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Integration of Photocatalysis and Microfiltration in Removing Effluent Organic Matter from Treated Sewage Effluent

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An integration of photocatalysis with low-pressure submerged membrane has attracted growing interest for its synergic advantages in water and wastewater treatment. In this study, the adsorption and photocatalytic oxidation of organic compounds by UV light responsive titanium dioxide (TiO_2) were investigated. First, the adsorption behavior of the TiO_2 was examined by the adsorption isotherm and kinetics experiments. The photocatalytic reactivity of the catalysts was then compared at different operating conditions. The results indicate that the Freundlich model described well the adsorption capacity of both materials. The photocatalytic kinetics showed that the highest removal of effluent organic matter (EfOM) was achieved at an optimum concentration of 1.0 g/L of TiO_2. In addition, it was found that the pre-photosensitization with titanium dioxide/ultra-voilet radiation (TiO_2/UV) could effectively reduce membrane fouling and enhance the permeate flux of the submerged membrane reactor when it was used as a post-treatment. An increase of 10% in organic removal efficiency was achieved by the posttreatment of membrane filtration. The sustainable flux of the membrane reactor increased from 25 up to 40 L/m^2.h when the pretreatment of photocatalysis was used.

Keywords biological treated sewage effluent; integrated system; organic matter; photocatalysis; submerged membrane reactor; titanium dioxide-tio$_2$; UV light responsive titanium dioxide

INTRODUCTION

Organic pollutants found in biologically treated wastewater are usually referred to as effluent organic matter (EfOM). As the practice of water recycling is becoming increasingly important, EfOM and persistent trace organic pollutants present in the water cycle have to be removed completely due to public health concerns. A number of separate and combined treatments such as flocculation (1,2,3), adsorption (4,5), ion exchange (6,7), membrane filtration (8,9), and advanced oxidation (10,11,12) have been employed to remove these dissolved organic pollutants.

Advanced oxidation processes are characterized by the generation of the highly reactive hydroxyl radical ($\cdot$OH) that can tackle and mineralize dissolved organic pollutants. Among the advanced treatment technologies, TiO_2/UV photocatalysis has attracted intensive studies in recent years due to its non-toxic and no sludge disposal properties (10). In the photo-oxidation process, hydroxyl radicals ($\cdot$OH) are generated when the catalyst (TiO_2) is illuminated by ultraviolet (UV) light. As a result, organic compounds are mineralized into CO_2, H_2O and inorganic constituents (12,13). Titanium dioxide is the most commonly used photocatalyst material due to its strong redox ability, chemical stability, and availability at low cost (14). Nevertheless, photocatalysis has disadvantages in energy consumption and the recovery of the photocatalyst (15).

Recently, the idea of integrating photocatalysis with low-pressure submerged membrane has attracted growing attention. In the combined system, microfiltration not only aids in the separation of suspended catalysts but also improves the effluent quality by selective separation of pollutants at molecular level. In return, semiconductor photocatalysis helps to reduce membrane fouling by photo-mineralizing compounds causing membrane fouling (12,16).

The aim of this study is to evaluate the performance of the photocatalysis -membrane hybrid system in removing effluent organic matter from biologically treated sewage effluent (BTSE). The effects of operating parameters (e.g., titanium dioxide (TiO_2) concentration, ultraviolet (UV) light intensity, and irradiation time) were investigated. The performance of the slurry reactor was also assessed in terms of transmembrane pressure (TMP).

EXPERIMENTAL

Materials

Synthetic wastewater. The synthetic wastewater used in this study represents the biologically treated sewage effluent. The synthetic wastewater consists of persistent organic compounds such as humic acid, tannic acid, lignin, polysaccharide, and other high molecular

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TABLE 1
Constituents of the synthetic wastewater (17)

Compounds	Weight (mg)/L
Beef extract	18
Peptone	27
Humic acid	42
Tannic acid	42
Sodium lignin sulfonate	24
Sodium lauryle sulphate	9.4
Acacia gum powder	47
Arabic acid	50
$(NH_4)_2SO_4$	71
K_2HPO_4	70
NH_4HCO_3	188
$MgSO_4 \cdot 3H_2O$	7.1

weight carbohydrates (Table 1). The feed was characterized in terms of dissolved organic carbon (DOC) using the Multi N/C 2000 analyzer (Analytik Jena AG). Within the duration of the experiments, the quality of the synthetic wastewater varied slightly with DOC concentration between 10–11 mg/L.

