The Upper Bound of Thin-film Composite (TFC) Polyamide Membranes for Desalination

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Abstract

16 Membrane separation properties are constrained by a tradeoff relationship between

17 permeability and selectivity. This tradeoff relationship has been well established for

18 gas separation membranes in the form of the Robeson's upper bound. In contrast, the

19 upper bound relationship is much less established for thin-film composite (TFC)

20 polyamide membranes used for desalination. In this work, we analyzed the tradeoff

21 between the water permeance and the water/NaCl selectivity for TFC membranes

22 gathered from more than 300 published papers. A clear upper bound behavior

23 relationship is established, and the various effects of membrane synthesis conditions

24 and modifications are reviewed in relation to this permeance-selectivity tradeoff. Our

25 work provides a critical tool for the evaluation and benchmarking of future membrane

26 development works in the context of desalination and water reuse.

27

28 Keywords: Upper bound; permeance-selectivity tradeoff; Thin-film composite

29 polyamide; Reverse osmosis membranes; Desalination

30

1. Introduction

Membrane-based desalination and water reuse have gained increasing popularity in arid regions to cope with water scarcity [1, 2]. These applications typically use thin-film composite (TFC) reverse osmosis (RO) and nanofiltration (NF) membranes, where a thin polyamide rejection layer is synthesized on top of a porous substrate by an interfacial polymerization (IP) reaction [3]. TFC membranes with greater water permeability can significantly reduce the specific energy consumption, whereas increasing their salt rejection is beneficial to improve the product water quality [4]. Nevertheless, there exists a strong tradeoff between membrane water permeability and selectivity: increasing water permeability generally leads to reduced salt rejection [5-7].

Historically, the tradeoff between membrane permeability and selectively was first introduced in the context of gas separation. In 1991, Robeson [8] published his classical work on the "upper bound" for the separation factor and permeability for two-gas systems (e.g., O_2/N_2, CO_2/CH_4, etc.), which quickly became a standard benchmark for gas separation membranes. Owing to the huge success of this seminal work, Robeson [9] published a follow-up paper in 2008 to update the upper bound by including several newly developed membrane materials (e.g., ladder-type and perfluorinated polymers). To date, the 2008 Robeson's upper bound (Fig. 1a) is regarded as the golden ruler to gauge nearly all new membrane development works in

52   the gas separation field [10-17].

53

54   Compared to the huge success of the Robeson's upper bound in gas separation, the

55   upper bound is much less established for desalination membranes. For example, Tang

56   and coworkers [7] reported the tradeoff relationship between water permeability

57   coefficient and NaCl rejection based on 11 commercial TFC polyamide RO and NF

58   membranes. In 2011, Geise et al. [5] formalized the theoretical framework for the

59   upper bound of desalination membranes on the basis of classical solution-diffusion

60   theory [18] for the first time. These authors then provided a log-log upper bound plot

61   of the intrinsic water/NaCl permeability selectivity (P_w/P_s) vs. the intrinsic water

62   permeability P_w for a set of 26 membranes of various chemistries (Fig. 1b). In their

63   approach, the calculation of P_w and the intrinsic NaCl permeability P_s requires the

64   determination of the exact thickness of rejection layers, which is often challenging for

65   TFC polyamide membrane due to the nanosized voids contained in their

66   "ridge-and-valley" surface roughness structures [19-22]. In a more recent review

67   paper, Werber et al. [6] reported the tradeoff relationship in the form of water-salt

68   permselectivity A/B vs. the water permeability coefficient A (Fig. 1c), which provides

69   a simpler way to evaluate polyamide RO membranes. Nevertheless, all the existing

70   attempts relied on relative small-sized data sets. A more comprehensive survey of the

71   literature is yet to be performed to establish the state-of-the-art upper bound for

72   desalination membranes.

73

74 In this study, we analyzed the separation performance of TFC RO and NF membranes,

75 both commercial ones and those prepared in-house, using a dataset collected from

76 more than three hundred of papers published in the last three decades. On this basis,

77 we formulated their upper bound relationship, which could serve as a standard

78 reference in the field of desalination membranes much like the Robeson's upper

79 bound for gas separation membranes. We further examined the effect of various

80 membrane synthesis conditions on the resulting separation performance using the

81 upper bound as a reference framework. Our study provides a new critical tool for the

82 evaluation of membrane development works.

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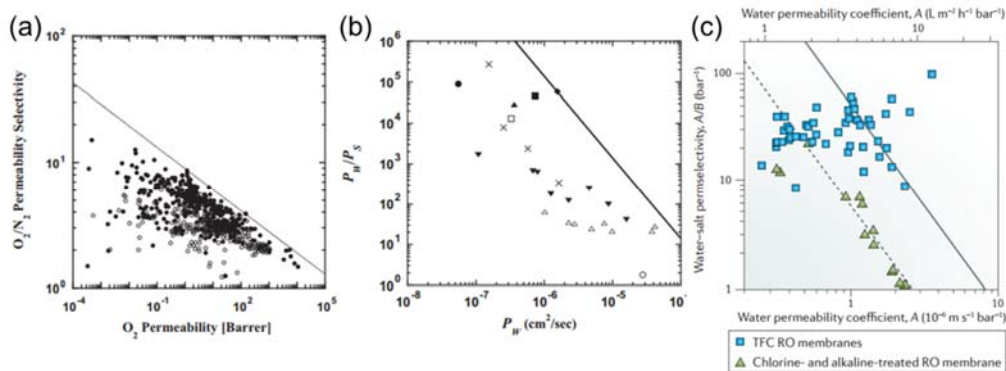
84

85 Fig. 1. The tradeoff between membrane permeability and selectivity reported in the literature.
86 (a) The Robeson upper bound for gas separation membranes (O_2/N_2 selectivity vs. O_2
87 permeability) [5, 8]; (b) the tradeoff between membrane intrinsic selectivity P_w/P_s and
88 intrinsic water permeability P_w for desalination membranes [5]; and (c) the tradeoff between
89 membrane water/NaCl selectivity A/B and water permeability coefficient A for desalination
90 membranes [6]. Copyright permissions for (a), (b), and (c) have been obtained from
91 references [8], [5] and [6] respectively.

92

2. Theoretical background

The transport of water and solutes through a dense polyamide rejection layer can be described by the solution-diffusion model [18, 23, 24]:

$$J_w = A(\Delta P - \Delta \pi) \tag{1}$$

$$J_s = B\Delta C \tag{2}$$

where J_w and J_s are the water flux and solute flux, respectively; ΔP, $\Delta \pi$, and ΔC are the differences in hydraulic pressure, osmotic pressure, and solute concentration across the membrane, respectively. The water permeability coefficient A (also known as the water permeance) and solute permeability coefficient B are related to the intrinsic membrane properties by [25]:

$$A = \frac{D_w K_w V_w}{lRT} = \frac{P_w V_w}{lRT} \tag{3}$$

and $$B = \frac{D_s K_s}{l} = \frac{P_s}{l} \tag{4}$$

where D_w and D_s are the membrane diffusivities to water and solute, respectively; K_w and K_s are the membrane solubilities to water and solute, respectively; V_w is the molar of water; R is gas constant (8.31 Jmol^{-1}K^{-1}); T is the absolute temperature (K); l is the thickness of membrane rejection layer. The determination of intrinsic water and NaCl permeabilities (P_w and P_s, respectively) requires the thickness of the polyamide rejection layer, which is often unavailable in the published papers. Therefore, we adopt A and B values for the further development of the upper bound relationship. It is worthwhile to note that the intrinsic water/solute permeability selectivity (P_w/P_s) reported by Geise et al. [5] is related to the A/B ratio by:

114 $$\frac{A}{B} = \frac{P_w}{P_s}\frac{V_w}{RT}$$ (5)

115 In Equations (1) and (2), $\Delta\pi$ and ΔC are related to the bulk solution properties:

116 $$\Delta\pi = f_{cp}(\pi_f - \pi_p)$$ (6)

117 and $$\Delta C = f_{cp}(C_f - C_p)$$ (7)

118 where π_f and π_p are the osmotic pressures of the feed and permeate water, respectively;

119 C_f and C_p are the solute concentrations of the feed and permeate water, respectively;

120 the factor f_{cp} describes the effect of concentration polarization and is calculated using

121 J_w and the overall mass transfer coefficient K [25]:

122 $$f_{cp} = \exp(\frac{J_w}{K})$$ (8)

123 The solute concentration in permeate water is related to J_s and J_w by:

124 $$C_p = \frac{J_s}{J_w}$$ (9)

125 Substituting Equations (2) and (7) into Equation (9) results in:

126 $$C_p = \frac{Bf_{cp}(C_f - C_p)}{J_w}$$ (10)

127 Furthermore, the apparent solute rejection R for a membrane process is given by [25]:

128 $$R = \frac{C_f - C_p}{C_f} = 1 - \frac{C_p}{C_f}$$ (11)

129 Substituting Equation (10) into Equation (11) leads to:

130 $$R = 1 - \frac{Bf_{cp}(C_f - C_p)}{J_w C_f} = 1 - \frac{Bf_{cp}}{J_w}R$$ (12)

131 Therefore,

132 $$R = \frac{J_w}{J_w + Bf_{cp}}$$ (13)

133   Equation (13) clearly shows that the solute rejection in a dense membrane is achieved

134   by the competition between water transport (J_w) and solute transport (Bf_{cp}). According

135   to the solution-diffusion theory, water and solute molecules diffuse through the dense

136   rejection layer independent of each. Fast diffusion of water molecules and slow

137   diffusion of solute molecules result in increased rejection, whereas slow diffusion of

138   water molecules and fast diffusion of solute molecules lead to a loss of rejection. The

139   enhanced rejection at greater water flux is also commonly known as the dilution effect

140   [25, 26]. Even with a fixed solute flux, a higher water flux dilutes the solute

141   concentration in the permeate solution (Equation (9)) and thus enhances membrane

142   rejection.

143

144   By substituting Equation (1) into Equation (13), we can further obtain a relationship

145   between solution rejection and the water-salt permselectivity A/B ratio:

146
$$R = \frac{\dfrac{A}{B}(\Delta P - f_{cp}\Delta \pi_b)}{\dfrac{A}{B}(\Delta P - f_{cp}\Delta \pi_b) + f_{cp}} \tag{14}$$

147   where $\Delta \pi_b$ is the bulk osmotic pressure difference across the membrane (π_f - π_p).

148   From Equation (14), salt rejection (R) is not only related to the A/B ratio but also

149   affected by the operational conditions (e.g., applied pressure and concentration

150   polarization).

151

152   In general, the water flux J_w and the apparent solute rejection R can be determined

153   experimentally. However, the experimental conditions reported in the publications

154   surveyed by us often do not contain sufficient information (e.g., the geometry of

155   spacer and the dimensions of the flow channel) to accurately determine the

156   concentration polarization factor f_{cp} [27-31]. Therefore, we simplified Equations (1),

157   (13) and (14) by assuming $f_{cp} = 1$ for the determination of the values of A and A/B in

158   the current study. This assumption is reasonable when $J_w << K$ (e.g., by using spacers

159   and high crossflow velocities to increase the mass transfer in the feed channels) [28].

