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Role of photo-oxidation and adsorption at water back-flushing in hybrid water treatment of multi-channels alumina MF and PP beads coated with photocatalyst

Gyu Myung Gyeong, Jin Yong Park*

Department of Environmental Sciences & Biotechnology, Hallym University, Gangwon 200-702, Korea, Tel. +82 33 248 2152; Fax: +82 33 256 3420; email: jypark@hallym.ac.kr

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ABSTRACT

The effect of organic matters on membrane fouling was observed in a hybrid process of seven-channel alumina microfiltration (MF) and polypropylene (PP) beads coated with TiO_2 photocatalyst for advanced water treatment. As a result, the organic matter, like humic acid, could be one of the main factors affecting membrane fouling because the final resistance of membrane fouling decreased significantly due to reduction in humic acid concentration. The treatment efficiencies of turbidity were as high as 96.6–96.9% independent of humic acid concentration, but that of dissolved organic matters was the maximum, 65.4% at humic acid 6 mg/L. Treatment portions of membrane filtration, photocatalyst adsorption, and photo-oxidation were investigated by comparing the treatment efficiencies of (MF), (MF + TiO_2), and (MF + TiO_2 + UV) processes. The membrane fouling resistance was the minimum, and the final permeate flux and the total permeate volume were the maximum at (MF + TiO_2 + UV) process. It means that the photo-oxidation could control membrane fouling, but the photocatalyst adsorption did not participate to reduce membrane fouling in our hybrid water treatment process. The photo-oxidation and the adsorption by PP beads coated with photocatalyst could play a much greater role to remove organic matter than turbid materials.

Keywords: Microfiltration; Photocatalyst; Hybrid process; Ceramic membrane; Water treatment; Photo-oxidation

1. Introduction

Photocatalytic oxidation, a new type of water pollution control technology, with the characteristics of high efficiency, low energy consumption, and a wide range of application, can oxidize most organic compounds, especially non-biodegradable organic contaminants, by mineralizing them to small inorganic molecules. For this reason, photocatalytic oxidation technology has broad prospects for application. Among various semiconductor photocatalysts, there is a general consensus among researchers that TiO_2 is

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*Corresponding author.

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more superior because of its high activity, large stability to light illumination, and low price [1–4]. In photocatalytic degradation, two modes of TiO_2 application are adopted: (1) TiO_2 immobilized on support materials and (2) TiO_2 suspended in aqueous medium [5,6]. Application of TiO_2 in suspension instead of immobilizing the TiO_2 on solid carriers has shown an improvement in organic degradation efficiencies due to the uniform distribution and large specific surface area. However, classical solid–liquid separation processes, such as sedimentation, centrifugation, and coagulation used for separation of the fine TiO_2 particles (typically less than 1 μm), are not effective [7]. In addition to the low reutilization rate, there is also a chance of secondary pollution caused by fine TiO_2 particles in the effluent. Therefore, the recovery of the photocatalysts is one of the main concerns that affect its engineering application on a large scale. A lot of investigations have been conducted aiming at solving this problem [8–12].

In recent years, titanium microsphere has also been proposed as a means to recover TiO_2 photocatalyst. Generally, the core-shell structured TiO_2 microspheres, with a mesoporous surface made of nano-TiO_2, have low density, high specific surface area, and large size favorable for separation [13–16]. But the preparation method and operating conditions have great effect on particle morphology associated with photocatalysis, and many problems existing in the current preparation methods need to be solved by optimizing the process conditions or developing new ones [17].

Membrane separation process for separation and purification has been developed rapidly during the past few decades. It can simultaneously separate and concentrate all pollutants in water by the retention of its microspores without secondary pollution and phase change. In addition, with the advantage of low-energy consumption, its equipment are compact, easy to operate, and capable of continuous operation at room temperature [18]. However, membrane fouling due to the adsorption precipitation of organic and inorganic compounds onto membranes leads to a decrease in the permeate flux, an increase in membrane cleaning costs, and a reduction of the life of the membrane. Although considerable progress has been made in membrane fouling [19,20], techniques for controlling membrane fouling remain inadequate, which is the major obstacle in the successful implementation of membrane separation technology. The TiO_2 photocatalysis-membrane separation coupling technology emerged recently can solve the two problems mentioned above effectively [21]. The coupling technology not only keeps the characteristics and capacity of the two technologies, but also produces some synergistic effects to overcome the drawbacks of the single technology. On the one hand, the pollutants are oxidized by the photocatalysis, and the selected membranes show the capability not only to retain the photocatalyst, but also to reject partially organic species by controlling the residence time in the reacting system. In other words, the membrane is also a selective barrier for the molecules to be degraded, thus the coupling technology could enhance the photocatalytic efficiency and achieve excellent effluent quality. On the other hand, the coupling of photocatalysis and membrane separation could solve or alleviate the problem of flux decline associated with membrane fouling [22].

