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Toward Resource Recovery from Wastewater: Extraction of Phosphorus from Digested Sludge Using a Hybrid Forward Osmosis–Membrane Distillation Process

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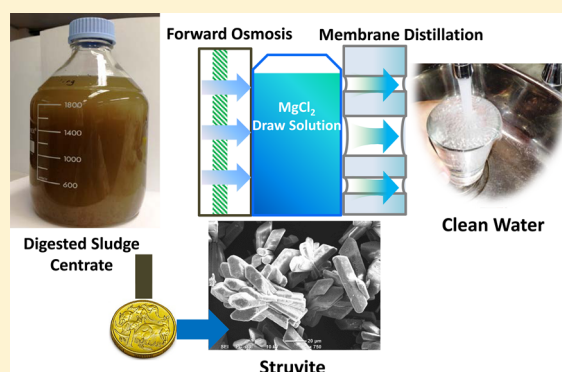
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S Supporting Information

ABSTRACT: We demonstrate the simultaneous extraction of phosphorus and clean water from digested sludge centrate using a forward osmosis (FO)–membrane distillation (MD) hybrid process. In this FO–MD hybrid process, FO concentrates orthophosphate and ammonium for subsequent phosphorus recovery in the form of struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$), while MD is used to recover the draw solution and extract clean water from the digested sludge centrate. A decline in water flux was observed during the FO process, but fouling was largely reversible after a brief, simple membrane flushing using deionized water. The FO process also provides an effective pretreatment capacity to the subsequent MD process, which exhibited stable water flux. The use of MgCl_2 as the draw solute for the FO process is another novel aspect of the system. The reverse salt flux of magnesium to the concentrated digested sludge across the FO membrane and the diffusion of protons away from the digested sludge create favorable conditions for the formation of struvite crystals. The precipitates obtained in the hybrid process were verified to be struvite crystals by examining the crystal morphology, element composition, and crystal structure. Results reported here highlight the potential and robustness of the FO–MD hybrid process for extracting phosphorus from wastewater.



INTRODUCTION

Phosphorus is an essential nutrient for plants. The flow of phosphorus from minable phosphate rocks to farm land and ultimately the natural waterway has accelerated in the past few decades because of the industrialization of agricultural production.¹ Thus, the global phosphorus reserve is being depleted at a rate that could seriously threaten food security.² Phosphorus is also a major contaminant, and its release to the aquatic environment is responsible for algal bloom and other severe ecological impacts.³ A promising approach to ensuring a renewable supply of phosphorus and protecting the environment is to extract phosphate ions from livestock and human wastes in the form of struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) mineral,⁴ which is an excellent slow release fertilizer.⁵

Previous studies have demonstrated the extraction of phosphorus as struvite from wastewater,^{6,7} anaerobically digested sludge,^{8–12} and urine.^{13,14} Several innovative techniques for recovering struvite from wastewater have also been proposed. Cusick and Logan¹⁵ reported the recovery of struvite and simultaneously produced valuable hydrogen gas using a microbial electrolysis cell. Zhang et al.¹⁶ employed an

electrodialysis process to fractionate phosphate for subsequent struvite precipitation. The formation of struvite requires the addition of magnesium, which is a high-value commodity. However, the demand for magnesium can be reduced if phosphate and ammonium can be enriched from a source solution.

Forward osmosis (FO) has the potential to concentrate phosphate, ammonium, and magnesium in the source solution to facilitate struvite recovery. High rates of rejection of phosphate and ammonium from activated sludge¹⁷ and wastewater¹⁸ by FO membranes have been reported. At the same time, the use of magnesium-based salts (e.g., MgCl_2) as the draw solute can enhance struvite formation because of the inherent bidirectional diffusion of ions in the FO process.¹⁹ Reverse diffusion of magnesium into the feed solution will increase the magnesium ion concentration of the feed side,

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while the coupled diffusion of protons from the feed to the draw solution will increase in the feed solution pH,²⁰ with both conditions favoring struvite precipitation. FO can not only serve as a stand-alone process in osmotic dilution to extract clean water^{21,22} but can also be integrated with other processes to recover clean water and to concentrate and recycle the draw solute.²³ In a recent study, Xie et al.²⁴ coupled FO with membrane distillation (MD) for direct sewer mining, demonstrating stable water flux and high-quality product water by this hybrid FO–MD system.

