N NaOH solution for 30 min, following by rinsing with deionized water. The coagulant used in the coagulation process was polyaluminum chloride (PACl). The concentration of PACl stock solution was 20 g/L and it was diluted to 20 mg/L for use in the experiments.

Pre-treatment methods

Two different pre-treatment techniques, coagulation and the MF process, were conducted prior to the UF process. They compared the efficiency of turbidity and DOM reduction. The coagulation process employed 20 mg/L of PACl as a coagulant and was operated at pH 7 (an optimized condition) by the jar test apparatus with 100 rpm rapid mixing for 1 min and then 30 rpm slow mixing for 30 min. After that, it was left to settle for 1 h before the supernatant was collected for analysis. For the MF process, raw water was sucked from the outside to the inside of the membrane at a transmembrane pressure of −0.18 bar. The pre-treated water was analyzed for water qualities and DOM reduction prior to the UF process.

Ultrafiltration set-up

The laboratory-scaled UF dead-end filtration was conducted as shown in Figure 1. The UF membrane was immersed in a reactor and water was drawn in through the filtering membrane by a suction pump at all times. The permeate flux
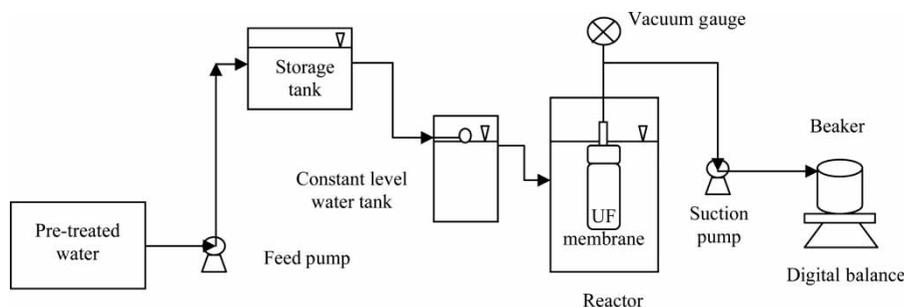
Figure 1 | Schematic diagram of the ultrafiltration (UF) process.

was measured as a function of time at a constant pressure of −0.2 bar for 120 min and the permeate volume was determined using a digital balance.

Resin fractionation

The resin adsorption procedure as proposed by Marhaba *et al.* (2003) was used to isolate DOM into two fractions, HPO and HPI, by using a DAX-8 resin. The water samples were filtered through a pre-combusted 0.7 μm GF/F filter and acidified to pH 2. The filtrated samples were then gravity-fed through the DAX-8 columns at a flow rate of approximately 12 bed volumes/h. The effluent from the column was defined as the HPI fraction. After all samples were run through the columns, each column was separately back-eluted with 0.1 N NaOH at 0.25 bed volumes/h, followed by 0.01 N NaOH at 1.25 bed volumes/h. The elution sample obtained was defined as the HPO fraction. Mass balance in terms of DOC was calculated to ensure that it reached an acceptable recovery level (90–110%). The HPI fraction was adjusted back to the ambient pH of the water with NaOH. The HPO fraction was reconstituted back to its equivalent DOC with deionized water (DI) and then to the ambient pH with H_2SO_4.

Analytical methods

The water samples were analyzed to determine their pH, turbidity, alkalinity, conductivity and DOM concentration in accordance with *Standard Methods for the Examination of Water and Wastewater* (APHA 1998). The DOC was determined by TOC Analyzer (O.I. analytical 1010). UV absorbance was measured by a spectrophotometer,

spectronic Genesys 10 UV at 254 nm. At least two replications of each measurement of the UV-254 analysis were performed. The pH in all water samples was adjusted to 7. The fluorescence excitation–emission matrix (FEEM) of the samples was generated by using a fluorescence spectrophotometer (JASCO FP-6200). Prior to the fluorescence measurement, pH of all samples was adjusted to neutral in the same manner as that in the UV measurement. The neutralized solution was buffered by a phosphate solution before incubation at $25 \pm 2\,^{\circ}C$ in amber bottles with PTFE liners. At the end of the 7-d reaction period, samples were checked to have a remaining free residual chlorine between 3–5 mg/L. The residual chlorine was measured according to the procedures mentioned in Standard Method 4500-Cl G, the *N,N*-dechthyl-p-phenylenediamine colorimetric method. The level of chlorine was then represented by the light absorbance at 515 nm using the spectrophotometer, spectronic unicam Genesys 10 UV with matched quartz cells that provided a path length of 10 mm. THMs were extracted with pentane in accordance with Standard Method 6232B (*Standard Methods*, APHA 1998). An Agilent Gas Chromatography-6890 with an electron capture detector (ECD) (Agilent Technologies Inc., Wilmington, DE, USA) and chromatographic column (J&W Science DB-624, DE, USA) with an 0.2 mm × 25 m 1.12 μm film used to analyze THMs.