In this study, the effect of organic matters on membrane fouling was observed in a hybrid process of seven-channel alumina MF and PP beads coated with TiO_2 photocatalyst for advanced water treatment. The one hybrid module of ceramic MF membrane and the PP beads fluidized between the gap of ceramic membrane and the acryl module case was the first trial in the hybrid water treatment process field of MF and photocatalyst. Also, treatment portions of membrane filtration, photocatalyst adsorption, and photo-oxidation were investigated by comparing the treatment efficiencies of (MF), ($MF + TiO_2$), and ($MF + TiO_2 + UV$) processes. Periodic water back-flushing using permeate water was performed for 10 s per 10 min filtration to reduce membrane fouling.

2. Experiments

2.1. Materials

The multi-channel ceramic MF membrane (HC10) used in the study was coated with α-alumina on a supporting layer of α-alumina, and its pore size was 1.0 μm. We purchased the membrane from Dongseo Inc. in Korea, and the specifications of the multi-channel ceramic membrane were arranged in Table 1.

The photocatalyst used was 4–6 mm PP beads coated with TiO_2 powder made by Prof. Kim's group [23] using chemical vapor deposition method, which was presented in Table 2. Instead of natural organic matters (NOM) and fine inorganic particles in natural water source, a quantity of humic acid sodium salt (Aldrich) and kaolin (Sigma-Aldrich) was dissolved in distilled water. It was then utilized as synthetic water in our experiment. The humic acid was selected as an organic matter representative because it was known that humic acid was the major humic material of river or lake. Kaolin concentration was fixed at 30 mg/L and humic acid concentration was changed from 2 to 10 mg/L in the synthetic feed water to investigate the effect of organic matters. The humic acid

Table 1
Specifications of the multi-channel ceramic microfiltration (HC10) used in this study

Membrane	HC10
Pore size (μm)	1.0
No. of channels	7
Outer diameter (mm)	20
Inner diameter (mm)	4
Length (mm)	245
Surface area (cm^2)	215.4
Material	α-alumina coating on α-alumina support
Company	Dongseo Inc. in Republic of Korea

Table 2
Specification of the TiO_2-coated PP beads used in this study

Material of the beads	Polypropylene (PP)
TiO_2 coating method	Chemical vapor deposition
Diameter (mm)	4–6
Weight (mg)	21.8–48.3
Average weight (mg)	39.9

concentration was fixed at 6 mg/L for researching a portion of the treatment efficiencies of multi-channel ceramic MF, adsorption, and photo-oxidation. UV light with 352 nm was radiated from outside of the acryl module by two UV lamps (F8T5BLB, Sankyo, Japan) having 8 W power.

2.2. Hybrid membrane module

For eliminating the turbidity and NOM, the hybrid module was constructed by packing PP beads coated with TiO_2 powder between the module inside and outside of a ceramic membrane. In addition, 100 meshes (0.150 mm), which was extremely smaller than 4–6 mm particle size of the PP beads utilized here, was installed at the outlet of the hybrid module to prevent PP beads loss into the treated water tank.

