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Optimization of Dead-end Membrane Filtration to Treat Tunneling Wastewater using the Sustainable Flux

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Abstract: The first object of this study is to confirm the feasibility of sustainable flux in a dead-end mode. The second object is to identify the performance of the MF system operating under sustainable flux and simultaneously using the sustainable flux as a control parameter to evaluate the effects of pre-coagulation via comparing the different operating conditions such as various ranges of the coagulant dose, various pH of the raw water, and comparing the effects of online coagulation with conventional coagulation conditions that with sedimentation. According to the experimental results, the feasibility of sustainable flux in dead-end microfiltration (DEMF) was confirmed. In addition, it was observed that the pH control was the most effective method to improve membrane performance compared with other strategies such as pre-coagulation, and sedimentation in case the raw water with preexistent high concentration metal ions. Relative long-term experiments also confirmed the feasibility of the sustainable flux and showed the importance of backwash to improve the membrane performance.

Keywords: Optimization, sustainable flux, dead-end microfiltration, pretreatment

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INTRODUCTION

Low-pressure membrane filtration-microfiltration (MF) and ultrafiltration (UF)—have been widely used in recent years as an alternative to conventional drinking water treatment to satisfy the requirements of increasing demand for water supply and more stringent water quality regulations. MF has been known to be effective for removal of particulates in macromolecule size range, and it has been primarily employed in the concentration of emulsions, the removal of bacteria and protozoa, and the removal of particulate turbidity or clarification (1). But it is poor for the removal of dissolved organic materials that generally were considered as the main fouling materials. Membrane fouling is the main impediment to constrain the further application of membrane filtration. According to previous studies (2–4), the fouling mechanisms in various membrane technologies such as MF, UF, NF, and RO are of different types and characters. In the MF process, it mainly can be attributed to internal pore fouling rather than concentration polarization and surface fouling due to adsorption and gel layer formation. To ameliorate this problem, variety strategies have been investigated. They include:

1. Pretreatment such as pre-coagulation, powdered activated carbon (PAC), granular activated carbon (GAC), and ozonation
2. Periodical direct cleaning of membranes
3. Optimization of operational conditions, such as operation under critical flux, critical transmembrane pressure (TMP) condition, intermittent suction operation
4. Developing hydrodynamic controls to provide surface shear such as cross-flow circulation, vibration of membrane system, applying vigorous bubbles, etc.

The effects of coagulation on membrane performance have been extensively studied in previous researches. Ben Aim's group (5–9) demonstrated that the coagulation-membrane filtration hybrid process has better performance both in removing NOM and reducing membrane fouling than the unit membrane process. But the inconsistent results still existed. Judd and Hillis (10) investigated the optimization of the coagulation-MF hybrid process and noted that the effect of coagulant dose is much more significant than a change in pH in a certain range and a low coagulant dose seemed to have a slightly detrimental effect on membrane performance. In contrast, Choi and Dempsey (11) presented that an acidic under-dosed condition improved UF performance in both water quality and fouling control based on an investigation of in-line coagulation with UF process for a range of conditions such as acidic under-dosed, alkaline under-dosed, charge neutralization, and sweep-floc. Furthermore, hydraulic cleaning of the membrane resulted in a better recovery of the membrane performance for the under-dosed conditions.

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So, then object of this study is to confirm the effects of different coagulation conditions on membrane performance.

The correlation between imposed flux and fouling has been investigated in much previous research (12–31) and it can generally be described via the concept of critical flux. Field et al. (12) introduced the concept of the critical flux for cross-flow microfiltration (CFMF) firstly by demonstrating that there is a permeate flux below which fouling will not be observed and above which fouling will occur. However, this definition has been shown to be more applicable in the theoretical viewpoint rather than the real application. Choi et al. (13) confirmed that there is perceptible fouling existence during filtration of natural water, even operating the system under considerable low flux (sub-critical flux). This encourages the appearance of the idea of "sustainable flux" (14, 22). The term "sustainable flux" was used in this paper to describe the weak form of critical flux (15, 16) that there is flux if operating above it the membrane system will have a sharp fouling increase rate ($\Delta TMP/\Delta t$) and operating below it the membrane system will have a much slower fouling increase rate that is sustainable in real practices. Zhang et al. (32) investigated the effects of pH, ionic strength, and temperature on sustainable flux based on a statistical analysis. The results demonstrated that the operating temperature and mixture properties (pH and ionic strength) all had statistically significant effects on the sustainable flux and the temperature was the most crucial parameter among them. Metsammuren et al. (33) also reported that there is an increase in the weak form of critical flux from 60 to 105 $Lm^{-2}h^{-1}$ as pH is increased from 7 to 8. Many other studies (34–37) were undertaken to evaluate the effects of the hydrodynamic condition and membrane properties on critical flux. But there is a rare study, which in a comprehensive viewpoint investigated the effects of pretreatment on sustainable flux. The current study tries to compensate this deficiency and identify the optimal pretreatment condition using sustainable flux as a control parameter.