RESULTS AND DISCUSSION

Characteristics of raw water

A summary of the characteristics of raw water from the Sri-Trang Reservoir is presented in Table 1.

Table 1 | Characteristics of raw water from Sri-Trang Reservoir

Parameters	Range	Average	S.D.
pH	6.98–7.04	7.01	±0.03
Alkalinity (mg CaCO$_3$/L)	8.0–9.0	8.33	±0.58
Conductivity (μS/cm)	48.8–51.1	49.87	±1.16
Turbidity (NTU)	7.85–8.21	8.03	±0.18
DO (mg/L)	7.06–7.87	7.49	±0.41
UV-254 (cm^{-1})	0.042–0.056	0.05	±0.01
DOC (mg/L)	3.9–4.2	4.08	±0.18
SUVA (L/mg-m)	1.08–1.36	1.24	±0.14

As shown in Table 1, the average pH value of the water samples from the Sri-Trang Reservoir indicated that it was nearly neutral whereas its alkalinity was slightly low. In order to prevent pH drop by alkalinity consuming during the coagulation/flocculation process, the conventional water coagulation that utilizes PACl as the coagulant generally requires additional alkalinity, especially in the case of low alkalinity raw water. Interestingly, its turbidity and SUVA also were very low (lower than 9 NTU and 2 L/mg-m, respectively). If the SUVA of any raw water is ≥2.0 L/mg-m, the coagulation process might not be proposed as the appropriate process to reduce DOM levels in water (USEPA 1999). The raw water that has low turbidity may affect floc formation in the coagulation/flocculation process whereas low SUVA implies a low aromatic characteristic (Korshin *et al.* 1997). This result implied that the coagulation process may not be efficient enough to remove DOM from this raw water source.

For the percent distribution of DOM fractions in the Sri-Trang Reservoir's raw water, it was observed that HPO and HPI fractions accounted for 56 and 44% of total DOC, respectively. As reported by Mash *et al.* (2004), major DOM fractions in raw waters from reservoirs were HPO fractions with a percent distribution of 50–65%. HPO is primarily made up of humic substances, they are mainly derived from soil and are also produced within natural water and sediments by chemical and biological processes such as the decomposition of vegetation (AWWA 1993).

Removal of DOM (DOC and UV-254)

This subsection is aimed at analyzing the effectiveness of MF combined with UF in comparison with coagulation

with a PACl dose of 20 mg/L combined with UF in order to reduce the DOM in the water samples.

The results from Figure 2 illustrate that the hybrid process MF combined with UF, and the coagulation as a pretreatment combined with UF, could reduce DOM in terms of DOC by 58 and 64% and could reduce DOM in terms of UV-254 by 50 and 64%, respectively. The results also show that both the hybrid UF processes were effective in reducing HPO fraction but less effective for HPI fractions. The HPO:HPI fraction ratio in raw water which had a percent distribution of 56:44% had changed to 35:65% in treated water. As reported by Kueseng *et al.* (2011), treated water from the Sri-Trang Reservoir by coagulation had a higher HPO fraction reduction than the HPI fraction reduction.

