Treatment of highly-colored surface water by a hybrid microfiltration membrane system incorporating ion-exchange

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ABSTRACT

This study investigated the performance of a hybrid membrane filtration system to produce industrial water from highly-colored surface water. The system consists of a membrane filtration process with appropriate pretreatments, including coagulation, pre-chlorination, and anion exchange (IE) process. The results of the pilot-scale experiments revealed that the hybrid system can produce treated water with color of around 5 Pt-Co, dissolved manganese concentration of no more than 0.05 mg/L, and a silt density index (SDI) of no more than 5 when sufficient coagulant and sodium hypochlorite were dosed. Although the IE process effectively reduced the color of the water, a moderate increase in the color of the IE effluent was observed when there was a significant increase in the color of the raw water. This resulted in a severe membrane fouling, which was likely to be attributed to the excess production of inorganic sludge associated with the increased coagulant dosage required to achieve sufficient reduction of color. Such severe membrane fouling can be controlled by optimising the backwashing and relaxation frequencies during the membrane filtration. These results indicate that the hybrid system proposed is a suitable technology for treating highly-colored surface water.

Key words | color reduction, industrial water, ion exchange, membrane filtration, surface water

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INTRODUCTION

For logistics management, many industrial estates have been located along the coastal region in eastern Thailand. These favourable locations have attracted many industries onto the estates. The expansion of industrial estates has resulted in an increase in demand and diversity of industrial water supply. Taking into account the potential uses of water in an industrial estate such as for washing purposes (e.g., manufacturers of automotive parts) and feed water for a polishing process using reverse osmosis (RO) membrane (e.g., electronics and food & beverage industries), an industrial water which can be directly applied to a polishing process using RO membrane and has low color content is needed for an industrial water treatment plant. Water treatment utilizing low-pressure membranes (microfiltration (MF) and ultrafiltration (UF) membranes) is thought to be a suitable technology for such purposes due to its capability to completely remove particulate matter. Therefore, applying low-pressure membranes is thought to be advantageous for improving treated water quality and simplifying pretreatment of a polishing process using an RO membrane. However, constituents smaller than the pore of the membrane such as small-sized natural organic matter (NOM) and dissolved metals cannot be effectively removed by the membrane filtration process alone (Laîné *et al.* 2000; Suzuki *et al.* 2001). To remove such compounds, appropriate combinations with other unit processes are required. For the membrane-based water treatment plants in the South-East Asian countries such as Thailand, special

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attention needs to be paid to the removal of colored NOM and dissolved manganese.

In the coastal region of eastern Thailand, surface water is commonly used as a source water for industrial water production. Generally, such surface water contains high concentrations of colored NOM. Due to the small size of colored NOM, its removal in the low-pressure membrane filtration process is commonly difficult. For sufficient removal of colored NOM in membrane-based water treatment, appropriate combinations with other treatment methods should be established. Candidates of add-on treatment processes for improving color removal in a membrane-based water treatment plant include ozonation or an advanced oxidation process (Azbar *et al.* 2004; Mezzanotte *et al.* 2013), coagulation (Leiknes *et al.* 2004), ion-exchange (Johnson & Singer 2004), and activated carbon (Hargesheimer & Watson 1996). On this basis, we previously investigated the performance of a hybrid membrane filtration system utilizing MF membrane (denoted as hybrid MF system hereafter), which consists of membrane filtration and appropriate pretreatments for removing colored NOM and dissolved manganese (Suzuki *et al.* 2012). The pretreatments investigated in our previous study were anion-exchange (denoted as IE hereafter), coagulation, activated carbon, pre-chlorination, and biological oxidation. The results revealed that combining IE, coagulation, pre-chlorination, and membrane filtration was an effective method for the treatment of highly-colored surface water (Suzuki *et al.* 2012). In this hybrid MF system, the amount of coagulant used can be significantly reduced compared with one without IE process at the same level of color reduction. Application of IE was also reported to be beneficial for mitigating membrane fouling (Jutaporn *et al.* 2016). This effect is particularly pronounced when combined with coagulation prior to membrane filtration (Kimura & Oki 2017).