On the other hand, Bessiere et al. (17) presented that critical flux cannot be observed if the critical factor is a critical wall shear stress. But a "critical filtered volume" has been found instead of it. However, considering dispersive forces such as those of Brownian diffusion, surface interaction, which are crucial parameters correlated with critical flux, still existed in the dead-end mode, there is a theoretic possibility that critical flux or sustainable flux can be found in the dead-end mode. Due to this hypothesis, to confirm whether the concept of critical flux that deduced from CFMF will still be applicable under the dead-end microfiltration (DEMF) condition is another object in this study.

In summary, the first object of this study is to confirm the feasibility of the sustainable flux in the dead-end mode. The second object is to identify the performance of the MF system operating under sustainable flux and simultaneously using it as a control parameter to evaluate the effects of pre-coagulation via comparing the different operating conditions such as

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various ranges of the coagulant dose, various solution pH, and comparing the effects of online coagulation with conventional coagulation conditions that with sedimentation.

MATERIALS AND METHODS

DEMF System

Polyvinylidene fluoride hollow fiber membrane (PVDF) with an average pore size of 0.1 μm and 17.3 cm^2 effective filtrated area was used throughout. A single hollow fiber was sealed by epoxy resin into a plastic cylindrical casing with 6 mm inner diameter to form a DEMF module.

The DEMF system was installed vertically (Fig. 1). Constant flow was pumped into the membrane module via a variable speed peristaltic pump. The increment of the transmembrane pressure (TMP) were recorded every 3 minutes by a pressure gauge ISE 40-01-22 and transferred to the computer by data acquisition Agilent 34970A. A hotplate and magnetic stirrer was used to ensure the supply of the homogeneous feed solution during membrane filtration. The system was automatically controlled via the programmable logic controller (PLC).

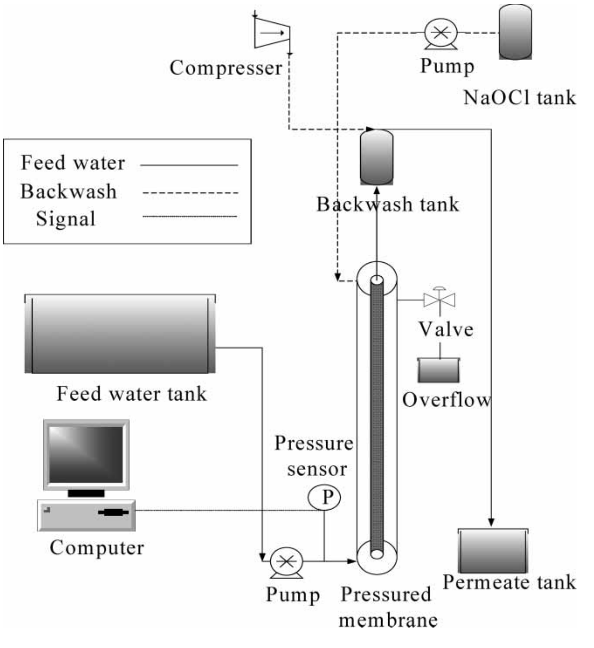

Figure 1. Schematic diagram of the bench-scale DEMF system used in this study.

