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Membrane-based Processes for Wastewater Nutrient Recovery: Technology, Challenges, and Future Direction

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26   **ABSTRACT**

27   Wastewater nutrient recovery holds promise for a more sustainable agriculture. We

28   critically review three emerging membrane processes – forward osmosis (FO), membrane

29   distillation (MD) and electrodialysis (ED) – that can advance wastewater nutrient recovery.

30   Challenges associated with wastewater nutrient recovery were identified. The advantages and

31   challenges of applying FO, MD, and ED technologies to wastewater nutrient recovery are

32   discussed, and directions for future research and development are identified. Emphasis is given

33   to exploration the unique mass transfer properties of these FO, MD, and ED processes in the

34   context of wastewater nutrient recovery. We highlight that hybridising these membrane

35   processes with existing nutrient precipitation process will lead to better management of and

36   more diverse pathways for near complete nutrient recovery in wastewater treatment facilities.

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40   **Keywords**: nutrient recovery, phosphate recovery, forward osmosis, membrane distillation,

41   electrodialysis, ammonia recovery

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1. Introduction

We face a major grand challenge in the twenty-first century: sustainably meeting food demands while simultaneously reducing agriculture's environmental harm (Foley et al. 2011, West et al. 2014). This challenge is being exemplified as an annual increase of 4% in fertiliser demand to feed an additional 2.3 billion people by 2050, thereby requiring a sustained supply of fertilisers (Elser and Bennett 2011).

Current fertiliser production heavily relies on the consumption of non-renewable energy and finite mineral resources. For example, the generation of ammonia from air in the Haber-Bosch process requires 35-50 MJ per kg nitrogen in the form of fossil fuel for energy supply (Desloover et al. 2012), which accounts for 2% of the world energy use. Phosphorus mining leads to a huge amount of gypsum by-products that are contaminated with heavy metals and radioactive elements (Ashley et al. 2011). More alarming, the forecasted phosphorus production peak is approaching in 2030, with an accelerated depletion of minable phosphorus rock (Elser and Bennett 2011).

The use of fertiliser to meet food demand also carries a heavy burden for wastewater treatment processes. It is estimated that 30% of nitrogen and 16% of phosphorus in fertilisers ends up in wastewater (Rahman et al. 2014, Verstraete et al. 2009). Consequently, wastewater treatment facilities consume up to 4% of the annual electrical energy used in the United States (Energy 2006, EPA and Water 2006), more than 77% of which is used for activated sludge aeration for nitrification (McCarty et al. 2011, Svardal and Kroiss 2011). The removal of nitrogen from wastewater requires substantial energy, 45 MJ per kg nitrogen, only to release it back as gaseous nitrogen into the atmosphere. This energy-intensive nutrient removal also contributes to greenhouse gas emissions of 0.9 kg CO_2 per cubic litre of treated wastewater (Hall et al. 2011, Rothausen and Conway 2011). The large energy and environmental footprint of nutrient removal from wastewater, in turn, adversely impacts the sustainability of fertiliser production for food security. As a result, wastewater nutrient recovery is anticipated to become a promising strategy to sustain fertiliser and food production, and at the same time, potentially to bring benefits to wastewater treatment facilities (Grant et al. 2012, Guest et al. 2009, Verstraete et al. 2009).

High-rejection membrane processes, such as nanofiltration (NF) and reverse osmosis (RO), have demonstrated huge potential in wastewater nutrient recovery. For example, RO was applied for urine concentration in a source-separation toilet system, achieving a concentration

74 factor of five and high rejection of ammonium, phosphate and potassium (Maurer et al. 2006).
75 NF separation also exhibited medium to high rejection of a range of nutrients, such as urea
76 (Pronk et al. 2006b), ammonium, phosphate and potassium (Blöcher et al. 2012, Niewersch et
77 al. 2014). Despite the potential of NF and RO processes in wastewater nutrient recovery,
78 current pressure-driven membrane processes are not without limitations. NF and RO processes
79 are prone to membrane fouling in wastewater nutrient recovery where the feed streams are
80 challenging and difficult to treat, such as urine and digested sludge. Fouling of NF and RO
81 membranes impairs membrane performance and shortens membrane lifetime, thereby reducing
82 productivity in nutrient recovery. Hence, a critical need exists for robust separation processes
83 for nutrient recovery from challenging wastewater streams.

84 We critically review membrane processes that can potentially advance the reclamation
85 of nutrients from wastewater and illustrate the challenges for membrane processes in
86 wastewater nutrient recovery. Emerging membrane processes — forward osmosis (FO),
87 membrane distillation (MD), and electrodialysis (ED) — are discussed and evaluated based on
88 their applications, nutrient recovery potential, and process limitations. Unique challenges
89 associated with the agricultural application of recovered nutrients are also elucidated.

90 **2. Existing technology illustrates challenges for wastewater nutrient recovery**

91 Struvite ($MgNH_4PO_4 \cdot 6H_2O$) precipitation is widely accepted as the most promising
92 technology in wastewater nutrient recovery (de-Bashan and Bashan 2004). Struvite is a slow-
93 release fertiliser, applicable to crops in soils with relatively low pH value. In the process of
94 nutrient recovery via struvite precipitation, an alkaline solution is obtained either by addition
95 of basic solution or aeration stripping of CO_2, and followed by the introduction of magnesium
96 salts for struvite precipitation (Doyle and Parsons 2002). Previous studies have demonstrated
97 nutrient recovery via struvite precipitation from various nutrient-rich streams, such as
98 wastewater (Gerardo et al. 2013, Ichihashi and Hirooka 2012), anaerobically digested sludge
99 (Battistoni et al. 2005, Lahav et al. 2013, Marti et al. 2008, Pastor et al. 2010, Quintana et al.
100 2003), and urine (Ronteltap et al. 2010, Triger et al. 2012). Despite the struvite precipitation
101 reaching commercial implementation for nutrient recovery (de-Bashan and Bashan 2004), two
102 critical challenges remain in wastewater nutrient recovery via struvite precipitation.

103 First, the efficiency of nutrient recovery via struvite precipitation is limited by the
104 phosphorus concentration in wastewater. The driving force and kinetics for struvite
105 precipitation are significantly influenced by the phosphorus concentration. Extensive

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106 experimental results showed that effective struvite precipitation could only be achieved when

107 the phosphorus concentration was above 100 mg/L (Figure 1A) (Çelen et al. 2007, Guadie et

108 al. 2014, Jaffer et al. 2002, Liu et al. 2011, Münch and Barr 2001, Pastor et al. 2008, Pastor et

109 al. 2010, Ronteltap et al. 2010, Song et al. 2011). Low phosphorus concentration resulted in

110 either low (<40%) struvite recovery or a longer precipitation reaction time, which substantially

111 impaired the economic feasibility of nutrient recovery via struvite precipitation. The demand

112 for high phosphorus concentration is challenging for wastewater where typical phosphorus

113 concentrations for wastewater influent and digested sludge supernatant were 6 and 56 mg/L,

114 respectively (Jaffer et al. 2002, Münch and Barr 2001). As a result, it is desirable to enrich

115 nutrients in the waste stream prior to struvite precipitation, thereby significantly enhancing the

116 struvite precipitation potential and efficiency.

117   Second, struvite precipitation for nutrient recovery is also challenged by the presence

118 of toxic heavy metal ions and emerging organic contaminants in wastewater (Pronk et al.