160   Thus,

161    $$J_w = A(\Delta P - \Delta \pi_b)$$ (15)

162    $$R = \frac{J_w}{J_w + B}$$ (16)

163    $$R = \frac{\frac{A}{B}(\Delta P - \Delta \pi_b)}{\frac{A}{B}(\Delta P - \Delta \pi_b) + 1}$$ (17)

164   Based on the limited data available in the literature [27-31], we performed a

165   comparison of the A and A/B values with and without the simplication of $f_{cp} = 1$ (Fig.

166   A1, Supporting Information Appendix A). Readers are cautioned that this simplication

167   leads to a slightly underestimated water permeance (i.e., A value). In addition, the

168   water/NaCl selectivity (i.e., A/B ratio) is also underestimated.

169

3. The Upper Bound

Fig. 2 presents the dependence of NaCl transmission (1 - R) and water/NaCl permselectivity (A/B) on water permeance A based on a set of 1204 data points collected from the literature [19, 20, 27-378]. There is a general trend of increased NaCl transmission (i.e., decreased NaCl rejection) for more permeable membranes, which has been well documented in the existing literature [5-7]. According to Equations (16) and (17), the solute rejection of a membrane depends not only on its permselectivity but also on the operational conditions (e.g., water flux or applied pressure). This relationship is further illustrated in Fig. 3. As shown in Fig. 3(a), for any given water flux and membrane water permeance A, increasing its water/NaCl permselectivity A/B results in improved NaCl rejection. Each shaded area in Fig. 3(a) represents the combinations of water permeance A, water/NaCl permselectivity A/B, and water flux J_w in order to achieve a given NaCl rejection. For example, to achieve a targeted rejection of 99% at a fixed flux of 10 Lm^{-2}h^{-1}, any A and A/B combinations yielding a constant B value of 0.1 Lm^{-2}h^{-1} would satisfy the requirement (Equation (16)). These combinations are represented by the line passing through A = 2 Lm^{-2}h^{-1}bar^{-1} and A/B = 20 bar^{-1} (the solid circle in Fig. 3) with a constant slope of 1. For the same membrane, if a higher water flux of 40 Lm^{-2}h^{-1} is used, an improved rejection of 99.75% can be achieved according to Equation (16). Alternatively, to maintain the same rejection of 99% at the water flux of 40 Lm^{-2}h^{-1}, a less selective

190 membrane can be allowed ($A = 2$ Lm^{-2}h^{-1}bar^{-1} and $A/B = 5$ bar^{-1}, represented by the

191 empty circle in Fig. 3(a)).

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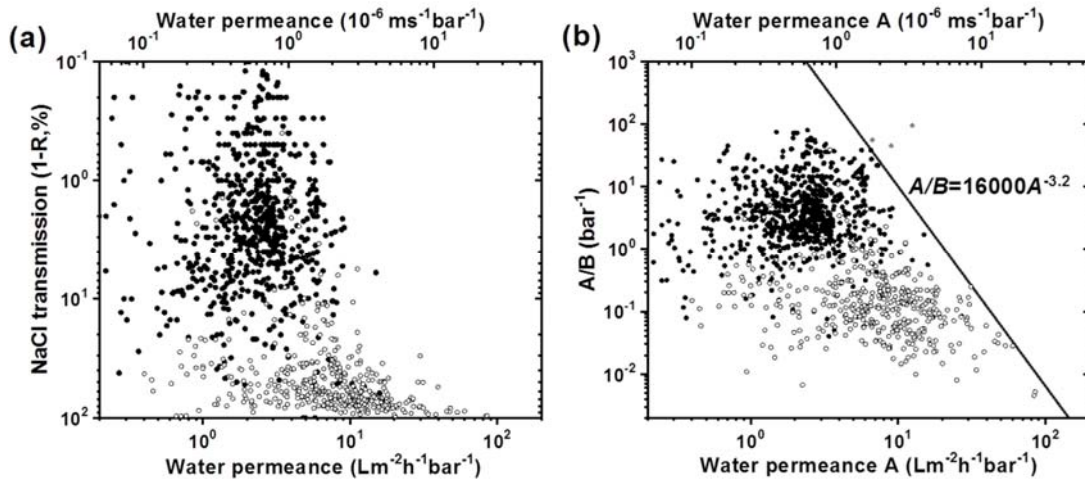
193
194 Fig. 2. The permeance-selectivity tradeoff for TFC polyamide membranes: (a) Correlation
195 between water permeance and NaCl transmission (1-R, %), (b) upper bound of water/NaCl
196 selectivity A/B and water permeance. The solid points are for RO and empty points are for NF.
197 The grey points were obtained from [6], which makes further reference to a conference paper
198 [379]. Nevertheless, the original source could not be verified. Therefore, these data points
199 were excluded for the purpose of establishing the upper bound line.

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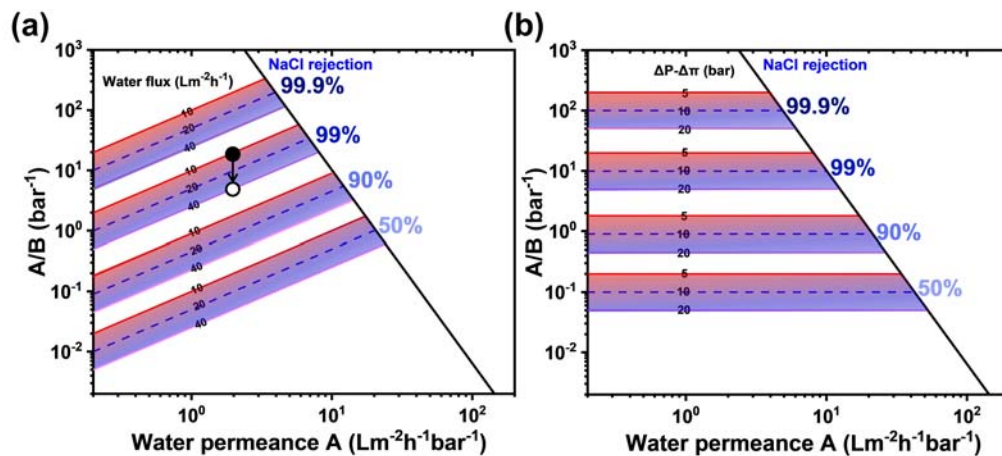
201
202 Fig. 3. Effect of membrane separation properties and operational conditions on salt rejection.
203 (a) Combination of water flux, salt rejection, water permeance and water/NaCl selectivity

204 based on Eq. (16); (b) Combination of net driving force (ΔP-$\Delta \pi$), salt rejection, water
205 permeance and water/NaCl selectivity based on Eq. (17).

206

207 It is worthwhile to note that many publications reporting the synthesis of polyamide

208 membranes often evaluate membrane performance at fixed pressure instead of fixed

209 flux. In Fig. 3(b), each shaded area represents the combination of water permanence A,

210 water/NaCl permselectivity A/B, and net driving force ΔP-$\Delta \pi$ for achieving a given

211 NaCl rejection. When a fixed net driving force is used, the NaCl rejection is solely

212 dependent on the water/NaCl permselectivity A/B, and high rejection is obtained for

213 membranes with greater A/B. For a given membrane, increasing the net driving force

214 enhances its rejection as a result of increased water flux (the dilution effect, see Fig.

215 3(a)). On the other hand, membranes with lower selectivity can be compensated by

216 the use of greater net driving force to achieve the same NaCl rejection (e.g., $A/B = 20$

217 bar^{-1} and ΔP-$\Delta \pi = 5$ bar vs. $A/B = 5$ bar^{-1} and ΔP-$\Delta \pi = 20$ bar for an NaCl rejection of

218 99%).

219

220 Due to the inherent dependence of rejection on water flux in addition to membrane

221 intrinsic separation properties (Fig. 3(a)), the plot of the NaCl transmission (1 - R) vs.

222 the water permeance A is less preferred for the establishment of the upper bound. A

223 more preferred way is to plot the water/NaCl permselectivity A/B vs. the water

224 permeance A. Fig. 2b shows a clear tradeoff behavior, with the upper bound line given

225 by:

226 $$\frac{A}{B} = 16000A^{-3.2} \quad \text{(with } A/B \text{ in bar}^{-1} \text{ and } A \text{ in Lm}^{-2}\text{h}^{-1}\text{bar}^{-1}) \quad (18)$$

227 For the first time, we established the upper bound relationship based on a

228 comprehensive dataset, which can be used as a standard reference for benchmarking

229 membrane separation properties in future studies.

230

231 Fig. 4 further presents the separation properties of commercial seawater RO (SWRO),

232 brackish water RO (BWRO), and NF polyamide membranes in relation to the upper

233 bound. The empty symbols in Fig. 4 represent membranes synthesized at bench scales.

234 The area under the upper bound line is further divided into three regions based on the

235 threshold rejection of 99% and 90% (assuming a water flux of 20 $\text{Lm}^{-2}\text{h}^{-1}$). As

236 expected, most of the SWRO membranes have rejections of > 99% whereas the

237 majority of NF membranes have rejections of < 90%. Even though the separation

238 performances of commercial BWRO and NF membranes are reasonably close to the

239 upper bound line, the data points for SWRO fall far below this line. This difference

240 shows a great margin for further improving SWRO membranes.

241

242 It is worthwhile to note that the upper bound in Fig. 2b was developed without the

243 explicit treatment of concentration polarization effect (refer to Section 2 and Eq.

244 12-17 for further information). Although a thorough review of the mass transfer and

245 concentration polarization in the bulk solution [380-383] is beyond the scope of the

246 current study, concentration polarization could have important effect on the apparent

247 membrane performance. Therefore, the development of novel strategies for enhanced

248 mass transfer (e.g., by using novel spacers [384-387]) is critical along with the

249 development of high performance membranes. It is also recommended that future

250 membrane development studies should explicitly report the mass transfer coefficient

251 and membrane *A* and *B* values in addition to their routine report of water flux and

252 apparent salt rejection for a more wholistic assessment of membrane separation