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Raw Water

The effluent came from the wastewater treatment plant treating tunneling wastewater that was produced during the drilling operation. The raw water used in this study was collected at the pre-sedimentation basin. It was characterized in terms of the pH (pH meter 410A), UV-254 (Shimadzu UV-2101 UV/Vis Spectrophotometer), DOC (Total organic carbon analyzer, Multi N/C 300), turbidity (ASI-5000A Autosampler, Dr.mini DMT 110), metal ions (X-Ray Fluorescence Spectrometer, PW2400), and SUVA (the specific ultraviolet absorbance divided by the concentration of DOC) as an indication of hydrophobicity. Table 1 showed the detailed characteristics of the raw water. The SUVA value is 9.5 here. According to Pikkarainen et al. (15), if the SUVA value is higher than 4 then it means that the aquatic humics existing in the water mostly consisted of high hydrophobicity and high molecular weight (MW) substance, such as humic and fulvic acids, high MW alkyl monocarboxylic and aromatic acids, proteins, and hydrocarbons etc. Also, a relatively high concentration of metal ion includes Fe, Al, Mn, Ca were observed in this raw water.

Experimental Procedure and Methods

Alum was used as a coagulant in this study. The unit was mg/L as $Al_2(SO_4)_3 \cdot 14H_2O$ (MW = 594 g/mole). Jar tests were conducted in a standard 6-position gang stirrer (Programmable Jartester, Phipps, and Bird, Richmond, VA) with 2-L square acrylic Gator jars. During coagulation, rapid mixing was given for 2 minutes at 150 rpm, which was followed by slowly mixing for 15 minutes at 30 rpm. Samples were taken from 5 cm above the jar bottom after the addition of the coagulant and after 60 minutes of quiescent setting, respectively. The final pH was adjusted to 7 and 5 by the addition of 0.1 N sodium hydroxide (NaOH) and hydrochloric acid (HCl) to the solution.

Table 1. Characteristics of the raw water over the experimental period

Parameters	Range	Average
pH	10.78 ~ 10.82	10.8
UV-254 (cm^{-1})	0.276 ~ 0.282	0.279
DOC (mg/L)	2.90 ~ 2.94	2.92
SUVA (UV254/DOC) × 100	—	9.55
Turbidity (NTU)	84.2 ~ 89	86.6
Alkalinity (mg/L as $CaCO_3$)	147.1 ~ 152.3	149.7
Total Mg (mg/L)	0.295–0.677	0.486
Total Al (mg/L)	1.692 ~ 5.033	3.3625
Total Ca (mg/L)	2.075 ~ 13.270	7.6725
Total Fe (mg/L)	0.004 ~ 1.135	0.5695

In this study, all the experiments were conducted in the constant flux mode. The protocol for the sustainable flux determination was the "flux-step" method that operation in a range of fluxes changed from 30 $Lm^{-2}h^{-1}$ to 200 $Lm^{-2}h^{-1}$ (30, 50, 72, 80, 120, 200 $Lm^{-2}h^{-1}$). Each flux was kept constant for 30 minutes, which is consistent with the most practical applications that have a backwash every half hour. To completely avoid the pre-operation effects, a virgin membrane fibers was used in each test. To reduce an experimental error, only the membrane fibers, which the deviation of initial TMP was less than 5% with deionized water, were used and each test was repeated at least three times. The water quality was measured after coagulation, sedimentation, and after being filtrated through the membrane.

RESULTS AND DISCUSSION

Jar Tests Results

The typical relationship between residual turbidity, and UV_{254}, and the alum dose was shown in Fig. 2.1–2.3. When coagulation was performed without the pH adjustment (Fig. 2.1), the turbidity removal did not change significantly up to 120 mg/L. The residual turbidity was sharply reduced (removal rate around 98%) when the coagulant dose was higher than 140 mg/L. A further coagulant dose could not improve the turbidity removal rate. UV_{254} The removal rate had the similar trend compared with the turbidity removal. The highest removal rate (85%) occurred at 140 mg/L of the coagulant and further addition did not help to increase the UV_{254} removal rate.

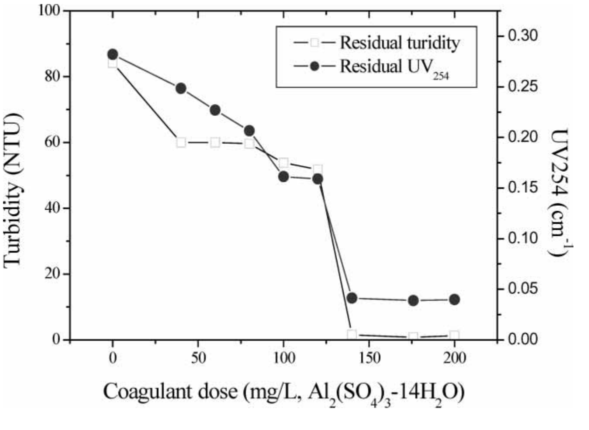

Figure 2.1. Residual turbidity and UV254 as a function of coagulant dose without pH control.