119 2006b), which substantially compromise struvite purity and safe agricultural application. For

120 example, a close examination of recovered struvite crystals revealed the presence of toxic

121 heavy metals, with arsenic concentration up to 570 mg/kg (Figure 1B) (Lin et al. 2013, Ma and

122 Rouff 2012, Pizzol et al. 2014, Rouff 2012, Rouff and Juarez 2014). The presence of such

123 contaminants in struvite fertiliser is strictly regulated and excessive amounts can result in the

124 fertiliser being banned from agricultural application.

125   Alternative nutrient recovery approaches with better selectivity should be considered

126 to improve nutrient product quality. For example, instead of struvite precipitation, ammonium

127 can be recovered under alkaline condition by membrane distillation as 10% ammonia solution

128 (Bonmatí and Flotats 2003, Jorgensen and Weatherley 2003); and phosphorus can be

129 fractionated as phosphoric acid by electrodialysis (Wang et al. 2013, Zhang et al. 2013a). These

130 nutrient recovery technologies targeting specific nutrient ions demonstrated better selectivity

131 and resulted in nutrient products with higher quality.

132   **[Figure 1]**

133 **3. Emerging membrane processes advance wastewater nutrient recovery**

134   The challenges of higher nutrient enrichment and membrane selectivity discussed

135 above (Section 2) open opportunities for emerging membrane processes to advance wastewater

136 nutrient recovery. Forward osmosis (FO), membrane distillation (MD) and electrodialysis (ED)

137 are three membrane-based processes best suited to overcome the challenges in wastewater

138   nutrient recovery, and could potentially represent a paradigm shift in wastewater nutrient

139   management (Table 1). As described herein, these technologies can achieve a high

140   concentration factor for struvite precipitation, their selectivity is conducive to the fraction of

141   valuable nutrient substances in various forms, and their energy requirements and associated

142   costs are competitive with more conventional, pressure-driven membrane processes. A process

143   overview of the three technologies is presented below and the advantages and disadvantages

144   of each for wastewater nutrient recovery are discussed.

145   **[Table 1]**

146   *3.1. Forward osmosis*

147   Forward osmosis (FO) could substantially enhance wastewater nutrient recovery via

148   struvite precipitation by its unique mass transfer properties: lack of hydraulic pressure and the

149   occurrence of reverse draw solute flux. In FO, a semipermeable membrane is placed between

150   two solutions of different concentrations: a concentrated draw solution and a more dilute feed

151   solution. Instead of hydraulic pressure, FO employs an osmotic pressure difference to drive the

152   permeation of water across the membrane. As a result, FO has demonstrated a lower fouling

153   propensity and higher fouling reversibility in comparison with pressure-driven RO membrane

154   filtration (Lee et al. 2010, Mi and Elimelech 2010). Consequently, FO enables concentration

155   of a range of challenging, nutrient-rich streams, achieving high enrichment factors for streams

156   (Table 1), such as anaerobically digested sludge (Holloway et al. 2007), activated sludge

157   (Achilli et al. 2009, Cornelissen et al. 2008) and raw sewage (Cath et al. 2005, Xie et al. 2013,

158   2014a, Xue et al. 2015).

159   Reverse draw solute diffusion, an inherent phenomenon commonly considered

160   detrimental to FO (Boo et al. 2012, Xie et al. 2014b), can be beneficial by elevating struvite

161   precipitation potential via supplementing magnesium cation into the feed when magnesium-

162   based draw solution is used (Figure 2). Recent studies demonstrated this proof-of-concept of

163   FO in nutrient recovery (Xie et al. 2013, 2014a). Feed sludge centrate was concentrated by FO

164   driven by $MgCl_2$ draw solution and achieved a concentration factor of five, resulting in a high

165   strength stream comprising ammonium (1210 mg/L), phosphate (615 mg/L), and magnesium

166   from reverse magnesium flux. As a result, the $MgCl_2$ draw solution not only provides the

167   driving force for nutrient enrichment, but also can be incorporated into the nutrient precipitate

168   making beneficial use of lost draw solution. These unique mass transfer properties of FO have

169   motivated nutrient recovery from various waste streams such as urine (Gormly and Flynn 2007,

170   Michael et al. 2012, Zhang et al. 2014a), sewage (Ge et al. 2012, Hancock et al. 2013, Phuntsho

171   et al. 2012, Wang et al. 2011a, Xie et al. 2013, Zhang et al. 2014b, Zhang et al. 2013b), and

172   sludge (Hau et al. 2014, Holloway et al. 2007, Nguyen et al. 2013).

173   Experimental results from aforementioned literature were corroborated by mathematic

174   modelling, illustrating promising potential and capacity of FO process in wastewater nutrient

175   recovery. For instance, Xue et al. (2015) calculated theoretic water recovery of 93% in a

176   seawater draw FO process, thereby achieving ten-fold concentration of ammonium and

177   phosphate in the secondary treated municipal wastewater. This high nutrient enrichment factor

178   also agreed with solution-diffusion model in FO filtration (Zhang et al. 2014a), yielding 50−80%

179   rejection of ammonium and above 90% rejection of phosphate and potassium.

180   Despite the feasibility of nutrient recovery by FO, membrane performance is

181   constrained by the water permeability – solute selectivity tradeoff (Yip and Elimelech 2011),

182   an intrinsic property of water transport through polymeric membrane. This restricts attainment

183   of increased water permeability to FO membrane materials with decreased selectivity (Freeman

184   1999, Geise et al. 2011), which hinders achievement of high nutrient concentration factor. For

185   nutrient recovery, a membrane with high solute selectivity effectively enriches ammonium and

186   phosphate, and hence, yields a high strength nutrient-rich stream. However, lack of sufficient

187   cations, particularly magnesium supplemented into this stream via reverse salt flux reduces

188   struvite precipitation potential. By contrast, a membrane with high water permeability produces

189   a higher water flux and the concomitant decline in membrane selectivity simultaneously

190   provides more draw solution cations to the feed by higher reverse salt flux, and causes

191   significant loss of nutrient solutes into the draw. Such detrimental effects work against the

192   benefit of a more permeable but less selective membrane to enhance struvite product yield.

193   Therefore, further understanding membrane permeability – selectivity tradeoff is crucial to

194   nutrient recovery by FO process.

195   **[Figure 2]**

196   *3.2. Membrane distillation*

197   Membrane distillation (MD) is a thermally-driven membrane process that can utilise

198   low-grade heat to drive separation (Alkhudhiri et al. 2012, Alklaibi and Lior 2005). In MD, the

199   aqueous feed stream is separated from the distillate by a hydrophobic, microporous membrane.

200   Liquid is unable to penetrate the membrane pores due to the hydrophobic nature of the

201   membrane, and a difference in the partial vapour pressure drives the transport of water vapour

202   across the membrane pores. Because water is transported through the membrane only in a
203   vapour phase, MD can offer complete rejection of all non-volatile constituents in the feed
204   solution. More importantly, MD could achieve high water recovery because water vapour
205   transport through MD membrane is not significantly influenced by the feed osmotic pressure.

206   Due to this unique transport mechanism, MD processes have been explored for the
207   recovery of valuable components. Based on the volatility and vapour pressure, these
208   components can be concentrated either in the feed stream or permeate streams. For example,
209   non-volatile inorganic nutrient ions, such as potassium and phosphate, can be concentrated in
210   the feed stream to facilitate subsequent nutrient precipitation. Indeed, the MD process achieved
211   a high concentration factor of three for seawater RO brine volume reduction (Martinetti et al.
212   2009). Similar high enrichment performance could also be observed for mineral acids (Elkina
213   et al. 2013, Tomaszewska et al. 1995, Tomaszewska 2000) and fruit juices (Mohammadi and
214   Bakhteyari 2006). Concentration of sulphuric acid by MD from 16% to 40% was reported with
215   a separation coefficient of above 98% (Tomaszewska and Mientka 2009).