In Thailand, seasonal variation in surface water quality is intense. The quality of water tends to deteriorate in the rainy season (typically from middle of May to middle of October). Such variation should be overcome by adjusting pretreatment conditions (e.g., adjustment of coagulant dosage according to the color of raw water). Such adjustments in pretreatment conditions may also affect the development of membrane fouling. Detailed knowledge about appropriate pretreatment conditions in relation to the seasonal variation in surface water quality has yet to be available. This information is critically important for designing a water treatment plant utilizing highly-colored surface water with significant seasonal variation as raw water. In this study, we investigated the performance of the hybrid MF system through an onsite continuous operation of a pilot-scale experimental apparatus. Appropriate adjustments of pretreatment conditions for the seasonal variation in raw water qualities are discussed. In the pilot-testing, approaches for controlling membrane fouling were also investigated.

MATERIALS AND METHODS

Pilot-scale hybrid MF system

A pilot-scale hybrid MF system was continuously operated at Amata Nakorn Industrial Estate, Thailand, using raw water delivered from the reservoir nearby. The schematic description of the experimental apparatus is shown in Figure S1 (available with the online version of this paper). The system consisted of a submerged membrane filtration process combined with appropriate pretreatments. The membrane used was a hollow-fiber membrane fabricated from polytetrafluoroethylene (PTFE) (nominal pore size of 0.1 μm). The hollow-fiber membranes were bundled to a membrane element with an effective surface area of 20 m^2 per element. The membrane unit, comprised of the membrane element mentioned above, was submerged in a membrane separation tank and membrane filtration was performed by a suction pump, with a constant flow rate. Thus, the development of membrane fouling is reflected in an increase in trans-membrane pressure (TMP). Correction of the TMP value due to the change in water viscosity in relation to water temperature was not performed, because the water temperature in the membrane separation tank was relatively stable throughout the experiment (30–40 °C). Coarse bubbles were intermittently introduced to the membrane separation tank during the membrane filtration to eliminate foulants accumulated on the membrane surface. Periodic physical membrane cleaning comprised of backwashing and relaxation was also performed at certain intervals. During this physical membrane cleaning, coarse bubbles were continuously introduced.

The feed water for the MF process was pumped to the membrane separation tank after the pretreatments with prechlorination followed by coagulation with polyaluminum chloride (PACl). The chemical dosages were adjusted to satisfy our target of treated water qualities (i.e., color: no more than 5 Pt-Co, dissolved manganese: no more than 0.05 mg/L, and silt density index (SDI): no more than 5); the targets of color and dissolved manganese concentration were selected based on the Japanese drinking water quality standard. Therefore, the chemical dosages increased when the quality of the raw water deteriorated. In our preliminary test, the change in color was not completely explained by the change in total organic matter content evaluated by total organic carbon concentration (data not shown). Because the target of treated water quality was defined by the color of the treated water, it was reasonable to monitor the color in treated water and adjust the operating condition according to the change in this index. When the membrane fouling became severe, a chemically enhanced backwashing (CEB) was performed by introducing permeate containing sulfuric acid from the permeate side of the membrane. The aim of the CEB is in the context of maintenance cleaning. When the restoration of the membrane permeability by the CEB was insufficient, an intensified chemical membrane cleaning was performed by submerging the membrane element in a chemical cleaning solution for 24 h (denoted as cleaning in place or CIP hereafter). The CIP aimed to eliminate the entire membrane fouling, and therefore, it can be referred to as a recovery cleaning. The details of the membrane cleaning conditions are summarized in Table S1 (available with the online version of this paper).