Photocatalyst. Photooxidation was conducted with powdered P25 Degussa TiO_2 obtained from Degussa Company (Germany). The TiO_2 particle is comprised of a 70:30 anatase/rutile ratio and has an approximately 50 m^2/g of active surface area. In the aqueous solution, TiO_2 particles aggregate and form large size agglomerations ranging from 0.2 to 1.2 µm for pH of 3 to 8, respectively (18). The characteristics of P25 can be found elsewhere (Table 2) (19).

Experiments

Adsorption equilibrium. In the adsorption equilibrium experiment, a certain amount of P25 varying from 0 to 2.5 g/L was added into 100 mL of synthetic wastewater. The flasks were continuously shaken for 72 hours at 13.61 rad/s (130 rpm) at 25°C (Ratek platform mixer). Upon the completion, the supernatants were collected and analyzed in terms of dissolved organic carbon (DOC) (Multi N/C 2000 Analyzer).

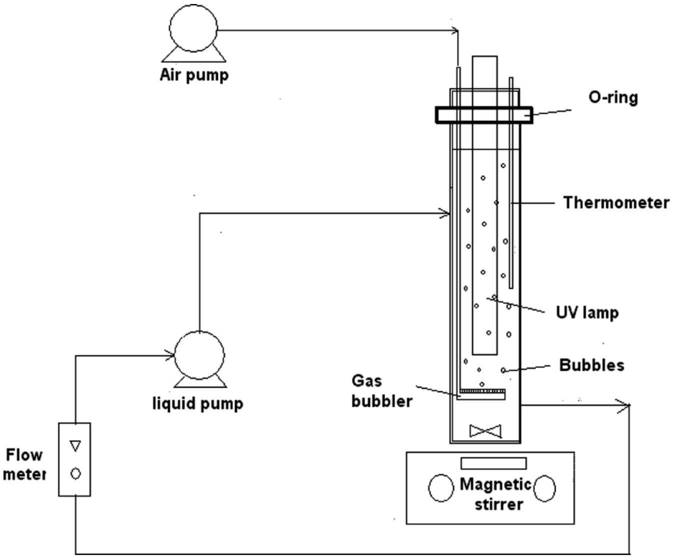

FIG. 1. Schematic of the batch photoreactor.

Adsorption kinetics. The batch experiments were carried out using 1 L of synthetic wastewater at 25°C for 6 hours. The concentrations of P25 studied were 0.1, 0.5, and 1.0 g/L. The stirring speed was maintained at 11.6 rad/s (110 rpm). The adsorption capacity of titanium dioxide was determined against Langmuir, Sips, and Freundlich models.

Photocatalytic oxidation. The photoreactor consisted of a 1.5 L annular reactor, an UV lamp (352 nm, FL10BL, Sankyo Denki, Japan), air sparging, a magnetic stirrer, and a recirculation cooling water column (Fig. 1). The

TABLE 2
Physico-chemical properties of P25 Degussa

Specification	P25 Degussa TiO_2
Type	Powdered
Structure	Non-porous
Components	65% anatase, 25% rutile, 0.2% SiO_2, 0.3% Al_2O_3, 0.3% HCl, 0.01% Fe_2O_3
Average aggregate particle diameter	0.3 µm
Primary crystal size	21 nm
Mean pore diameter	6.9 nm
Band gap	3.03 (from 500 to 300 nm) with UV-Vis
Apparent density	130 kg/m^3
Specific surface area (BET)	$49.32 \pm 0.18\ m^2/g$
Product code	Degussa, Frankfurt am Main, Germany