It is of interest here to point out that the DOC and the UV-254 removal efficiencies of the hybrid coagulation–UF process were higher than that of the MF–UF process. The reason for this phenomenon is that UV-254 could detect DOM in the form of most aromatic organic substances
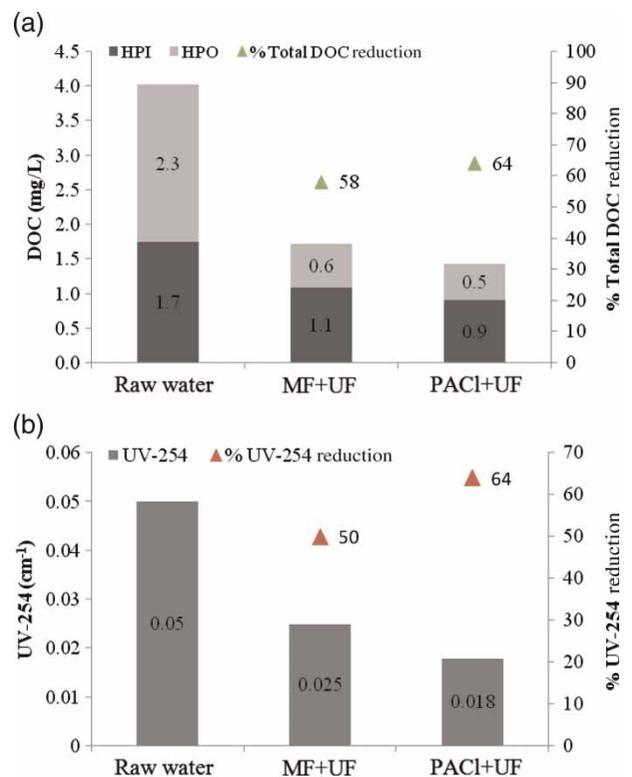
Figure 2 | Change in (a) DOC and (b) UV-254 of raw waters and treated waters by the UF process.

(HPO) which could be reduced effectively by coagulation (AWWA 1993). Some tiny organic substances had been adsorbed onto the aluminum flocs and settled down under gravity prior to the UF process. In addition, residual aromatic organic substances, such as humic acid-like substances, have a particle size in the range of 0.0025–0.01 μm, which is larger and/or smaller than the UF membrane pore size of 0.008 μm, and hence the UF process displays good effectiveness in the removal of humic acid-like substances. These removed DOM significantly indicated rejection not only by pore size exclusion but also by other separation mechanisms such as adsorption onto the membrane surface, adsorption onto particles in the cake layers or sieving, as a result of physical constriction of the membrane pores due to fouling. However, its efficiency was not 100% due to some particles of humic acid-like substances which, if smaller than 0.008 μm, will be able to pass through the UF membrane pore size. On the other hand, DOM removal by the MF–UF process relies on the size sieving mechanism. MF with a pore size of 0.1 μm, which is larger than the size distribution of organic substances, is not sufficient to reduce DOC and UV-254 and will not be suited to be used as a pretreatment prior to UF.

DOM characterization and DOM removal efficiency by FEEM

Examples of FEEM peaks obtained from this study were depicted in Figure 3. Figure 3(a) indicates that raw waters from the Sri-Trang Reservoir contained not only the humic acid-like substances (peak B: $280\,nm_{Ex}/410–430\,nm_{Em}$)

and fulvic acid-like substances (peak C: $330\,nm_{Ex}/390–410\,nm_{Em}$) but also tryptophan-like substances (peak A: $280\,nm_{Ex}/360–370\,nm_{Em}$).

If tryptophan-like substances are present, it is most likely that the raw water is contaminated with municipality discharge (Musikavong et al. 2007). These three fluorescent peaks were categorized in accordance with the boundaries of the FEEM peak created by Chen et al. (2003).

Musikavong et al. (2007) suggested that the fluorescent intensity in the QSU units of each fluorescent peak could be further utilized to determine the quantity of all fluorescent organic matter in water by adding the fluorescent intensities of all FEEM peaks. For total fluorescent organic matter reduction, it was found that both the hybrid coagulation–UF process and hybrid MF–UF process could reduce the total fluorescent organic matter by around 61%. The results imply that humic and fulvic acid-like substances and tryptophan-like substances could be moderately reduced by the hybrid UF processes. However, when the reduction efficiency of each fraction is considered, the reduction of all fluorescent intensities will be slightly different.

