



# Performance of two photocatalytic membrane reactors for treatment of primary and secondary effluents



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## ABSTRACT

The performance of two photocatalytic membrane reactors (PMRs), utilizing ultrafiltration (UF) – PMR1, or direct contact membrane distillation (DCMD) – PMR2, during treatment of primary (PE) and secondary (SE) effluents of municipal wastewater treatment plant is presented. Additionally, single UF and DCMD were also examined. TiO<sub>2</sub> Aeroxide® P25 (0.5–1.5 g/dm<sup>3</sup>) was applied as a photocatalyst. Photocatalysis contributed to an improvement of the permeate flux in PMR1 compared to UF alone for 25–38% in case of PE and for 33% when SE were used. Based on SEM, AFM and contact angle measurements the flux improvement was attributed to the porous structure of TiO<sub>2</sub> cake and hydrophilicity of the TiO<sub>2</sub> – covered membrane. On the opposite, no flux decline during treatment of SE in PMR2 was found. However, in case of PE a decrease of the flux for ca. 40–50%, regardless of the presence or absence of TiO<sub>2</sub>, was observed. That was due to the formed dense fouling layer, which contributed to the increase of mass and thermal resistances. Permeate quality was higher in PMR2 than in PMR1. However, since mineralization of dissolved organic carbon in feed was very low, the treatment efficiency was attributed mainly to membrane separation rather than to photocatalysis.

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## 1. Introduction

Photocatalytic membrane reactors (PMRs) applied for treatment of aqueous solutions have gained significant attention of researchers in recent years. These hybrid systems couple advantages of both membrane separation and photocatalytic degradation of contaminants. Two main groups of the PMRs can be distinguished: (1) with photocatalytic membranes [1–7] and (2) with a photocatalyst suspended in the reaction solution [8–21].

Amongst various configurations of the PMRs described in the literature the systems utilizing pressure driven membrane processes, such as microfiltration (MF) [2,9–14,20–24], ultrafiltration (UF) [7–9,15–17,21,25–27] and nanofiltration (NF) [28] have mainly been investigated. Such systems were applied for removal of various contaminants, including pharmaceuticals [28,29], 4-chlorophenol [30], dyes [31,32], natural organic matter (NOM) [21,22,26] and others from water. Despite numerous advantages of these PMRs they exhibit also some drawbacks, mainly low permeate quality, especially in case of MF/UF [33], and membrane fouling due to the presence of photocatalyst (TiO<sub>2</sub>) particles [10,15,17,27,34,35]. However, most of the works concerning the

decline of flux caused by TiO<sub>2</sub> refer to the experiments during which either a suspension of the photocatalyst in distilled water [15] or in model solutions of various organic contaminants in pure water [21] were applied. In the former group of works the researchers focused mainly on the influence of photocatalyst loading on the permeate flux. Generally, an increase of the photocatalyst concentration in feed solution caused the flux decline as a result of the formation of a cake layer on a membrane surface [15,36].

Literature data which present the results of application of the hybrid systems for removal of NOM, e.g. fulvic (FA) and humic (HA) acids reveal that in the presence of TiO<sub>2</sub> and UV light an improvement of permeate flux compared to the single membrane operations can be obtained. Lee et al. [27] observed that when a mixture of HA and TiO<sub>2</sub> without UV irradiation was applied during UF, a significant permeate flux decline occurred due to formation of a dense filtration cake with humic acids molecules deposited in the void space of the TiO<sub>2</sub> particle layers. On the opposite, in the presence of UV light no flux decline was observed during 6 h of experiment. Since mineralization was not very efficient in the system, the authors explained the observed phenomenon in terms of decomposition of HA into non-aromatic by-products or compounds with less adsorptive properties than the initial contaminants [27]. The examples presented above can lead to a conclusion that the influence of TiO<sub>2</sub> on permeate flux in PMRs is dual. First, in the presence of TiO<sub>2</sub> a deterioration of the flux resulting from the formation

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**List of symbols and abbreviations**

|            |   |
|------------|---|
| AFM        | atomic force microscopy   |
| CSTR       | continuous stirred-tank reactor                                   |
| DCMD       | direct contact membrane distillation                              |
| DOC        | dissolved organic carbon  |
| DRS        | diffuse reflectance spectroscopy                                  |
| EDX        | energy-dispersive X-ray spectroscopy                              |
| FA         | fulvic acid   |
| FTIR       | Fourier transform infrared spectroscopy                           |
| HA         | humic acid  |
| J          | permeate flux   |
| MD         | membrane distillation   |
| MF         | microfiltration   |
| NF         | nanofiltration  |
| NOM        | natural organic matter  |
| PE         | primary effluent  |
| PES        | polyethersulfone  |
| PMR        | photocatalytic membrane reactor                                   |
| PMR1       | PMR coupling photocatalysis with UF                               |
| PMR2       | PMR coupling photocatalysis with DCMD                             |
| PP         | polypropylene   |
| PWF        | pure water flux   |
| $R_a$      | mean roughness  |
| SD         | standard deviation  |
| SE         | secondary effluent  |
| SEM        | scanning electron microscopy                                      |
| STP        | sewage treatment plant  |
| TDS        | total dissolved solids  |
| TIC        | total inorganic carbon  |
| TOC        | total organic carbon  |
| UF         | ultrafiltration   |
| UHR FE-SEM | ultra-high resolution field emission scanning electron microscope |

*List of units*

|     |  |
|-----|--|
| Da  | dalton, non-SI unit of mass, equal to the unified atomic mass unit |
| NTU | Nephelometric Turbidity Unit                                       |

of a cake layer on a membrane surface can take place. Second, when the feed solution contains NOM, especially humic and fulvic acid, application of  $\text{TiO}_2$  and UV irradiation can lead to improvement of the flux due to decomposition of organic molecules.

