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A hybrid photocatalysis and ceramic membrane filtration process for humic acid degradation: Effect of pore size and transmembrane pressure

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ABSTRACT

A hybrid photocatalysis with ceramic membrane filtration system is demonstrated for degradation of humic acids (HAs), which are typical refractory components of natural organic matter (NOM) present in water and wastewater. More specifically, the combination of chemical oxidation photocatalysis process with physical separation via a ceramic membrane filtration was explored. The effects of operating parameters such as transmembrane pressure and the membrane pore size on the permeate flux and organic removal was investigated. The interaction between the two solutes in the system, humic acids and TiO_2 photocatalyst, played an important role in the observed flux decline during ceramic ultrafiltration (UF) and microfiltration (MF). Results showed that the MF membrane showed flux rates that were about 30% lower than the ones achieved with UF membranes. The dissolved organic carbon (DOC) removal was found to be higher in UF membrane (> 70%) compared to MF membrane (50%). Finally from the liquid chromatography (LC) analysis showed that after photocatalytic treatment, there is a change in the molecular weight distribution of the organic compounds and preferential adsorption of those compounds by TiO_2 results in different fouling mechanisms in UF and MF membranes. It can be concluded that the use of ceramic membrane not only acts as a barrier in recovering the TiO_2 photocatalyst but also assists in DOC reduction.

Keywords: Ceramic membrane; Photocatalytic processes; Humic acid; Membrane pore size; Transmembrane pressure

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1. Introduction

Heterogeneous photocatalysis using TiO_2 as a photocatalyst has proven to be an effective method for the treatment of organic compounds present in water and wastewaters. Although photocatalytic oxidation process is efficient in mineralising various organic compounds there remains two key challenges. Firstly the recovery and recycle of photocatalyst is needed and secondly the intermediate oxidation products formed during the photocatalytic oxidation process are freely transported into final stream [1]. Both the above challenges can be resolved using membrane separation process as they not only aid in the separation of suspended photocatalyst, but also improve the effluent quality by separation of organic compounds. In recent years hybrid

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photocatalysis-membrane system have been developed to effectively separate the TiO_2 photocatalyst and also enhances the effluent quality by selective separation of intermediates and byproducts that are produced from the photocatalytic oxidation [2]. Amongst various configurations of the hybrid system described in the literature the systems utilizing pressure driven membrane processes, using polymeric membranes such as microfiltration (MF) [3–6] and ultrafiltration (UF) [1,4,7,8] have mainly been investigated. Such systems were applied for removal of various contaminants, including pharmaceuticals [9,10], and natural organic matter (NOM) [4,7] from water. Recently, Ho et al., utilized a hybrid photocatalysis–MF process for the treatment of a synthetic wastewater [11]. The treated wastewater after photodegradation in a photoreactor was allowed to settle for 1 h in a settling tank, in which turbidity of the supernatant was reduced from 878 NTU to 30 NTU, and after that it was pumped into a submerged membrane reactor. The authors found that such a system allowed to reduce the membrane fouling and enhance the filtration flux. Similar conclusions were drawn by Pidou et al. [12] who observed that photocatalytic pretreatment of synthetic grey water allowed to reduce membrane fouling. However, they also reported that a minimum UV residence time of 120 min in the continuous stirred-tank reactor (CSTR) was necessary.

Even though numerous advantages of these hybrid system, they reveal also some problems, mainly low permeate quality, especially in case of polymeric MF/UF [13], and membrane fouling due to the presence of photocatalyst (TiO_2) particles [6,14] . Recently ceramic membranes are promising as an alternative to polymeric membranes due to high stability to high temperature and pressure, high mechanical strength and abrasion resistance, chemical and biological inertia for wide pH range adaptability, long service life and good recoverability [15–17]. Therefore the merger of ceramic membranes with photocatalysis is considered a novel and unique opportunity for wastewater treatment [18]. Recently Dow et al. [19] studied the use of ozone–coagulation–ceramic membranes at pilot scale where it was demonstrated that at the high flux obtainable with ozone– coagulation–ceramic membranes and the cost was comparable to polymeric membranes with pre-coagulation. In addition, the inclusion of advanced oxidation processes such as ozonation or photocatalytic oxidation as a pre-treatment step with ceramic membranes, can significantly reduce fouling by organic compounds and improve water quality that in turn reduces the operating cost. Additionally, ceramic ultrafiltration provides an additional barrier against pathogenic microbes such as virus rejection. Therefore this study explores the feasibly of combining a batch recirculated photoreactor with a tubular ceramic membrane ultra and microfiltration for heterogeneous photocatalyst applications.