Performance of hybrid MF system incorporating IE process

A pilot-scale hybrid MF system incorporating the IE process was continuously operated to investigate its treatability and development of membrane fouling. This continuous operation was designated as Run 1. One membrane element was submerged in a membrane separation tank with an effective volume of 0.17 m^3 in Run 1. The ion-exchange resin used in this study was a strong base anion-exchange resin (Type I) with a polyacrylic macroporous structure. Anionic organic matter contained in raw water is exchanged by a chloride ion associated with the functional group of the resin. The resin concentration in the contact tank and the contact time in the IE process were 20–30% and approximately 10 min, respectively. The IE process had a flow to the resin bed volume ratio in the range of 1,500–2,000. The physical membrane cleaning (30 seconds backwashing followed by 90 seconds relaxation) was performed every 20 to 30 minutes. The intermittent aeration cycle was 60 sec-aeration/180 sec-stop. The air-flow rate was controlled so that the average specific air demand per membrane surface area (SAD_m) became 0.05 m^3/m^2/hr. The membrane flux and pH in the coagulation tank were set at 0.8 m^3/m^2/day and 7.2, respectively. To investigate the adaptability of the hybrid MF system proposed to the seasonal variation in raw water quality, Run 1 was continued for approximately ten months including both rainy and dry seasons. According to the changes in raw water qualities, chemical dosages were changed six times during Run 1. Therefore, Run 1 was further divided into seven sub-periods (Periods 1-1–1-7) based on the chemical dosages adopted in each run (Table 1). In Run 1, Periods 1-1, 1-2, and 1-7 were performed in a rainy season.

Measures for controlling membrane fouling during high-color periods

As discussed below, the results obtained in Run 1 revealed that membrane fouling became severe when the value of color in the raw water increased. An additional continuous experiment (Run 2) was therefore performed to investigate measures for controlling membrane fouling during high-color periods. The entire experimental period of Run 2 was dry-season. Therefore, the hybrid MF system was operated in the absence of the IE process to simulate the effluent of the IE process during high-color periods in Run 2. The water qualities in Run 2 are also summarized in Table 1. As discussed later, the worst situation with regard to the raw water qualities was simulated in Run 2. Therefore, it was not necessary to continue this run throughout the year. As a result, the experimental period of Run 2 was apparently shorter than that of Run 1.

In Run 2, the pilot-scale hybrid MF system was operated with six membrane elements submerged in a membrane separation tank with an effective volume of 1.55 m^3. The influence of the difference in the number of membrane

Table 1 | Averages of chemical dosages in each period

Run	Period	Initiation	Termination	PACl	NaClO
Run 1	1-1	Aug. 23, 2013	Sept. 18, 2013	341 ± 59	9.3 ± 3.2
	1-2	Sept. 19, 2013	Nov. 29, 2013	169 ± 27	10.8 ± 3.5
	1-3	Nov. 30, 2013	Dec. 27, 2013	107 ± 23	5.7 ± 2.3
	1-4	Dec. 28, 2013	Feb. 3, 2014	116 ± 11	4.1 ± 1.0
	1-5	Feb. 4, 2014	Apr. 11, 2014	66 ± 23	4.8 ± 2.4
	1-6	Apr. 12, 2014	May 16, 2014	75 ± 14	17.4 ± 9.7
	1-7	May 17, 2014	Jun. 22, 2014	56 ± 10	14.3 ± 8.5
Run 2	2-1	Jan. 23, 2015	Feb. 13, 2015	275 ± 37	10.0 ± 1.8
	2-2	Feb. 14, 2015	Mar. 13, 2015	229 ± 58	13.7 ± 4.8

Dosages are expressed as average ± standard deviation. PACl dosages are measured in mg/L; sodium hypochlorite dosages are measured in available chlorine.