253 performance.
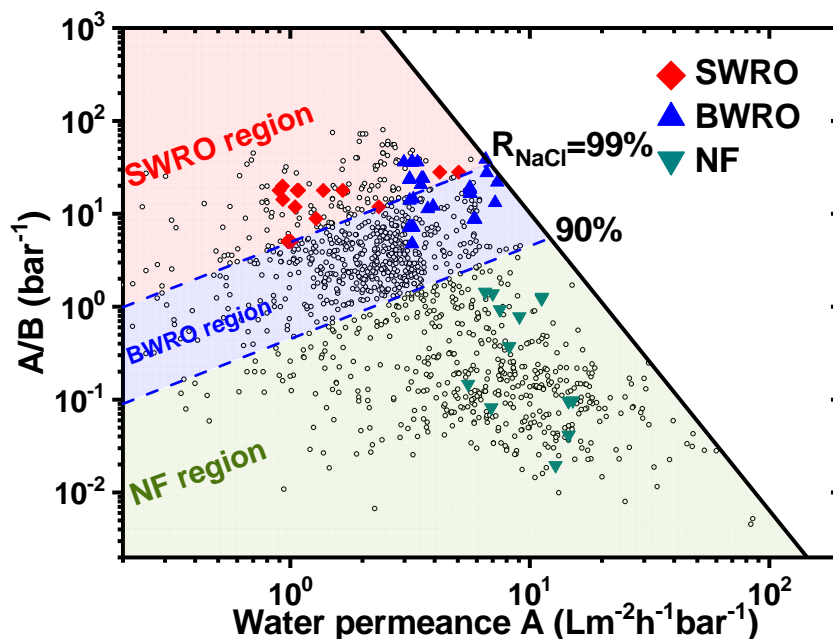
254
255 Fig.4. Water permeance and selectivity of commercial seawater RO (SWRO), brackish water
256 RO (BWRO) and NF membranes in the water permeance and selectivity diagram. Blue dash
257 lines correspond to the NaCl rejection of 99% and 90%, respectively, at a flux of 20 $Lm^{-2}h^{-1}$.
258 The empty symbols in Fig. 4 represent membranes synthesized at bench scales. The
259 separation performance of commercial membranes was collected based on manufacture's
260 specifications provided by DuPont Filmtech, GE-Osmonics (Desal), Nitto Hydranautics and
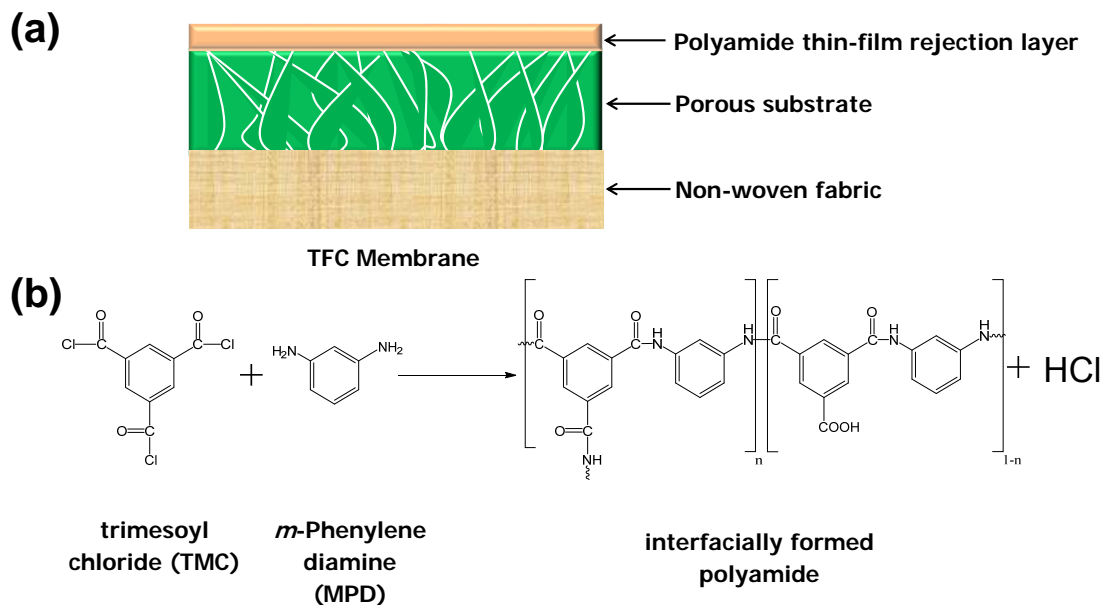261 Toray Industries [375-378].

262
263

4. Major factors affecting the separation performance of TFC

4.1. Monomers

4.1.1. *Monomer types*

The state-of-the-art TFC RO membrane is composed of a dense crosslinked polyamide, a porous ultrafiltration support layer and a non-woven fabric layer to provide strong mechanical stability (Fig. 5a). The polyamide layer, which largely determines the water flux and salt rejection [93], is usually prepared by an interfacial polymerization reaction between *m*-phenylenediamine (MPD in aqueous phase) and trimesoyl chloride (TMC in organic phase, Fig. 5b). At the meantime, a wide range of alternative monomers/reactants has also been reported in the literature. Table 1 provides a summary of different types of monomers/reactants and the typical membrane performance. More comprehensive information on the impact of monomer on membrane performance is provided in Table B1 of the Supporting Information.

(a)

Polyamide thin-film rejection layer

Porous substrate

Non-woven fabric

TFC Membrane

(b)

trimesoyl
chloride (TMC)

m-Phenylene
diamine
(MPD)

interfacially formed
polyamide

$+ HCl$

278

279   Fig. 5. Structure and preparation of TFC polyamide membrane: (a) typical membrane

280   structure; (b) interfacial polymerization reaction between MPD and TMC.

281

282   The separation performance of membranes prepared by TMC/MPD chemistry is

283   shown in Fig. 6. In general, this membrane chemistry can provide high NaCl rejection

284   (typically > 90% and often > 99%) along with moderate water permeance of 1-10 L

285    $m^{-2}h^{-1}bar^{-1}$. Indeed, this chemistry has been the most commonly used one for

286   commercial SWRO and BWRO membranes [3, 105]. Since both TMC and MPD

287   contain a benzene ring, membrane prepared using this chemistry are often known as

288   fully aromatic polyamide membranes [388]. The TMC/MPD chemistry typically

289   results in highly crosslinked polyamide rejection layer that is a pre-requisite of

290   achieving high NaCl rejection [7, 342, 389]. This densely crosslinked structure can be

291   explained by the high electron density of the benzene ring that makes the amine

292   functional groups of MPD more reactive compared to aliphatic diamines [20]. In

16

293   addition to MPD, other aromatic diamines (see Table 1) can also react with TMC to

294   form highly-crosslinked fully aromatic polyamide rejection layers with NaCl rejection

295   of >90% (Fig. 6). Likewise, fully aromatic polyamide can be prepared by reacting

296   MPD with other aromatic acryl chlorides (Table 1). In general, these fully aromatic

297   polyamide membranes are located in SWRO or BWRO regions of the upper bound
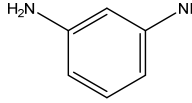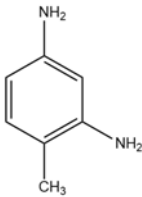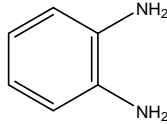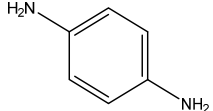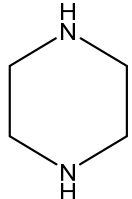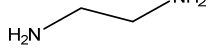
298   diagram (Fig. 6).

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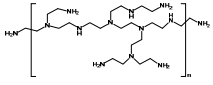
300   In contrast to the relatively high NaCl rejection of fully aromatic polyamide

301   membranes, semi-aromatic polyamide membranes prepared by the reaction of TMC

302   with aliphatic diamines (Table 1) have much lower salt rejection (typically <90%, see

303   Fig. 6) due to the relatively low reactivity of the amine functional groups [20].

304   Therefore, these semi-aromatic polyamide membranes are generally not suitable for

305   RO applications. Instead, they are more commonly used as high-permeability NF

306   membranes. Piperazine (PIP) is one of the most commonly used aliphatic diamine for

307   the fabrication of semi-aromatic polyamide nanofiltration membrane. For example,

308   the widely reported commercial NF membrane NF270 (DuPont FilmTech®) is

309   prepared by a TMC/PIP chemistry. In addition to aliphatic amine monomers,

310   polymer-based aliphatic amines can also be used. For example, polyethyleneimine

311   (PEI), which contains abundant amine groups, can react with TMC to form positively

312   charged TFC membranes with high water permeance but low NaCl rejection [314].

313   Other monomers in an aqueous phase, such as alcohols or alcohol-like monomers

314   (containing hydroxyl groups), could also react with TMC to form polyester-based

315   rejection layer, which has much looser structure and thus low NaCl rejection (Fig. 6).

316

317   Table 1. Main types of monomers.

	Type of monomers	Examples	Chemical formula	Membrane types	Remarks
Aqueous phase	Aromatic diamine	MPD [105]		Most widely used chemistry for SWRO and BWRO. Also used for tight NF membranes.	Typical NaCl rejection > 90%; rejection of >99% can also be achieved; low to moderate water permeance
		Homologue: MMPD [390]			
		OPD: 1,2-phenylene diamine		Allotrope of MPD, to fabricate BWRO and tight NF membranes.	Moderate NaCl rejection 85-91%; moderate water permeance [304]
		PPD: 1,4-phenylene diamine			
	Aliphatic diamine	PIP [343]		Most widely used chemistry for NF membranes.	Negatively charged NF; typical salt rejection s followed: $Na_2SO_4 \approx MgSO_4 > MgCl_2 \approx NaCl$
		EDA [273]		NF membranes	High water permeance; low salt rejection
	Aliphatic polymer-based	PEI [314]		NF membranes	Often resulting in positively charged NF membranes; Rejection

				Rejection/Property notes
	multi-amine			typically follows $MgSO_4 \approx MgCl_2 > Na_2SO_4 \approx NaCl$
	Alcohol or hydroxyl groups containing chemicals	PVA: Polyvinyl alcohol [283]	Polyester-based rejection layer; resulting in loose NF membranes	Relatively high water permeance and low salt rejections
		TEOA [178]		
	Others	Melamine [301]	Loose NF membranes	Negative charge, relatively high water permeance, and moderate salt rejection
		Sulfonated diamine [303]	SWRO and BWRO membranes	Low water permeance; high boron rejection
			NF membranes	Enhanced antifouling properties
		Disulfonated diamine [28]	Tight NF membranes	High water permeance, but low NaCl rejection
Organic phase	Aromatic acryl chloride.	TMC [105]	Most widely used chemical for fabricating SWRO and BWRO. Also used for tight NF membranes.	Typical NaCl rejection > 90%; rejection of > 99% can also be achieved; low to moderate water permeance
		Tri-functional acid chloride BTRC: [294]	React with MPD to fabricate SW/BW RO membranes	Low water permeance; high NaCl rejection (>98%)

		Tri-functional acid chloride CFIC [173, 297]			
		Tetra-functional acid chloride BTEC: [294]			
	Bi/mono-functional aromatic acryl chloride	PC: phthaloyl chloride		NF membranes [304]	Moderate water permeance; low NaCl rejection (< 50%)
		IPC: isophthaloyl chloride			
		TPC: terephthaloyl chloride			
		BC: Benzoyl chloride			
	Cycloaliphatic acryl chloride	HTC: cyclohexane-1,3,5-tricarbonyl chloride [390]		SW/BW membranes	Moderate water permeance and high NaCl rejection (>95%); high chlorine resistance
	Others	CC: cyanuric chloride[287]		NF membranes	High permeance; low salt rejection
		GA: Glutaraldehyde [283]		NF membranes	Often used a crosslinking agent

