



## Effect of interactions between $\text{Co}^{2+}$ and surface goethite layer on the performance of $\alpha\text{-FeOOH}$ coated hollow fiber ceramic ultrafiltration membranes



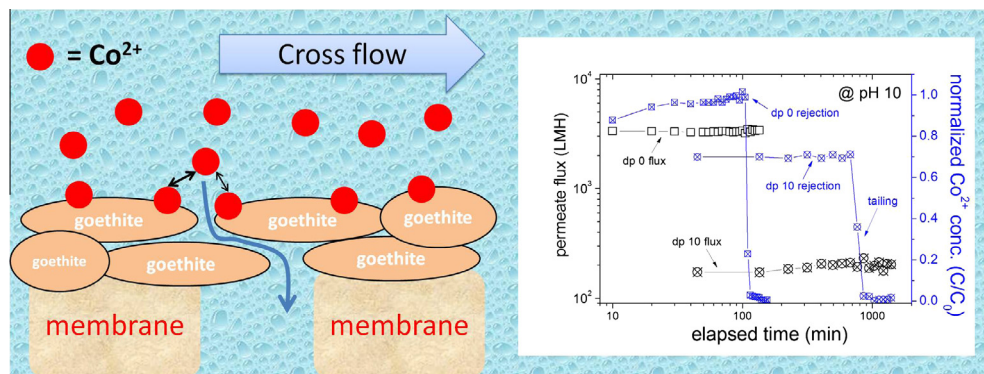
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### GRAPHICAL ABSTRACT



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### ABSTRACT

The consideration of water energy nexus inspires the environmental engineering community to pursue a more sustainable strategy in the wastewater treatment. One potential response would be to enhance the performance of the low-pressure driven filtration system. To reach this objective, it is essential to have a better understanding regarding the surface interaction between the target substance and the surface of membrane. In this study, the hollow fiber ceramic membranes were coated with a goethite layer in order to enhance the  $\text{Co}^{2+}$  rejection. Experimental results indicate that higher  $\text{Co}^{2+}$  rejections are always accompanied with the significant reduction in the permeability. Based on the consideration of electroviscous effect, the surface interactions including the induced changes in viscosity, pore radius and Donnan effect in the goethite layer are likely responsible for the pH dependent behaviors in the rejection and permeability. These results could be valuable references to develop the filtration system with high rejection along with acceptable degree of permeability in the future.

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## 1. Introduction

Membrane technology is known capable of separating target substances out of a large matrix on an industrial scale. Regarding wastewater treatment, a variety of membranes are commercially available, whose nominal pore size ranges from angstroms (reverse osmosis membranes) to micrometers (microfiltration membranes), which allows environmental engineers to design and optimize the treatment protocol for different wastewaters. Among these available membranes, the utilization of RO filtration is known to reject mineral ions excellently at much smaller costs and much higher efficiency. The consideration of water energy nexus [1], however, inspires the environmental engineering community to pursue a more sustainable strategy in the wastewater treatment. One potential response would be to enhance the rejection efficiency of the low-pressure driven filtration system as it greatly reduces the energy consumption during the filtration process. To improve the efficiency, surface modification by coating a thin layer of functional material is frequently used in the material chemistry community. For example, a surface polyvinyl alcohol layer was found to greatly enhance the surface hydrophilicity of fully aromatic polyamide (PA) membranes [2]. Ultrafiltration of Cr(VI) using an analcime-C zeolite-coated ceramic membrane was reported to have a rejection of up to 84% compared with the unmodified membranes [3]. The surface hydrophobicity of titania ceramic membranes could be adjusted by grafting with different lengths of fluorine alkyl chains of perfluoroalkylsilanes because perfluoroalkylsilane molecules with shorter fluorocarbon chains are primarily attached via siloxane and geminal silanol bonds, while those with longer chains are mainly associated through single and geminal silanol bonds [4]. Surface deposition with a diatomite-kaolin composite layer was reported to significantly reduce the pore size of sintered diatomite membranes while retaining an acceptable level of permeability [5].