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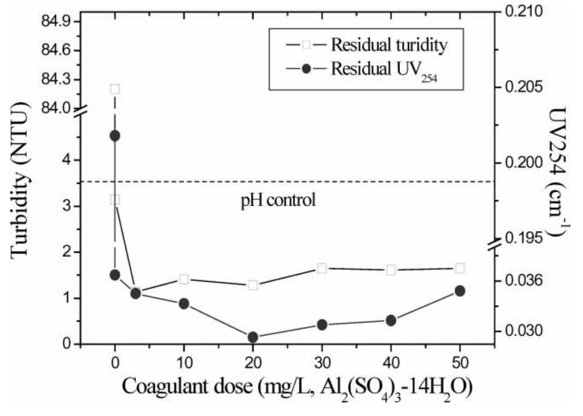

Figure 2.2. Residual turbidity and UV254 as a function of coagulant dose at pH 7.

When coagulation was performed with pH adjustment to 7 (see Fig. 2.2), even without adding the coagulant, the turbidity removal rate reached 96.3%. There is a slight improvement (98.6%) in the turbidity removal rate at 3 mg/L of alum. But further addition did not help the turbidity removal. The UV_{254} removal rate reached 81.8% after the pH was adjusted to 7. The highest removal rate (85.5%) occurred when adding 20 mg/L of the coagulant. Further addition deteriorated the UV_{254} removal rate.

When coagulation was performed with pH adjustment to 5 (see Fig. 2.3), similar to the above situation, the turbidity removal rate reached 77.3% even without adding the coagulant. As the coagulant dose was increased, the turbidity removal rate improved, and reached 91% at 20 mg/L. However, further addition deteriorated the removal (75.3% at 30 mg/L and

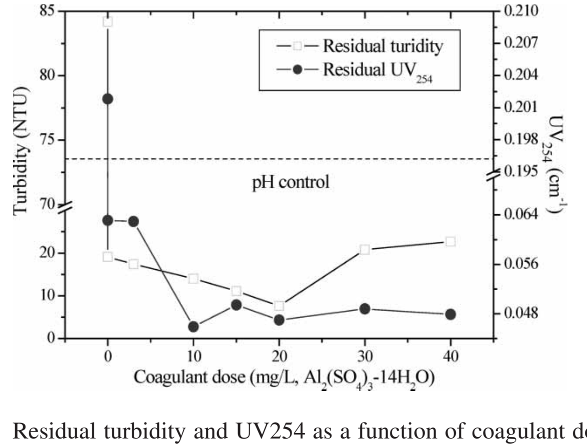

Figure 2.3. Residual turbidity and UV254 as a function of coagulant dose at pH 5.

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73.4% at 40 mg/L). The highest UV_{254} removal rate (77.3%) occurred at 10 mg/L of the coagulant. Further addition deteriorated the UV_{254} removal rate.

To identify the coagulation mechanisms, the contour diagram of zeta potential (ZP) and the percentage removal of the turbidity as a function of the aluminum dosage and the pH were developed (Fig. 3.1 and Fig. 3.2). Figure 3.1 showed two zones with more than 90% removal of the turbidity. Zone 1, which was located in the range of about pH 5, represented the charge-neutralization condition because the corresponding ZP value is approximately zero (−5 to +5 mV) in this region (see Fig. 3.2). It is well-known that the colloids or particles re-stabilize when the ZP value is higher than +5 mV unless a voluminous or sweep-floc is formed. The majority of zone 2 was located in the positive ZP region (see Fig. 3.2) with the pH range from 5.5 to 8. Since the aluminum hydroxide precipitate could form in this region, it was considered as the sweep-floc zone. Similar figures have been used by Lee et al. (18). The major difference from them was the location of these zones. Figure. 3.1 and Fig. 3.2 showed that there was a high removal rate of turbidity (more than 90%) with a pH adjustment from 5 to 7 even without or with a very small quantity of the coagulant. High concentration of preexisted aluminum in the raw water was suspected to cause the excellent turbidity removal without the coagulant (Table 1).