Our first paper described the influence of several experimental variables including solution pH and salinity on the removal of model humic acid (HA) solution using TiO_2/UV photocatalytic oxidation process [20]. Our second paper described the combined operation of the integrated photoreactor-ceramic UF process system on the removal of model humic acid solution under several experimental variables such as photocatalyst dose and solution salinity [21]. The main objectives of this study were HA removal and improving permeate flux. The results revealed that this hybrid system results in synergistic effects including oxidation by the photocatalytic reaction, rejection of HA by ceramic membrane filtration and separation of TiO_2 particles from permeate by membrane rejection. The results showed that relatively high removal of total organic carbon (TOC) and UV absorbance, removal of HA fouling potential and the complete recovery of TiO_2 slurry using this hybrid system were achieved. However the steady state permeate flux decreases slightly with the presence of NaCl concentrations and TOC removal, and the reduction in UV absorbance decreased slightly with increasing NaCl concentrations due to interfacial effects on the ceramic membrane filter. This study is an extension of our previous studies where the objective of this work was to investigate the effects of transmembrane pressure (TMP) and membrane pore size on the ceramic membrane performance. The overall removal efficiency is assessed in terms of both DOC concentration and UV254 absorption. As per our previous studies, humic acid and TiO_2 were used as the test pollutant and photocatalyst respectively.

2. Methods

2.1. Materials

Microfiltration (MF) and ultrafiltration (UF) ceramic membranes were used in this study. The ultrafiltration membrane used was a tubular titania ceramic membrane (Schumasiv^{TM}, Pall Co., Japan) made of a titania (TiO_2) coating on an alumina (α-Al_2O_3) support with pore size of 5 nm (approximate molecular weight cut off of 10 KDa) and an effective surface area of 48.38 cm^2. The microfiltration membrane used was a zirconia membrane (SchumasivTM, Pall Co., Japan) made of a zirconia (ZrO_2) coating on an alumina (α-Al_2O_3) support with pore size of 100 nm (approximate molecular weight cut off of 200 KDa) and an effective surface area of 50 cm^2. TiO_2 powder (P25 from Degussa) used in these experiments has a 50 m^2/g of active surface area and is composed of a 80/20 anatase/rutile ratio. In aqueous dispersions, TiO_2 forms large agglomerations and was measured as 300 nm. HA stock solution used in this work was prepared by mixing 6 g of HA powder (Fluka AG, Cheische Fabrik. CH-9470 Buchs, Switzerland) in 2 L deionized water, stirred over a period of 2 days with a magnetic stirrer. The feed solution (HA_0) containing 20 mg/L HA was prepared by appropriate dilution of stock solution. Six 18W UV-A lamps (NEC Blacklight Lamp) with a spectral irradiance of the UV ranges from 320–400 nm (UV-A, Photoelectric Instrument Factory of Beijing Normal University, China) were employed as UV light source.

2.2. Apparatus

The experimental rig (Fig. 1) was composed with a 2 L photocatalytic reactor, UV lamp and a cross flow filtration unit. The photocatalytic reactor with an illuminated surface area of the reactor was 713 cm^2 and made up of stainless steel with baffle plates, such that the water flowed in a zigzag manner through 5 open channels. A peristaltic pump (Masterflex 7592-45, Cole-Parmer, USA) was used to circulate the TiO_2 slurry at a flow rate of 0.5 L/min in the photocatalytic reactor and 1.2 L/min in the UF membrane and 0.8 L/min in the MF membrane.