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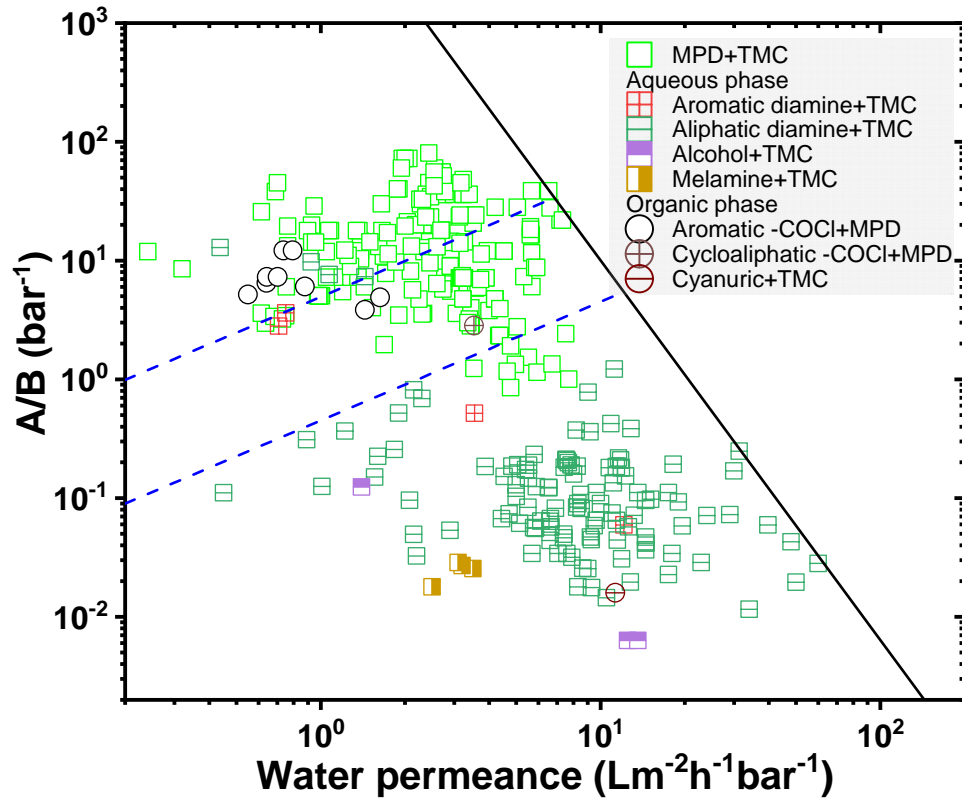
Fig. 6. The impact of monomer types on the upper bound correlation based on Table 1. Blue dash lines correspond to the NaCl rejection of 99% and 90%, respectively, at a flux of 20 $Lm^{-2}h^{-1}$.

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324

325 **4.1.2.** *Monomer concentration and reaction conditions*

326 Monomer concentration plays an important role in determining membrane separation

327 performance due to its effect on monomer diffusivity and reaction rate. This section

328 focuses on the effect of monomer concentration of MPD, PIP, and TMC due to the

329 wide coverage in the literature on these monomers and their commercial success.

330 Effects of other reaction conditions are also briefly mentioned.

331

332 Table 2 presents a summary of some recently published literature relating membrane

333 separation performances to monomer concentrations in the aqueous and organic phase,

334 with a more comprehensive summary presented in Table B2 in the Supporting

335 Information. Increasing monomer concentration in the aqueous phase, regardless of

336 whether MPD or PIP was used, generally results in significantly decreased water

337 permeance and increased salt rejection (Table 2). This can be attributed to the

338 enhanced crosslinking degree of the polyamide rejection layer caused by the greater

339 presence of the amine monomers [119, 183]. Likewise, the increased reaction time,

340 increased immersion time in the aqueous amine solution and higher temperature of

341 interfacial polymerization can also result in a denser and more crosslinked rejection

342 layer [58, 173, 182, 193].

343

344 The effect of TMC concentration in the organic phase appears to be more disparate.

345   Some studies reported that the increased TMC concentration could result in less

346   crosslinked polyamide film with reduced salt rejection [28, 183], while other studies

347   showed enhanced salt rejection at higher TMC concentration [182, 343]. These

348   contradictions may be reconciled by recognizing a "two-stage" effect of TMC

349   concentration on membrane separation performance [391]. When TMC concentration

350   is low (e.g., 0.01-0.1 wt.%), increasing its concentration could potentially enhance the

351   crosslinking degree and/or the thickness of the polyamide rejection layer, resulting in

352   increased salt rejection and decreased water flux [119, 183]. However, a much higher

353   TMC concentration can lead to the formation of a less crosslinked TFC membrane:

354   the high abundance of TMC over MPD caused more acryl chloride groups to

355   hydrolyze instead of forming polyamide bonds with MPD [119, 183]. This effect

356   leads to higher water permeance yet reduced NaCl rejection. Therefore, maintaining

357   an optimal stoichiometry is critical for the preparation of high-rejection membranes.

358   Based on experience, an MPD/TMC concentration ratio of 10 – 20 is often reported in

359   the literature [28]. Theoretically, this optimum ratio will also depend on the affinity of

360   MPD to the substrate and the desorption and diffusivity of MPD. For example, the use

361   of an intermediate layer   (e.g., formed tannic acid-Fe^{3+} complex [349] or

362   polydopamine[347]) enhances amine monomer sorption and regulate its diffusion,

363   which led to a tighter rejection layer compared to the control membrane.

364

365   The pH of the aqueous solution could have a profound impact on membrane

366 performance. Higher pH can effectively remove proton, a product produced during the

367 IP reaction (Fig. 5b), thus resulting in a more crosslinked polyamide rejection layer

368 [173, 390]. In addition, controlling the temperature of the aqueous or organic phase is

369 an effective strategy to regulate the IP reaction. For instance, Khorsidi et al. [200]

370 decreased the temperature of the organic phase to react with MPD, which could

371 significantly reduce the diffusion rate of MPD. The formed polyamide layer under the

372 low temperature of the organic phase (e.g., -20 °C) was much thinner and more

373 flattened compared to that of the control membrane prepared under room temperature.

374 As a result, membrane water permeance increased with slightly decreased NaCl

375 rejection. In another study, Liu et al. [344] also decreased the temperature of the

376 organic phase to react with PIP. Their low-temperature approach not only reduced the

377 thickness and roughness of the rejection layer but also generated a Janus polyamide

378 layer with opposite charge due to the reduced diffusion of PIP. The resulting

379 membrane showed approximately 3-fold higher water permeance with similar salt

380 rejection. Despite advances, a more systematic and fundamental framework is yet to

381 be established for the control of the IP reaction.

382

383 **Table 2.** Monomer concentration.

Application	Effect of monomer concentration (aqueous phase)	Effect of monomer concentration (organic phase)	Ref.
RO/FO	Increasing MPD concentration from 0.5 to 2 wt.%: water permeance ↓ and R_{NaCl} ↑	Increasing TMC concentration from 0.05 to 0.1 w/v%: water permeance ↓ and R_{NaCl}↓; increasing TMC concentration from 0.1 to 1 w/v%: water permeance ↑ and R_{NaCl}↓	[183]
RO	Increasing MPD concentration from 0.5 to 3 w/v%: water permeance ↗ ↘ and R_{NaCl} no	Increasing TMC concentration from 0.025 to 0.13 w/v %: water permeance ↗ ↘ and	[28]

	major change	R_{NaCl} no major change	
RO	Increasing MPD concentration from 0.01 to 1 wt.% (MPD:TMC molar ratio=10): water permeance ↓ and NaCl rejection ↑	Increasing TMC concentration from 0.001 to 0.1 wt.% (MPD:TMC molar ratio=10): water permeance ↓ and R_{NaCl} ↑↑	[177]
NF	Increasing PIP concentration from 0.25 to 1.5 wt.%: water permeance ↓ and R_{Na2SO4} ↑	Increasing TMC concentration from 0.05 to 0.3 wt.% and reaction time from 10 to 240: water permeance ↓; R_{Na2SO4} ↑	[58]
NF	Increasing PIP concentration from 0.1 to 2% and immersion time from 0.5 to 4 min: water permeance ↓; R_{Na2SO4} ↑	Increasing TMC concentration from 0.01 to 0.2% and reaction time from 0.5 to 3 min: water permeance ↓ and R_{Na2SO4} ↑	[161]
NF	Increasing PIP concentration from 0.03 to 0.5 wt.%: water permeance ↓ and R_{MgSO4} ↑	Increasing TMC concentration from 0.05 to 0.3 w/v% and reaction time from 10 to 110 s: water permeance ↓ and R_{MgSO4} rejection ↑	[182]
NF	Increasing PIP concentration from 0.5 to 2 g/L: water permeance ↓ and R_{Na2SO4} ↑	Increasing TMC concentration from 0.5 to 2.5 g/L and reaction time from 0.5 and 4 min: water permeance ↓ and R_{Na2SO4} ↑	[343]
NF	Increasing PIP concentration from 0.1 to 2 wt.%: water permeance ↓ and R_{Na2SO4} ↑	- -	[348]
NF	Increasing PIP concentration from 0.1 to 2.4 wt.%: water permeance ↓ and R_{Na2SO4} ↑	Increasing TMC concentration from 0.00625 to 0.025 w/v%: water permeance ↓ and R_{Na2SO4} ↑; increasing TMC concentration from 0.025 to 0.1 w/v%: water permeance ↑ and R_{Na2SO4} ↓	[392]

384

4.2. Additives

4.2.1. *Additives in aqueous solution*

Incorporating additives in the aqueous phase during interfacial polymerization reaction could significantly affect monomer diffusion rate, monomer solubility and miscibility between the aqueous and organic phase [335]. Subsequently, various additives have been reported for optimizing membrane separation performances [105]. The major types of additives and their main impact on membrane performance are summarized in Table 3, and more detailed information is provided in Table B3 in the Supporting Information.

Proton acceptor is an import type of additives that are often used in the aqueous phase (Table 3). As shown in Fig. 5b, the IP reaction generates hydrochloric acid as a reaction byproduct, whose accumulation could result in a poor crosslinking of the polyamide rejection layer. This issue can be addressed by the addition of proton acceptors, such as $NaOH/Na_2CO_3$ [178], $NaHCO_3$ [19, 20] and triethylamine (TEA) [333], to neutralize H^+ generated during membrane formation. The resulting membranes tend to have more crosslinked rejection layers with improved NaCl rejection [178, 333]. For example, Fig. 7a shows improved selectivity by the addition of TEA [333] and $NaHCO_3$ [19, 20]. Furthermore, the addition of $NaHCO_3$ led to the formation of nanosized CO_2 bubbles, whose encapsulation in the polyamide resulted in a rougher membrane with a greatly increased surface area – a phenomenon referred

406 as nano-foaming by Tang and co-workers [20]. However, excessive addition of proton

407 acceptors could potentially hydrolyze TMC, resulting in poor crosslinking degree,

408 increased water flux and decreased NaCl rejection [178, 333].

409

410 Aside from the addition of bases as proton acceptors, acids can also be incorporated

411 into the aqueous phase to control the IP reaction (Table 3). Camphor sulfonic acid

412 (CSA), a relatively strong acid, is often added into the aqueous solution to improve

413 membrane water permeance (Fig. 7a). Rahimpour et al. [333] reported that the

414 addition of CSA into MPD solution could generate a less crosslinked polyamide RO

415 membrane, thus resulting in increased water flux and decreased NaCl rejection. In

416 addition, membrane surface hydrophilicity was improved due to the abundant sulfonyl

417 hydroxide groups in CSA [329]. Likewise, incorporating acid such as

418 3,5-diaminobenzoic acid into PIP aqueous solution result in a more permeable NF

419 membrane with decreased NaCl salt rejection [324]. Interestingly, the rejection of

420 divalent ions (e.g., Na_2SO_4 or Mg_2SO_4) was improved as a result of the enhanced

421 Donnan exclusion effect due to the increased membrane surface charge.

422

423 Surfactants, such as sodium dodecyl sulfate (SDS), contains a hydrophobic tail and a

424 hydrophilic head [393]. Thus, the addition of SDS into the aqueous phase can

425 significantly reduce the surface/interfacial tension between the aqueous and organic

426 phases [394]. Often, membrane separation performances are not significantly affected

427 by the addition of SDS alone in the aqueous solution [394]. In contrast, adding SDS

428 and TEA together can significantly enhance membrane separation performance [394].