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Raw Water Sustainable Flux Test

First, permeability of the raw water through DEMF was evaluated. The result based on this test can be used as a control parameter for comparing and

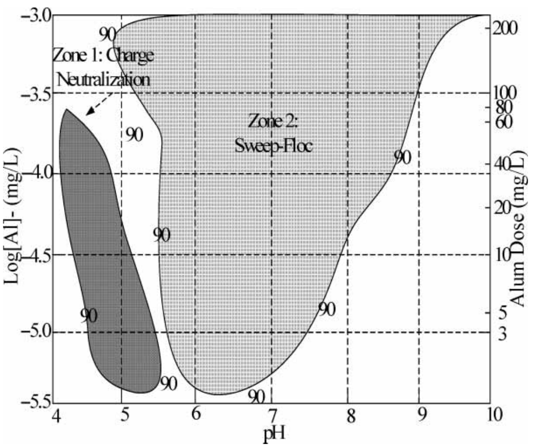

Figure 3.1. Contour diagram of percentage of turbidity removal as a function of alum dosage and pH.

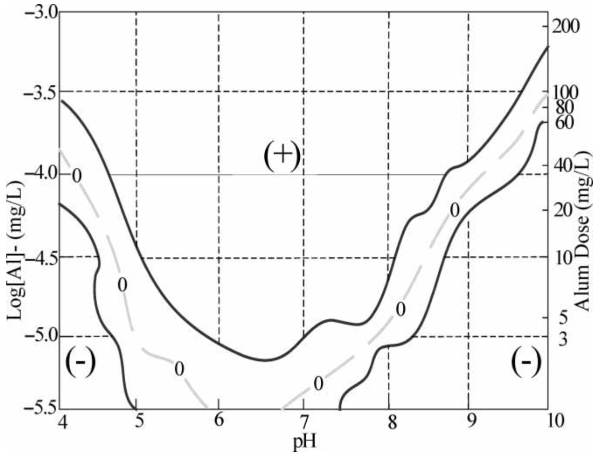

Figure 3.2. Contour diagram of zeta potential (ZP) as a function of alum dosage and pH.

evaluating the effects of other parameters. The experimental results, which were based on the "flux-steps" protocol, were shown in Fig. 4.1–4.3. Figure 4.1 showed the change in TMP as a function of flux. According to Fig. 4.1, the TMP increase was insignificant at low fluxes. TMP increased significantly when the flux exceeded a certain value. The sustainable flux was estimated using the mean TMP versus flux shown in Fig 4.2 that is consistent with the protocols used by Chan et al. (19), Huisman et al. (20), and Choi et al. (13). The critical point was taken at the flux of 80 $Lm^{-2}h^{-1}$ in this study due

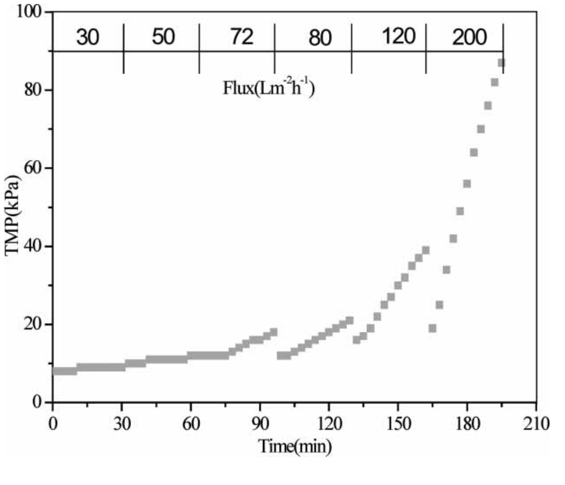

Figure 4.1. TMP as a function of operating time under different flux conditions.

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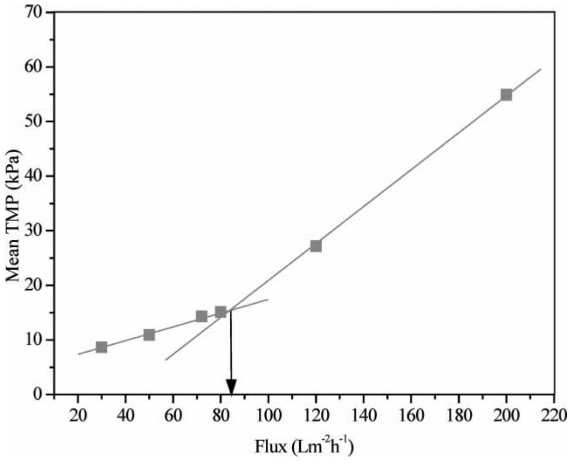

Figure 4.2. Mean TMP as a function of flux.