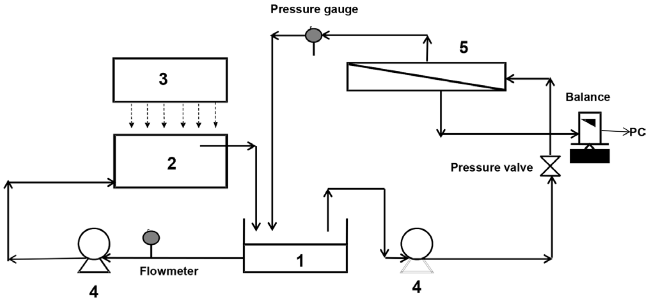

Fig. 1. Schematic diagram of the photocatalysis/ ceramic membrane system. (1) Feed tank (2) Photoreactor (3) UV lamp (4) Peristaltic pump (5) Ceramic membrane.

2.3. Experimental procedures

HA and TiO_2 slurry were mixed by magnetic stirrer in the dark for 15 min in order to obtain adsorption equilibrium before each photocatalytic treatment. The solution (2L), to be treated, was pumped from the feed tank into the photocatalytic reactor and was irradiated by UV for 1 hr. All the experiments were conducted at a temperature of at 20ºC and at UV intensity of 3.4 mW/cm^2. After photocatalytic reactions, the treated slurry from the batch photocatalytic reactor was feed into the tubular ceramic ultra and microfiltration membrane unit for removal and recovery of the TiO_2 particles. The permeate flux was continuously measured using a balance via data acquisition software. All experiments were performed at a temperature of 20ºC. The permeability of the ceramic ultrafiltration and microfiltration membranes were 0.29 and 2.6 $L/hr\ m^2$ kPa respectively.

2.4. Analyses

2.4.1. DOC, UV_{254} and turbidity measurements

Samples were collected at designated time intervals and filtered through 0.45 µm filter. The Shimadzu TOC V-CSH analyser was used to measure DOC concentration of the samples. The aromatic organic constituent of the water sample was measured by the absorption of the filtered sample at a wavelength of 254 nm against organic-free water as blank (UV_{254}–UV absorbing, Method 10054, HACH). The turbidity of the water sample was measured using HACH 2100 portable turbidimeter. The result shows that the ceramic UF and MF membranes are very effective for the separation of TiO_2 photocatalyst which were less than 0.15 and 0.2 NTU respectively.

2.4.2. Liquid chromatography (LC)

LC analyses were carried out using a TSK gel column (G3000 SW, C-No. SW 3600482) using a phosphate buffer (10 mM KH_2PO_4 + 10 mM Na_2HPO_4, 0.04 M, pH 6.8) as the mobile phase. The column was operated with a flow-rate of 0.5 mL/min and a 50 µL injection volume. This was coupled with a UV-visible photodiode array (λ = 200–800 nm). The LC column was calibrated using polystyrene sulphonate (PSS) compounds with molecular weight standards of 3420, 4600, 6200, 15,650 and 39,000 Da.

3. Results and discussions

3.1. Effect of pore size on the permeate flux and HA degradation

Fig. 2 shows the effect ceramic membrane pore sizes (UF and MF) on the permeate flux after UV/TiO_2 photocatalytic oxidation reaction. The initial pure water flux for ZrO_2 (MF) membrane was 270 $L\cdot/m^2\cdot h^1$ under TMP of 20 kPa and TiO_2 (UF) membrane was 28 $L\ /\cdot m^2\cdot h^1$ under TMP of 100 kPa. It is interesting to note that after 60 min filtration of TiO_2 suspension after photocatalytic treatment, ZrO_2 membrane flux reduced from 270 $L\cdot/m^2\cdot h^1$ to 164 $L\cdot/m^2\cdot h^1$ and TiO_2 membrane flux is reduced from 28 $L\ /\cdot m^2\cdot h^1$ to 27.4 $L\ /\cdot m^2\cdot h^1$. The observed differences in the normalised permeate flux behavior during TiO_2 and ZrO_2 membrane filtration was associated with the composition of humic acid after photocatalytic treatment and TiO_2 concentrations. Photocatalytic oxidation is very efficient in destroying aromatic structures in organic matter and increasing the hydrophilicity of the organics. The smaller the membrane pores, the more difficult for particles to penetrate them, resulting in less severe pore blocking in TiO_2 membrane compared to ZrO_2 membrane. A previous study also showed that the surface chemistry of ZrO_2 membrane had a significant influence on the filtration performance and organic fouling [22]. In particular, hydrophilic organic matter with low molecular weight had a specific contribution to organic fouling of ZrO_2 membranes. The results confirmed that smaller molecules have a stronger affinity to ZrO_2 membranes either forming a cake layer or causing pore blockage leading to higher flux decline. The TiO_2 membrane showed better effects in terms of permeate flux and turbidity removal rate. The turbidity of the TiO_2 suspension after photocatalytic treatment reduced from 2589 to 0.12 NTU and 0.25 NTU for the TiO_2 and ZrO_2 membranes respectively.