In this study, we demonstrate for the first time the extraction of phosphorus from anaerobically digested sludge by an FO–MD hybrid system with a MgCl_2 draw solution. System efficiency was examined in terms of nutrient concentration and product water recovery. The bidirectional diffusion of magnesium and protons, which facilitates struvite precipitation, was also quantified.

MATERIALS AND METHODS

FO–MD System. A lab-scale FO–MD system was employed to extract water and phosphorus from digested sludge. The FO–MD system comprised two identical membrane cells (for the FO and direct contact MD process, respectively), four circulation pumps, and a temperature control unit (Figure S1 of the Supporting Information).

Digested Sludge and Membranes. Anaerobically digested sludge was collected from an anaerobic digester of a sewage treatment plant with biological nutrient removal (BNR) in Wollongong, New South Wales, Australia. The centrate was obtained by screening the sludge through a 0.5 mm sieve and then centrifuging at 3750 rpm for 20 min (Allegra X-12R, Beckman Coulter, Fullerton, CA). Key physicochemical properties of the digested sludge centrate are summarized in Table 1.

Table 1. Key Physicochemical Properties of the Digested Sludge Centrate (average \pm standard deviation from duplicate measurements)

solid content (mg/L)	1800 \pm 100
turbidity (NTU)	524 \pm 9
electrical conductivity (mS/cm)	6.72 \pm 0.12
pH	7.72 \pm 0.02
total organic carbon (mg/L)	647 \pm 20
total nitrogen (mg/L)	783 \pm 11
ammonium (mg/L)	538 \pm 25
phosphate (mg/L)	223 \pm 12
magnesium (mg/L)	10.5 \pm 2.1

A flat-sheet, cellulose triacetate membrane from Hydration Technology Innovations (Albany, OR) was used for the FO process. A microporous, hydrophobic membrane from Porous Membrane Technology (Ningbo, China) was used for the MD process. Key properties of the FO and MD membranes are summarized in Table S1 of the Supporting Information.

Experimental Protocol for Water and Phosphorus Recovery. Digested sludge centrate was processed continuously by the FO–MD hybrid system until 1 L of permeate had been produced. Initial volumes for digested sludge centrate, draw, and distillate solutions were 1.5, 2, and 1 L, respectively. For the FO process, a 1.5 M MgCl_2 draw solution at 40 °C generated a water flux of 9 L $\text{m}^{-2} \text{h}^{-1}$. In the MD process, the same water flux was obtained at draw and distillate temper-

atures of 40 and 20 °C, respectively. Water fluxes for the FO and MD processes were recorded continuously. Digested sludge centrate, draw, and distillate samples were taken at specific time intervals for further analysis. All experiments with the FO–MD hybrid system were conducted in duplicate.

To mitigate FO membrane fouling and balance the water fluxes of the FO and MD processes, the FO system was flushed using deionized water for 15 min at a cross-flow velocity of 18 cm/s (twice the value used in the FO–MD process) when the FO water flux decreased to 50% of its initial value. After 500 mL of distillate had been obtained, the distillate temperature was also increased from 20 to 30 °C to reduce the MD water flux to match that of the FO process.

Digested sludge centrate was also treated directly by the MD process alone, where feed and distillate temperatures were 40 and 20 °C, respectively. Initial volumes for feed and distillate solutions were 1.5 and 1 L, respectively. Membrane flushing was also conducted in the MD-only process. The fouled MD membrane was flushed with deionized water using the aforementioned protocol for the FO process (i.e., cross-flow velocity of 18 cm/s at 40 °C for 15 min).

The recovery of phosphorus from digested sludge centrate as struvite was performed at the conclusion of each FO–MD experiment. The concentrated digested sludge centrate was first filtered by a 0.45 μm filter paper (GCS0, Advantec) and then adjusted to obtain a molar ratio of magnesium (Mg^{2+}) to orthophosphate (PO_4^{3-}) of 2:1 by adding appropriate volumes of a 1 M MgCl_2 stock solution to the concentrated centrate. Next, the pH of the concentrated centrate was increased to 9.5 by adding a small volume of 1 M NaOH to form struvite crystals. The solution was mixed by a magnetic stirrer, and the pH was continuously monitored until no further crystal formation was observed. The crystals obtained were washed with deionized water twice and dried in a desiccator at room temperature (approximately 22 °C).