elements on the treated water quality was likely to be insignificant because both the pre-chlorination and coagulation processes were designed based on the same criteria. On the other hand, the difference in the number of membrane elements may affect the development of membrane fouling. Because the experimental apparatus used in Run 2 had closer structure to a real treatment plant than the one used in Run 1, with regard to the development of membrane fouling, the data obtained in Run 2 are thought to be more realistic than those obtained in Run 1. The intermittent aeration cycle was changed to 45 sec-aeration/135 sec-stop. This alternation did not change the net air-flow rate compared with the operating conditions in Run 1. In Run 2, the pilot-scale hybrid MF system was operated with different physical cleaning cycles: 20 min-filtration/80 sec-backwashing/20 sec-relaxation in Period 2-1 and 15 min-filtration/60 sec-backwashing/15 sec-relaxation in Period 2-2. At the initiation of Run 2, CEB was performed (all six membrane elements were cleaned at the same time) to eliminate the membrane fouling developed prior to this run.

Analytical methods

The color of water was measured using a digital colorimeter (WA-PT-4DG, Kyoritsu Chemical-Check Lab., Corp., Tokyo, Japan) at a wavelength of 390 nm. Manganese concentration was determined by the 1-(2-pyridylazo)-2-naphthol PAN method (HACH, Loveland, USA) using a HACH spectrometer (DR2000). The SDI was measured in accordance with the method provided by the American Society for Testing and Materials (ASTM D4189–95).

RESULTS AND DISCUSSION

Performance of the hybrid MF system incorporating IE process

Table 2 lists the qualities of raw water, IE effluent, and treated water. The raw water quality varied significantly during the continuous experiment. Both color and dissolved manganese concentration in the raw water increased during the rainy season at the beginning of Run 1 (Periods 1-1 and 1-2). The significant variation of color in the raw water was partially alleviated by the IE process. However, the value of color determined for the effluent of the IE process also moderately increased during the rainy season. The increase in dissolved manganese concentration in the raw water was almost directly reflected that of the IE (anion exchange) effluent. Despite such significant changes in the qualities of IE effluent, the treated water qualities generally satisfied or at least were comparable to the targets of the treated water quality as a result of the adjustment of chemical dosages. In addition to the Periods 1-1 and 1-2, Period 1-7 was also assigned to a rainy season and, therefore, the average values of color in the raw water and the effluent of the IE process were higher than those in the previous period (Period 1-6). Nevertheless, we intentionally reduced the chemical dosages to investigate the effect of reducing chemical dosages on treated water quality. As a result, the color of the treated water in this period was higher than those in the other periods, indicating that the selection of appropriate chemical dosages is critically important to achieve sufficient treated water quality in the hybrid MF

Table 2 | Characteristics of raw water (RW), IE effluent (IEW) and treated water (TW)

Period		1-1	1-2	1-3	1-4	1-5	1-6	1-7	2-1	2-2
Color[a] (Pt-Co)	RW	61.3 ± 6.7	52.5 ± 7.9	38.3 ± 2.0	31.4 ± 1.7	30.6 ± 2.7	34.2 ± 6.7	37.1 ± 4.1	28.8 ± 3.2	26.8 ± 2.1
	IEW	29.6 ± 9.4	31.4 ± 9.6	20.5 ± 2.7	15.1 ± 2.6	11.8 ± 3.0	12.6 ± 3.5	17.7 ± 4.9	N.A.	N.A.
	TW	4.4 ± 1.4	4.9 ± 2.1	5.5 ± 1.6	4.0 ± 1.0	4.4 ± 1.5	5.1 ± 1.7	7.1 ± 3.5	3.8 ± 0.7	3.9 ± 1.6
	n	14	30	15	16	29	19	20	13	12
Mn[b] (mg/L)	RW	0.19 ± 0.11	0.09 ± 0.09	0.05 ± 0.05	0.01 ± 0.01	0.02 ± 0.01	0.05 ± 0.02	0.10 ± 0.08	0.03 ± 0.05	0.02 ± 0.01
	IEW	0.15 ± 0.07	0.09 ± 0.09	0.03 ± 0.04	0.01 ± 0.01	0.03 ± 0.03	0.04 ± 0.05	0.08 ± 0.07	N.A.	N.A.
	TW	0.03 ± 0.03	N.D.	N.D.	N.D.	0.00 ± 0.01	0.01 ± 0.02	0.02 ± 0.04	N.D	N.D
	n	9	9	9	5	10	10	14	12	12
SDI (−)	TW	4.6 ± 0.6	4.5 ± 0.7	4.2 ± 0.7	3.7 ± 0.1	4.4 ± 1.0	4.1 ± 0.6	3.8 ± 0.7	3.8 ± 0.3	3.9 ± 0.5
	n	7	21	12	11	19	9	9	9	9