216   Ammonia recovery can be one important application of MD process in wastewater
217   nutrient recovery where ammonia is more volatile than water and can be enriched in the
218   permeate stream of MD processes (du Preez et al. 2005, Zarebska et al. 2014, Zhao et al. 2013).
219   Ammonia recovery exemplifies the selectivity of MD membrane process, an approach that
220   differs from aiming for a high nutrient concentration factor. MD processes were configured as
221   vacuum MD, gas sweeping MD and direct contact MD for ammonia recovery from varying
222   waste streams, such as urine (Zhao et al. 2013), wastewater (Ahn et al. 2011, Ding et al. 2006,
223   El-Bourawi et al. 2007, Qu et al. 2013, Xie et al. 2009), and swine manure (Thygesen et al.
224   2014, Zarebska et al. 2014). These MD processes achieved more than 96% ammonia recovery
225   in the form of aqueous solution, which can be conveniently processed as commercial fertiliser.
226   More importantly, in direct contact MD, low concentration of sulphuric acid was used as
227   stripping solution on the permeate side to further enhance the capture of ammonia vapour. The
228   application of an acidic stripping solution in the MD permeate stream substantially improves
229   ammonia recovery to 99%, with ammonium sulphate being produced as fertiliser.

230   Varying composition of nutrient-rich waste stream can pose distinctive challenges to
231   MD in nutrient recovery (Meng et al. 2014, Tijing et al. 2015, Van der Bruggen 2013). Volatile
232   organic compounds, such as volatile fatty acids that exert partial vapour pressures comparable
233   to or higher than water are transported across the MD membrane with the water vapour, causing
234   contamination of the permeate stream and jeopardising the quality of recovered ammonia

fertiliser. Certain components in wastewater, such as surfactants, can lower the liquid surface tension of the feed solution and cause wetting of the membrane pores. Membrane pore wetting will result in a direct liquid flow from feed through the wetted pores, substantially deteriorating distillate quality. To restore the vapour-liquid interface at the pores, the wetted membrane must be taken out of operation and dried completely, resulting in process downtime and potential membrane degradation. Dissolved organic matters and colloids present in the nutrient-rich waste streams can lead to MD membrane fouling. Fouling clogs membrane pores, which leads to flux decline and pore wetting and imposes an additional hindrance to heat and mass transfer, thereby diminishing the MD process productivity in nutrient recovery. MD membrane fouling in ammonia wastewater recovery was initiated by adsorption of peptides and proteins on the MD membrane surface. As a result, the Gibbs free energy and hydrophobicity of the MD membrane surface reduced, hindering ammonia vapour permeation (Thygesen et al. 2014, Zarebska et al. 2014).

[Figure 3]

Fabrication of MD membranes with special wettability, such as a superhydrophobic or omniphobic property, imparts membrane anti-fouling property and mitigates deleterious membrane fouling and wetting, thereby improving the nutrient recovery efficiency of MD in processing challenging waste streams (Liao et al. 2013, 2014, Lin et al. 2014). Increasing the hydrophobicity of an MD membrane leads to higher liquid entry pressure and consequently more resistance to pore wetting. For example, Razmjou et al. (2012) fabricated a superhydrophobic polyvinylidene fluoride (PVDF) MD membrane with TiO_2 nanoparticles providing hierarchical structures with multilevel roughness on the membrane surface. The resultant MD membrane, possessing high liquid entry pressure of 195 kPa, demonstrated a much higher water flux recovery after humic acid fouling in comparison to the pristine PVDF membrane. Another strategy for preventing membrane fouling and wetting is fabrication of MD membrane with an omniphobic property that repels both water and low surface tension liquids such as surfactants. Indeed, Lin and coworkers (2014) fabricated an omniphobic MD membrane with silica nanoparticles via surface fluorination and polymer coating, and demonstrated anti-wetting MD membrane performance, maintaining water flux and salt rejection, even with the presence of surfactant. The advancement of MD membrane fabrication with special wettability can open up vast opportunities for MD application for beneficial nutrient recovery, particularly ammonia, from challenging waste streams.

3.3. Electrodialysis

Electrodialysis (ED), which arranges ion-exchange membranes alternately in a direct current field (Xu and Huang 2008), could selectively fraction nutrients from wastewater streams into high quality nutrient products. The current field is the driving force in an ED process where cations and anions migrate towards the cathode and anodes, respectively. Ion separation in the ED process is achieved by ion-exchange membranes that comprise cation-selective, anion-selective, and bipolar membranes. Cation- and anion-selective membranes are widely used in conventional ED to hinder the passage of co-ions (anions and cations, respectively) by virtue of Donnan repulsion. When bipolar membranes comprising a cation-selective layer and an anion-selective layer are used in an ED process, dissociation of solvent molecules, such as water, into H^+ and OH^- can be realised.

The unique ion separation mechanism of the ED process provides a selective mechanism for wastewater nutrient recovery. ED process selectively partitioned phosphate from wastewater effluent containing various ions as a concentrated phosphate solution, achieving a concentration factor of up to 7 (Zhang et al. 2012, Zhang et al. 2013a). Similar selective phosphate enrichment by the ED process was also observed in urine nutrient recovery, resulting in a purified phosphate concentrate (Escher et al. 2006, Pronk et al. 2006a). Phosphate selectivity in an ED process can be further enhanced by either adjusting the feed stream to the alkaline pH range or increasing current density (Tran et al. 2014, Tran et al. 2015). Better performance was expected based on the ED separation mechanisms where multivalent phosphate migrates more slowly than monovalent ions under the current field (Zhang et al. 2012).

Nutrient recovery efficiency and product purity could be significantly improved when a bipolar membrane was employed in an ED process. The ED process with bipolar membrane integrates solvent (water) and salt dissociation (Bailly 2002); it provides H^+ and OH^- *in situ* without the introduction of salts (Huang and Xu 2006, Huang et al. 2006, Huang et al. 2007). The combination of H^+ and anions in certain chambers leads to production of acid, while the combination of OH^- ions and cations in other chambers leads to production of the corresponding base. As a result, this ED process with a bipolar membrane concept could diversify the final products and enhance purity for nutrient recovery. For example, Wang et al. (2013) employed the ED process with a bipolar membrane to convert phosphate in sludge supernatant to purified phosphoric acid of 0.075 mol/L.

Despite the high purity and diverse product extracted by the ED process, it suffers from membrane fouling during wastewater nutrient recovery. The build-up of fouling layers in the

301 ED process increases the cell resistance (current drop), decreases migration yield and ion

302 selectivity, and eventually alters membranes due to irreversible fouling (Mondor et al. 2009).

303 Unlike fouling in RO and FO membranes, fouling of the ion-exchange membrane in the ED

304 process is significantly dependent on the charge of the membrane (Wang et al. 2011b).

305 Specifically, more severe fouling was observed in an anion-selective membrane when

306 negatively charged humic substance, protein and surfactant were presented (James Watkins

307 and Pfromm 1999, Lee et al. 2009, Lindstrand et al. 2000). By contrast, a cation-selective

308 membrane could be hampered by calcium-dominated scaling (Ayala-Bribiesca et al. 2006,

309 Bazinet and Araya-Farias 2005). Abating ED membrane fouling could be achieved by

310 periodically reversing the polarity of electrodes, decreasing current density, improving

311 hydraulic conditions in stack compartment by increasing flowrate or gasket with flow pattern,

312 and in-place cleaning with acidic or basic solutions (Lee et al. 2002, Mondor et al. 2009, Ruiz

313 et al. 2007).