2.3. Experimental procedure

The advanced water treatment system utilizing a hybrid module (6) of multi-channel ceramic microfiltration (MF) and PP beads coated with TiO_2 photocatalyst is demonstrated in Fig. 1, which was utilized at our previous study [24]. We performed cross-flow filtration for the multi-channel ceramic membrane and periodic water back-flushing utilizing permeated water. The hybrid module (6) filled up with 40 g/L of PP beads was coated with TiO_2 photocatalyst between the gap of ceramic membrane and the acryl module case, and it was installed in the advanced water treatment system. Then the feed tank (1) was filled with 10 L of prepared synthetic water composed of humic acid and kaolin, and the temperature of the feed water was constantly maintained by using a constant temperature circulator (3) (Model 1146, VWR, USA). Also, the synthetic feed water was continuously mixed by a stirrer (4) in order to maintain the homogeneous condition of the feed water, and it was caused to flow into the inside of the multi-channel ceramic membrane by a pump (2) (Procon, Standex Co., USA). The feed flow rate was measured by a flow meter (5) (NP-127, Tokyo Keiso, Japan). The flow rate and pressure of the feed water that flowed into the hybrid module were constantly maintained by controlling valves (9) of both the bypass pipe of the pump (2) and the concentrate pipe. The permeate flux treated by both the multi-channel ceramic membrane and PP beads coated with photocatalyst was measured by an electric balance (11) (Ohaus, USA). The permeate water flowed into the back-washing tank (13) when the permeate flux had not been measured. After the treated water was over a certain level in the back-washing tank (13), it was recycled to the feed tank (1) to maintain a constant concentration of the feed water during operation.

Kaolin was fixed at 30 mg/L and humic acid was changed from 2 to 10 mg/L in the synthetic feed water to investigate the effect of organic matters. The back-flushing time (BT) and filtration time (FT) were fixed at 10 s and 10 min, respectively. Only MF process without PP beads coated with TiO_2 photocatalyst and UV light (MF), and MF process with PP beads ($MF + TiO_2$) were, respectively, operated at 6 mg/L of humic acid and compared with the hybrid process of MF and PP beads with UV ($MF + TiO_2 + UV$) for researching a portion of the treatment efficiencies of multi-channel ceramic MF, adsorption, and photo-oxidation.

We observed the resistance of the membrane fouling (R_f) and permeate flux (J) during a total FT of 180 min under each condition. Under all experimental conditions, transmembrane pressure (TMP) was maintained constant at 0.8 bar, the water back-flushing pressure at 1.0 bar, the feed flow rate at 1.0 L/min, and the feed water temperature at 20 ℃. Periodic water back-flushing using permeate water was performed during 10 s per 10 min filtration.

The quality of feed water and treated water was analyzed in order to evaluate the treatment efficiencies of turbid materials and dissolved organic matters. Turbidity was measured by a turbidimeter (2100N, Hach, USA) and UV_{254} absorbance was analyzed by a UV

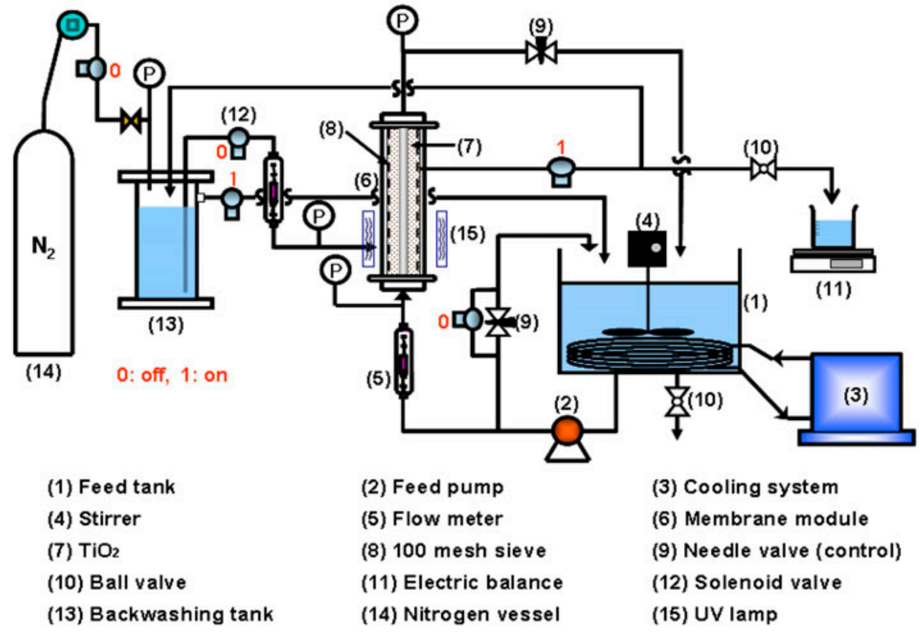

Fig. 1. Apparatus of advanced water treatment system using hybrid module of ceramic microfiltration and PP beads coated with TiO_2 photocatalyst with periodic water back-flushing [24].

spectrophotometer (Genesys 10 UV, Thermo, USA). The detection limits of turbidimeter and UV spectrophotometer were 0–4,000 NTU (±0.001 NTU) and -0.1–$3.0\,cm^{-1}$ ($\pm 0.001\,cm^{-1}$). Before the measurement of UV_{254} absorbance, the sample was filtered by 0.2 μm syringe filter to remove turbid materials.

