



# Ceramic membrane separation coupled with catalytic ozonation for tertiary treatment of dyestuff wastewater in a pilot-scale study



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## HIGHLIGHTS

- Coupling catalytic ozonation with ceramic membrane separation was piloted.
- Catalytic ozonation effectively mitigates membrane fouling.
- COD, SS, *E. coli* and chromaticity color were effectively removed.
- Effluent did not show genotoxicity risk to aquatic organism.

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## ABSTRACT

An apparatus coupling catalytic ozonation with ceramic membrane separation was designed and piloted for the tertiary treatment of dyestuff wastewater from Anaerobic–Anoxic–Oxic effluent. The apparatus's water treatment capacity was about 10 t/d at the average permeate flux of 110 L/(m<sup>2</sup>·h). After the treatment of the dyestuff wastewater, COD<sub>Cr</sub> decreased from 100 ± 20 mg/L to <10 mg/L, suspended solids drastically reduced from 20 ± 5 mg/L to <0.1 mg/L and *Escherichia coli* was completely removed from the initial concentration of >2.4 × 10<sup>6</sup> MPN/L. Chroma also got a large decrement from 90 ± 10 to <2 at dilution times. Furthermore, genotoxicity showed an effective abatement detected by comet assay. The high performance of the designed apparatus in the treatment of the dyestuff wastewater was associated with the synergistic effects of catalytic ozonation and membrane separation. This work indicates that the system coupling catalytic ozonation with membrane separation will be an alternative approach for the advanced wastewater treatment in practical application.

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## 1. Introduction

Dyestuffs are widely used in many industries such as textile, leather and rubber industry, but cause serious environmental problems such as a threat to the growth of aquatic lives and the self cleaning of waters when released into rivers and lakes due to their characters of high chemical oxygen demands (COD), suspended solids (SS) and intense color etc. [1]. Hence, the treatment of dyestuff wastewater has received considerable concerns in the past decades. Conventional biological treatment process is an economical alternative compared with other treatment options [2]. However, the treatment of dyestuff wastewater is not effective due to the stable nature of dyestuffs that are associated with a large degree of aromatic structure [3]. Moreover, secondary effluent of

dyestuff wastewater still retains residue dyestuff and some microbes, which present potential risks to aquatic organisms and human health if directly discharged into natural water bodies [4]. Thus, it is necessary to treat the dyestuff wastewater with advanced technologies associated with traditional wastewater treatment processes to improve water quality and minimize the potential risks.

Over the last decades, membrane separation technology has gained increasing attention in the advanced wastewater treatment. Although membrane separation process can availably block substances with sizes larger than the membrane pores, it is difficult to reject substances smaller than the membrane pores, and its application is restricted by membrane fouling problem [5,6]. To address this challenge, researchers have tried to integrate some advanced oxidation technologies with membrane separation. For instance, coupling ozonation with membrane separation (OMS) has shown beneficial effect in increasing recalcitrant pollutants

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removal [7] and mitigating membrane fouling [8], and displayed good capability in reducing organic compounds, chroma and recalcitrant micropollutants during the treatment of drinking water or natural water [9–11]. Barry et al. [12] proposed that preozonation could mitigate the membrane fouling behavior caused by biological colloids. Qi et al. [13] reported that ozonation as a pre-treatment increased the pollutant removal. Byun et al. [14] found that preozonation significantly improved the permeate flux of nanofiltration. Besides, the treatment efficiency achieved by OMS mainly depends upon the characteristics of the feed water [15]. Given the complex composition of secondary effluent, the problem of membrane fouling would be worse, and it is not economical to use OMS in the secondary effluent treatment unless the problem is effectively solved [16,17].

Alternatively, membrane fouling can be further mitigated when membrane separation is combined with catalytic ozonation due to the enhanced oxidation of foulants by the generation of reactive oxygen radicals such as hydroxyl radicals [18]. However, in previous studies, catalytic ozonation was used as a pre-treatment step of membrane separation, that was, isolated from the membrane separation [19,20]. In such situation, the produced reactive oxygen radicals decayed before they reacted with the precipitates on membrane surfaces due to their short half-life, which indicated that membrane fouling could not be effectively mitigated [21]. Catalysts coated on ceramic membrane make it possible for catalytic ozonation and membrane separation process to proceed simultaneously, which means that membrane foulants can be timely decomposed before they accumulate and deposit on the membrane surface [22]. Therefore, employing an efficient ozonation catalyst of Ti–Mn that was used in our previous work [23], an apparatus coupling catalytic ozonation with ceramic membrane separation was designed for the tertiary treatment of dyestuff wastewater.