n: number of measurements. Values and concentrations are expressed as average ± standard deviation.
[a]True color.
[b]Dissolved form, N.D. is not detectable. N.A. is not analysed.

system proposed. The values of the SDI of the treated water suggest that this water can be directly applied to a polishing process using a RO membrane, because some RO membranes can be operated under SDI values between 4 and 5. Based on these results, it can be said that the hybrid MF system is capable of stably producing high-quality industrial water despite significant seasonal variation in raw water quality as long as the chemical dosages are appropriately adjusted.

Coagulant dosages in membrane-based water treatment processes utilizing PACl as coagulant reported in previous studies range from 6 to 94.5 mg/L as PACl (1 mg/L of Al roughly corresponds to 18.9 mg/L as PACl) (Kimura *et al.* 2008; Wray *et al.* 2016; Kimura & Oki 2017). The coagulant dosages adopted in the continuous experiment utilizing the IE process as pretreatment carried out in a dry season (Periods 1-3 to 1-6) were in the range or slightly exceeded the upper limit of the range of previously reported coagulant dosages mentioned above. On the other hand, substantially higher coagulant dosages were required to satisfy the targets of treated water in the continuous experiments carried out in a rainy season (Periods 1-1 and 1-2) even though the pretreatment utilizing the IE process was performed in these periods. The primary purpose of injecting sodium hypochlorite was oxidizing dissolved manganese to facilitate its removal in an MF process. In the chlorination process of surface water, hypochlorite ion injected would also be consumed by organic matter and/or suspended solids contained in raw water. The actual sodium hypochlorite dosages adopted in the continuous experiment (Table 1) was required to ensure sufficient removal of dissolved manganese in the hybrid MF system proposed.

On the other hand, the development of membrane fouling was significantly affected by the seasonal variation in the raw water quality. Figure 1 shows the changes in TMP recorded in Run 1. In Figure 1, the changes in color of raw water and the coagulant dosage are also presented. During Run 1, CEB and CIP were performed 3 and 2 times, respectively. As explained previously, only acid cleaning solutions were used in these chemical membrane cleanings. Because membrane permeability was almost completely restored by these acid chemical cleanings, membrane fouling developed in this continuous operation was mainly caused by inorganic matter, unlike in previous studies in which organic matter dominantly contributed to the development of membrane fouling (Yamamura *et al.* 2007; Zondervan & Roffel 2007). During Periods 1-1 and 1-2 (rainy season), the rate of increase in TMP was significantly higher than in the other periods. By visual inspections, browny sludge, which is thought to be inorganic sludge, significantly accumulated in the membrane element. The values of color in the raw water in these periods were apparently higher than those in the other periods. To achieve sufficient color reduction, the coagulant dosages were increased during these periods. The increase in sludge production associated with high coagulant dosage was likely