Fig. 3(a) shows that the DOC removal over the duration of hybrid treatment. The first 15 min of the reaction shows the adsorption of HA on the TiO_2 surface in the dark, ranging from 29 to 38% DOC removal and after 15 min, UV exposure commenced and after 1 hr of UV irradiation, the

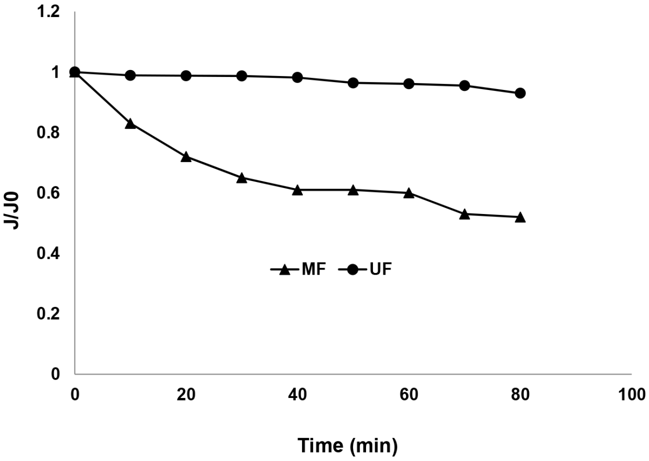

Fig. 2. Normalised flux vs time for different pore sizes of ceramic membranes (TiO_2 concentration: 0.5 g/L, HA concentration: 20 mg/L, TMP: 100 kPa (UF) & 20 kPa (MF), pH: 7.5; UV intensity: 3.4 mW/cm^2).

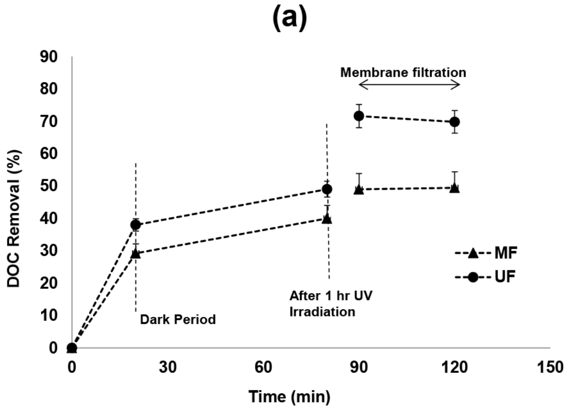

Fig. 3. (a) Dissolved organic carbon (DOC) removal efficiency; (b) reduction in UV absorbance of the photocatalytic—ceramic membrane integrated system for different pore sizes (HA concentration: 20 mg/L, TiO_2 concentration: 0.5 g/L, TMP: 100 kPa (UF) & 20 kPa (MF), pH: 7.5; UV intensity: 3.4 mW/cm^2).

DOC removal was 40 to 49%. The DOC removal by photocatalysis was low, which suggests the need for higher TiO_2 concentrations. The DOC removal after 1 hr ceramic membrane filtration was 70 and 50% for TiO_2 and ZrO_2 membranes respectively.