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to the phenomena of the TMP "jump" that occurred when the flux is higher than this point. The change of the TMP over time (ΔTMP/Δt) against permeate flux was also plotted, which confirmed the sustainable flux of 80 $Lm^{-2}\,h^{-1}$. Here, the ΔTMP/Δt (0.165 kPa/min) that under the flux of 80 $Lm^{-2}\,h^{-1}$ satisfied the statistical requirements suggested by Choi et al. (13) that f-test with $p = 0.05$ (non-zero slopes) or t-test with $p = 0.05$ (significant deviation from previous values for ΔTMP/Δt). The same methods were used to determine the sustainable flux throughout this study.

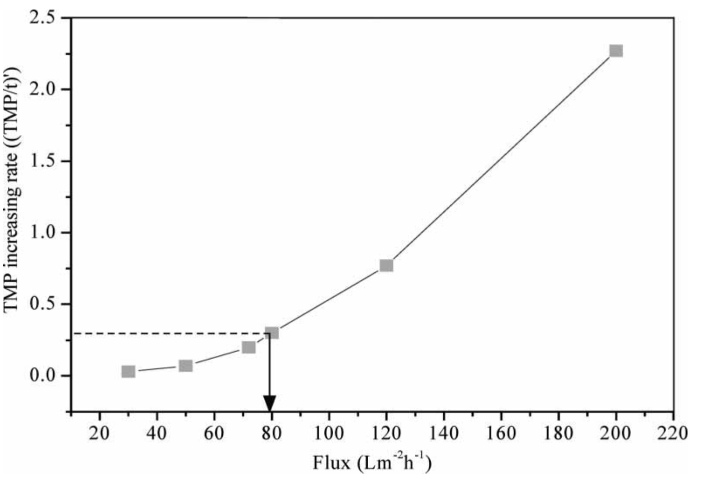

Figure 4.3. TMP increasing rate (ΔTMP/Δt) as a function of flux.

Table 2. Water qualities of the raw water, the settled, and the permeate through membrane

	Conditions	Feed Water		
		Raw water	After pH adjustment	
pH	Raw water	10.8	5.1	7.1
	Settled	10.8	5.5	7.3
	Permeate	10.6	5.7	7.5
Turbidity (NTU)	Raw water	89	49.5	36
	Settled	—	5.08	3.14
	Permeate	0.04	0.04	0.04
UV-254 (cm^{-1})	Raw water	0.281	0.060	0.056
	Settled	—	0.053	0.049
	Permeate	0.060	0.040	0.038

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Effect of pH Control on the Sustainable Flux

The pH of the raw water was adjusted to 5 and 7, respectively, by the addition of 0.1N hydrochloric acid (HCl) and sodium hydroxide (NaOH). The corresponding water characteristics and aluminum concentration in the raw water were presented in Tables 2 and 3. The sustainable fluxes were about 150 $Lm^{-2}h^{-1}$ under both conditions (Fig. 5.1 and Fig. 5.2). They were approximately two times higher than the sustainable flux of raw water. This improvement was mainly due to the amelioration of the water quality after the pH control. From Table 2, it can be seen that more than 44% of the turbidity was removed after the pH was adjusted to 5, and the removal improved to 90% after settling. Similar trends were observed for the UV_{254} removal. Generally, aluminium species in natural soil exist as two main configurations: alumina silicate that consist of feldspar, mica, kaolinite ($Al_2Si_2O_5(OH)_4$), alunite ($K_2Al_3(OH)_6(SO_4)_3$), and soluble aluminium that can be divided further as exchanged state, organic complex state, amorphous state, and crystal state. With the H^+ concentration increase, some aluminum ions were released from the alumina silicate via the displacement reaction. Those new Al^+ acted as coagulant and contributed to the water quality improvement.