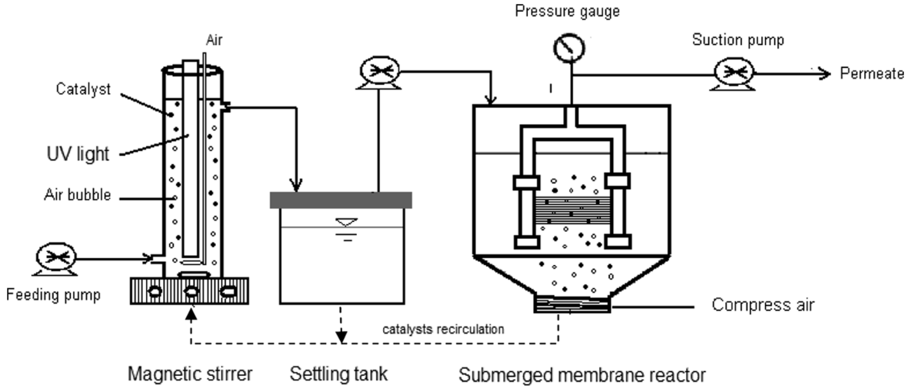

FIG. 2. Schematic diagram of the continuous-mode photocatalysis – membrane hybrid system.

illumination intensity of the reactor varied from 46.6 to 276.7 mW/cm^2. Air sparging was equipped to supply oxygen into the reactor at the rate of 10 L/min. In the first stage of this study, the effect of several parameters were examined including catalyst doses, illumination intensity as well as feed flow rate to optimize the operating condition of the photoreactor.

Membrane reactor. The membrane reactor was used to separate TiO_2 from the slurry. The polyethylene hydrophilic coated membrane with the internal pore size of 0.1 µm was used (Mitsubishi-Rayon, Tokyo, Japan). The effective area of the membrane was equal to 0.05 m^2. The photomineralized solution collected from the photoreactor was pumped into the submerged membrane reactor after undergoing a settling for 1 hour (Fig. 2). To evaluate the performance of the membrane system used, transmembrane pressure (TMP) was recorded and the permeate was collected for dissolved organic carbon (DOC) analysis. The effectiveness of TiO_2 separation by the membrane was determined on the basis of turbidity removal. Turbidity was measured using a HACH 2100 N IS turbidimeter.

RESULTS AND DISCUSSION

Adsorption Equilibrium

The adsorption equilibrium experimental results and the model simulation are shown in Fig. 2. As can be seen, the Freundlich model described well the adsorption equilibrium behavior of P25 in treating synthetic wastewater in this study. The Freundlich isotherm is given below (Eq. (1) with Fig. 3). A comparison made with Freundlich, Langmuir, and Sip's isotherms showed that Freundlich was the best among the three isotherms for the wastewater and P25 investigated in this study.

Adsorption Kinetics

Figure 4 shows the adsorption kinetics at different TiO_2 doses. The rate of adsorption was rapid in the first one hour of solution-adsorbent contact and reached the equilibrium after two hours. The amount of organics adsorbed increases proportionally with the amount of TiO_2 added. A significant improvement (by 30%) was observed when P25 dose was increased from 0.1 to 0.5 g/L, followed by another 10% when doubled to 1.0 g/L. The overall DOC removal by TiO_2 adsorption was less than 50%. In comparison with other popular adsorbents such as granular and powdered activated carbon (GAC and PAC), Kim et al. (20) reported a ten-fold higher in the mass transfer coefficient of PAC as compared to TiO_2 in the removal of metsulfuron-methyl. The results indicate that surface adsorption is not the main mechanism