Analytical Methods. Key water quality parameters for digested sludge centrate, draw, and distillate samples were measured according to standard methods. Specifically, ammonium (NH_4^+) and orthophosphate (PO_4^{3-}) were assessed using a Flow Injection Analysis system (QuickChem 8500, Lachat, Loveland, CO). Total organic carbon (TOC) and total nitrogen (TN) were measured by a TOC/TN analyzer (TOC-V_{CSH}, Shimadzu, Kyoto, Japan). The magnesium (Mg^{2+}) concentration was determined by atomic absorption spectrometry (SpectrAA-220, Varian). The solution pH and electrical conductivity were measured by an Orion 4-Star Plus pH/conductivity meter (Thermo Scientific, Waltham, MA).

The crystals obtained were characterized using scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS) (JCM-6000, JEOL, Tokyo, Japan) and X-ray diffraction (GBC MMA, Hampshire, IL). An X-ray diffraction (XRD) spectrum of the struvite crystal standard with a purity of 99.998% (Alfa Aesar, Lancashire, U.K.) was also obtained and used as a reference to verify the struvite crystals obtained from our study.

RESULTS AND DISCUSSION

The FO–MD Process Produces High-Quality Water from Digested Sludge Centrate. FO pretreatment ensures stable water production by the MD process (Figure 1). In particular, organic fouling of the hydrophobic MD membrane was effectively suppressed.²⁵ By contrast, when the digested sludge centrate was directly applied to the MD process, the

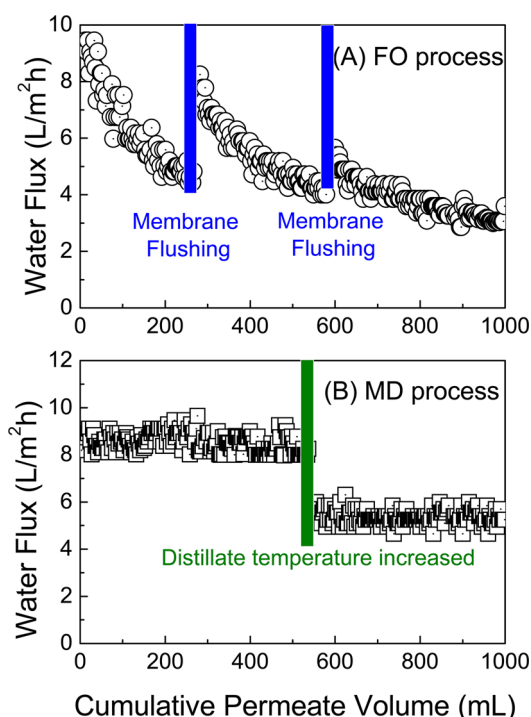


Figure 1. Production of water by the FO–MD system: (A) FO and (B) MD process. Experimental conditions: digested sludge centrate feed (Table 1); draw solution of 1.5 M MgCl_2 ; temperatures of feed, draw, and distillate of 20, 40, and 20 °C, respectively; and cross-flow rates of 1 L/min (corresponding to cross-flow velocity of 9 cm/s) for the feed, draw, and distillate. FO membrane flushing was conducted when the water flux decreased to 50% of its initial value. Deionized water was used to flush the fouled FO membrane for 15 min at a cross-flow velocity of 18 cm/s. The distillate temperature was increased from 20 to 30 °C, after 500 mL of permeate had been produced, to reduce MD water flux to maintain similar water fluxes for the FO and MD processes.

water flux decreased from 10 to 2 $\text{L m}^{-2} \text{h}^{-1}$ because of severe membrane fouling. Fouling of the MD membrane with the digested sludge centrate was irreversible as membrane flushing with deionized water was not able to recover the water flux (Figure S2 of the Supporting Information). Given the very high total solid content (1800 mg/L) and TOC (647 mg/L) of the digested sludge centrate (Table 1), significant water flux decline was also observed for the FO process. However, in the case of FO, membrane fouling was largely reversible following a simple, brief flushing with deionized water, which resulted in 82 and 68% water flux recovery for the first and second membrane flushing cycles, respectively (Figure 1A). The results confirm the weak propensity of membrane fouling in FO, which is mostly reversible, particularly at relatively low initial water flux.^{26,27} The decline in water flux in FO could be attributed to both feed salinity buildup and membrane fouling. Reverse diffusion of MgCl_2 draw solute elevated feed salinity, thereby reducing the overall driving force (i.e., effective osmotic pressure difference) in FO. In addition, the FO membrane autopsy suggested the formation of a dark fouling layer, which consisted predominately of phosphorus, sulfur, oxygen, and magnesium (Figure S3 of the Supporting Information).

