



Micropollutants removal from water using microfiltration membrane modified with ZIF-8 metal organic frameworks (MOFs)



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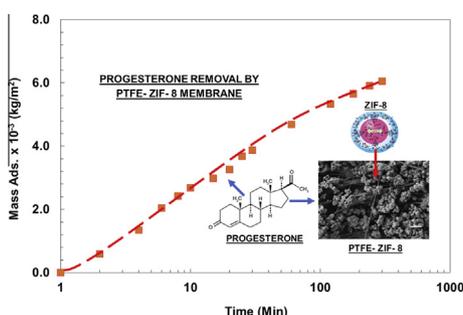
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HIGHLIGHTS

- PTFE membrane is modified with ZIF-8 MOF for adsorption of micropollutants.
- Membrane characterization before and after particles incorporation is done.
- Adsorption properties are measured using progesterone as a model pollutant.
- The modification increased adsorption by ~40% and almost doubled permeability.
- A model is presented that described adsorption characteristics adequately.

GRAPHICAL ABSTRACT



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ABSTRACT

A polytetrafluoroethylene (PTFE) double layer microfiltration membrane has been modified with zeolite imidazolate metal organic frameworks-8 (ZIF-8) and tested for micropollutants removal from water. The physical properties of the modified membrane and its removal characteristics are tested using progesterone as a model pollutant. Compared to the unmodified membrane, incorporation of ZIF-8 onto the polymer matrix resulted in close to 40% increase in the membrane adsorption capacity and almost doubling its water permeability which could result in significant reduction in processing specific energy consumption. The membrane maintained near 95% of its original removal efficiency after three regeneration cycles using polyethylene glycol-400. Modeling of the adsorption results agreed satisfactorily with the experimental measurements. The synergy provided by using ZIF-8 particles and the significant regeneration volume reduction ratio demonstrates the effectiveness of the approach for increasing the overall micropollutants removal capacity and allows for its further consideration for development of viable continuous membrane adsorption techniques for decontamination of water resources.

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1. Introduction

Micropollutants such as pharmaceuticals, pesticides, and endocrine disrupting chemicals are currently the focus of a growing

body of scientific research due to their potential risk to both human and aquatic environment [1–5]. Micropollutants may be released to the environment through sewage discharge due to the relatively low removal efficiency of such compounds by conventional systems [6], and different approaches have been investigated to address this issue including biological, advanced oxidation, and physical processes [7]. For the latter, the main

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applied strategies involve using either packed bed-type adsorption, or filtration separation techniques. In the first, adsorbing materials such as activated carbon, chitosan and carbon nanotubes have been tested. This approach however faces several difficulties such as recovery of the adsorbing material, high pressure drop and clogging, and slow mass transfer [8,9]. In the second type, and since packing is not necessary, problems due to channeling, bed compression, and intraparticle diffusion can be avoided. It also provides relatively shorter bed height and larger pores for convective flow, as well as simpler linear scale-up by using more membranes or a spiral-wound configuration. However, conversion to equivalent bed volume may be necessary if the technique is to be compared to packed bed adsorption.

In membrane processes, both the removal mechanism and its efficiency varies depending on the physicochemical properties of the compound and the membrane, as well as the hydrodynamics of the process [10,11]. In previous studies, micropollutants removal by size exclusion using nanofiltration membranes have been investigated, and the effect of sorption on pollutants breakthrough was reported [12–16]. Such unwanted mechanism may present an opportunity for micropollutants removal based on novel highly adsorptive materials using ultrafiltration or microfiltration (MF) membrane platform. This difference, together with the membrane permeability, fouling tolerance, and the required transmembrane pressure, can be key factors in determining the overall process productivity and specific energy consumption. In this regard, MF based materials may present an attractive choice due to their low cost and least pumping energy, which is a major cost element in membrane separation processes.

Removal of estrogens pollutants from water resources using MF membranes has been successfully demonstrated and sorption capacity $\sim 0.87 \mu\text{g}/\text{cm}^2$ using a $0.45\text{-}\mu\text{m}$ polyamide membrane has been reported [17]. In a comparative pollutants adsorption study using different types of membranes, it was demonstrated that membranes with larger pore sizes and higher porosity have higher compound adsorption compared to tighter membranes. This was explained by the fact that solute adsorption is not only restricted to membrane surface but can also occur in the skin and support layers as well as the pores [12]. Accordingly, larger pore sizes and higher porosity membranes would have more access to adsorption sites compared to tighter membranes with limited access [18,19]. To further augment a membrane adsorptive properties, incorporation of functional particles onto its polymeric matrix have been successfully demonstrated for separation applications of biomolecules and pollutants [20–23].