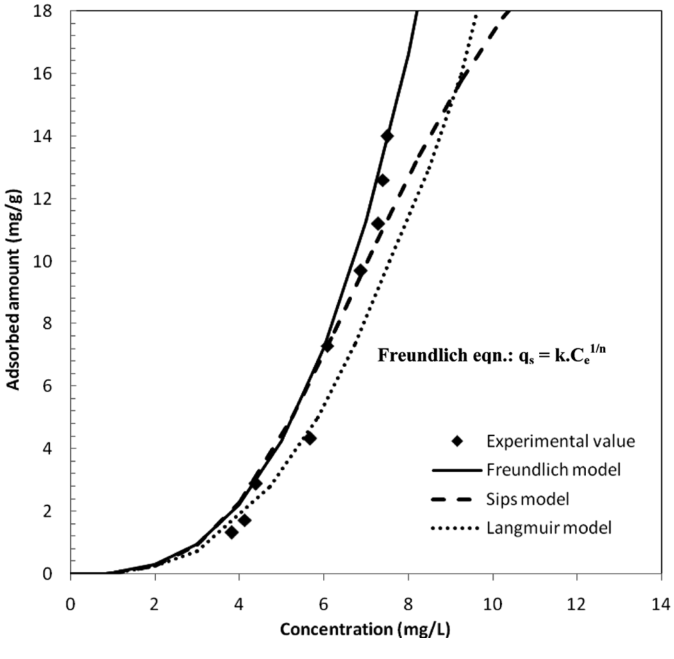

FIG. 3. Adsorption isotherm of synthetic wastewater (initial DOC = 10.34 mg/L, contact time = 72 hours, mixing rate = 130 rpm, temperature = 25°C).

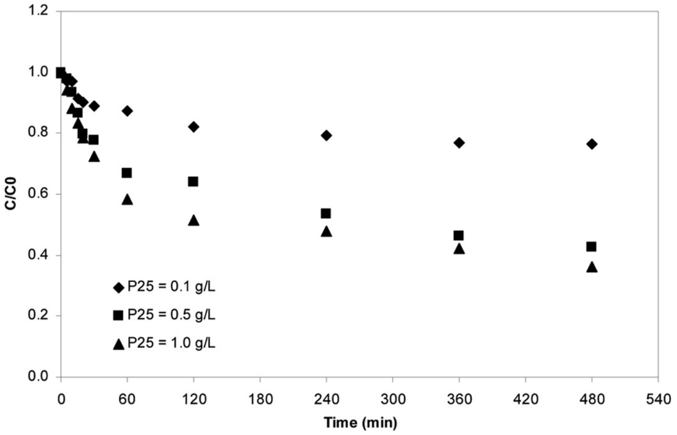

FIG. 4. Adsorption kinetics of synthetic wastewater at different P25 doses (initial DOC = 10.34 mg/L; stirring speed 11.6 rad/s; C = effluent DOC concentration, mg/L and C0 = influent DOC concentration, mg/L).

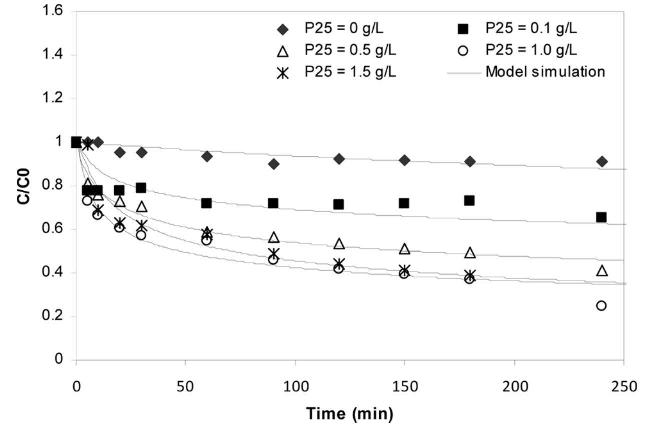

FIG. 5. DOC removal efficiency of effluent at different P25 doses (lamp surface = 184.6 mW/cm^2; initial DOC = 10–11 mg/L).

of TiO_2 since its surface area is relatively small (only 50 m^2/g) compared to GAC and PAC (300–1500 m^2/g).

Photocatalysis Degradation

Effect of Photocatalyst Concentration

In the UV photocatalysis process, the semiconductor catalyst dose is one of the most important parameters that affect the photocatalytic oxidation rate. The main functions of TiO_2 are to absorb light and produce the electron hole pairs, e^-/h^+, which react with contaminants and break them down into small and harmless species. Therefore, the availability of photocatalysts for the illumination process has a significant effect on the organic mineralization rate. However, excessive loading might weaken the penetration of light into the slurry that results in less effective reaction.