314   **[Figure 4]**

315 **4. Path forward**

316 *4.1. 1+1>2*

317   The emerging membrane processes discussed above have demonstrated their capacity

318 to advance wastewater nutrient recovery by either maximising nutrient concentration factors,

319 such as FO and MD, or enhancing nutrient selectivity, such as MD and ED. Technological

320 advancements could be further achieved by hybridising these membrane processes. Hybrid

321 membrane processes complement each other, thereby maximising overall nutrient recovery

322 efficiency.

323   Integration of FO with other processes could not only re-concentrate diluted draw

324 solution for sustainable process performance, but also complement wastewater nutrient

325 recovery with freshwater production. This concept motivates coupling FO with different

326 membrane processes, such as RO, MD, and ED (Figure 5). For example, an FO-RO hybrid

327 system can achieve high rejections of phosphate and ammonium (99.9% and 92%, respectively)

328 from wastewater effluent (Hancock et al. 2013, Holloway et al. 2007) or nutrient-rich sludge

329 (Nguyen et al. 2013). More importantly, this hybrid system also simultaneously produces high

330 quality permeate water. In an FO-MD hybrid system, FO concentrated orthophosphate and

331 ammonium for subsequent phosphorus recovery in the form of struvite ($MgNH_4PO_4 \cdot 6H_2O$),

332 while MD was used to recover the draw solution and extract clean water from the digested

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333   sludge centrate (Xie et al. 2013, 2014a). The MD unit in the hybrid system can also be powered

334   by solar energy, thereby reducing the overall operating cost for wastewater nutrient recovery

335   (Qtaishat and Banat 2013). Similarly, ED can also levy solar photovoltaic energy to re-

336   concentrate draw solution in an FO-ED hybrid system (Zhang et al. 2013b), thereby

337   simultaneously concentrating nutrient from the feed waste, and producing freshwater from the

338   draw solution.

339   Coupling FO with a membrane bioreactor (MBR) is also becoming attractive for

340   wastewater nutrient recovery (Holloway et al. 2014, Holloway et al. 2015). This osmotic MBR

341   (OMBR) concept substantially benefits from the high nutrient rejection by the FO membrane

342   in MBR, the high concentration factor due to low FO fouling propensity, and the supply of

343   magnesium ions to facilitate nutrient precipitation via reverse draw solution diffusion (Figure

344   3). Recently, Qiu and Ting (2014) applied an OMBR using $MgCl_2$ draw solution to directly

345   extract phosphorus from wastewater, achieving 95% phosphorus recovery via calcium

346   phosphate precipitation. Subsequently, Qiu and coworkers employed an OMBR with seawater

347   brine draw to achieve 90% phosphorus recovery in the form of amorphous calcium phosphate

348   (Qiu et al. 2015).

349   **[Figure 5]**

350   Despite the versatility and robustness of hybridized FO system for wastewater nutrient

351   recovery, this technology is not without limitations. One significant hindrance is contaminant

352   accumulation in the draw solution. In the closed-loop FO hybrid system, contaminants that

353   permeate through the FO but not the downstream RO or MD process can accumulate in the

354   draw solution, leading to a build-up of unfavourable contaminants in the draw solution

355   (D'Haese et al. 2013, Shaffer et al. 2012). Significant accumulation of organic foulants in the

356   draw solution was observed in an FO-RO hybrid system (Coday et al. 2015, Hancock et al.

357   2013). Similarly, this detrimental effect was also found in an FO-MD hybrid system where

358   micropollutant concentrations increased in the draw solution as the cumulative permeate

359   volume increased (Xie et al. 2013, 2014a). Therefore, it is of paramount importance to manage

360   this detrimental contaminant accumulation to ensure system performance and reliability.

361   Struvite precipitation, a key step for phosphorus nutrient recovery, could also benefit

362   from coupling with membrane processes to improve precipitation efficiency. For example, the

363   ED process was operated in tandem with struvite precipitation reactor, which enhanced the

364   selective capture of phosphate from the effluent of the struvite reactor (Zhang et al. 2013a). As

365 a result, the phosphate from struvite reactor effluent was further concentrated in the ED stack

366 and recirculated into the struvite reactor, thereby improving the overall phosphorus recovery

367 to 97%.

368   Ammonia recovery, which could result in high quality liquid fertiliser, can be

369 substantially advanced by hybrid membrane processes. In an ED-RO hybrid system, the

370 ammonium was fractioned by a cation-selective membrane in an ED unit where the

371 ammonium-rich stream from concentrate compartments was further concentrated by an RO

372 membrane. This ED-RO hybrid process produced highly concentrated ammonium solution up

373 to 13 g/L (Mondor et al. 2008), which is beneficial in agricultural application. On the other

374 hand, the volatile ammonia could be captured in an ED-MD hybrid process (Ali et al. 2004,

375 Graillon et al. 1996, Udert and Wächter 2012). For instance, an ED process with bipolar

376 membranes produced ammonia from an ammonium nitrate waste stream via splitting water

377 solvent. The produced ammonia was recovered by stripping under vacuum membrane

378 distillation, achieving an ammonia concentration of 2 mol/L.

379 *4.2. Decentralised or centralised?*

380   Key nutrient concentrations – ammonium and phosphate – decrease along the sewer

381 system from individual households to centralised wastewater treatment facilities, with

382 phosphate concentration being 100 times higher from a single household compared to the

383 concentration at the wastewater treatment plant (Carroll et al. 2006, Chanan and Woods 2006,

384 Maurer et al. 2003, Tchobanoglous et al. 1991). This significant variation of nutrient

385 concentration gradient unlocks opportunities for tailoring nutrient recovery approaches with

386 varying membrane processes for both decentralised (e.g., single household) and centralised

387 (e.g., wastewater treatment plant) applications.

388   On-site urine separation and recovery via struvite precipitation is one promising

389 strategy for decentralised, small-scale wastewater nutrient recovery (Larsen et al. 2009). In

390 particular, the urine stream contributes a large proportion of nutrients from households (81%

391 nitrogen, 50% phosphorus, and 55% potassium), but less than 1% of the total volume of

392 municipal wastewater (Karak and Bhattacharyya 2011). More importantly, via nutrient

393 recovery, on-site urine separation can significantly reduce nutrient loadings to the wastewater

394 treatment plants and downstream effluent-receiving water bodies (Ishii and Boyer 2013,

395 Wilsenach and Loosdrecht 2003). As such, on-site nutrient recovery from urine can be more

396 energetically efficient than nutrient removal and recovery in centralised wastewater treatment

397   process, although at relatively low economic efficacy of such small-scale systems. However,

398   the deployment of urine source separation requires substantial change to existing infrastructure,

399   such as varying flush water from urine diverting toilets (Wilsenach and Van Loosdrecht 2004),

400   proper urine storage for urea hydrolysis (Ishii and Boyer 2015), and precipitation in urine-

401   separating toilets (Udert et al. 2003). In addition, the deployment of on-site nutrient recovery

402   from urine also encounters varying degree of acceptance. For example, fertiliser produced by

403   urine was less accepted by farmer in comparison with the public where more than 50% farmers

404   have concerns in technical feasibility as well as nutrient product quality (Lienert and Larsen

405   2010).