Thus, this study was in connection with our previous study, but explored the apparatus in the tertiary treatment of dyestuff wastewater, and identified that the apparatus showed good performance in terms of the quality of the effluent and the permeate flux. Firstly, Ti–Mn catalyst was successfully coated on the tubular ceramic membrane and characterized. Secondly, the system coupling ceramic membrane separation with catalytic ozonation performed well in  $COD_{Cr}$  removal and mitigating membrane fouling for the tertiary treatment of dyestuff wastewater in bench scale. Lastly, the designed apparatus on the optimal conditions (operating pressure of 0.2 MPa, ozone dose of 2.5 mg/L) was practically applied in the dyestuff wastewater treatment for 30 days, when permeate flux, effluent quality including  $COD_{Cr}$ , chroma, SS, *Escherichia coli* as well as the genotoxicity were evaluated.

## 2. Experimental

### 2.1. Membrane preparation

The membrane was prepared as reported in our previous work [23]. In brief, commercial ceramic membranes from Guangzhou JieXi LiShun Co., Ltd., China were immersed into rutile  $TiO_2$  suspension (10 wt%). After immersion, the membranes were dried at room temperature and were sintered at 1273 K for 1 h to get the intermediate layer. Then the membranes were immersed into the pre-prepared titanium and manganese sol (45 g pluronic F127, 71.5 mL acetic acid, 250 mL titanium (IV) isopropoxide, and 38.45 mL manganese (II) nitrate solution (50%) were one by one dissolved in 1000 mL ethanol under vigorous stirring), dried at 323 K for 12 h and then calcined at 823 K for 2 h to obtain the catalytic layer.

### 2.2. Feed wastewater

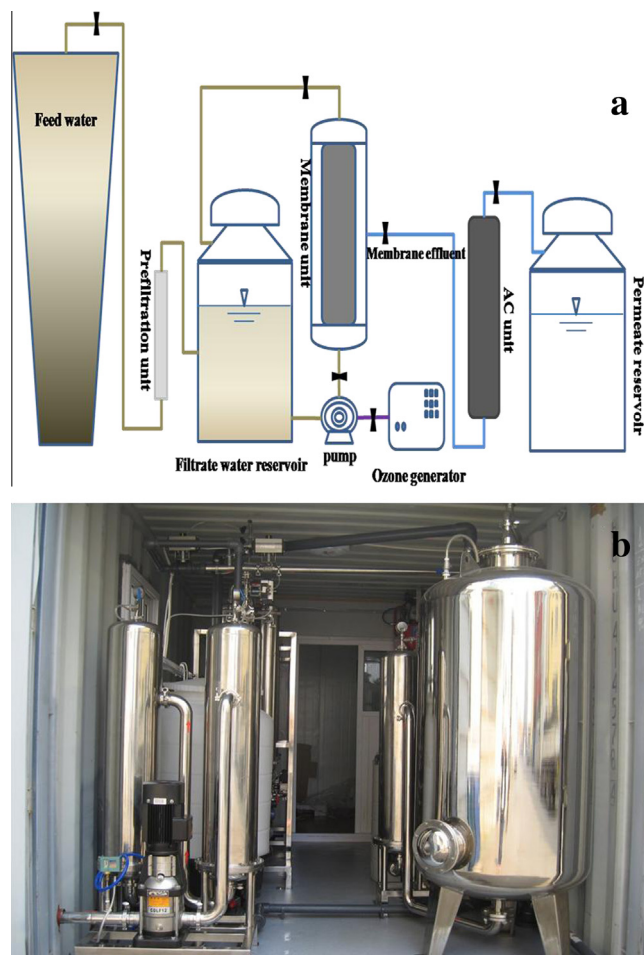
Feed water was obtained from a dyestuff wastewater treatment plant with Anaerobic–Anoxic–Oxic process in Northeast China. Secondary effluent of the dyestuff wastewater was collected and stored at 4 °C for bench scale experiments, and it was directly pumped on site for pilot scale tests. Parameters for water quality of the secondary effluent are given in Table 1.

### 2.3. Experimental units

Two devices were used in this experiment for both the bench and pilot scales, which were of the same principle but different scales. Fig. 1 shows the schematic representation and on-site picture of the apparatus, mainly consisting of three units. First, the pre-filtration unit (5  $\mu$ m fiber filter) was used to remove particulate matter from wastewater to inhibit membrane fouling. Second, the

**Table 1**  
Parameters of the feed water.

Parameters	Values
pH	7.5 $\pm$ 0.5
COD (mg/L)	100 $\pm$ 20
UV <sub>254</sub> (cm <sup>-1</sup> )	1.6 $\pm$ 0.2
SS (mg/L)	20 $\pm$ 5
Chroma (dilution times)	90 $\pm$ 10
<i>E. coli</i> (MPN/L)	>2.4 $\times$ 10 <sup>6</sup>



**Fig. 1.** (a) Schematic representation and (b) On-site picture of the apparatus.

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