The FO–MD hybrid system effectively rejected inorganic salts (indicated by electrical conductivity, ammonium, and orthophosphate) and organic matter (indicated by TOC and TN measurements), thereby leading to high-quality product

water (Table S2 of the Supporting Information). This high product water quality was mainly attributed to the nearly complete rejection by the MD process in which only water vapor is transported through the membrane pores.²⁸ However, such a high level of MD rejection also resulted in the accumulation of contaminants (e.g., ammonium and orthophosphate) in the draw solution during the operation of the FO–MD system (Figure S4 of the Supporting Information).²⁴ Mitigation strategies for reducing the rate of accumulation of contaminants in the draw solution have been proposed in our recent work.^{24,29}

FO Concentrates Ammonium and Phosphate. FO exhibited a high rate of rejection of ammonium (>90%) and orthophosphate (>97%) (Figure S5 of the Supporting Information), thereby effectively concentrating these nutrients in the digested sludge centrate (Figure 2A). This is consistent

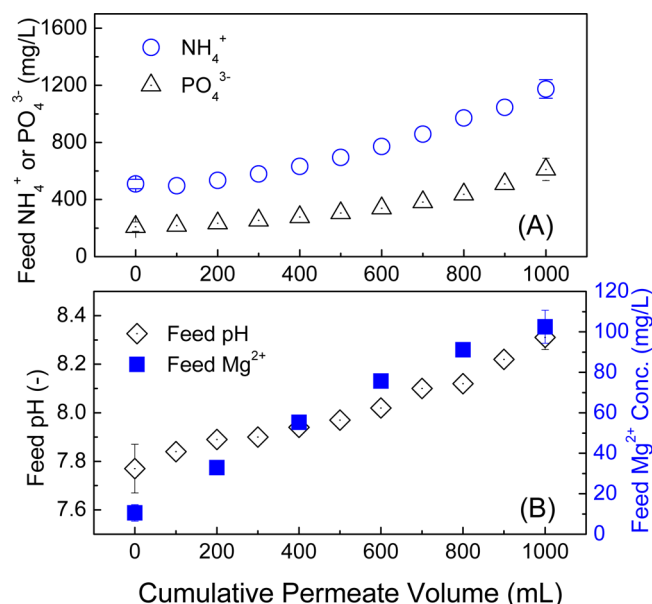


Figure 2. (A) Concentration of feed NH_4^+ and PO_4^{3-} by the FO–MD process. (B) Concentration of feed Mg^{2+} and increase in pH as a function of cumulative permeate volume. Experimental conditions are described in the legend of Figure 1.

with previous FO studies.^{17,30} The enrichment of ammonium and orthophosphate, which are two key constituents for struvite, substantially elevated the precipitation potential and product yield of struvite.

Bidirectional Diffusion of Magnesium and Protons Facilitates the Extraction of Phosphorus as Struvite. An appropriate magnesium concentration and an alkaline solution are two key factors for the extraction of phosphorus as struvite from the nutrient-concentrated digested sludge centrate.³¹ Here, the bidirectional ion diffusion, which is a unique mass transfer phenomenon in FO,^{19,32–34} can be utilized to facilitate phosphorus extraction. Specifically, forward proton diffusion elevated the feed solution pH, and at the same time, reverse magnesium diffusion enhanced the feed magnesium concentration (Figure 2B). The increase in feed pH was mainly driven by maintaining solution electroneutrality.³⁵ Similar pH variation was observed by Hancock et al.,²⁰ who examined the bidirectional ion diffusion in an FO process. More importantly, reverse magnesium permeation (reverse magnesium flux of 12 $\text{mmol m}^{-2} \text{h}^{-1}$) substantially increased the feed magnesium