Table 3. Aluminum concentration in the raw water before and after pH adjustment

Parameters	Total Al (mg/L)	Soluble Al (mg/L)
Raw water (pH 10.8)	1.692 ~ 5.033	0.947 ~ 1.058
After pH adjustment (pH 7)	1.842 ~ 4.992	0.276 ~ 0.801

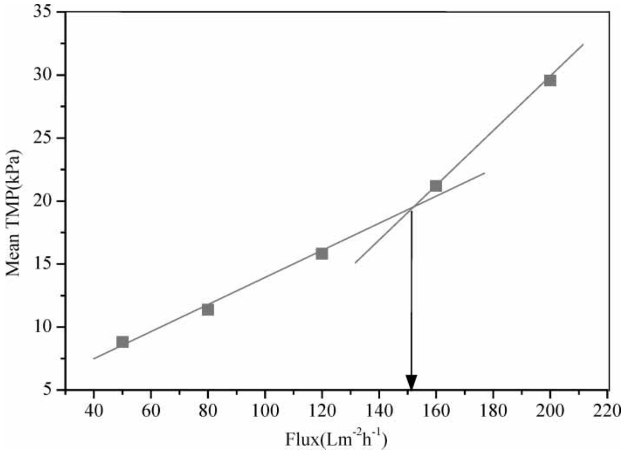

Figure 5.1. Mean TMP as a function of flux after the pH adjustment to 5.1.

Effect of Coagulation on Sustainable Flux

It was mentioned before that preexisted aluminum significantly improved the water quality after the pH control. Whether adding in more alum would help to improve the membrane performance was examined. Alum was added at 3, 20, and 200 mg/L. Table 4 clearly showed that further addition of alum failed to improve the membrane performance. The preexisted aluminum in the raw water was plenitudinous to induce coagulation.

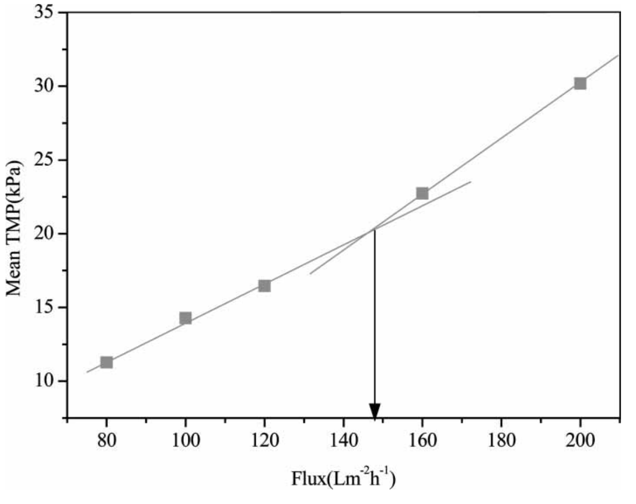

Figure 5.2. Mean TMP as a function of flux after the pH adjustment to 7.1.

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Table 4. The experimental results for further alum addition after the pH adjustment to 5.1

Coagulants dose (mg/L)	pH	Sedimentation	Sustainable flux ($Lm^{-2}h^{-1}$)	ΔTMP/Δt	Zeta potential (mV)
0	5.1	Without	150	0.165	−3.5
3	5.1	Without	150	0.165	−0.7
20	5.1	Without	130	0.165	1.2
200	5.1	Without	150	0.165	9.5

Effect of Sedimentation on Sustainable Flux

To examine the effects of sedimentation, two experimental groups were compared. The results were presented in the Table 5. According to Table 6, under group I condition the sustainable flux increased from 130 $Lm^{-2}h^{-1}$ without sedimentation to 219 $Lm^{-2}h^{-1}$ with sedimentation. Under group II condition, the sustainable flux increased from 150 $Lm^{-2}h^{-1}$ without sedimentation to 350 $Lm^{-2}h^{-1}$ with sedimentation. Those increase mainly due to the removal of the turbidity and UV-254 by sedimentation (See Table 2). It also can be seen that there was much more increase of sustainable flux under group II condition compared with the group II condition. This result suggested that sedimentation was more effective under sweep-floc condition.