Fig. 3(b) represents the relative UV absorbance removal over the duration of the hybrid treatment. The reduction in UV absorbance after photocatalytic treatment was 55 to 56%. The reduction in UV absorbance after 1 hr of ceramic membrane filtration was 97 and 77% for 5 nm and 100 nm respectively (Fig. 3(b)). The DOC and UV 254 removal by the membrane alone was 30 and 19% and 93 and 48% for the TiO_2 and ZrO_2 membranes respectively. The lower removal of DOC and higher removal of UV 254 indicates that HA was not completely mineralised by photocatalytic oxidation but the aromatic structures of the HA were partially broken or changed to various forms with different adsorptive properties [1]. Results showed that membrane pore size and surface chemistry plays a role in the removal of DOC and UV absorbance through the rejection of HA adsorbed to TiO_2 particles, which is either adsorbed on the TiO_2 particle surfaces or dissolved in the concentrate phase.

3.2. *Effect of TMP on the permeate flux and HA degradation*

Fig. 4 presents an effect of TMP on the steady state permeate flux of ceramic ultrafiltration after TiO_2/UV photocatalytic treatment. The steady state flux increases with TMP for the all TiO_2 concentrations (0.1, 0.5 and 1 g/L). The overall flux was increasing linearly with increasing pressure in the range of TMP used. Further the steady state permeate flux shows closer agreement with the non-fouled membrane (clean water flux) performance. In addition, regression analysis conducted on the plots in Fig. 4 led to a value of 0.28. 0.26 and 0.25 l/hr m^2 kPa for the slope of the straight line for the TiO_2 concentrations of 0.1, 0.5 and 1 g/L respectively. These values are well compared to the value of the hydraulic permeability for clean water obtained for the ceramic membrane (0.29 l/hr m^2 kPa). This result also confirms that no significant fouling on ceramic ultrafiltration membrane at different catalysts loading and under different TMP [23,24].

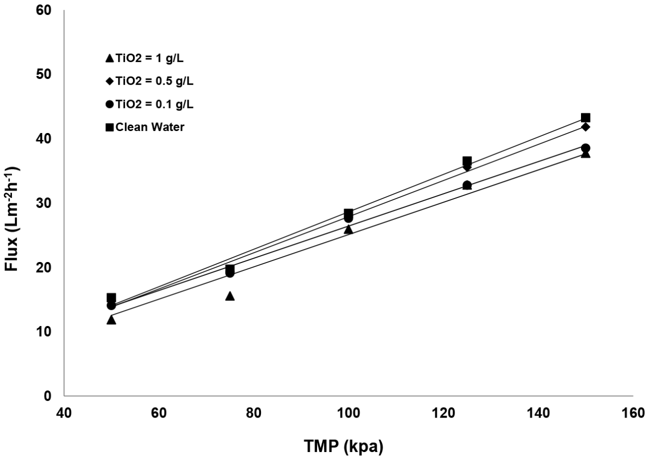

Fig. 4. Effect of TMP on the UF permeate flux under different TiO_2 concentrations (HA concentration: 20 mg/L, pH: 7.5; UV intensity: 3.4 mW/cm^2).

Previous studies have shown that coupling photocatalysis and pressure-driven membrane processes using polymeric membranes resulted in a decrease of the permeate flux and do not have the capacity to remove low-molecular organic compounds present in wastewaters as well as the products of their photo degradation [23,24]. Whereas in this study using the hybrid photocatalysis–ceramic UF process there was no significant flux decline observed. Moreover, no significant influence of photocatalyst concentration on permeate flux was found in this case.

The synergic effects of the integrated photocatalysis and ceramic ultrafiltration membrane system were evaluated in terms of DOC removal efficiency under different TMP under different TiO_2 concentrations (Fig. 5).