406   The emerging membrane processes discussed above also exhibit satisfactory

407   performance in urine separation. For instance, the FO process mined macronutrients (nitrogen,

408   phosphate and potassium) from urine after hydrolysis, achieving significant volume reduction,

409   and high rejection of ammonium (50-80%), phosphate and potassium (>90%) (Zhang et al.

410   2014a). A higher ammonia separation factor from urine could be observed in a vacuum MD

411   process where rejection of ammonia reached 99% (El-Bourawi et al. 2007, Zhao et al. 2013).

412   The ED process is also capable of recovering and concentrating nutrient ions from urine

413   contaminated by micropollutants (Pronk et al. 2006a).

414   Wastewater nutrient recovery has been practised in centralised wastewater treatment

415   facilities (Cote et al. 2013, Kuzma et al. 2012). However, the benefit of upgrading conventional

416   wastewater treatment process goes beyond economic gains (McConville et al. 2014), since re-

417   engineering the existing nutrient recovery process by a suite of membrane processes offers

418   more sustainable sewage management and nutrient cycling.

419   *4.3. Bioavailability for recovered nutrients*

420   The reviewed three emerging membrane processes for wastewater nutrient recovery

421   could utilise a range of renewable energy to further enhance the process sustainability and

422   substantially reduce the carbon footprint. For instance, via powering the ED process by solar

423   energy, the operating cost for an FO-ED hybrid process was €3.32 to 4.92 per cubic meter

424   treated water (considering the investment for membranes and solar panel) for a small size

425   (Zhang et al. 2013b). In addition, levying waste heat generated by a power plant (Zhou et al.

426   2015) or introducing the biogas produced by the wastewater treatment plant (Qin and He 2014)

427   can be an important alternative to drive the MD process with less cost in wastewater nutrient

428   recovery. Furthermore, a life cycle assessment showed that more than 25% reduction of the

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429 environmental impact could be achieved when incorporating FO process into traditional
430 seawater desalination or wastewater reclamation process (Hancock et al. 2012).

431 Apart from the potential energy consumption, producing agriculturally applicable
432 fertiliser is the final goal for wastewater nutrient recovery. In addition to the technological
433 aspects of wastewater nutrient recovery, attention should also be paid to the agronomic efficacy
434 and crop uptake of fertiliser produced from recovered nutrients (Withers et al. 2014).
435 Phosphorus plant availability of struvite precipitate recovered from a waste stream was
436 compared with a well-established, water-soluble fertiliser, triple superphosphate, using pot
437 experiments with isotope ^{32}P-labelled soil, suggesting negligible difference in plant phosphorus
438 nutrition and growth (Achat et al. 2014). However, despite the abundance of phosphorus
439 availability for plant growth, the recovered nutrient fertiliser showed poor nitrogen uptake for
440 plant growth (Ganrot et al. 2007, Matassa et al. 2015). In addition, phosphorus recovered in the
441 form of amorphous calcium phosphate precipitate, exhibited less water solubility, thereby
442 hindering crop uptake (Plaza et al. 2007). These results show that wastewater nutrient recovery
443 is a multi-dimensional challenge, requiring a suitable market for distributing recovered nutrient
444 product with proven agronomic efficacy.

445 **5. Conclusion**

446 Three emerging membrane processes – FO, MD and ED – can advance wastewater
447 nutrient recovery with their unique mass transfer properties. FO, demonstrating low fouling
448 propensity and supplementing magnesium ion via reverse salt flux, is able to maximise nutrient
449 enrichment prior to struvite precipitation. MD, driven by vapour pressure difference, is not
450 only capable of achieving a high concentration factor, but also can recover volatile ammonia
451 as a high quality fertiliser. ED can selectively partition phosphate with an anion-selective
452 membrane, or produce phosphoric acid or ammonia with a bipolar membrane that splits water
453 solvent into proton and hydroxide. In addition, integration of these membrane processes with
454 existing nutrient precipitation processes could substantially improve nutrient recovery
455 efficiency, and diversify the nutrient product that can be extracted, even achieving a near
456 complete wastewater nutrient recovery. For the future, detailed techno-economic analysis of
457 these hybridised membrane-based processes in wastewater nutrient recovery should be
458 performed, such as process energy demand, CO_2 footprint, system robustness, operating costs,
459 product quality and market demands.

460 **6. Acknowledgements**

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463 **7. References**

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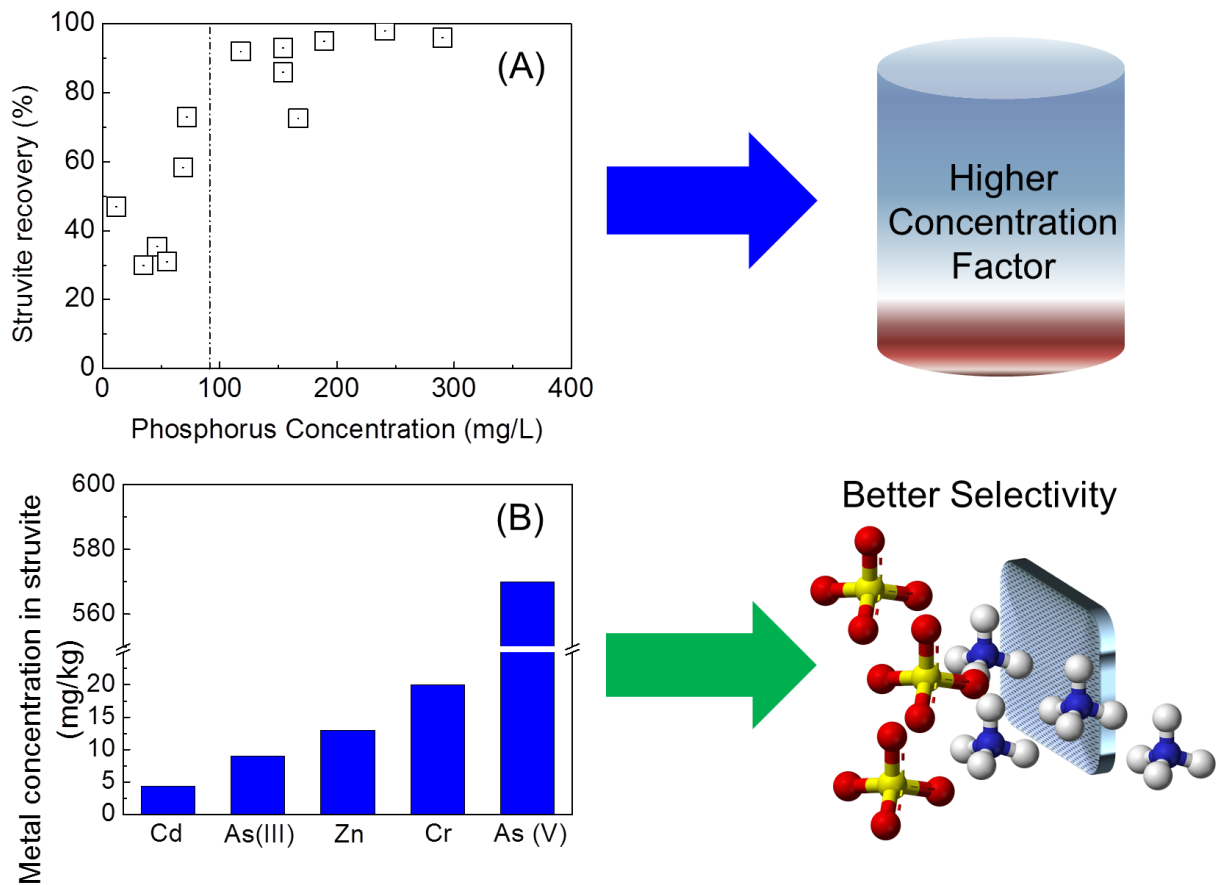
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871 **Figure 1:** Illustrations of the critical challenges in wastewater nutrient recovery. (A) Struvite
872 precipitation efficiency as a function of initial phosphate concentration; there is a critical need
873 for membrane processes enabling a higher concentration factor. Data points were summarised
874 from literatures (Çelen et al. 2007, Guadie et al. 2014, Jaffer et al. 2002, Liu et al. 2011, Münch
875 and Barr 2001, Pastor et al. 2008, Pastor et al. 2010, Ronteltap et al. 2010, Song et al. 2011)
876 (B) Presence of toxic heavy metal ions in struvite precipitates from waste streams; there is a
877 critical need for membrane processes with high selectivity. Data points were collected from
878 literatures (Lin et al. 2013, Ma and Rouff 2012, Pizzol et al. 2014, Rouff 2012, Rouff and
879 Juarez 2014).