The functionality of a membrane, such as the selectivity and rejection capacity, could be enhanced by its surface modification, but the consequential trade off in permeability loss is inevitable. This is because the modification layer is to some degree similar to the fouling layer that greatly increases the hydraulic resistance of the modified membranes [6]. Moreover, because solutes travel much slower in the tortuous paths of the decoration layer due to the surface interaction effect, their accumulation due to hindered back diffusion to the bulk solution would generate an extra concentration gradient due to the concentration polarization effect. This phenomenon is known as cake-enhanced osmotic pressure (CEOP), which further compromises the permeability of modified membranes [7]. The CEOP effect in a surface-modified membrane is also expected to be more significant than in a fouled membrane because the modification layer typically is securely bound to the membrane surface by the subsequent thermal treatment. Thus, the modification layer is more rigid and resistant to the hydraulic flow than the fouling layer, causing difficulty for the exposure of the solutes in the modification layer to the shear by tangential flow [8].

The balance between permeability loss and performance enhancement is always an issue for engineers but is an interesting research topic for scientists. This is because once the dominant mechanism that controls this trade-off is fully understood, a great leap in performance enhancement along with an acceptable degree of permeability loss can be expected. Pursuing this objective relies on acquiring knowledge regarding the surface interactions between the solute and its surrounding tortuous path in the decoration layer. For example, mineral salt rejections by nanofiltration using a positively charged alumina membrane were shown to follow the order of divalent cation, monoanion > monocation,

monoanion ~ dication, dianion > monocation, dianion. The authors concluded that this phenomenon was closely correlated with the relative ratio of the coulombic, dielectric and hydration interactions between the membrane surface and the ionic species [9]. Donnan exclusion and the formation of an electrical double layer in the tortuous paths are proposed to explain the higher rejection of trications ( $\text{La}^{3+}$ ) than in dications ( $\text{Mg}^{2+}$ ) by the mesoporous  $\gamma\text{-Al}_2\text{O}_3$  membrane with a nominal pore size of 3.4–8.7 nm [10]. These studies have unambiguously addressed that the knowledge of surface interactions between target substances and the membrane surface is the key to meet the merit of water energy nexus consideration in the membrane process system.

In this study, a thin goethite layer was coated on the inner walls of mullite-based hollow fiber ceramic membranes through the simple dip coating method. Factors including the number of dip coating cycles and operation pH were discussed. Unlike previous reports, we focus on the discussion regarding the surface interaction effect between  $\text{Co}^{2+}$  and surface of goethite layer on the rejection and permeability. The knowledge acquired would be important reference for the development of an appropriate surface modification protocol to establish a filtration system possessing high performance and permeability, including the most frequently used organic membranes.

## 2. Experimental

### 2.1. Chemicals and the preparation of goethite-coated hollow fiber ceramic ultrafiltration membranes

All of the chemicals used in this study were ACS grade and were purchased from Sigma–Aldrich. Milli-Q water (18.2 M $\Omega$ ) was used without any further purification. Mullite-based hollow fiber microfiltration membranes were prepared based on the phase inversion method. Their inner diameter and wall thickness was approximately 1.0 and 0.5 mm, respectively, with a nominal pore diameter of approximately 1.02  $\mu\text{m}$  [11]. The goethite nanoparticles were prepared via precipitation by the dropwise addition of 3.0 M NaOH to a 1.0 M  $\text{Fe}^{3+}$  solution (prepared by dissolving an aliquot amount of  $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ) until the supernatant became transparent [12]. Goethite precipitates were aged for 24 h and then were collected by centrifugation. Then, the precipitates were dispersed again in Milli-Q water to remove residues; this cleaning process was repeated three times. The goethite suspension used in the dip coating process was composed of 0.4 g goethite nanoparticles (dried at 60 °C) and 10 mL Milli-Q water (solid/liquid ratio, 40 g/L) in a polypropylene centrifuge tube [13]. Prior to each dip coating step, the outer walls of these membranes were covered with a thick layer of Teflon tape because the decoration was performed to modify the inner walls, which would be the filter area exposed to feed water in the subsequent filtration experiments. To deposit the goethite layer as homogeneously as possible, membranes with a relatively short length (35–55 mm) were immersed in the pre-ultrasonicated goethite suspension for one minute. At the end of the immersion, these membranes were air dried and then subjected to a short period of thermal treatment (100 °C, one hour) to secure the deposited goethite nanoparticles. The dip coating process was repeated 1–15 times, and the membranes were named, for example, dp 15, representing the 15 cycles of the dip coating process.

### 2.2. Characterization

The goethite phase was confirmed by X-ray diffraction (XRD) analysis with Cu K $\alpha$ 1 radiation (0.154056 nm, D2 Phaser diffractometer, Bruker). The specific surface area and porous property

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