The removal efficiency of THMFP

THMFP has commonly been used to determine THMs at the completion of reaction conditions between DOM and the excess amount of chlorine. Waters with high THMFP values could potentially form a high level of THMs. In addition, reduction of THMFP by water treatment processes can be used to represent the
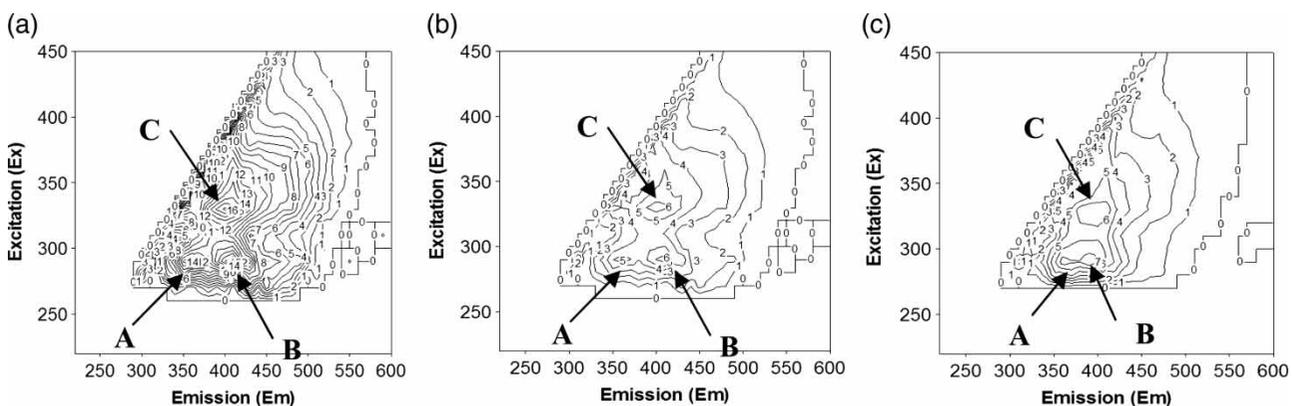

Figure 3 │ FEEM and fluorescent peaks of (a) raw water, (b) MF–UF and (c) PACl–UF in the contour graph with 2 QSU contour interval.

reduction of DOM, which has an active ability in forming THMs. THMFP, therefore, is considered to be an appropriate indicator and was utilized to monitor the highest possible concentrations of THMs in water. THMFP was determined from the summation of chloroform formation potential ($CHCl_3$-FP), bromodichloroform formation potential ($CHClBr_2$-FP), dibromochloroform formation potential ($CHClBr_2$-FP) and bromoform formation potential ($CHBr_3$-FP).

Results from THMFP evaluation of each water sample (Figure 4) revealed that only chloroform and bromodichloroform were formed during chlorination, and chloroform was the major THM species in the water samples. This observation corresponds well with results from previous research (Rodriguez *et al.* 2003). Figure 4 also showed that the THMFP value of the Sri-Trang Reservoir's raw water was 437 μg/L while 56 and 54 μg/L of THMFP were respectively observed from treated waters by the hybrid MF–UF process and the hybrid coagulation–UF process, which could reduce THMFP in raw waters by approximately 87 and 88%, respectively. According to a report by WHO (2006), which the Thailand Metropolitan Waterworks Authority employed as a water quality standard for tap water in the country, the maximum acceptable levels for $CHCl_3$ and $CHCl_2Br$ are 300 and 60 μg/L, respectively. From these results, it can be stated that the THMFP values of treated waters by the hybrid UF processes in this study comply with the

WHO standard and also the standard of Thailand's Metropolitan Water Work Authority.

Moreover, these results indicate that the main precursor of THMFP for all water samples was the HPO fraction which agrees well with the study of Kueseng *et al.* (2011). To better understand the reactivity of each organic fraction on the formation of THMs, the specific THMFP (STHMFP) was observed in this analysis. Total THMFP has been defined as the ratio between THMFP from each organic fraction and the DOC of the water samples. This gives an overall picture of how THMs were formed in the water samples. A specific THMFP, on the other hand, is the ratio between THMFP and the DOC of each organic fraction. From the results, the HPO fraction was observed to give higher STHMFP than the HPI fraction for all samples (e.g. STHMFP of MF–UF: $STHMFP_{HPI} = 20$ and $STHMFP_{HP0} = 57$) despite the quantity of the HPI fraction that was larger than the HPO fraction after the UF experiment (Figure 1). This indicates that HPO is strongly reactive with chlorine in THM formation. In addition, when the ratio between THMFP and the total remaining FEEM intensity of the water sample was considered, it was found that the ratio of treated water by the hybrid coagulation–UF process was lower than that of water treated by the hybrid MF–UF process. Thus it could be concluded that the hybrid coagulation–UF process can reduce THMFP better than the hybrid MF–UF process.
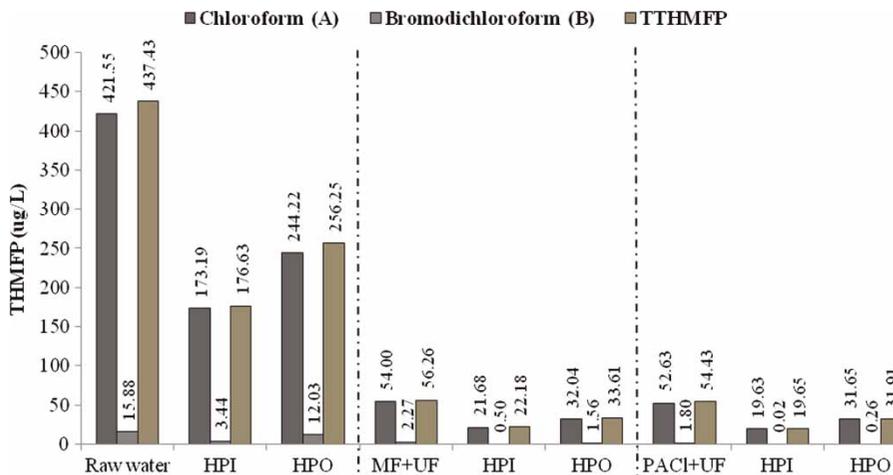