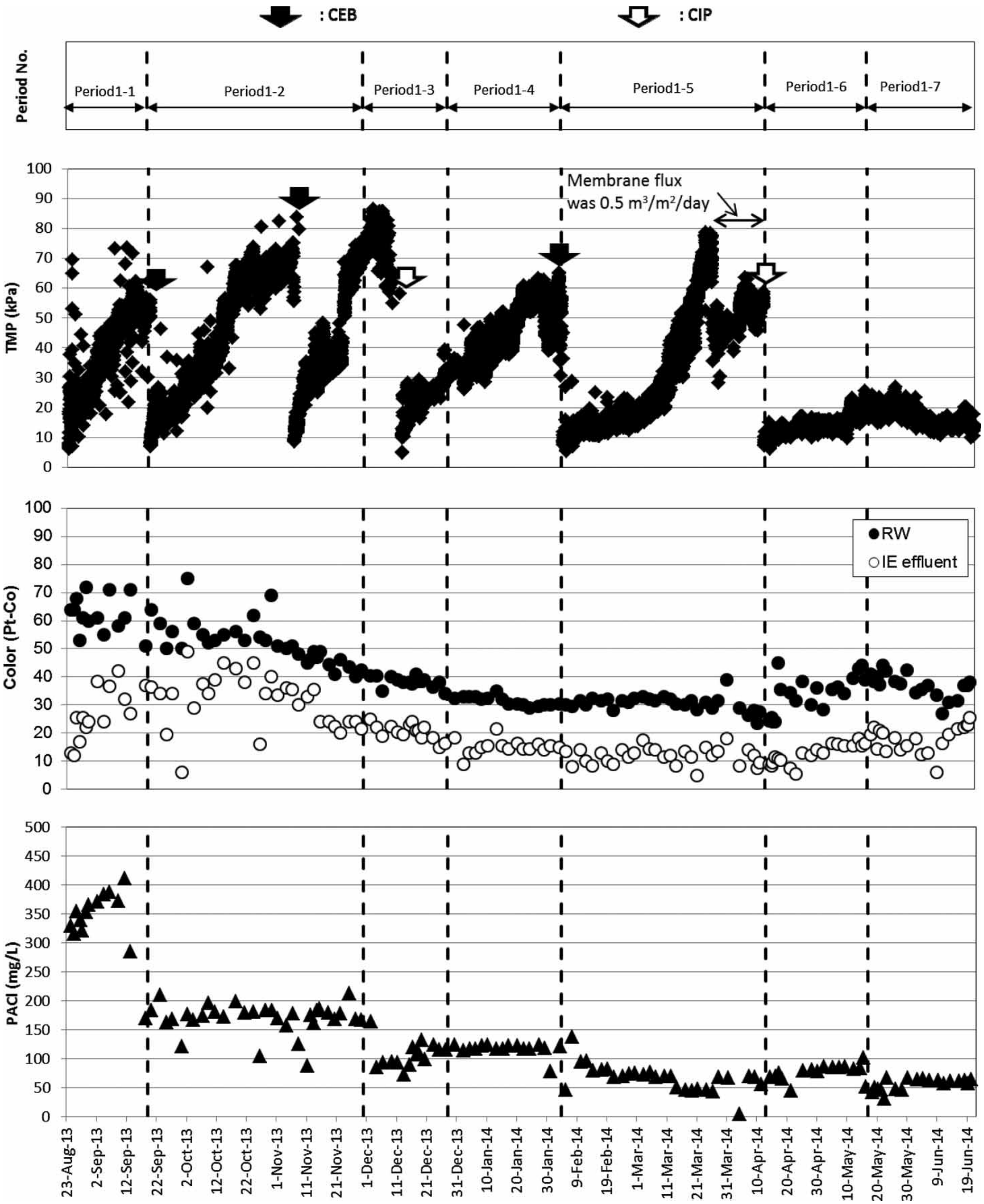

Figure 1 | Changes in TMP, color of raw water and IE effluent (provided as true color), and coagulant dosage in Run 1.

to accelerate the development of membrane fouling. Establishing strategies for controlling membrane fouling in the high-color period is important for stable operation of the hybrid MF system in a region, like Thailand, where the raw water has high color.

The rate of increase in TMP increased again in the middle of March (dry season). By a visual inspection, it was suggested that this rapid increase in TMP is attributed to ferric or manganese oxides due to a failure in manganese removal in the hybrid MF system. One possible explanation for the failure in manganese removal may be an insufficient accumulation of manganese oxide in the membrane separation tank. After the recovery cleaning performed on April 12th, 2014, the rate of increase in TMP became quite low, suggesting that the development of membrane fouling during the dry season can be controlled unless undesirable upset in the removal of dissolved manganese happens. Controlling sludge discharge could be one possibility to keep manganese removal efficiency when the accumulation of manganese oxide in the membrane separation tank is insufficient.