3. Results and discussions

The effect of organic matters on membrane fouling was investigated in the hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst for advanced water treatment. Also, treatment portions by membrane filtration, photocatalyst adsorption, and photo-oxidation were investigated by comparing the treatment efficiencies of (MF), ($MF + TiO_2$), and ($MF + TiO_2 + UV$) processes. Resistances of membrane, boundary layer, and membrane fouling (R_m, R_b, and R_f) were calculated from permeate flux (J) data using the resistance-in-series filtration equation as the same method as our previous study [25].

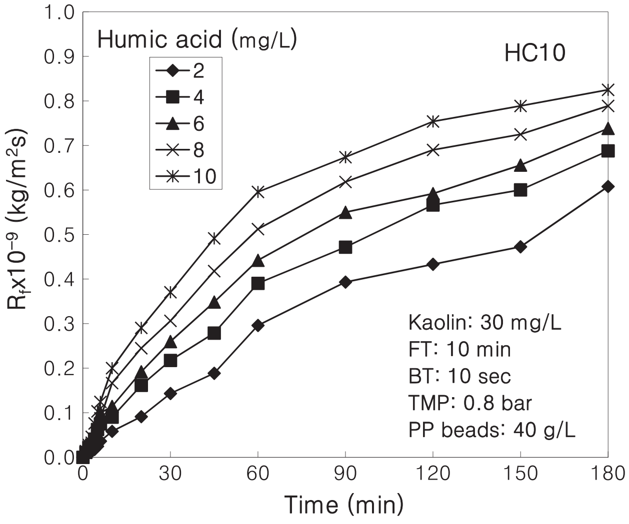

Fig. 2. Effect of humic acid on resistance of membrane fouling in hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst.

3.1. Effect of organic matters

As shown in Fig. 2, the resistances of membrane fouling (R_f) were highly influenced by humic acid, and R_f increased dramatically as rising concentration of humic acid from 2 to 10 mg/L. It means that the organic matter as humic acid could make membrane fouling more severe on the surface and inside the ceramic membrane as increasing the humic acid concentration in water. After 180 min of operation, the $R_{f,180}$ value at 10 mg/L of humic acid was $0.825 \times 10^9\,kg/m^2s$, which was 1.34 times higher than $0.616 \times 10^9\,kg/m^2s$ of the $R_{f,180}$ value at 2 mg/L as summarized in Table 3.

Fig. 3 showed the dimensionless permeate flux (J_{180}/J_0), where J_0 is the initial permeate flux which

Table 3
Effect of humic acid on filtration factors for hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst (BT 10 s, FT 10 min)

Humic acid (mg/L)	2	4	6	8	10
$R_m \times 10^{-9}$ (kg/m^{2}s)	0.374	0.369	0.370	0.369	0.368
$R_b \times 10^{-9}$ (kg/m^{2}s)	0.008	0.006	0.005	0.013	0.021
$R_{f,180} \times 10^{-9}$ (kg/m^{2}s)	0.616	0.688	0.738	0.789	0.825
J_0 (L/m^{2}hr)	740	753	754	738	741
J_{180} (L/m^{2}hr)	285	266	254	241	234
J_{180}/J_0	0.386	0.353	0.337	0.326	0.316
V_T (L)	26.5	23.8	22.4	20.7	19.4

was estimated by extrapolation using initial two data at 1 and 2 min. The J_{180}/J_0 tended to decrease as increasing humic acid concentration from 2 to 10 mg/L because of the membrane fouling reduction by the fewer organic macromolecules, which could be a rather complex combination process involving the adsorption of macromolecules to the membrane surface and within the membrane pores, and formation of a gel-like cake layer. Thus, the J_{180}/J_0 value of 0.386 at 2 mg/L of humic acid was 1.22 times higher than 0.316 at 10 mg/L as shown in Table 3. Furthermore, the total permeate volume (V_T) of 26.5 L at 2 mg/L of humic acid was 1.37 times higher than 19.4 L of V_T at 10 mg/L. Finally, we could find out that NOM, like humic acid, should be one of the main factors affecting membrane fouling in our hybrid process of multi-channel ceramic MF membrane and PP beads coated with TiO_2 photocatalyst.