Despite numerous works on PMRs utilizing pressure driven membrane techniques which were used for organics removal from aqueous solutions there are very few reports on application of these systems for treatment of real water or wastewater. Kim et al. [10] used a PMR utilizing submerged MF membranes for treatment of seawater and lake water. The authors found that degradation of organic matter in the seawater was marginal whilst in case of the lake water almost 80% of total organic carbon (TOC) was removed. The low mineralization rate in the former case was attributed to the presence of chloride ions in seawater which were responsible for scavenging of the photoinduced radicals thus decreasing the photodegradation efficiency. Moreover, the authors did not observe any significant membrane fouling during 4 h of experiments [10]. A PMR utilizing the submerged MF membranes was also applied for treatment of river water [37]. The authors found that at a flux of  $1 \times 10^{-5}$  m/s a one month of operation without cleaning was possible. An increase of the flux up to  $1.5 \times 10^{-5}$  m/s required application of backwashing. Nonetheless, after 33 days of operation a severe membrane fouling was observed and chemical cleaning of the module was necessary. Another configuration of PMR, in which ceramic MF membranes were used, was applied

for treatment of Colorado River water [17]. However, the authors focused mainly on the efficiency of removal of selected pharmaceuticals and endocrine disrupting compounds and did not present any data on permeate flux behaviour during the process. A hybrid photocatalysis–MF process was also used for treatment of a synthetic wastewater [38]. However, the authors did not in fact applied a PMR but a photocatalysis–membrane integrated system. 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 ca. 30 NTU, and after that it was pumped into a submerged membrane reactor. Such a system allowed to reduce the membrane fouling and enhance the filtration flux [38]. Similar conclusions were drawn by Pidou et al. [39] who observed that photocatalytic pretreatment of synthetic greywater 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. It should be stressed, however, that the latter two configurations were integrated photocatalysis–membrane systems, but not PMRs.

Treatment of real wastewater with application of a UF  $\text{TiO}_2/\text{Al}_2\text{O}_3$  photocatalytic membrane was reported by Choi et al. [6]. The wastewater was obtained by filtration (1.2 mm glass fibre filter) of an activated sludge from a municipal wastewater treatment plant. The authors found that the composite membrane exhibited high water permeability, reliable organic retention, and anti-biofouling properties. Similarly as in case of the PMRs with suspended photocatalyst, the improved performance of the system was explained in terms of photocatalytic action of  $\text{TiO}_2$ . It was concluded that while the organic contaminants and microorganisms attached at the  $\text{TiO}_2$  membrane surface and interacted to form an adsorption fouling layer they were decomposed or their attachment strength was weakened.

Although the majority of works are focused on the hybrid systems utilizing MF, UF or NF, it should be noted that there are also some other configurations of PMRs [40–42]. Our group has proposed a PMR in which the photodegradation was combined with direct contact membrane distillation (DCMD) [43]. DCMD is a process in which only water vapour, volatile compounds and gases present in feed are transported through a microporous and hydrophobic membrane. The driving force, which is a vapour pressure difference on both sides of the membrane, depends on the temperature and composition of the solutions in the layers adjacent to the membrane. The volatile species evaporate from warm feed solution, diffuse and/or convect across the membrane pores and subsequently are condensed or dissolved in cold distillate. Due to the hydrophobic nature of the membrane the mass transfer of the liquid phase is prevented. In DCMD the rejection of non-volatile components, such as salts, amounts to (theoretically) 100% [44–46]. We have found that the main advantages of the PMR utilizing DCMD are a lack of membrane fouling due to presence of  $\text{TiO}_2$  particles and higher permeate quality compared to the PMRs in which the pressure driven membrane techniques are applied. The PMR utilizing DCMD was used for treatment of tap water contaminated with ibuprofen [47]. During the long term experiments a significant permeate flux decline took place due to scaling caused by  $\text{CaCO}_3$  crystals. However, we have concluded that the  $\text{TiO}_2$  layer on a membrane surface might prevent from deposition of  $\text{CaCO}_3$  inside the membrane pores thus protecting the membrane from damage and elongating its lifetime. Moreover, we have observed that application of cleaning with HCl solution allowed to dissolve the  $\text{CaCO}_3$  scale deposit and recover the permeate flux.

The literature overview presented above revealed that application of PMRs could be a promising way of removal of organic contaminants from aqueous solutions. However, further studies on these hybrid systems are necessary, especially with reference to treatment of real water and wastewater.

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