Strategies for controlling severe membrane fouling in high-color periods

As mentioned above, the results obtained in Run 1 revealed that the treated water of the hybrid MF system could satisfy the target of treated water quality as long as chemical dosages are appropriately adjusted. However, these results also indicate that controlling membrane fouling in a high-color period is critically important for stably operating the hybrid MF system in an area where there is a rainy season, like in Thailand. Therefore, we performed an additional continuous experiment (Run 2) to investigate strategies for controlling membrane fouling in high-color periods. As mentioned previously, the coagulant dosages in Periods 1-1 and 1-2 were substantially higher than those reported in previous studies (Kimura *et al.* 2008; Wray *et al.* 2016; Kimura & Oki 2017). Therefore, investigation on strategies for controlling membrane fouling in the operation of a submerged membrane filtration process in such high coagulant dosages is likely to give us important knowledge required to design and operate a submerged membrane filtration process treating highly-colored surface water. To simulate the IE effluent in the rainy season, we omitted the IE process in Run 2. The chemical dosages and the water qualities in Run 2 are also summarized in Tables 1 and 2. The average values of color in the raw water in Run 2 were comparable to those of the IE effluent in Periods 1-1 and 1-2 of Run 1 (Table 2). Therefore, high coagulant dosages comparable to Periods 1-1 and 1-2 (Table 1) were required to achieve sufficient color reduction in Run 2. Owing to the high chemical dosages, the treated water quality in Run 2 completely satisfied the targets of treated water quality. Therefore, it can be said that the operating situations in a high-color period of the hybrid MF system incorporating IE process, which was the worst situation with regard to the raw water quality, were successfully reproduced by omitting the IE process in the dry season (from January to April). After comparison of the experimental results obtained in Periods 1-5–1-6 (the dry season in Run 1) and Run 2 (Table 1), it is obvious that the coagulant dosage required to achieve comparable treated water quality can be significantly reduced by incorporating the IE process in the hybrid MF system. This point is one of the most important advantages of this combination.

The values of SDI determined in Run 2 (Period 2-1: 3.8 ± 0.3, Period 2-2: 3.9 ± 0.5) were slightly lower than those in Run 1. This improvement could be attributed to the increase in the coagulant dosage. These results suggest that further reduction in SDI of the treated water of the hybrid MF system is possible by optimizing coagulation conditions.

As mentioned above, evaluating the effectiveness of a fouling control strategy based on the experimental data obtained in Run 2 is thought to be valid for considering operating conditions in high-color periods. Figure 2 shows the change in TMP in Run 2. In Period 2-1, intermittent aeration cycle and backwashing duration were changed from Run 1. The fraction of aeration time in the intermittent aeration cycle in Run 2 (25%) was the same as that adopted in Run 1. The rate of increase in TMP during Period 2-1 was approximately 1.17 kPa/day and the operation did not seem to be stable. In Period 2-2, the membrane filtration cycle was changed (frequency of backwashing and relaxation was increased and backwashing duration was decreased). The intermittent aeration cycle in Period 2-2 was the same as that in Period 2-1. This change was very effective for