In the first series of the experiments, an optimal dose of TiO_2 for the annular reactor was investigated. Previous studies reported an average TiO_2 loading of 1.0 g/L (12, 21). In this study, the dose of TiO_2 was varied between 0 and 1.5 g/L (Fig. 5). As expected, the results were quite different between tests carried out with and without TiO_2. When no catalyst was used, UV lamp alone failed to degrade persistent organic pollutants in the wastewater. As the catalyst dose was increased, DOC removal efficiency increased proportionally. A considerable improvement from 40 to 62% in terms of DOC removal efficiency was obtained when TiO_2 doses increased from 0.1 to 1.0 g/L. However, there was no significant improvement when the TiO_2 dose was increased beyond this value. This was a result of excessive loading, which prevents the penetration of UV light into the bulk of the solution, making the photoactivity of the catalyst less effective (13).

Past studies reported that the degradation rate of organic contaminants treated by illuminated TiO_2 can be fitted well with the Langmuir-Hinshelwood (L-H) kinetics (22,23). In this study, A slightly modified version of the Langmuir-Hinshelwood kinetics (similar to a Sips isotherm) was used to fit the experimental data.

$$\frac{dC}{dt} = -K_r\left(\frac{K_d C^n}{1 + K_d C^n}\right) \tag{2}$$

where C is the concentration at any time (mg/L), K_{ad} the adsorption coefficient and k is the reaction rate constant (h^{-1}). K_{ad} and k are the limiting rate constants of reaction at maximum coverage under the given experimental conditions and equilibrium constant for adsorption of organics onto TiO_2.

The simulation of the Langmuir-Hinshelwood model and a summary of the calculations of rate constant (k) and removal efficiency (R_{eff}) are shown in Fig. 4 and Table 3, respectively. The plot shows that the slope increases with the increase in catalyst doses. Under these operating conditions, an optimum TiO_2 dose of 1.0 g/L was obtained and selected for the subsequent experiments.

Effect of Light Intensity

Besides the photocatalyst dose, the type and intensity of the light source used to irradiate the reactor also play a crucial role in the photooxidation process. In this study, the lamps were chosen with the same wavelength of 352 nm (UV-B). The capacity of the light source was varied in order to determine the effect of light intensity on DOC removal efficiency (Figure 6). Minor variations were observed within the first 30 minutes, during which higher intensity led to faster degradation rate. However, the eventual efficiencies after a 3-hour operation varied slightly with the increase in the light intensity. It indicated that the annular configuration of the photoreactor allows UV light to uniformly penetrate through the bulk solution.

TABLE 3
Optimization of catalyst concentrations in the photocatalytic oxidation process

	k (h^{-1})	K_{ad}	R^2	R_{eff} (%)
P25 concentration (mg/L) (at 184.6 mW/cm^2)				
0.0 g/L	0.0015	0.0087	0.9947	9
0.1 g/L	0.0073	0.0675	0.9594	35
0.5 g/L	0.0135	0.0633	0.9920	59
1.0 g/L	0.0193	0.0870	0.9799	62
1.5 g/L	0.0187	0.1331	0.9691	63
UV light intensity (mW/cm^2) (at 1.0 g/L of TiO_2)				
46.6	0.0165	0.8235	0.9988	53
92.3	0.0172	0.7456	0.9980	60
184.6	0.0183	0.8730	0.9962	61
277.0	0.0179	0.7809	0.9985	63

As all particles absorbed photons and released electron-hole pairs the rate became steady. It was demonstrated by the rate constant k which varied only marginally from 0.0165 to 0.0183 h^{-1} in response to the increase in intensity. Due to the energy considerations, the optimal intensity of 92.3 mW/cm^2 was chosen in the subsequent experiments.

Effect of the Flow Rate

In the continuous system, the detention time (T) for reaction to occur inside the column is governed by the feed flow (θ).

$$T = \frac{V}{\theta} \tag{3}$$

where V is the volume of the photoreactor (1.5 L).