429 In addition to surfactant, hydrophilic additives/salts can also be incorporated in the

430 aqueous phase to participate in the IP reaction to enhance membrane performance

431 (Table 3). For example, the use of TEACSA [209, 334], ABA-TEA salt [115, 335],

432 2-(2-hydroxyethyl) pyridine, 4-(2-hydroxyethyl) morpholine [357], $CaCl_2$ [359] and

433 taurine [40] have been shown to not only enhance membrane hydrophilicity, but only

434 provide additional water transport pathway and enhanced membrane charge.

435 Subsequently, membrane permeance and selectivity can be increased at the same time

436 ([357] and Fig. 7b). Hao et al. [359] reported that the addition of Ca^{2+} in the

437 MPD/water solution resulted in a polyamide membrane with enhanced antifouling

438 property thanks to the sequestration of the carboxyl groups.

439

440 In addition to the use of water as the solvent for the amine monomer, organic solvents

441 (e.g., dimethyl sulfoxide (DMSO) [329, 330]) or aqueous co-solvents (e.g., alcohols

442 such as isopropyl alcohol (IPA) [327, 335], ethanol [326] and 2-ethyl-1,3-hexane diol

443 (EHD) [318]) can also be added into the aqueous phase to control the IP reaction

444 (Table 3 and Fig. 7c). For example, adding co-solvents, such as alcohols, reduces the

445 surface tension of the aqueous phase, thus helping MPD penetrating into the pores of

446 the relatively hydrophobic substrate to promote the IP reaction [327, 335].

447 Accordingly, the membrane roughness is significantly increased, leading to increased

448   water flux and nearly unchanged selectivity [327]. However, the addition of IPA at

449   high concentration (e.g., greater than 20%) would result in reduced NaCl rejection

450   [327], probably due to the formation of polyester-based rejection layer caused by the

451   poor crosslinking degree. The incorporation of another alcohol, 2-ethyl-1,3-hexane

452   diol (EHD), with a hydrophobic chain, can effectively facilitate the transport of MPD

453   to the organic phase, leading to the improved membrane selectivity [318].

454   Furthermore, the hydroxyl groups of EHD can form hydrogen bonds with MPD to

455   improve membrane water flux [318]. On the other hand, the incorporation of organic

456   solvent into the aqueous phase could significantly increase the miscibility of the

457   aqueous phase and organic phase to enhance the MPD diffusion rate. For example, the

458   incorporation of DMSO could lead to the TFC membrane with significantly enhanced

459   water flux (i.e., nearly 5 times with the concentration of 3 wt.% in the aqueous phase)

460   and slightly reduced NaCl rejection [330].

461

462   Other additives, such as oxidants (e.g., NaClO), can be added into the aqueous phase

463   to oxidize the amine monomer *in situ* during the IP reaction. Xu et al. incorporated

464   NaClO   [49]   into   PIP   aqueous   solution,   which   oxidized   PIP   into

465   1,1′-carbonylbis-piperazine. The oxidized monomer was then reacted with TMC to

466   generate polyamide nanofiltration membrane with both enhanced water permeance

467   and salt rejection.

468

469 Table 3 Main types of additives used for interfacial polymerization reaction and their effects
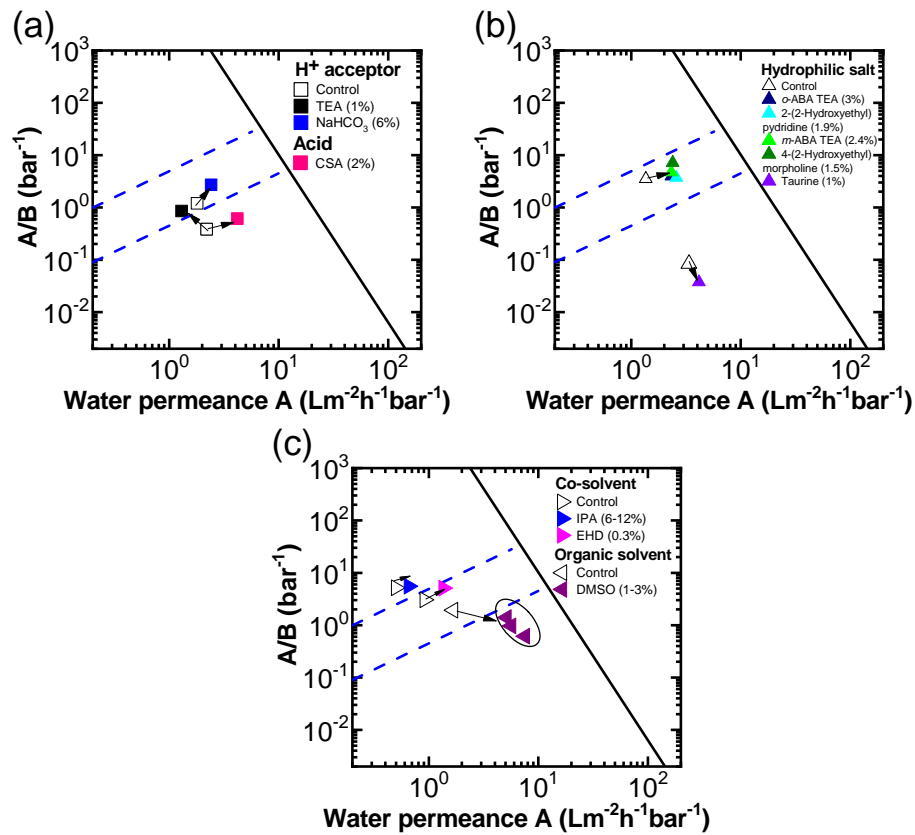
470 on membrane performance.

	Type of additives	Examples	Purpose	Membrane performance
Aqueous phase	H+ acceptor	NaOH [173, 390], NaHCO$_3$ [19], Na$_2$CO$_3$ [178] and triethylamine (TEA) [333]	Removal of hydrogen chloride of IP reaction; acting as a reaction accelerator	Resulting in enhanced crosslinked polyamide layer and ↑ salt rejection; overdosage could lead to hydrolysis of TMC, water permeance ↑ and salt rejection ↓
	Acid	Camphor sulfonic acid CSA [329, 333]	Relatively strong acid, acting as pH regulator	Less crosslinked polyamide layer; water permeance ↑ and salt rejection ↓; Membrane hydrophilicity ↑ due to the increased -COOH groups
		3,5-diaminobenzoic acid [324]	Amine and carboxylic groups reacting with TMC	Water permeance ↑, NaCl rejection ↓; enhanced surface charge: NaSO$_4$ rejection ↑
	Surfactants	Sodium dodecyl sulfate (SDS) [329, 393, 394]	Decreasing surface tension between the aqueous and organic phase	No major change in membrane separation performance; often couple with TEA
	Hydrophilic additives/salt	*o*-aminobenzoic acid-triethylamine salt; *m*-aminobenzoic acid-triethylamine salt, CaCl$_2$ [359], 2-(2-hydroxyethyl) pyridine; 4-(2-hydroxyethyl) morpholine and taurine [40, 115,	Enhancing hydrophilicity and charge repulsion; improving water transport; neutralized surface charge for enhanced antifouling properties	Water permeance ↑ and salt rejection ↑; creating more free volume in the polyamide rejection layer, allowing both water and salt pass through; overloading could cause ↓↓ NaCl rejection [357]

		209, 335, 357]		
	Organic solvent	DMSO [329, 330]	Increased miscibility of aqueous and the organic phase; enhanced MPD diffusion rate	Water permeance ↑ and NaCl rejection ↓; higher concentration (>1 wt.%) may cause NaCl rejection significant reduction
	Co-solvent (e.g., alcohols)	Isopropyl alcohol (IPA) [327, 335]	Reduce surface tension of the aqueous solution; form ester bond when reacting with TMC	When IPA concentration is between 5-20 wt.%, water permeance ↑ and NaCl rejection unchanged; when IPC concentration is greater than 20%, water permeance ↓ and NaCl rejection ↓; Increase polyamide surface roughness;
		Ethanol [326]	Ethanol can dissolve in both water and organic phase, which can form a miscible zone; facilitate MPD diffusion; reduce surface tension; help MPD solution penetrate substrate; formation of ester bond when reacting with TMC	Water permeance ↑ and salt rejection ↓ ; higher crosslinking degree
		2-ethyl-1,3-hexane diol (EHD) [318]	The hydrophobic alkyl group of EHD help transport MPD to TMC; hydroxyl group of EHD can form hydrogen bond with MPD	Water permeance ↑ and salt rejection ↑
	Oxidation agents	NaClO [49]	PIP could be oxidized to 1,1′-carbonylbis-piperazine to react with TMC;	Water permeance ↑ and salt rejection ↑
Organic	Surfactants	SDS,	Increased free volume in	Water permeance ↑ and

phase		Cetyltrimethylammonium bromide (CTAB) and Non-ionic (Triton X-100) [320]	the PA layer	NaCl and Na$_2$SO$_4$ rejection unchanged and MgCl$_2$ rejection ↑; higher concentration may deteriorate of the PA layer
	Co-solvents	Acetone [319]	Decreasing interfacial tension and solubility between two phases; formation of thin dense PA layer	Formation of thin dense PA layer; water permeance ↑ by 4-fold with slightly ↓ NaCl rejection
		Ethyl acetate, diethyl ether and toluene [317]	Resulting in a multilayered polyamide structure	No major change in membrane separation performance
		IPA and dimethyl formamide (DMF) [317]	Creating holes in polyamide rejection layer by adding IPA in the organic phase; DMF may dissolve the PSF substrate;	Water permeance significantly ↑ with NaCl rejection significantly ↓
		Tetraethyl orthosilicate (TEOS) [322]	Increased MPD diffusion that terminates the IP reaction earlier	Water permeance ↑ by 4-fold with slightly ↓ NaCl rejection
	Amine monomer inhibitor	1,3-propanesultone [323]	Attracting amine groups to the polyamide rejection layer; resulting in a multilayered polyamide structure that increases the membrane surface area	Water permeance ↑ by 40% with unchanged NaCl rejection
	Phosphate additives	Tributyl phosphate (TBP) and triphenyl phosphate (TPP) [318]	The phosphate group can form complex with acryl chloride groups of TMC *via* dipole-dipole interaction	The addition of TBP resulted in water permeance ↑ , and NaCl rejection decreased; The addition of TPP resulted in water permeance ↓ and NaCl rejection unchanged;

473

Fig. 7. The effect of additives in aqueous phase on membrane separation performance: (a) proton acceptor and acid; (b) hydrophilic salts; (c) co-solvent and organic solvent. Blue dash lines correspond to the NaCl rejection of 99% and 90%, respectively, at a flux of 20 $Lm^{-2}h^{-1}$.

477

478   **4.2.2. *Additives in organic solution***

479   The incorporation of additives in the organic phase can also significantly affect the IP

480   reaction and membrane performance by influencing the miscibility between water and

481   organic phases and/or monomer diffusion rate (Table 3 and S3). In this section,

482   various organic phase additives (e.g., co-solvents and surfactants) are summarized

483   (see Fig. 8).