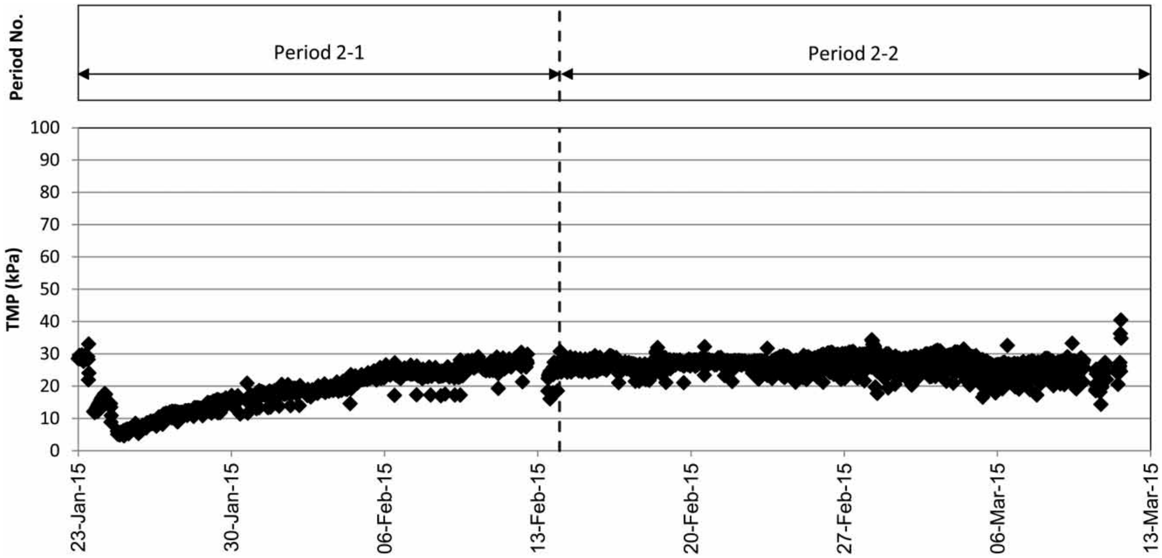

Figure 2 | Change in TMP during Run 2.

controlling membrane fouling, as the increase in TMP during Period 2-2 was marginal. These results indicate that implementing backwashing and relaxation with higher frequencies is a suitable approach for controlling severe membrane fouling in a high-color period. Taking into account the negligible increase in TMP during Period 2-2, it is suggested that the hybrid MF system can be stably operated even in a high-color period by selecting appropriate operating conditions.

In contrast, the changes in the intermittent aeration cycle and backwashing time in a single membrane filtration cycle were not likely to be influential factors on the development of membrane fouling. This fact implies that there is room for further energy saving by optimizing the aeration or backwashing schemes. For example, the results obtained in Period 2-2 revealed that an intermittent aeration cycle of 45 sec-aeration/135 sec-stop is totally sufficient for stable operation of the hybrid MF system. Applying this intermittent aeration cycle results in 75% reduction in energy consumption associated with the air-blower compared with the operation with continuous aeration. If the time fraction of aeration in an intermittent aeration cycle can be reduced further, the energy consumption associated with air-blower can also be reduced further. Further investigation of this issue is important to strengthen the economic competitiveness of the hybrid MF system proposed.

CONCLUSIONS

In this study, we investigated the performance of a hybrid MF system incorporating an IE process for producing high-quality industrial water from a surface water, occasionally containing high concentration of colored NOM. The treated water from the proposed treatment system generally satisfied the targets of treated water quality (color: no more than 5 Pt-Co, dissolved manganese: no more than 0.05 mg/L, and SDI: no more than 5). This result suggests that the treatment system proposed is capable of producing high-quality industrial water, which is suitable for washing purpose and could be directly applied to a polishing process using RO membrane. The application of a IE process was effective for reducing the coagulant dosage required for achieving sufficient color reduction. However, the value of color in the effluent of the IE process increased when there was a significant increase in the raw water color. In such periods, an increased dosage of coagulant was required to maintain the color of treated water at an acceptable level. The increase in coagulant dosage accelerated the development of membrane fouling through an increase in the production of inorganic sludge. Nevertheless, the severe membrane fouling during high-color periods can be successfully controlled by increasing frequency of backwashing and relaxation in a membrane filtration cycle. Based on the results obtained, it can be concluded that a hybrid MF system incorporating an IE process is capable of stably producing high-quality industrial water even if raw water is occasionally highly colored.

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