According to the results obtained in the batch experiments, the flow rate in the continuous system was adjusted from 38 to 100 mL/min so that the detention time ranged between 15 and 40 minutes. This range was chosen to allow sufficient time for the UV/TiO_2 to break down and mineralize pollutants. The results are presented in Fig. 7. As expected, lower flow rates (i.e., longer photooxidation time) yielded higher DOC removal. The effluent DOC was reduced by 50% at the rate of 50 mL/min after 60 minutes and became relatively stable after 3-hour operation. As the flow rate was increased, the reaction time within the reactor was shortened, and resulted in partial oxidation of pollutants only. However, when the flow rate was reduced to 38 mL/min, catalysts started to deposit inside the tube and became stagnant. This reduced the amount of catalysts fed into the reactor and therefore slowed down the photooxidation process. The optimal feed flow rate of 50 mL/min was chosen in consideration of DOC removal and the productivity of the system.

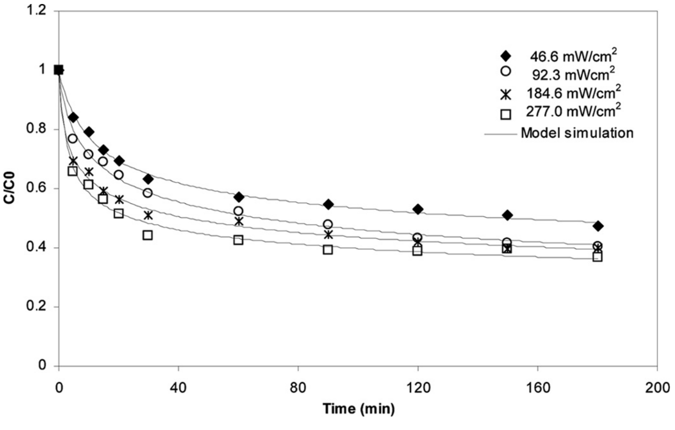

FIG. 6. DOC removal efficiency of TiO_2 at different light intensities (initial DOC = 10.34 mg/L; TiO_2 dose = 1.0 g/L).

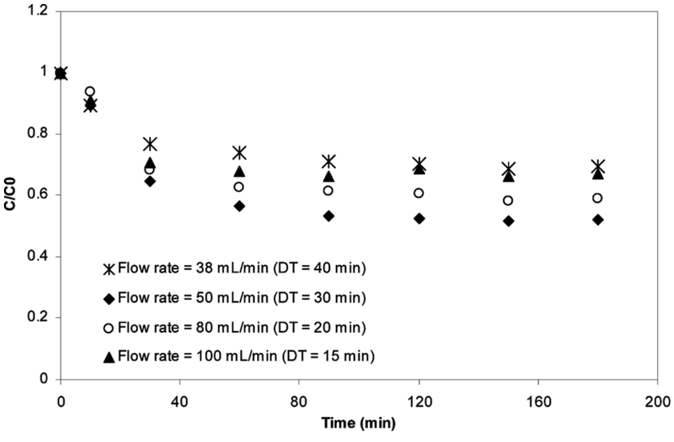

FIG. 7. DOC removal efficiency of the continuous photoreactor at different flow rate and corresponding detention time (DT) (TiO_2 dose = 1.0 g/L, light intensity = 92.3 mW/cm^2).

Photocatalysis – Membrane Integrated System

Previous studies (16,24,25) have shown that coupling photocatalysis and pressure-driven membrane processes resulted in a decrease of the permeate flux. Xi and Gieseen (25) reported a significant decrease in membrane permeability during microfiltration (MF) with an increase of TiO_2 concentration (ranging from 0.5 to 3.0 g/L). Nanofiltration, on the other hand, was found to have the least effect of the TiO_2 presence in the feed on permeate flux (16). Shon et al. (12) investigated the effect of settling time of TiO_2 suspension on permeate flux during MF. They reported an increase in permeable flux as the settling time was increased from 15 to 60 minutes.