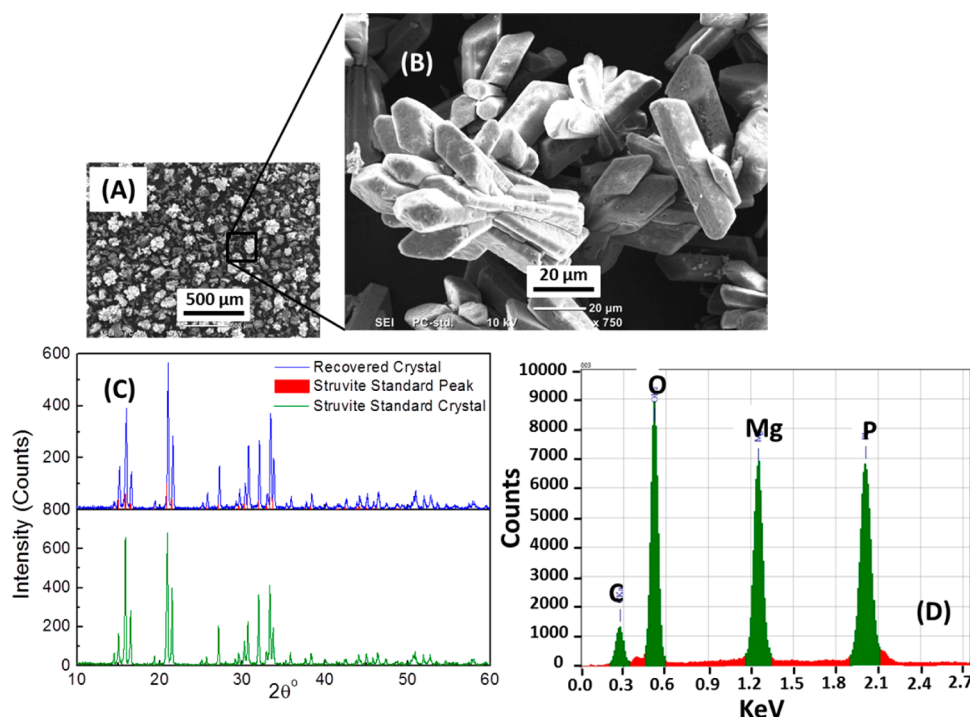


Figure 3. Struvite crystal recovered by the FO–MD process: (A and B) SEM images, (C) XRD analysis, and (D) EDS analysis. The solution pH was further increased to 9.5 by addition of base (NaOH). Continuous stirring was applied for 2 h, and crystals were recovered and dried in a desiccator at room temperature.

concentration, thereby supplementing magnesium for struvite formation.

The ionic product of ammonium, orthophosphate, and magnesium at the conclusion of the FO–MD experiment was $10^{-5.77} \text{ M}^3$, which was above the struvite conditional solubility product of $10^{-7.54} \text{ M}^3$ (Figure 2).³⁶ The molar ratio of magnesium to orthophosphate was further increased from 1:1.43 to 2:1 by addition of a small amount of magnesium (0.06 g of Mg^{2+} /g of struvite produced), thereby promoting struvite precipitation. Indeed, struvite formation was indicated by the continuous decrease in solution pH (Figure S6 of the Supporting Information). The solution pH variation also revealed that the struvite induction time was <2 min.

The precipitate was analyzed to identify the crystal morphology, element composition, and structure (Figure 3). The precipitated crystals showed a distinctive orthorhombic structure, with an average size of $40 \mu\text{m}$ (Figure 3A, B). The XRD spectrum obtained of the precipitate was identical to that of the reference pure struvite crystal standard (Figure 3C). The EDS spectrum showed that the three major peaks were for magnesium, phosphorus, and oxygen, which are the key elements of struvite. Apart from carbon (which was used to coat the sample), no other elements were detected. Thus, the results shown in Figure 3D confirm the purity of the obtained struvite crystal. It is noteworthy that struvite also contains nitrogen. However, nitrogen is a light element and cannot be detected by EDS analysis. The high struvite purity observed here could be attributed to the enrichment of phosphate and ammonium by the FO–MD hybrid system.

In summary, our results demonstrate the potential of the FO–MD hybrid system for simultaneously extracting phosphorus and clean water from digested sludge centrate. The fouling resistant FO process provides an effective pretreatment for MD to allow a stable water flux. In addition, the

bidirectional transport of magnesium (the draw solute) and protons across the FO membrane creates favorable conditions for phosphorus extraction in the form of struvite precipitate.

■ ASSOCIATED CONTENT

■ Supporting Information

Details of the description of the lab-scale FO–MD system and the schematic diagram (Figure S1), key properties of FO and MD membranes (Table S1), production of water by the MD-only process (Figure S2), FO membrane autopsy (Figure S3), product water quality by the FO–MD hybrid system (Table S2), rejection of ammonium and orthophosphate by the FO process (Figure S4), accumulation of ammonium and orthophosphate in the draw solution (Figure S5), and solution pH variation as a function of time during struvite formation (Figure S6). This material is available free of charge via the Internet at <http://pubs.acs.org>.

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Notes

The authors declare no competing financial interest.

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