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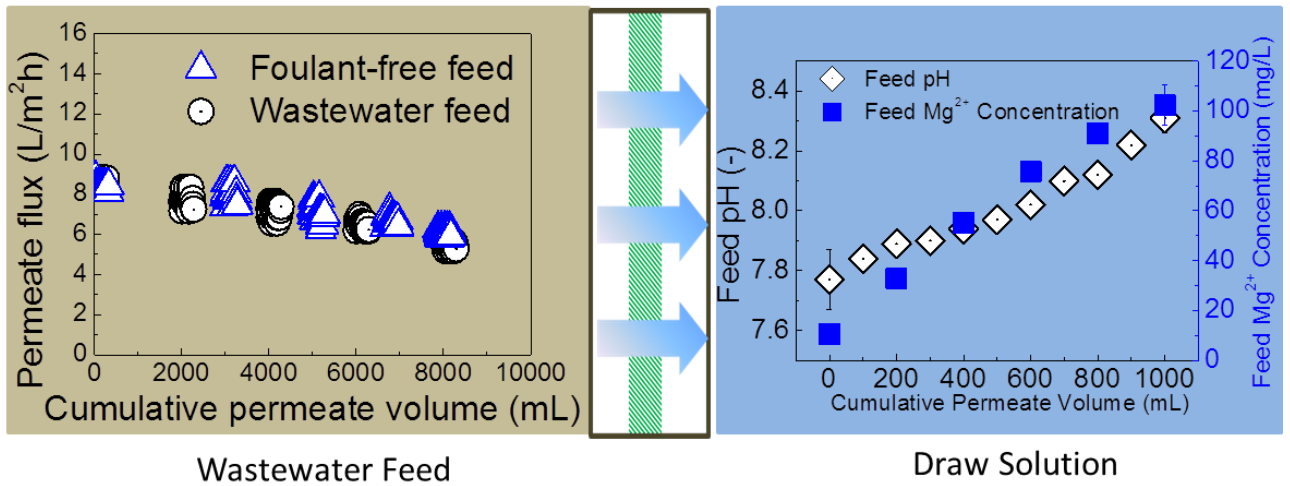
Figure 2: Unique mass transfer properties of forward osmosis (FO) enhance efficiency of nutrient recovery from wastewater. Data reproduced from (Xie et al. 2014a, Xie et al. 2014b).
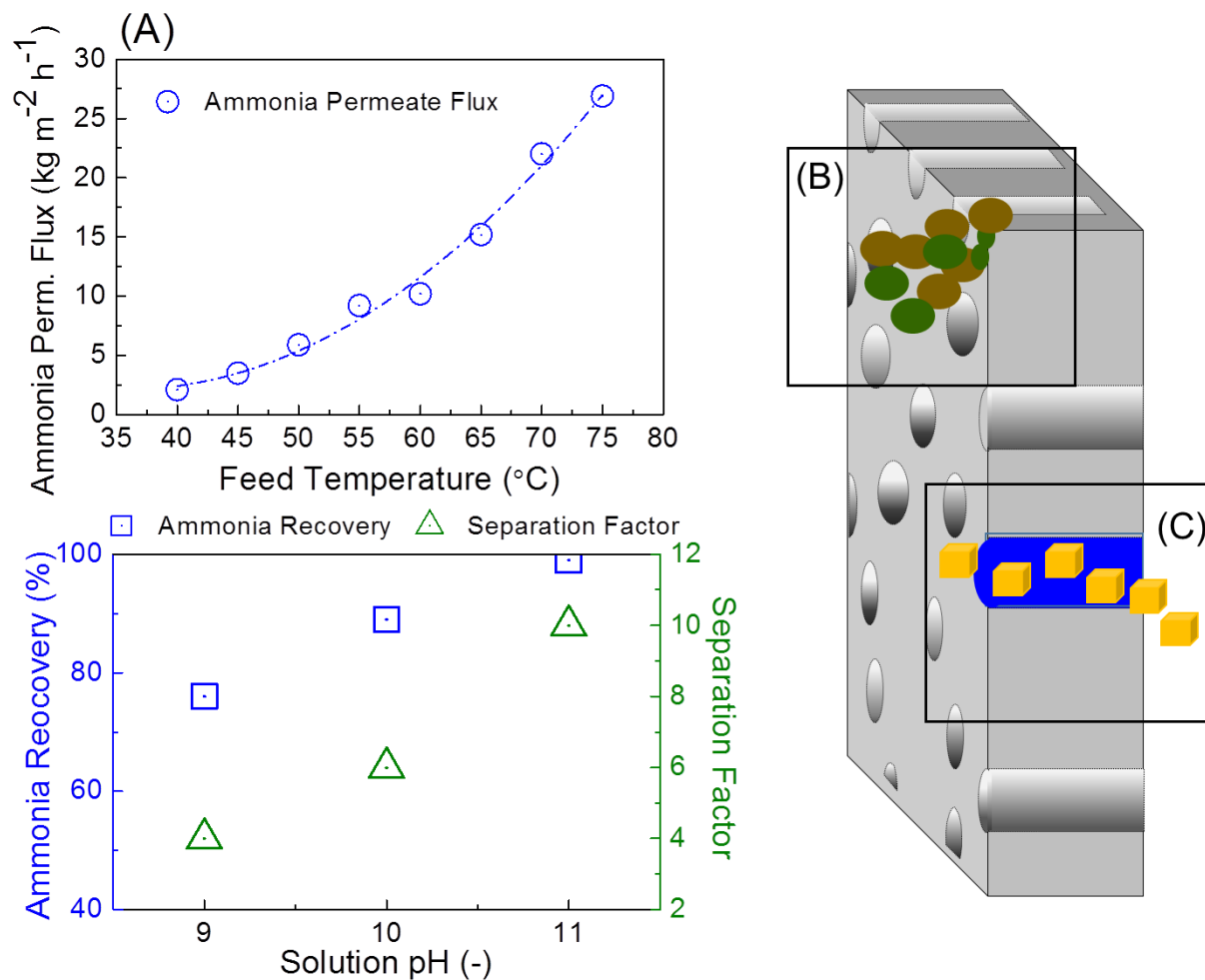
Figure 3: Conceptual illustration of membrane distillation (MD) for wastewater nutrient recovery. (A) Ammonia vapour selectively permeates through the membrane pores as a function of feed temperature (upper panel) and solution pH (lower panel where separation factor was calculated as the ratio of ammonia concentration in the feed and permeate); experimental data were reproduced from references (Ding et al. 2006, El-Bourawi et al. 2007, Qu et al. 2013, Xie et al. 2009). (B) Fouling of MD membrane leading to detrimental effect on process productivity (such as flux decline). (C) Wetting of MD membrane pores and permeate quality, such as feed solute (yellow cubes) flowing directly across membrane.