484

485   The effect of organic phase co-solvents is shown in Fig. 8a. Kamada et al. [317]

486   reported the incorporation of different types of co-solvents into the organic phase. For

487   example, the addition of acetone in the organic phase can effectively reduce the

488   monomers solubility difference in the two phases and simultaneously enhance the

489   monomer diffusion rate. The resulting membrane had much rougher surface together

490   with a multi-layered polyamide structure as well as increased water permeance. The

491   incorporation of another co-solvent, dimethylformamide (DMF), can significantly

492   enhance membrane water flux by approximately an order of magnitude but at the

493   expense of severely decreased salt rejection [317]. The added DMF might have

494   dissolved the polysulfone support layer during the IP reaction, which significantly

495   reduced membrane mass transfer resistance [317].

496

497   Surfactants, such as cationic cetyltrimethylammonium bromide (CTAB), non-ionic

498   (Triton X-100) and anionic sodium dodecyl sulfate (SDS), can be added into the

499 organic phase to affect the interfacial polymerization reaction (Table 3 and Fig. 8b).

500 Mansourpanah et al. [320] incorporated SDS in the organic phase of TMC that was

501 used to react with PIP. The modified membrane had more free volume in its rejection

502 layer due to the strong repulsion force between polyamide chains and the SDS

503 molecules, resulting in enhanced membrane water permeance. The incorporation of

504 CTAB (positive charge) and Triton X-100 (through H-bonding) could further

505 significantly enhance the absorbed amount of PIP, leading to a denser polyamide

506 rejection layer [320]. Incorporating other additives, such as phosphate additive [318]

507 or 1,3-propanesultone (PS) could enhance membrane separation performance through

508 dipole-dipole interaction with TMC or H-bonding ([323] and Fig. 8c).

509
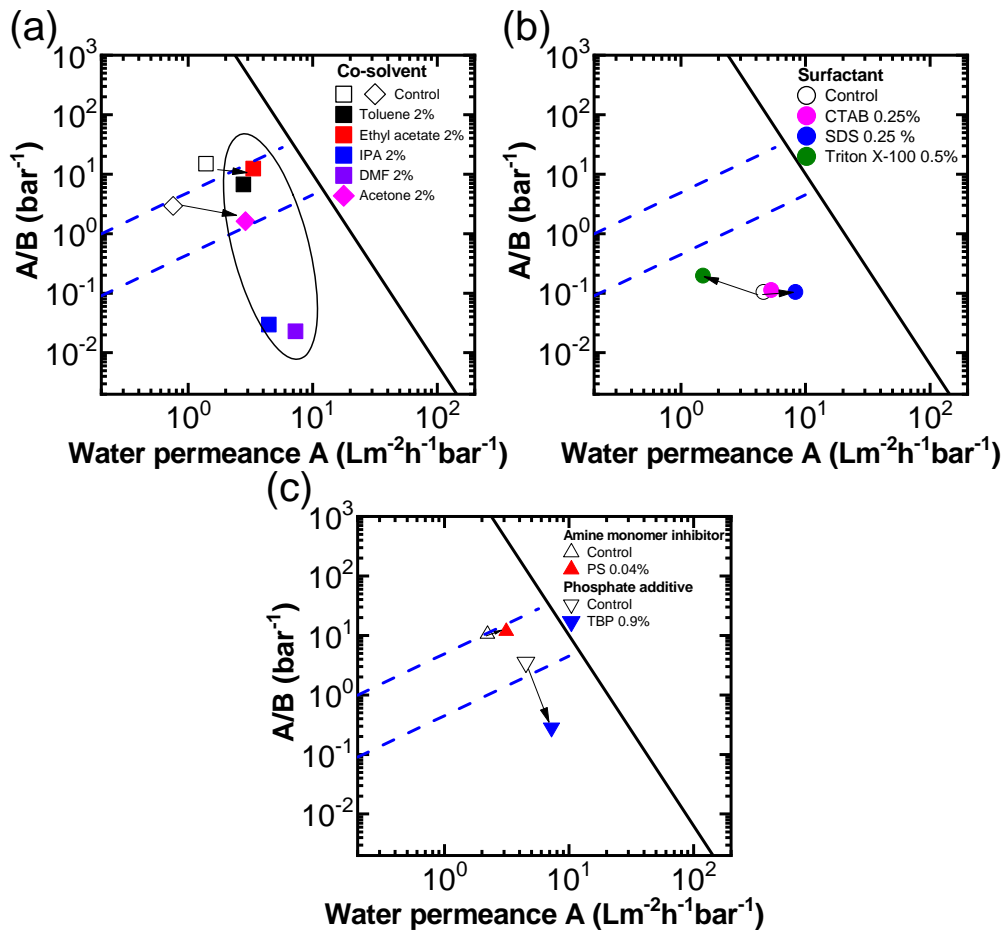
Fig. 8. Effect of additives in organic phase on membrane separation performance: (a) co-solvent; (b) surfactant; (c) amine monomer inhibitor. Blue dash lines correspond to the NaCl rejection of 99% and 90%, respectively, at a flux of 20 $Lm^{-2}h^{-1}$.

4.3. Substrate

The traditional wisdom believes that the substrate of a TFC membrane has little effect on its overall transport properties since the transport resistance to both water and solutes should be dominated by that of the polyamide layer according to the resistance-in-series model. Nevertheless, recent studies show increasing evidence that membrane substrate can play a critical role in the separation properties of TFC membranes [127, 131, 355, 395, 396].

Several studies have investigated the role of substrate pore size and porosity. For example, Ramon et al. [397] modeled the transport properties of TFC membranes and concluded that greater pore density is preferred to maximize the overall membrane permeance by reducing overall transport distance of water molecules. While Ramon's model implicitly assumes that the transport properties of the polyamide rejection film are not affected by the substrate properties, recent experimental studies revealed great influence of the substrate on the morphology and properties of the polyamide film [120, 355, 395]. Several experimental studies [61, 83, 355] show improved water permeance by using substrates with larger pores, although oversized pores (e.g., approximately 450 nm [131]) could lead to defects formation in the rejection layer with significantly decreased membrane selectivity. In a more recent study, Li et al. [355] reported that membrane permeance and selectivity both improved due to the increased pore number density, despite the reduction of substrate surface pore size

536   from 50.7 to 24.7 nm. Existing literature seems to suggest that there exists a

537   competing effect between pore size and pore number density on affecting membrane

538   performance. Therefore, more mechanistic studies are still needed to better understand

539   the underlining mechanisms and to guide the further optimization of substrates.

540

541

4.4. Membrane modification

4.4.1. *Surface coating*

Table 4 summaries the major types of membrane surface coatings and their impact on membrane performances, with the more detailed information provided in Table B4 in the Supporting Information. These coatings are generally adopted to achieve one or a combination of the following functions:

1. Enhancing anti-adhesion and anti-fouling properties. TFC PA membranes are known to have high fouling propensity due to (a) their ridge-and-valley surface roughness that traps colloids and macromolecules [398], (b) their relatively hydrophobic surfaces that attracts hydrophobic foulants through hydrophobic interactions [399], and (c) the abundance of negatively charged carboxylic groups on its surface that are prone to bridging with foulants through divalent metal ions such as Ca^{2+} as well as attachment by cationic foulants [271]. In commercial practice, a neutral hydrophilic coating such as polyvinyl alcohol (PVA) [222] is often applied to enhance membrane antifouling properties by neutralizing its surface charge, improving membrane surface hydrophilicity, and/or reducing surface roughness. Other hydrophilic coatings, such as polydopamine (PDA) [29, 400, 401], zwitterionic polymers [228], and tannic acid (TA)/Fe^{3+} complex [360], can also significantly enhance membrane anti-adhesion and anti-fouling properties.

563

2. Imparting antimicrobial and antibiofouling surface properties. Chemicals and materials with antimicrobial/biocidal effects, such as quaternary amines [402], chitosan [238], AgNPs [219, 403] and CuNPs [404], have widely reported in the literature. In addition, carbon-based materials such as carbon nanotubes [405] and graphene oxide [235, 406] sheets have also been applied to improve membrane anti-biofouling performance.

570

3. Improving the rejection of solutes. In recent years, the surface coating has also been applied to improve the rejection of specific solutes (e.g., small molecular weight organic micropollutants). For example, Guo et al. [220] reported that a hydrophilic PDA coating can significantly enhance the rejection of hydrophobic endocrine disrupting chemicals (EDCs) due to the reduced hydrophobic interaction between these solutes with the hydrophilic membrane surface. Zhou et al. [407] studied TiO_2 coating by atomic layer deposition (ALD) on TFC RO membrane of 10-100 cycles (one cycles could deposit approximately 0.1 nm thick TiO_2 layer). These coated membranes showed enhanced rejection of pharmaceutical compounds thanks to the enhanced the tightened size exclusion effect. Often, surface coatings can also lead to enhanced NaCl rejection (e.g., PVA [7] and PDA [408]), though a relatively thick and loose coating can

583   potentially result in severe cake enhanced concentration polarization [409] and

584   thus reduced solute rejection.

585

586 4. Increasing chlorine resistance. Anti-chlorine coatings are often studied, such as

587   spin coating graphene oxide (GO) layers onto the TFC membrane surface [235,

588   406]. Some other examples include PDA-grafted-PEI [410] and terpolymer [411]

589   coatings.

590

591 Despite their various functions, Fig. 9 shows that surface coating generally reduces

592 the membrane permeance due to the additional membrane hydraulic resistance of the

593 coating layer. On the other hand, a well-designed (e.g., thin and highly-selective)

594 coating can significantly enhance the *A/B* value of the coated membrane.
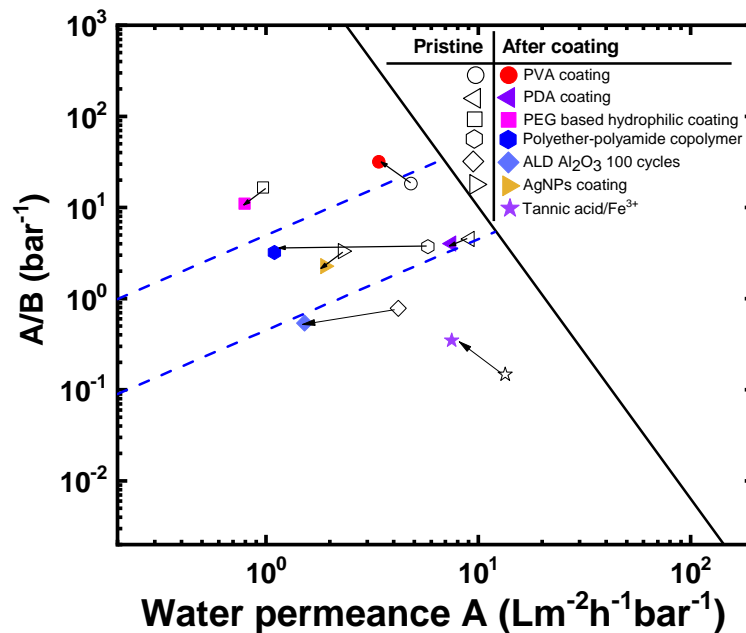
595

596   Fig. 9. Effect of surface coating on membrane separation performance. Blue dash lines
597   correspond to the NaCl rejection of 99% and 90%, respectively, at a flux of 20 Lm^{-2}h^{-1}.