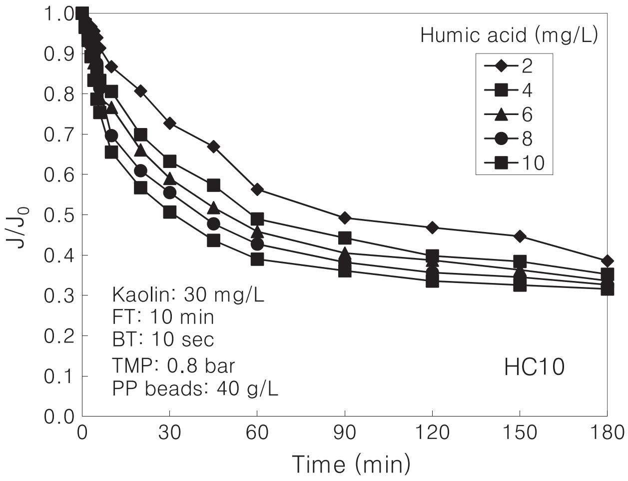

Fig. 3. Effect of humic acid on dimensionless permeate flux in hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst.

As arranged in Table 4, the treatment efficiency of turbidity was almost constant between 96.6 and 96.9% independent of humic acid concentration. It means that the organic matters could not affect the treatment of suspended particles like kaolin in our hybrid process. But the treatment efficiency of UV_{254} absorbance, which means the concentration of organic matters, was the maximum 65.4% at 6 mg/L of humic acid as shown in Table 5. The treated water quality of organic matters increased less slowly than the feed water quality from 2 to 6 mg/L of humic acid because most of the humic acid could be adsorbed on fouling materials inside the membrane or retained by cake layer on the membrane, and the remained things passed through the membrane adsorbed or oxidized by PP beads coated with TiO_2 photocatalyst. But the treated water quality of organic matters increased more rapidly than the feed water quality above 6 mg/L of humic acid because most of the organic matters passed through the membrane and could not be treated by adsorption or photo-oxidation by PP beads coated with TiO_2 photocatalyst. In our previous work [26] for the hybrid process of tubular ceramic UF membrane (pore size: 0.05 μm) and PP beads coated with TiO_2 photocatalyst, which was the same with PP beads used in this study, the treatment efficiency of organic matters was the maximum at 4 mg/L of humic acid, which was very similar with this result.

3.2. *Role of membrane filtration, adsorption, and photo-oxidation*

To investigate the role of membrane filtration, adsorption, and photo-oxidation in the hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst, the process with PP beads without UV light ($MF + TiO_2$) and only MF without any PP beads and UV (MF) were performed, respectively, at 6 mg/L of humic acid, and compared with the hybrid process of MF and PP beads with UV light ($MF + TiO_2 + UV$). The R_f values of ($MF + TiO_2 + UV$), ($MF + TiO_2$), and only MF process at 6 mg/L of humic acid were compared during 180 min of operation as shown in Fig. 4. The R_f could be maintained high at only MF and the ($MF + TiO_2$) processes, but dramatically the lowest at the ($MF + TiO_2 + UV$) process. It means that photo-oxidation by UV irradiation could reduce the membrane fouling strongly, but photocatalyst adsorption did not affect it in this hybrid water treatment process. As summarized in Table 6, the final R_f ($R_{f,180}$) after 180 min of operation was high at MF and ($MF + TiO_2$) processes, but the minimum 1.088×10^9 kg/m^{2}s at ($MF + TiO_2 + UV$) process.

Table 4
Water quality and treatment efficiency of turbidity in the hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst for effect of humic acid (BT 10 s, FT 10 min)

Experimental condition		Turbidity (NTU)				
		Feed water		Treated water		
Kaolin (mg/L)	Humic acid (mg/L)	Range	Average	Range	Average	Average treatment efficiency (%)
30	2	12.3–22.8	16.7	0.453–0.586	0.512	96.9
	4	12.8–25.4	17.6	0.488–0.664	0.563	96.8
	6	11.3–26.7	18.9	0.423–0.806	0.616	96.7
	8	14.5–25.8	20.1	0.433–0.867	0.678	96.6
	10	16.6–28.7	22.4	0.591–0.887	0.722	96.8