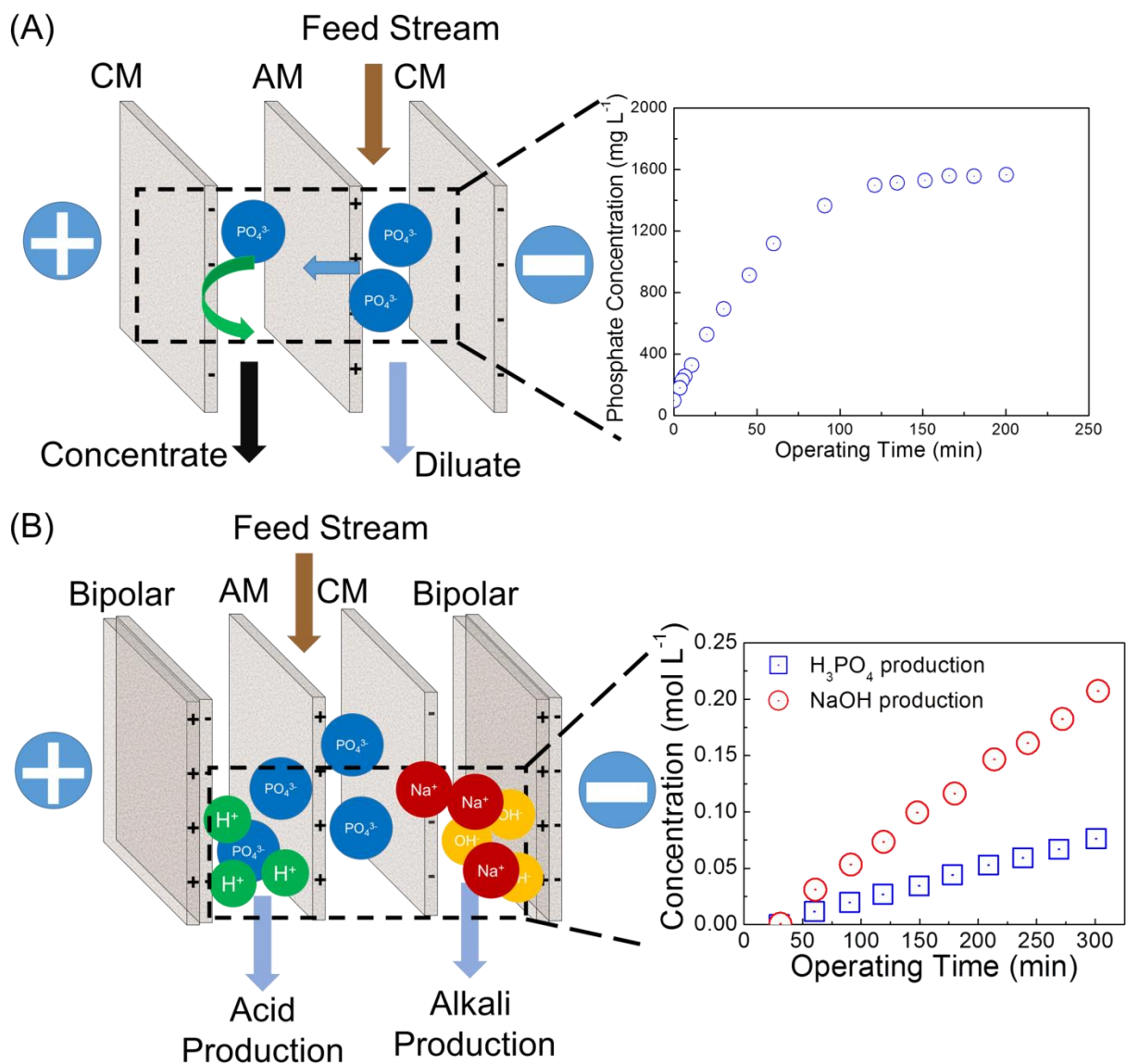
Figure 4: Conceptual illustration of electrodialysis (ED) for wastewater nutrient recovery. (A) Conventional ED process selectively concentrates phosphate in waste stream, where phosphate ion concentration in the concentrate stream increased as a function of time. (B) ED process with bipolar membrane selectively produces phosphoric acid from waste stream, where phosphoric acid concentration increases as a function of operating time. Data reproduced from Wang et al. 2013.
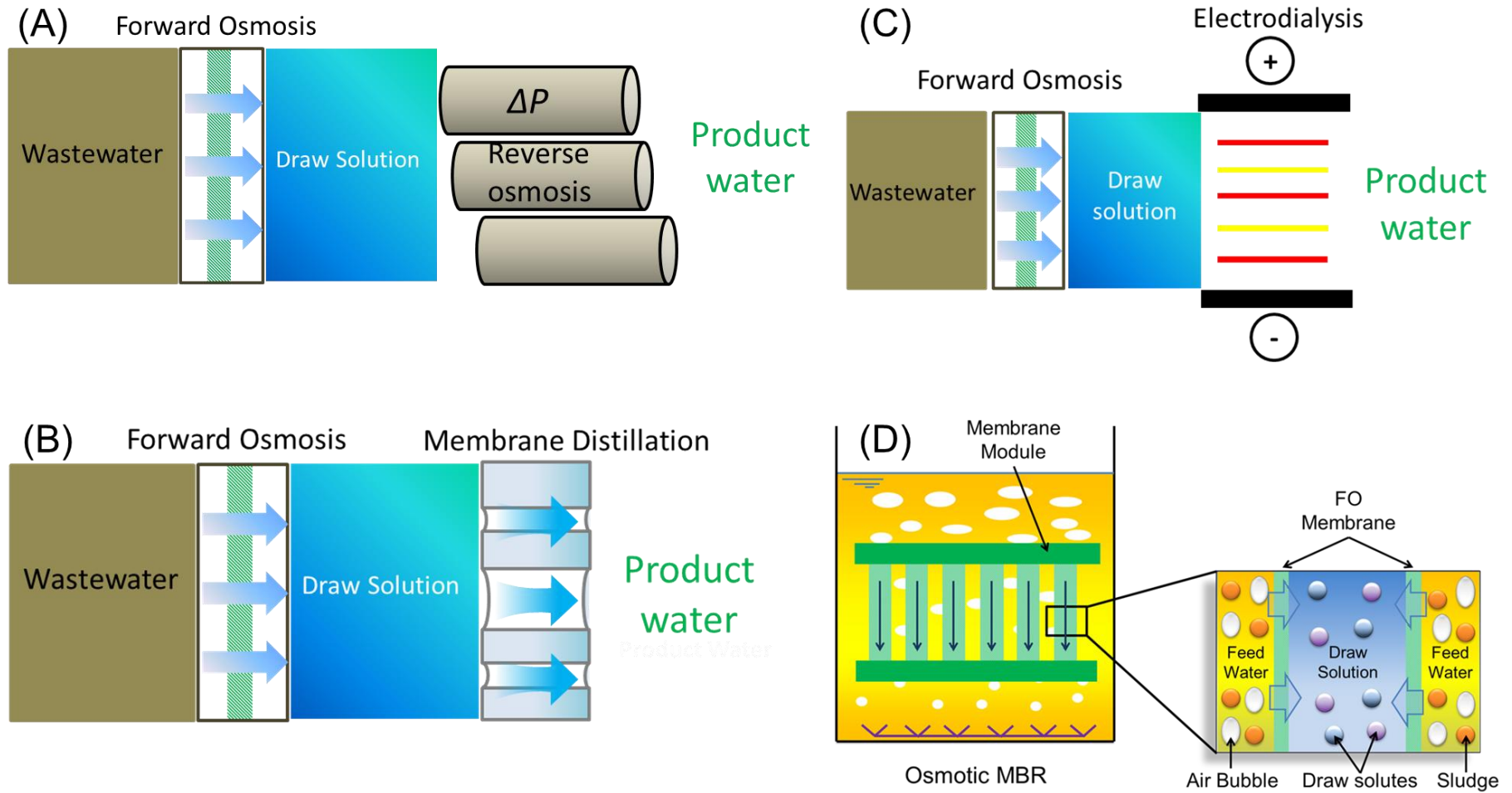
Figure 5: Conceptual illustrations of forward osmosis (FO)-based membrane processes for wastewater nutrient recovery: (A) FO-RO hybrid process; (B) FO-MD hybrid process; (C) FO-ED hybrid process; and (D) Osmotic MBR with FO membrane.