598

599   Table 4. Membrane surface treatment

Surface treatment	Purpose	Examples	Recipe and methods	Membrane performance
Surface coating	Enhancing anti-adhesion and anti-fouling properties	Polyvinyl alcohol (PVA)	25% in (IPA/water of 3:7), dried at 130 °C [222]	Surface charge, antifouling, and hydrophilicity ↑; water permeance ↓, NaCl rejection ↑;
			0.1 wt.% cross-linked by succinic acid (1:0.27% w/w) [231]	Surface roughness ↓; Hydrophilicity and antifouling properties ↑; water permeance ↓, NaCl rejection ↑
			GA cross-linking [239]	Hydrophilicity and antifouling properties ↑; water permeance ↓, NaCl rejection ↑
		Polycations:PEI [271]	0-2000 mg/L	Water permeance ↓, NaCl ↑, hydrophilicity and antifouling properties against cationic foulants ↑
		Polyelectrolytes *via* layer-by-layer assembly [224]	Poly(sodium 4-styrenesulfonate) and poly(allylamine hydrochloride); 0, 6, 12 layers (6 optimum)	Antifouling properties ↑; roughness ↓; water permeance ↓, NaCl rejection ↑
		Polydopamine (PDA)	2 g/L in Tris-HCl buffer for 1 or 42 h [400]	Hydrophilicity ↑; water permeance ↓, NaCl rejection unchanged

			0.1-8 g/L and coating time from 0-120 mins [29]	Hydrophilicity and antifouling properties ↑; water permeance and NaCl rejection slightly ↓
			PDA/zwitterionic polymer [228]	Hydrophilicity, surface charge and antimicrobial properties ↑; transport properties no major change
		Polyamideoamine (PAMAM) dendrimers and PMMA-PEG [234]	Coating time 30s	Hydrophilicity ↑ water permeance ↓, NaCl rejection slightly ↓
		Polyether-poly amide copolymer [229]	1 wt.%, dip-coating and dried at 60 °C	Hydrophilicity and antifouling ↑ roughness ↓; water permeance ↓, NaCl rejection ↑
	Imparting antimicrobial and antifouling surface properties	Silver nanoparticles (AgNPs)	AgNO$_3$/NaBH$_4$ 1, 2 and 5 mM [219]	Water permeance ↓, NaCl rejection ↑; hydrophilicity and antimicrobial properties ↑
			Arc plasma deposition 0-100 pulse shots [233]	Hydrophilicity and antimicrobial ↑ water permeance ↑, NaCl rejection ↓
			PDA *in situ* reduction; 2 g/L PDA with 4 g/L AgNO$_3$ [221, 236]	Hydrophilicity and antimicrobial properties ↑; water permeance ↓, NaCl rejection ↑
			AgNO$_3$/NaBH$_4$ +zwitterionic	Hydrophilicity and antimicrobial

			polymer [227]	properties ↑; water permeance ↓, NaCl rejection ↑
		CuNPs	Carboxylated chitosan (CCTS) /CuCl$_2$/GA [238]	Hydrophilicity and anti(bio)fouling properties ↑ water permeance ↓, NaCl rejection ↑
			CuSO$_4$/NaBH$_4$ (50 mM) [404]	Water permeance slightly ↑ and NaCl rejection slightly ↓
			PEI-CuNPs-PAA; spray or spin coating for 4-16 bilayers [230]	Antimicrobial properties ↑; water permeance ↓, NaCl rejection ↑
	Improving rejection of solutes	Polydopamine (PDA)	2 g/L PDA for 0.5-4 hr [220]	Hydrophilicity and antifouling properties ↑; water permeance and NaCl rejection unchanged, hydrophobic micropollutants rejection ↑
		Tannic acid-Fe^{3+}	TA: 2.4 mM with TA-Fe molar ratio 1:0.5-1:6 [360]	Water permeance ↓,; NaCl, neutral and hydrophobic trace organics rejection ↑
		Atomic layer deposition (ALD)	Al$_2$O$_3$ [232] or TiO$_2$ [407] 10-100 cycles	Hydrophilicity and antifouling ↑ roughness ↓; water permeance ↓, NaCl and pharmaceutical solutes rejection ↑
	Enhancing chlorine resistance	Graphene oxide (GO) [235, 406]	Spin coating; 0.075 g/L; 1-5 layers	Hydrophilicity and chlorine resistance ↑; water permeance ↓, NaCl rejection ↑
Other surface	*Ex situ*	PEG-NH$_2$	0.1 wt.% EDC	Water permeance ↓,

modification methods	membrane surface bonding (modifying the fabricated or commercial TFC membrane) for enhancing membrane hydrophilicty	grafting initiated by 1-Ethy-3-(3-di methyl amidopropyl) carbodiimide (EDC) [244]	solution at 4 ℃ for 3 h; then immersed in PEG-NH$_2$ solution at 4 ℃ for 24 h	hydrophilicity and antifouling properties ↑ with nearly constant NaCl rejection
		Polyvinylamine (PVAm) grafting initiated by EDC/ N-hydroxysuc cinimide (NHS) [265]	0.05-0.25 w/v% PVAm aqueous solutions for 4 h	Surface roughness ↓, antifouling , water permeance ↓ with no change of NaCl rejection and antifouling properties ↑
		Imidazolidinyl urea (IU) [266]	NHS/EDC/ethylene diamine (EDA) assisted grafting	Water permeance ↓, antibiofouling and chlorine resistance↑ with nearly constant NaCl rejection
		Jeffamine grafting initiated by EDC/NHS [30]	1-15% (2% optimal)	Hydrophilicity and antifouling properties ↑` water permeance and NaCl rejection ↓
		Acrylic acid grafting initiated by redox (K$_2$S$_2$O$_8$) [242]	Acrylic acid (1-50 g/L); -NH- groups in polyamide was activated	Hydrophilicity and antifouling ↑; Surface roughness ↓; water permeance and NaCl rejection ↑
	In situ surface bonding (also called second IP reaction or quenching process) for enhancing membrane hydrophilicty and membrane	PEI [260]	3 wt.% by quenching	Hydrophilicity ↑; FO/PRO: J$_v$ and J$_s$ ↑; RO: water permeance ↑; NaCl rejection ↓; antifouling property ↑
		MPD, serinol (SRN), N-methyl-D-gl ucamine (GCMN) and	0.15 wt% solution of the corresponding amino alcohol (GCMN, APD, or	For MPD: Hydrophilicity ↓ ; water permeance and NaCl rejection no change; boron

		(±)-3-amino-1, 2-propanediol (APD) [31]	SRN	rejection ↑ ; For the others: Hydrophilicity ↑ and water permeance ↑ ; NaCl and boron rejection ↓ (For APD NaCl rejection ↑)
		Methanol, ethanol, isopropanol, ethylene glycol; ammonium hydroxide and MPD [264]	Contacting time 60s and 50 ℃	Water permeance ↑and NaCl rejection ↓ except quenching MPD (unchanged membrane performance)
Post-treatment	Study degradation mechanisms and membrane separation performance	Chlorination (NaClO)	10-100 ppm; pH 4.7 to 10; immersion time 0-54 h [208]	pH < 7: water permeance and NaCl rejection ↓ pH > 7: water permeance and NaCl rejection ↗↘
			10, 100 and 1000 ppm [207]	pH <= 7: water permeance ↓; NaCl and boron rejection ↗↘ pH > 7: water permeance ↑; NaCl rejection ↗↘; boron rejection ↓
			300-3000 ppm [27]	Water permeance and NaCl rejection slightly ↑; water permeance greatly ↑; NaCl rejection ↓ w/ NaOH immersion
			1250 mg/L; 30-60 mins coating time; pH=11[212]	Hydrophilicity and water permeance ↑and NaCl rejection ↓; surface roughness ↓
	Improving membrane performance	Acid treatment	HCl, nitric acid; sulfuric acid, phosphoric; concentration varied from 1-10	Membrane water permeance ↑

			wt.% [206]	
			5 wt.% sulfamic acid; 0.01 wt.% Na_2CO_3 and 0.025 wt.% $NaHSO_3$ [215]	
			5 wt.% hydrofluoric acid (HF); 1 wt.% fluosilicic acid (FSA) [210]	
		Alkali treatment	pH=9 and 13 using NaOH [260]	Membrane water permeance ↑and NaCl rejection ↓
		Solvents treatment	Alcohol: IPA, glycerol, ethanol, benzyl alcohol [209, 211, 361]	Membrane water permeance ↑ and NaCl rejection ↓
			Organic solvent: DMF [361]	

600

601 **4.4.2.** *Other Surface modifications*

602 In addition to surface coating, other membrane surface modification techniques can

603 also be applied to enhance membrane hydrophilicity and antifouling properties.

604 Surface modification can be categorized into two groups: e*x situ* and *in situ* surface

605 modification. Their differences and the corresponding examples are presented in

606 Table 4, and more detailed information can be found in Table B4 in the Supporting

607 Information.

608

609 *Ex situ* surface modification is a "post-modifying" approach to chemically graft or

610 modify a TFC membrane that has been fabricated beforehand, such as modifying a

611 commercial TFC membrane. Since the membrane surface does not contain highly

612 reactive functional groups such as acryl chloride groups, *ex situ* modification often

613 requires the activation of groups in polyamide chains (e.g., -NH- or COOH) to further

614 react with other chemicals. For example, Xu et al. grafted imidazolidinyl urea (IU)

615 [266] on the surface of a TFC RO membrane with the aid of *N*-hydroxysuccinimide

616 (NHS)/*N*-(3-dimethylaminopropyl)-*N*-ethylcarbodiimide hydrochloride (EDC) to

617 activate the carboxyl groups. The modified membrane showed enhanced

618 hydrophilicity, excellent antibiofouling, and chlorine resistance properties. However,

619 the modified membrane had decreased water permeance, probably due to the strong

620 H-bonding among these IU on the membrane surface [266] (Fig. 10a).

621

622   *In situ* surface modification, also called second interfacial polymerization or

623   quenching [260, 264], is achieved by directly immersing a newly-fabricated TFC

624   membrane (without removing or hydrolyzing TMC) into a grafting solution with

625   desirable functional groups. In this case, the unreacted acryl chloride groups in the

626   TMC react with the grafting chemicals to endow the modified membrane with the

627   desired surface functional groups. Werber et al. [264] explored *in situ* surface

628   modification by immersing (i.e., quenching) a freshly-fabricated TFC membrane into

629   a set of alcohols or amine containing chemicals solutions, such as ethanol, ammonium

630   hydroxide, and MPD. Membrane quenched by the alcohols showed a significantly

631   enhanced water permeance and slightly decreased salt rejection compared to the

632   control [264] (Fig. 10b). This can be due to the enhanced membrane hydrophilicity

633   due to the grafted hydrophilic groups. In addition, membrane salt rejection decreased,

634   probably due to the formation of polyester bonds when quenching in alcohol solutions.

635   In contrast, quenching a 10 wt.% MPD solution could lead to a denser PA layer with

636   reduced water permeance (Fig. 10b).