Figure 4 | THMFP removals by the hybrid ultrafiltration process.

Effect of coagulation and MF on UF membrane flux decline

The effect of coagulation and the MF process on UF behavior is shown in Figure 5 where measured flux is plotted against time. Figure 5 shows that the rates of permeate flux decline were different according to the pre-treatment process. With the coagulation process, the UF flux decreased to about 9% of the initial flux after 120 min. In contrast, in the MF process, the membrane flux decreased to 24% of the initial flux in the same time. This suggests that the membrane flux declines slowly with 20 mg/L PACl coagulant and declines more with the MF process. This could be due to the fact that MF uses a size sieving machanism which has a pore size of 0.1 μm that could effectively reduce particles which are larger than 0.1 μm in water but with less effectiveness for particles smaller than 0.1 μm such as humic acid-like substances which have particle sizes in the range of 0.0025–0.01 μm. This finding indicates that fouling of the UF process is more likely by internal pore blocking by the penetration of small organic substances, resulting in a higher decrease of the membrane flux, whereas coagulation is a floc formation mechanism which could settle a wide range of organic matter particle sizes and only become supernatant after the floc sedimentation was fed to the UF process, thus results in the decrease of membrane fouling. Therefore, the coagulation–UF process strongly showed more effectiveness for DOM removal in this study. However, fouling occurred on the UF membrane surface after coagulation might be caused by the remaining light flocs, which is attributed to the cake/gel layer formation.
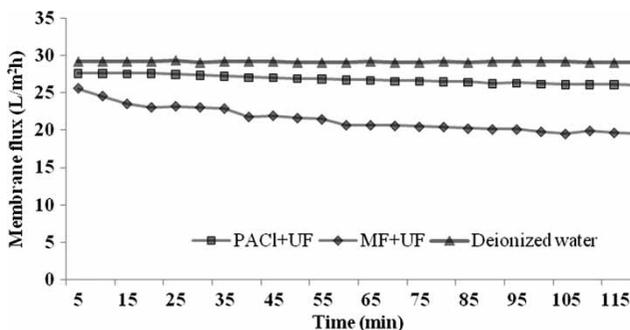

Figure 5 | Effect of coagulation and MF on membrane flux decline.

CONCLUSIONS

The hybrid coagulation–UF membrane process could reduce DOM in terms of UV-254 and DOC from the Sri-Trang Reservoir more effectively than the hybrid MF–UF membrane process. The use of coagulation as a pre-treatment produced a permeate flux higher than the pre-treatment with MF when they are compared for the same time. However, when THMFP reduction efficiency is considered, the hybrid MF–UF process displayed almost the same efficiency as the hybrid coagulation–UF membrane process. From these results, it can be concluded that the hybrid MF–UF process, with a pore size of 0.1 μm for the MF and 0.008 μm for the UF, has a potential to be an alternative process in the case of DOM reduction for the PSU water treatment plant that employs low turbidity raw water from the Sri-Trang Reservoir. Coagulant is not needed together with low chemical needs for the pretreatment with MF. From the resin adsorption technique using DAX-8, the HPO fraction was the major fraction of DOM in this raw water and it was the main precursor of THMFP.

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