Table 1: Summary of nutrient and water recovery by pressure- (nanofiltration and reverse osmosis), osmosis- (forward osmosis), thermally- (membrane distillation) and electrically- (electrodialysis) driven membrane processes in terms of process performance, membrane fouling, and operating cost and energy consumption.

Driving force	Source water	Nutrient recovered	Key membrane process	Performance	Membrane fouling	Operating cost and energy consumption
Pressure-driven	Urine (Maurer et al. 2006)	Ammonium, phosphate, potassium	RO	Concentration factor up to 5. Ammonium: 70%; phosphate: 73%; potassium: 71%.	Membrane scaling	Operating pressure of 50 bar
	Synthetic urine (Pronk et al. 2006b)	Urea, ammonium, phosphate	NF	Urea: 10% ammonium: 55% phosphate: 94%	N.A.[*]	Operating pressure of 20 bar
	Synthetic wastewater (Niewersch et al. 2014)	Phosphate, potassium	NF	Phosphoric acid: 50% potassium: 30%	N.A.	Operating pressure of 12 bar
	Digested sludge (Blöcher et al. 2012)	Phosphate	NF	phosphate: 50%	N.A.	Operating pressure of 25 bar Operating cost can be covered by phosphate recovery revenue
Osmosis-driven	Urine (Zhang et al. 2014a)	Ammonium, phosphate, potassium	FO	Ammonium: 50-80% phosphate: >90% potassium: >90%	N.A.	N.A.
	Urine (Gormly and Flynn 2007, Michael et al. 2012)	Water	FO (X-Pack™, Water Well®)	Total nitrogen >95% urea > 93% total organic carbon > 95%	N.A.	N.A.

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Synthetic wastewater (Phuntsho et al. 2012)	Water	FO with fertilizer draw solution	Diluted fertilizer for agricultural irrigation	N.A.	N.A.
Secondary treated effluent (Hancock et al. 2013)	Water	FO-RO	Nitrate >72% phosphate >99% dissolved organic carbon > 98%	Cake layer formation	N.A.
Raw sewage (Xie et al. 2013)	Water	FO-MD	Total organic carbon > 99% total nitrogen >99%	Cake layer formation	Draw solution temperature 40 °C
Secondary treated effluent (Zhang et al. 2013b)	Water	FO-ED	Total organic carbon >90% Near 100% rejection of heavy metal ions (Cd, As, Pb)	N.A.	€3.32-4.92 per m^3 product water
Activated sludge (Nguyen et al. 2013)	Water, ammonium, phosphate	FO	Ammonium >96%, phosphate >98% dissolved organic carbon > 99%	Cake formation	N.A.
Activated sludge (Hau et al. 2014)	Water, ammonium, phosphate	FO-NF	Ammonium >97% phosphate >99%	Cake formation	NF operating pressure: 80 psi
Activated sludge (Holloway et al. 2007)	Water, ammonium, phosphate	FO-RO	Ammonium >92.1% phosphate >99.8%	Pore blocking and surface fouling	4 kWh/m^3 at 75% water recovery
Anaerobic sludge (Xie et al. 2014a)	Water, phosphate	FO-MD	Ammonium >90% phosphate >97% struvite product	Cake formation	Draw solution temperature 40 °C

Thermally-driven	Urine(Zhao et al. 2013)	Water, ammonia, organic matters	Vacuum MD	Organic matter: >99% Ammonia: 41-75% Water: 32-49%	Organic fouling with salt crystallization	Feed temperature: 50-70 °C Vacuum pressure: 9.5kPa.
	Synthetic wastewater (Xie et al. 2009)	Ammonia	Sweep gas MD	Ammonia: >96%	N.A.	Feed temperature: 65°C sweep gas flowrate: 3 L/min
	Synthetic wastewater (El-Bourawi et al. 2007)	Ammonia	Vacuum MD	Ammonia: >90%	N.A.	Feed temperature: 50 °C Vacuum pressure: 6.3kPa.
	Synthetic wastewater (Ahn et al. 2011)	Ammonia	Direct contact MD	Ammonia: >92%	N.A.	Feed temperature: 35°C Ammonia stripping solution: 1 M H_2SO_4
	Synthetic wastewater (Qu et al. 2013)	Ammonia	Direct contact MD	Ammonia: >99%	N.A.	Feed temperature: 55°C Ammonia stripping solution: 0.1 M H_2SO_4
	Swine manure (Zarebska et al. 2014)	Ammonia	Direct contact MD	Ammonia: >99%	Organic fouling followed by pore wetting	Feed temperature: 40°C Ammonia stripping solution: 0.5 M H_2SO_4
	Swine manure (Thygesen et al. 2014)	Ammonia	Direct contact MD	Ammonia: >98%	Organic fouling	Feed temperature: 35°C Ammonia stripping solution: 0.5 M H_2SO_4
Electrically-driven	Urine (Pronk et al. 2006a)	Ammonium, phosphate, potassium	ED with ion exchange membrane	Concentration factors: ammonia (2.9), potassium (3.1), phosphate (2.7); Eliminating micropollutants	N.A.	Applied current density: 22.5 mA/cm² Current efficiency: 50%

	Municipal Wastewater (Zhang et al. 2013a)	Phosphate	ED with ion exchange membrane	Concentration factors: phosphate (6.5)	N.A.	Applied current density: 31.25 A/cm^2 Current efficiency: 72% Energy consumption: 16.7 kWh/(kg PO$_4^{3-}$)
	Synthetic wastewater (Wang et al. 2013)	Phosphate	ED with ion exchange membrane	Concentration factors: phosphate (4.2)	N.A.	Applied current density: 71.5 mA/cm^2
	Synthetic wastewater (Wang et al. 2013)	Phosphate	ED with bipolar membrane	Concentration factors: phosphate (16); product phosphorus acid of 0.075 mol/L	N.A.	Applied current density: 50 mA/cm^2 Current efficiency: 75% Energy consumption: 29.3 kWh/(kg H$_3$PO$_4$)
	Swine manure (Mondor et al. 2008, Mondor et al. 2009)	Ammonium	ED with ion exchange membrane	Concentration factors: ammonium (5.3)	Calcium and colloidal particle deposition	Applied current density: 2.7 A/cm^2 Current efficiency: 77.9%

[*] not applicable