Photocatalysis alone was able to remove about 78% of DOC when 1 g/L of TiO_2 was used. When it was coupled with ceramic membrane filtration, the organic degradation efficiency of the integrated system was not showed further improvement at all TMP. When 0.5 g/L of TiO_2 was used, the photocatalysis alone was able to remove about 52% of DOC and combined with ceramic membrane filtration the DOC removal was more than 75%

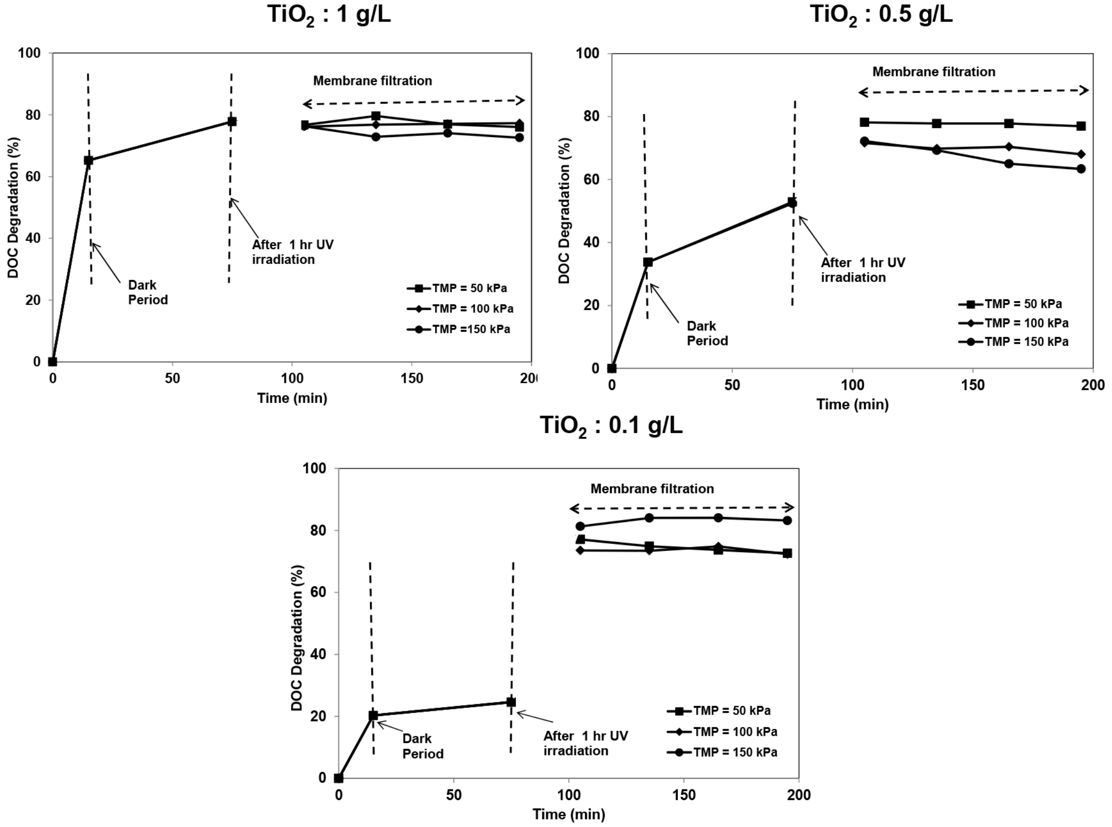

Fig. 5. DOC removal efficiency of the photocatalytic–ceramic ultrafiltration membrane integrated system under different TMP and various TiO_2 concentrations (HA concentration: 20 mg/L, pH: 7.5; UV intensity: 3.4 mW/cm^2).

under lower TMP and around 70% under higher TMP. While 0.1 g/l of TiO_2 was used, the photocatalysis alone was able to remove about 25% of DOC and combined with ceramic membrane filtration the DOC removal was more than 80% under higher TMP and around 75% under lower TMP. This result indicates that even at lower TiO_2 concentration, the overall DOC removal of the hybrid system increased to 75% compared to higher TiO_2 concentration, showing ceramic ultrafiltration could effectively remove the remaining DOC after the photocatalysis process at lower TMP.

Fig. 6 represents the relative UV absorbance during the entire treatment under different TMP. Photocatalysis alone was able to remove about 75% of UV absorbance when 1 g/L of TiO_2 was used. When it was coupled with ceramic membrane filtration, the reduction in UV absorbance was about 95% at all TMP and found more than 97% at lower TMP after 30 min of filtration. When 0.5 g/L of TiO_2 was used, the photocatalysis alone was able to remove about 50% of UV absorbance and combined with ceramic membrane filtration the reduction in UV absorbance was more than 95% under all TMP. While 0.1 g/L of TiO_2 was used, the photocatalysis alone was able to remove about 12% of UV absorbance and combined with ceramic membrane filtration the reduction in UV absorbance was more than 96% under all TMP. The result indicates that the ceramic membrane filtration could effectively remove relative UV absorbance even at low TiO_2 concentration and low TMP were used.