In this study, treated water upon photooxidation was collected and allowed to settle for 1 hour before the supernatant was pumped into the submerged membrane reactor. Gravity settling reduced the turbidity of the supernatant from 876 NTU (1.0 g/L of TiO_2 slurry) to approximately 29.7 NTU, whereas the turbidity of raw wastewater was 4.3 NTU. The critical flux (measured in terms of the development of transmembrane pressure (TMP)) of the influent and the effluent upon photooxidation was determined and compared. The results are presented in Fig. 8.

It can be seen from Fig. 8 that pre-photooxidation with TiO_2/UV could effectively reduce membrane fouling and enhance the filtration flux of the submerged membrane reactor. The critical flux increased from 25 $L/m^2 \cdot h$ for synthetic wastewater to 40 $L/m^2 \cdot h$ for supernatant collected after photomineralization. In addition, variation in the detention time of the photocatalytic oxidation also affected the filtration flux. The longer the detention time, the higher the degradation of organic pollutants, leading to a lower transmembrane pressure, thus a higher critical flux. In addition, the turbidity of the permeate was around 0.7 to 1.1 NTU.

Figure 9 illustrates the DOC removal efficiency of the hybrid system. MF alone was able to remove less than 30–35% of organic matters (in terms of DOC) in the synthetic wastewater. Although MF was integrated primarily for the detainment of the catalysts, it was found that an increase of 10% in organic removal was achieved after the filtration. This raised the total DOC removal of the hybrid system to 70–75% and enabled it to operate continuously and consistently at a flux of 40 $L/m^2 \cdot h$ for a period of 6 hours.

In addition, the high-performance liquid chromatography (HPLC) was employed in order to determine the removal of different molecular weight ranges of organics by photooxidation and membrane filtration. The results are presented in Fig. 10. The two main peaks detected were humic acids (HA) (1.5 kDa) and low MW acids (250–500 Da) It was found that MF and TiO_2 adsorption (without UV) were able to remove essentially humic acids. It is interesting to notice that the membrane pore size used in this study was 0.1 μm which is much larger than the equivalent size of HA. This suggested that the retention of HA was mainly due to adsorption onto the membrane surface. The subsequent accumulation on the membrane surface resulted in diminishing the effective membrane pore size, thus leading to further physical retention (12).

The most significant decrease in the intensity of the absorbance peak was observed when photocatalytic oxidation was employed (Figure 10). After 3 hours of UV radiation, the intensity peak corresponding at a MW of 1.5 kDa was reduced by almost 80%. Earlier studies (12,26) also indicated a significant degradation of organic matter in the range of 800–2000 Da by TiO_2 photocatalysis. However, the amount of low MW acids (250 Da) remained unchanged. This could be due to high competition of HA and larger MW for the generated radicals during the

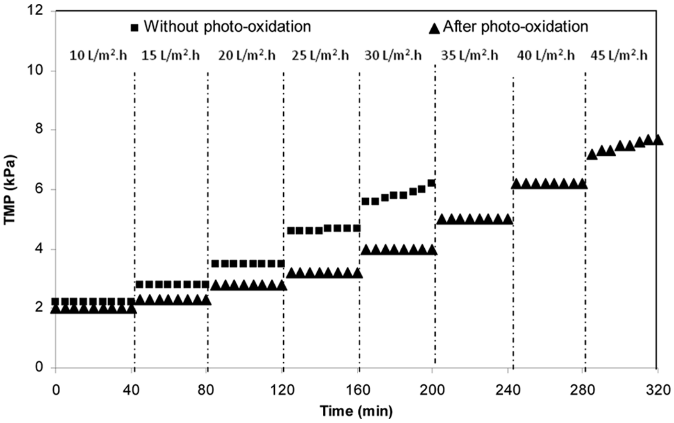

FIG. 8. Effect of pre-photooxidation on critical flux (membrane pore size = 0.1 μm; TiO_2 dose = 1.0 g/L; settling time of TiO_2 = 1 hour; light intensity = 92.3 mW/cm^2; feed flow-rate = 50 mL/min; settling time of TiO_2 = 1 hour).