637

638
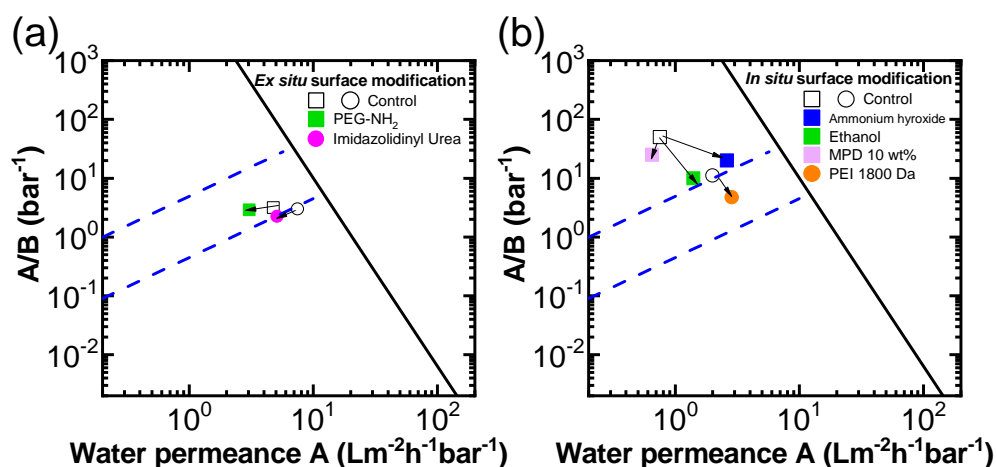
Fig. 10. Effect of surface modifications on membrane separation performance: (a) *ex situ* surface modification; (b) *in situ* surface modification. Blue dash lines correspond to the NaCl rejection of 99% and 90%, respectively, at a flux of 20 $Lm^{-2}h^{-1}$.

4.4.3. *Post-treatment*

Membrane post-treatment can significantly affect membrane separation performance. Table 4 summaries the major types of post-treatment, such as chlorine, acid or alkali and solvents (e.g., alcohols or organic solvents) treatment. This section mainly discusses the impact of post-treatment conditions on membrane separation performance, and more detailed information can be found in Table B4.

Chlorination, often with the addition of NaClO, can significantly alter the polyamide structure, thus leading to significant changes in membrane separation performance. Important chlorination mechanisms include intermolecular rearrangement [412], aromatic ring chlorination (both direct ring attach and Orton arrangement [413, 414]),

655   and direct formation of *N*-chloramide [415-417]. Many researchers [27, 207, 208, 356,

656   416] have studied membrane chlorination as well as its impact on the corresponding

657   membrane physiochemical properties. For example, Do et al. [207] explored the effect

658   of pH on the chlorination of a TFC NF membrane performance. When pH is below 7,

659   the exposure to chlorine led to a more hydrophobic membrane surface, with reduced

660   water permeance and enhanced salt rejection. This can be due to the chlorination

661   induced additional bonds *via* azo compounds on membrane surface [356]. However,

662   when pH is $\geqslant$7 [27, 207], membrane chlorination could induce hydrolysis of the

663   crosslinked polyamide which enhances membrane hydrophilicity as well as its water

664   permeance. A combination of high chlorine exposure and high pH often results in

665   severely impaired NaCl rejection [27, 207, 208, 356]. Nevertheless, Do et al. [207]

666   showed the possibility of achieving simultaneously enhanced permeance and

667   selectivity by controlling the chlorine-induced hydrolysis under moderate chlorine

668   concentrations (Fig. 11a).

669

670   In addition to chlorine treatment, acid or alkali are often used as conventional

671   post-treatment approaches to improve membrane separation performance [206, 210,

672   211, 215, 260]. Shen et al. [260] reported a permeance and salt rejection decline of an

673   acid (pH=1) or alkali (pH=13) post-treated TFC membrane (Fig. 11b), which can be

674   due to the hydrolysis of the polyamide rejection layer. Likewise, Kulkarni et al. used

675   hydrofluoric acid (HF) to post-treat the commercial TFC RO membrane. When HF

676   concentration was below 5 wt.%, the modified membrane showed both enhance water

677   permeance and salt rejection (Fig. 11b), thanks to the enhanced membrane

678   hydrophilicity. However, overdosage could severely deteriorate polyamide integrity

679   with decreased NaCl rejection. Solvent-based post-treatments, such as alcohols [209,

680   211, 361] or other organic solvents (e.g., DMF), can also result in membranes with

681   enhanced water permeance and nearly unchanged or slightly decreased NaCl rejection,

682   due to their swelling effect (Fig. 11c). In some cases, a solvent could also partially

683   dissolve the substrate, leading to significant changes in membrane performance [361].

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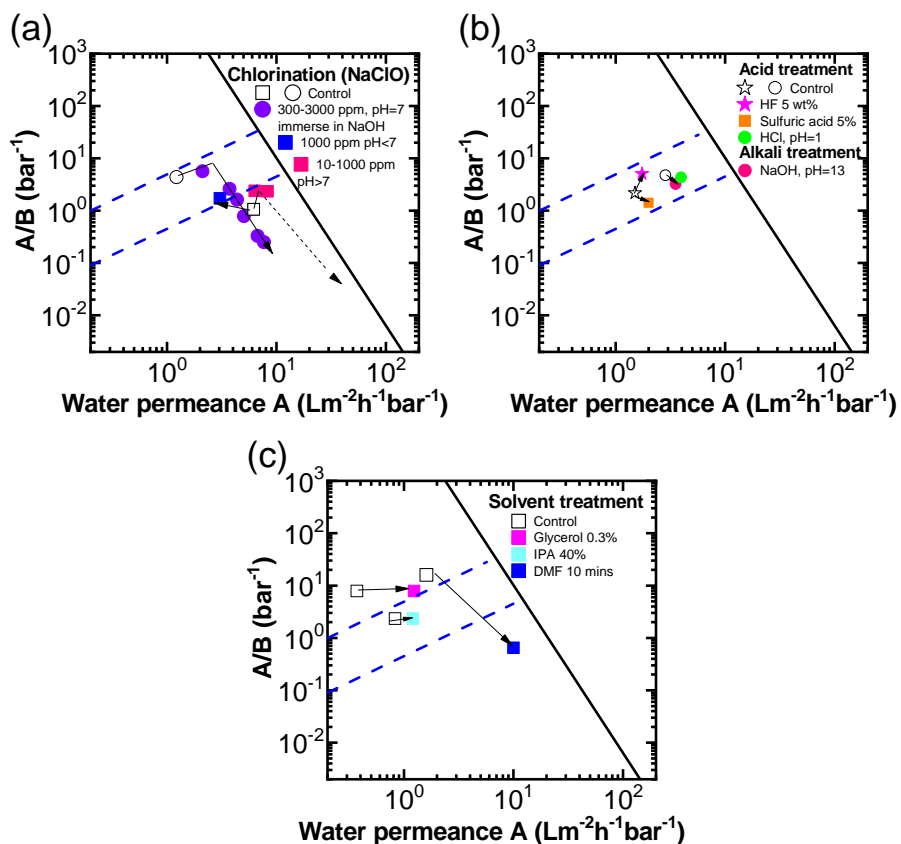
685

686   Fig. 11. Effect of post-treatment on membrane separation performance: (a) membrane
687   chlorination. The dashed black line represents the prediction of membrane permeance and
688   selectivity of a highly degraded TFC membrane [207]; (b) acid and alkali treatment; (c)
689   solvent treatment. Blue dash lines correspond to the NaCl rejection of 99% and 90%,
690   respectively, at a flux of 20 $Lm^{-2}h^{-1}$.

691

692

693

5. Implications

694

695   The current study establishes an upper bound relationship for water permeance and

696   water to NaCl selectivity for TFC polyamide membranes, which provides a useful

697   tool for benchmarking future membrane development. While the effect of

698   concentration polarization is not included in Fig. 2 due to general limitation of

699   literature data, additional analysis in Fig. A1 (Supporting Information Appendix A)

700   shows that both A and A/B could be underestimated by assuming f_{cp} = 1. Therefore,

701   future studies need to explicitly include f_{cp} (e.g., Equation 13) in their calculation of

702   membrane separation properties.

703

704   In developing strategies to achieve enhanced membrane performance, one needs to

705   pay attention to the crosslinking degree of the polyamide rejection layer, its effective

706   surface area for filtration, and the free volume and nanovoids contained in the

707   rejection layer. Increased rejection can be generally obtained with improved

708   crosslinking degree, e.g., with the use of H^+ acceptor such as TEA. Increasing

709   membrane surface roughness can lead to improved water permeance due to the

710   creation of additional effective filtration area. In a recent study, Ma et al. [19, 20]

711   reported the use of $NaHCO_3$ for the simultaneous enhancement of NaCl rejection and

712   water permeability, where HCO_3^- served the dual function of H^+ acceptor and

713   roughness promoter. Another effective strategy to break the permselectivity upper

714   bound is to increase the free volume within the polyamide network [418-421]. For

715 instance, the addition of DMSO [330] into the aqueous phase during interfacial

716 polymerization could effectively increase the aggregate pores size of polyamide

717 network due to the enhanced miscibility of the aqueous and the organic phase. The

718 optimum loading of DMSO could result in significantly enhanced water flux without

719 severe impact on the crosslinking degree. In parallel to enhancing free volume, the

720 incorporation of porous nanomaterials in the polyamide rejection layer can

721 significantly enhance membrane separation performance thanks to the additional

722 selective water pathways in these nanofillers [422, 423]. A recent work further shows

723 the creation of additional selective nanochannels along the surface of hydrophilic

724 nanofillers [358]. Several studies have also demonstrated the effective use of an

725 interlayer (e.g., polydopamine [347], tannic acid-Fe^{3+} complex [349], or carbon

726 nanotubes [424, 425]) to simultaneously enhance flux and rejection, thanks to the

727 optimized membrane structure and reduced defects.

728

729 In the membrane community, there have been overwhelming interests in

730 "next-generation" desalination membranes prepared from carbon nanotube,

731 nanoporous graphene, graphene oxide and aquaporin. Nevertheless, it is worthwhile

732 to highlight the general inadequacy of their NaCl rejection [3]. Ritt et al. [426] further

733 reveal that, even under ideal conditions, the separation performance of 2-D

734 materials-based membranes still cannot surpass the performance of TFC desalination

735 membranes due to their inherent defects. The failure of "next-generation" desalination

736   membranes in addressing the permselectivity underpins the great value of the TFC

737   polyamide membranes for delivering product water of highly reliable quality.

738   Therefore, future developments on novel materials and membranes for desalination

739   need to benchmark directly against the state-of-the-art upper bound.

740

6. Conclusion

741

742 This study analyzed the separation properties of TFC polyamide membranes gathered

743 from more than 300 technical papers published in the last three decades. The analysis

744 showed a clear permeance-selectivity tradeoff between the membrane water

745 permeance (A) and water/NaCl selectivity (A/B) for polyamide-based desalination

746 membranes so that membranes with higher water permeance tend to have lower

747 water/NaCl selectivity. This study further reviews the effect of various synthesis

748 conditions (monomer types and concentration, additives, etc.) and modification

749 methods on these important separation properties. An upper bound was identified for

750 the state-of-the-art TFC polyamide membranes, which is given in the form of $A/B =$

751 $16000A^{-3.2}$ (with A/B in bar^{-1} and A in $Lm^{-2}h^{-1}bar^{-1}$). This upper bound relationship can

752 serve as a useful benchmark for evaluating future membrane development works.

753

757

Appendices

758

759 Appendix A. The effect of concentration polarization on membrane separation

760 performance; Appendix B. Tables of the major factors affecting the separation

761 performance of TFC

762

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