Results show that certain combinations of TiO_2 concentrations and TMPs provide significant improvements in both the DOC and UV254 rejection rates. For example, rejection of DOC is less sensitive to the increase in TMP when 1 g/L of TiO_2 was used. Whereas the rejection of DOC decreases when TMP increases for 0.5 g/L of TiO_2 was used. In the case of 0.1g/L of TiO_2, rejection of DOC increases when TMP increases. On other hand there were more than 95% rejections of UV254 for all theTiO_2 concentration and at all TMPs. In ceramic UF membrane filtration with lower TiO_2 dose, increase in TMP increases the rejection of DOC possibly due to un-destructed aromatics forming a cake layer on the membrane, not affecting the flux but enhancing the rejection. The results show that small TiO_2 dosages and UF with lower TMPs are effective in removing HA from water.

3.3. Liquid chromatography (LC)

Fig. 7 shows LC chromatogram of untreated feed, feed treated with TiO_2 and UV, and after 5 nm and 100 nm membrane filtration. The chromatogram of untreated HA consists of two main peaks denoted as peak 1 and 3 at molecular weights of approximately 650 and 50,000 Da, respectively. After photocatalytic oxidation treatment, 50% of peak 3 was removed so the reactions oxidised the organics and two new peaks denoted as '4' and '5' corresponding to 340 and 220 Da appeared due to the breakdown of organics into