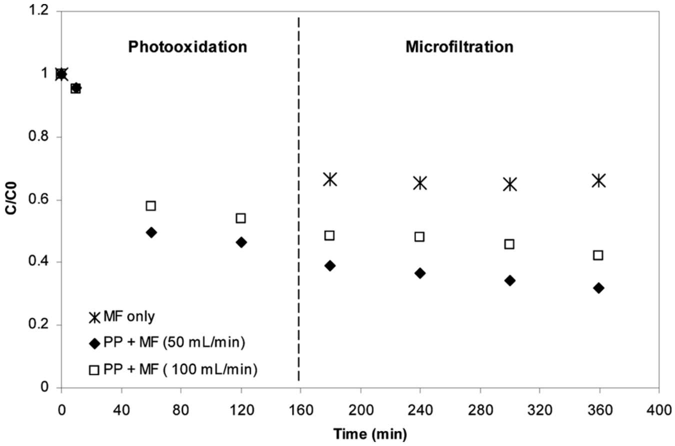

FIG. 9. DOC removal efficiency of the photocatalysis – membrane integrated system (TiO_2 dose = 1.0 g/L, light intensity = 92.3 mW/cm^2; C0 is influent concentration; C is effluent at time t).

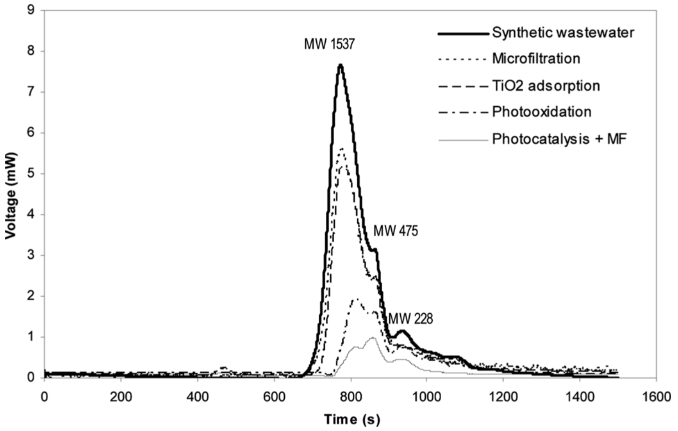

FIG. 10. Molecular weight distribution of organic matters in synthetic wastewater before and after treatment (initial DOC = 11–12 mg/L; TiO_2 dose = 1.0 g/L; filtration flux = 25 L/m^2 · h).

photooxidation process. Further separation with MF, as expected, did not help much in the removal of HA and low MW acids.

CONCLUSIONS

The study was conducted to investigate the effect of photocatalytic oxidation coupled with a low-pressure microfiltration in removing EfOM from the BTSE. The following conclusions were drawn out from the obtained results:

- The batch adsorption and photocatalysis studies showed that:
  - The adsorption equilibrium of P25 was well described by the Freundlich model while the photocatalytic kinetic processes could be predicted by a slightly modified Langmuir – Hinselwood model;
- A detailed study on photocatalysis revealed that:
  - The photocatalytic reaction was significantly affected by the TiO_2 dose and the optimal dose for this photoreactor was at 1.0 g/L. The UV light intensity showed only a marginal effect on the degradation of organic matters. In our experiments, an optimal intensity of 92.3 mW/cm^2 could be sufficient for the photodegradation;
  - Higher residence time in the photoreactor led to a higher removal efficiency. The suitable detention time for the photoreaction was found to be 30 minutes.
- Integrating photooxidation (for organic degradation) and membrane filtration (for separation of photocatalyst) showed that:
  - Pre-photooxidation with TiO_2/UV could effectively reduce the membrane fouling and enhance the filtration flux of the submerged membrane reactor. The critical flux of the membrane filtration unit increased from 25 L/m^2 · h to 40 L/m^2 · h when the BTSE underwent photooxidation.;
  - The integrated photocatalysis and submerged membrane system resulted in a DOC removal of 70–75% and operated consistently for 360 minutes (the total time of the experiment period).

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