Table 5
Water quality and treatment efficiency of organic matters (UV_{254} absorbance) in the hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst for effect of humic acid (BT 10 s, FT 10 min)

Experimental condition		UV_{254} absorbance (cm^{-1})				
		Feed water		Treated water		
Kaolin (mg/L)	Humic acid (mg/L)	Range	Average	Range	Average	Average treatment efficiency (%)
30	2	0.008–0.032	0.018	0.001–0.013	0.007	61.8
	4	0.026–0.051	0.037	0.006–0.021	0.013	63.6
	6	0.030–0.092	0.059	0.008–0.031	0.021	65.4
	8	0.047–0.125	0.086	0.018–0.051	0.033	61.4
	10	0.087–0.145	0.119	0.026–0.079	0.055	52.1

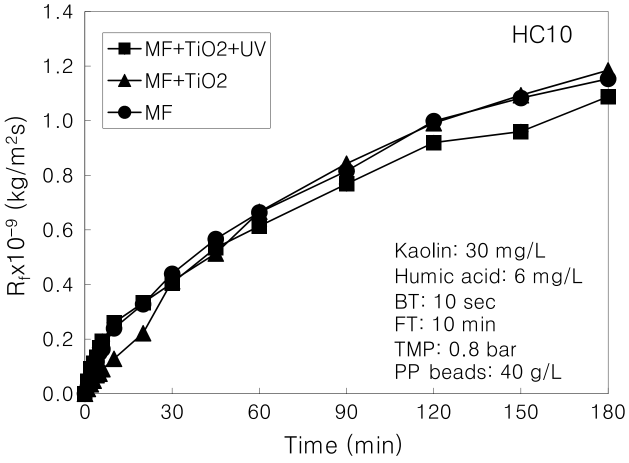

Fig. 4. Role of membrane filtration, adsorption, and photo-oxidation on resistance of membrane fouling in hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst.

As shown in Fig. 5, the J/J_0 values could maintain the highest at the ($MF + TiO_2 + V$) process and low at MF and the ($MF + TiO_2$) processes. It means that the dimensionless permeate flux at the ($MF + TiO_2 + UV$) could be higher than that at only MF and the ($MF + TiO_2$) processes because the photo-oxidation reduced the membrane fouling effectively. The final value ofJ/J_0 (J_{180}/J_0) after 180 min of operation was the maximum 0.275 at ($MF + TiO_2 + UV$) process, which was 1.07 times higher than the minimum 0.255 at only MF process as summarized in Table 6. The highest permeate flux could be maintained because the membrane fouling was controlled the more effectively by photo-oxidation at ($MF + TiO_2 + UV$) process than at only MF or ($MF + TiO_2$) processes. In our previous work [26] for the hybrid process of tubular ceramic UF membrane (pore size: 0.05 μm) and PP beads coated with TiO_2 photocatalyst, the maximum J_{180}/J_0 could be acquired at ($UF + TiO_2 + UV$) process, which was exactly the same result as the one obtained with this study.

The portions of treatment efficiency performed by membrane filtration, adsorption, and photo-oxidation, respectively, in our hybrid process could be calculated by reducing sequentially the turbidity or organic matter treatment efficiencies of ($MF + TiO_2 + UV$), ($MF + TiO_2$) and MF, which are summarized in Table 7. In turbidity treatment efficiency, the treatment portion

Table 6
Roles of membrane filtration, adsorption, and photo-oxidation on filtration factors in hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst at humic acid 6 mg/L

Process	$MF + TiO_2 + UV$	$MF + TiO_2$	MF
$R_m \times 10^{-9}$ (kg/m^{2}s)	0.351	0.369	0.379
$R_b \times 10^{-9}$ (kg/m^{2}s)	0.061	0.046	0.015
$R_{f,180} \times 10^{-9}$ (kg/m^{2}s)	1.088	1.184	1.153
J_0 (L/m^{2}hr)	684	681	716
J_{180} (L/m^{2}hr)	188	177	182
J_{180}/J_0	0.275	0.259	0.255
V_t (L)	17.1	17.2	16.9