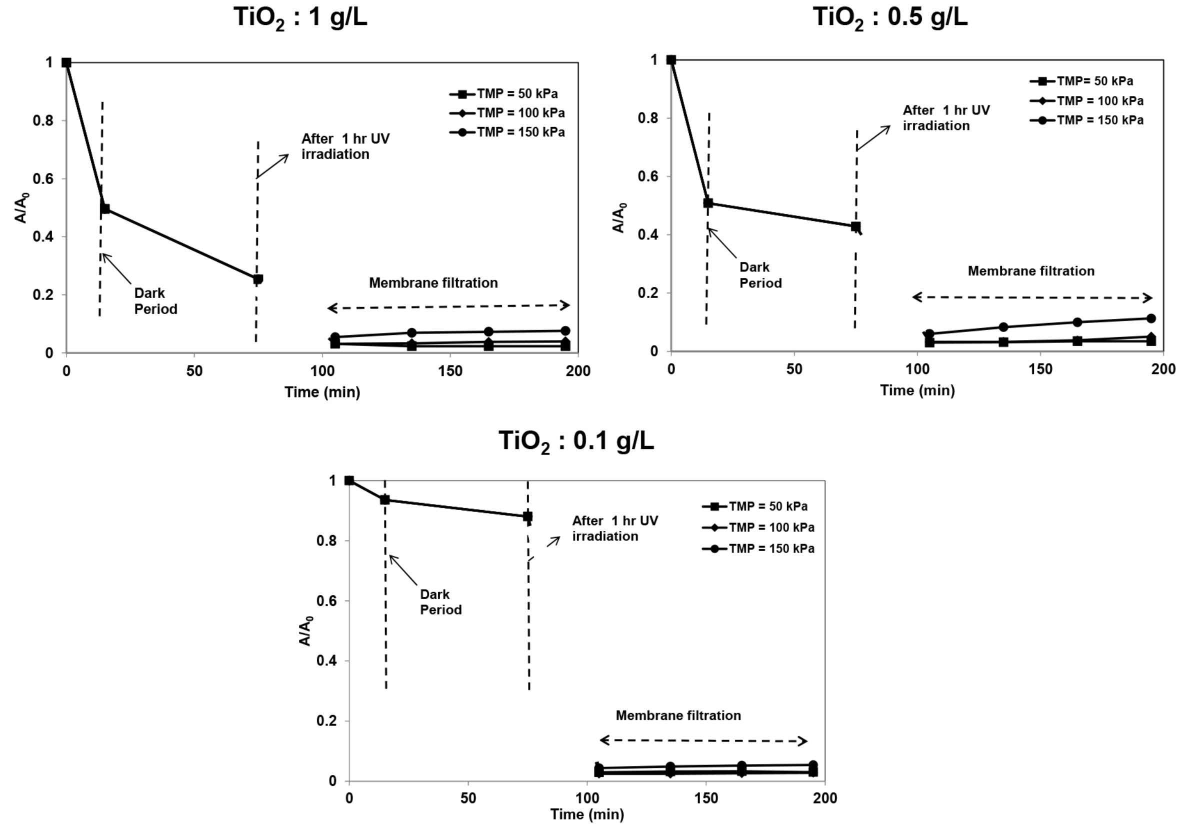

Fig. 6. Reduction in UV absorbance of the photocatalytic–UF ceramic membrane integrated system under different TMP and various TiO_2 concentrations (HA concentration: 20 mg/L, pH: 7.5; UV intensity: 3.4 mW/cm²).

smaller compounds. The increase in absorbance intensity of peak '2' from peak '1' of untreated HA sample after photocatalytic oxidation is possibly due to aggregation between small MW compounds and the successive degradation of by-products. Both the UF and MF membranes showed complete removal of larger molecular weight compounds (peak 2). UF membrane showed up to 50% removal of smaller molecular weight compounds whereas MF membranes showed very little removal of smaller molecular weight compounds (peak 4 and 5). As can be seen from the LC analysis, majority of the HA contains smaller molecular weight compounds in the range of 100–5000 Da. After photocatalytic treatment, there is a change in the molecular weight distribution of the compounds and preferential adsorption of those compounds by TiO_2 particles results in different kind of fouling in UF and MF membranes.

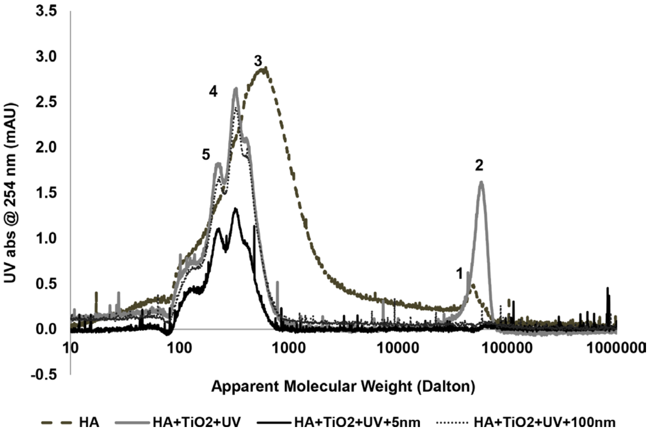

Fig. 7. LC analysis of HA at different stages of treatment (HA concentration: 20 mg/L, TiO_2 = 0.5 g/l, pH: 7.5; UV intensity: 3.4 mW/cm²).

4. Conclusions

A hybrid photocatalysis and ceramic membrane system was investigated by treating HA with different TMP and TiO_2 concentrations. The hybrid system results in synergistic effects including oxidation by the photocatalytic reaction, rejection of HA by ceramic membrane filtration and separation of TiO_2 particles from permeate by membrane rejection. The results reveal that the ceramic membrane filtration could effectively remove the remaining DOC and UV absorbance after the photocatalysis process even at lower TMP. The result indicates that the ceramic MF membranes showed higher flux decline compared to ceramic UF membrane after photocatalytic treatment. Results show that membrane filtration plays a role in the removal of DOC and UV absorbance through the rejection of HA adsorbed to TiO_2 particles, which is either adsorbed on the TiO_2 particle surfaces or dissolved in the concentrate phase. The LC analysis reveal that there is a change in the molecular weight distribution of the compounds after photocatalytic treatment leads to preferential adsorption of those compounds by TiO_2 particles as well as on membranes resulting different kind of fouling mechanisms on UF and MF membranes.

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