Inspired by the above, the main objectives of this research is to investigate the potential of developing an adsorptive membrane material that exhibits both high water permeability as well as micropollutants removal capacity. This was achieved by incorporating zeolite imidazolate metal organic frameworks (ZIF-8) particles onto the polymeric matrix of a highly porous double layer

polytetrafluoroethylene (PTFE) microfiltration membrane material. The membrane was selected based on chemical durability, commercial availability at low cost, as well as its relatively large thickness ($\sim 300 \mu\text{m}$) which can potentially provide large number of sorption sites and enhance its mechanical strength. The ZIF-8 particles used is a member of the recently developed metal organic frameworks (MOFs) family that gained interest in many separation processes [24,25]. It possess exceptional thermal and chemical stability as well as simple preparation method and low raw material cost. The possible advantage of its large surface area on adsorption enhancement could be further augmented by the potential formation of fast water transport channels akin to aquaporin in cell membranes for higher water permeability [26]. The developed PTFE-ZIF-8 membrane is tested for removing micropollutants from water using progesterone (PGS) as a model pollutant compound. Different concentrations were tested including values higher than typically found in water effluents to determine the membrane adsorption limits. Characterization and adsorption studies are performed for ZIF-8 particles and the membrane materials using batch stirring and flow through conditions, and the effect of the membrane regeneration on its removal efficiency is presented.

2. Experimental

2.1. Materials and methods

The PTFE membrane was kindly donated by Shanghai LingQiao E.P.E.W. Co., Ltd. (Shanghai, china), and was made of a MF layer on top of a looser support material. All other chemicals were purchased from Sigma–Aldrich. ZIF-8 MOFs was synthesized as reported before [27] by dissolving a solution of the metal precursor $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in methanol followed by addition of 2-methylimidazol organic ligand solution. The mixture was stirred for one hour at room temperature and the precipitated crystals were separated by centrifugation and dried in vacuum oven (120°C) for 24 h (Fig. 1). The modified membrane was prepared by solvent evaporation by mixing different weight percentages of ZIF-8 particles in deionized water at 50°C for 12 h followed by sonication for 30 min. The PTFE membrane sheets were then cut into a predetermined size and immersed in the ZIF-8 solution for 24 h followed by drying in a vacuum oven at 40°C for 3 h. Samples with up to 20 wt.% particle loading were prepared since higher loading led to particle agglomeration.

2.2. Material characterization

The material morphology was defined using *field emission scanning electron microscope (FESEM)*, Hitachi High-Technologies GmbH, Germany. Samples were prepared on aluminum stubs, coated with gold and measured at an accelerating voltage of 20 kV. *Energy dispersive X-ray spectroscopy (EDX)* was used for

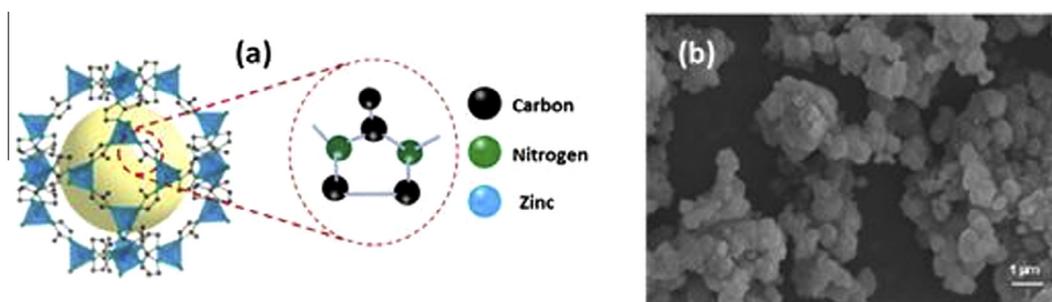


Fig. 1. Zeolite Imidazole Framework 8 (ZIF-8) (a) schematic crystalline structure, and (b) particles FESEM image.

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