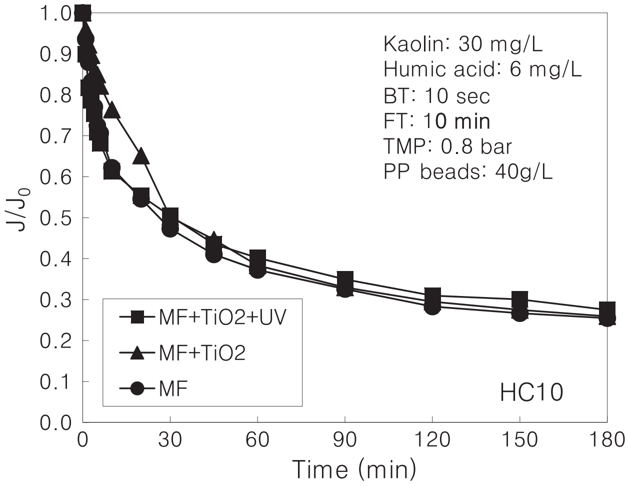

Fig. 5. Role of membrane filtration, adsorption, and photo-oxidation on dimensionless permeate flux in hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst.

Table 7
Treatment efficiency portions of membrane filtration, adsorption, and photo-oxidation in the hybrid process of multi-channel ceramic MF and PP beads coated with TiO_2 photocatalyst at humic acid 6 mg/L

Portion of treatment efficiency	Turbidity	Organic matters (UV_{254} absorbance)
Membrane filtration (%)	97.9	44.6
Adsorption (%)	0.0	0.5
Photo-oxidation (%)	0.6	3.2
Total treatment efficiency (%)	98.5	48.3

of membrane filtration was very high 97.9% at 6 mg/L of humic acid. The treatment portion of adsorption by PP beads coated with TiO_2 photocatalyst was 0.0% and that of photo-oxidation by PP beads and UV light was very low 0.6%. It means that the role of adsorption and photo-oxidation by PP beads coated with TiO_2 photocatalyst and UV was not important for treatment of the suspended particles like kaolin in our hybrid process. Kaolin could not be removed effectively by the adsorption and photo-oxidation by PP beads coated with TiO_2 because kaolin was inorganic and did not have electric charge. In the treatment efficiency of organic matter, the treatment portion of membrane filtration was still very high 44.6%, but that of adsorption was very low 0.5% and that of photo-oxidation was a little high 3.2% at 6 mg/L of humic acid. It proved that the role of photo-oxidation by PP beads coated with TiO_2 photocatalyst was more important than that of adsorption by PP beads for the organic matters treatment in our hybrid process. The photo-oxidation by the PP beads and UV irradiation could reduce the membrane fouling strongly because the photo-oxidation had the major role of organic matters reduction in this hybrid water treatment process. In our previous work [26] for the hybrid process of tubular ceramic UF membrane (pore size: 0.05 μm) and PP beads coated with TiO_2 photocatalyst, the role of adsorption was more important than that of photo-oxidation for both turbidity and organic matters treatment. It was thought that the particle size through UF membrane was much smaller than those through MF, and the smaller particles could be adsorbed easily on the surface of PP beads.

4. Conclusions

In this study, the effect of organic matters on membrane fouling was observed in a hybrid process of seven-channel alumina MF and PP beads coated with TiO_2 photocatalyst for advanced water treatment. Also, treatment portions by membrane filtration, photocatalyst adsorption, and photo-oxidation were investigated by comparing the treatment efficiencies of (MF), ($MF + TiO_2$), and ($MF + TiO_2 + UV$) processes. Finally, we could find out the following results.

(1) The organic matter, like humic acid, could be one of the main factors affecting membrane fouling because the final resistance of membrane fouling ($R_{f,180}$) value decreased significantly as reducing the humic acid concentration.
(2) The treatment efficiencies of turbidity were outstanding at 96.6–96.9% independent of humic acid concentration. But that of dissolved organic matters (UV_{254} absorbance) was the maximum 65.4% at humic acid 6 mg/L.

(3) As a result of investigating the role of membrane filtration, adsorption, and photo-oxidation, the membrane fouling resistance was the minimum, and the final permeate flux was the maximum at (MF + TiO_2 + UV) process. This result proved that photo-oxidation could control membrane fouling, but photocatalyst adsorption was not helpful to reduce membrane fouling in our hybrid water treatment process.

(4) The adsorption and photo-oxidation by PP beads coated with TiO_2 photocatalyst could play a much greater role in the removal of organic matter than that of turbidity in our hybrid water treatment process. Kaolin could not be removed effectively by the adsorption and photo-oxidation by PP beads coated with TiO_2 because kaolin was inorganic and did not have electric charge.

Acknowledgments

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