Optimum Operating Conditions

The sustainable fluxes with different experimental conditions were compared in order to find the optimal operating conditions. There was not much difference between the sustainable fluxes at pH 5 and 7. However, since pH 7 was closer to the original pH value, it became the better operating condition. Once

Table 5. Effect of sedimentation

Conditions	Aluminum dosage (mg/L)	pH	Sedimentation	Sustainable flux ($Lm^{-2}h^{-1}$)	ΔTMP/Δt	Zeta potential
Group I	20	5.1	Without	130	0.165	1.2
	20	5.1	With	219	0.165	1.2
Group II	200	7.8	Without	150	0.165	10.6
	200	7.8	With	350	0.165	10.6

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Table 6. The long-term experimental operating condition

Conditions	Coagulation	Sedimentation	pH	Backwash	Flux ($Lm^{-2}h^{-1}$)	$\Delta TMP/\Delta t$
I	Without	Without	7	With	150 (sustainable flux)	0.035
II	Without	Without	7	With	200 (supra-sustainable flux)	0.104
III	Without	Without	7	Without	150 (sustainable flux)	0.190
IV	Without	Without	7	Without	200 (supra-sustainable flux)	0.416

the pH was adjusted, the coagulant addition was unnecessary. Adding in extra coagulant did not improve the membrane performance even at a very high dose. Sedimentation increased the sustainable flux especially under the sweep-floc condition. But due to the disadvantage of the large footprint, adding in this process in a real situation needs a comprehensive economic analysis. In summary, the unit membrane filtration process with the strategy of the pH control to 7 was considered as the optimal operating condition to treat the tunneling wastewater in this study.

Long-term Operation

Relative long-term experiment was then conducted in order to confirm the feasibility of the sustainable flux in DEMF operation. They were evaluated under four different conditions, as shown in Table 6. The results fairly confirmed the feasibility of the sustainable flux at DEMF operation. The TMP increase rate was 0.190 at the sustainable flux that was slightly higher than the value under short-term test. When the flux was adjusted to the supra-sustainable condition, the TMP increase rate sharply reached to 0.416. On the other hand, Fig. 6 compared the membrane performance at different operating conditions. It clearly showed the importance of backwash. Backwash was beneficial for the operation at the sustainable flux as well as at the supra-sustainable flux.

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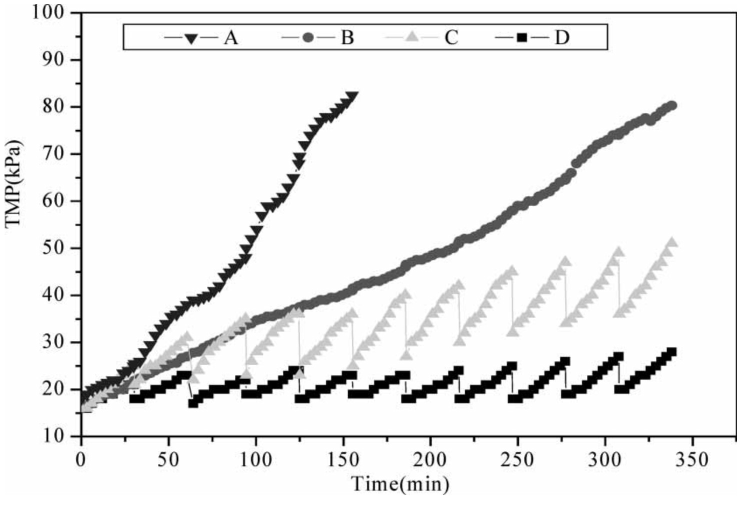

Figure 6. The TMP variation as a function of time under different operating conditions. (A) Operating under supra-sustainable flux condition without backwash. (B) Operating under sustainable flux without backwash. (C) Operating under supra-sustainable flux with backwash. (D) Operating under sustainable flux with backwash.

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CONCLUSION

The "*sustainable flux*" that originated from the term of "*critical flux*" was used as a control parameter to evaluate the performance of the hybrid membrane filtration process in this study. Based on the experimental results the subsequent conclusions were made:

a) This study confirmed the feasibility of the sustainable flux in DEMF mode.
b) Due to the raw water with high pH value, the maximum turbidity and UV_{254} removal needed a high addition of coagulant (140 mg/L) under without pH control procedure.
c) Due to the pre-existing aluminum in raw water, the pH control process effectively improved the water quality and the membrane performance.
d) The pre-existing aluminum in raw water was plenitudinous as coagulants and the addition of more dosage could not increase sustainable flux.
e) Sedimentation was more effective to improve membrane performance under the sweep-floc condition than the charge-neutralization condition.
f) The unit membrane filtration process with the strategy of pH control to 7 was considered as the optimal operating condition in this study.
g) Relative long-term experiments confirmed the feasibility of the sustainable flux at the DEMF mode and showed the importance of backwash under both the operating conditions.

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