Mixed polyamide-based composite nanofiltration hollow fiber membranes with improved low-pressure water softening capability

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(a)

(b)

(c)

Fig. 1. Morphology of PES hollow fiber substrate: (a) cross-section at 50X; (b) enlarged cross-section at 200X; (c) inner surface at 50,000x.

(a) PEI only

(b) PEI/PIP

(c) PIP only

Fig. 2. Cross-sectional and surface morphologies of IP thin film formed with (a) PEI only; (b) mixed PEI/PIP and (c) PIP only as the aqueous phase IP monomer.

Fig. 3. AFM images of the fiber inner surface: (a) PEI only membrane; (b) PIP only membrane; (c) PEI/PIP membrane and (d) PES hollow fiber substrate.

Fig. 4. FTIR spectra of PES substrate and composite hollow fiber membranes made using single or mixed amine monomers.

Fig. 5. Interfacial reaction scheme between (a) PEI, (b) PIP and (c) TMC; (d) possible structure of resultant polyamide/polypiperazinamide network

Fig. 6. Zeta potential of PES substrate and composite hollow fiber membranes made using single or mixed amine monomers.

Fig. 7. Effect of PEI/PIP mixing ratio on composite hollow fiber membrane performance (total amine concentration = 0.25% (w/v), amine solution pH = 11; rejection obtained via 1000 ppm MgSO_4 feed solution with operating pressure of 2 bar).

Fig. 8. Effect of total amine concentration on composite hollow fiber membrane performance (PEI/PIP mixing ratio = 95/5 (w/w), amine solution pH = 11; rejection obtained via 1000 ppm MgSO_4 feed solution with operating pressure of 2 bar).

Fig. 9. Effect of amine solution pH on composite hollow fiber membrane performance (total amine concentration = 0.25% (w/v), PEI/PIP mixing ratio = 95/5 (w/w); rejection obtained via 1000 ppm MgSO_4 feed solution with operating pressure of 2 bar).

Fig. 10. Rejection of three types of composite hollow fiber membranes to different salts (total amine concentration = 0.25% (w/v), amine solution pH = 11 for all three membranes and PEI/PIP mixing ratio = 95/5 (w/w) for PEI/PIP membrane; rejections obtained using 1000 ppm single salt feed solutions under operating pressure of 2 bar).

Table 1. Isoelectric point, contact angle and tensile strength of PES substrate and composite hollow fiber membranes made using single or mixed amine monomers.

Membrane	Isoelectric point (pH)	Contact angle (deg)	Tensile modulus (MPa)	Stress at break (MPa)	Strain at break (%)
Support fiber	< 3.5	75.4	88.2	4.9	63.5
PEI only	7.4	39.6	95.7	5.6	60.2
PEI/PIP	6.7	42.2	107.3	5.8	55.9
PIP only	5.9	47.7	99.6	5.9	58.4

Table 2. Rejection of current composite hollow fiber membranes to various neutral solutes

Solute	Molecular weight (Da)	Stokes radius (nm)	Solute rejection* (%)		
			PEI only	PEI/PIP	PIP only
Glucose	180	0.359	79.1	84.7	72.7
Sucrose	342	0.462	87.0	89.5	98.5
Raffinose	504	0.538	89.8	91.0	98.8

* Total amine concentration = 0.25% (w/v), amine solution pH = 11 for all three membranes and PEI/PIP mixing ratio = 95/5 (w/w) for PEI/PIP membrane; the solute rejection was obtained using 200 ppm single solute feed solutions under operating pressure of 2 bar.

Table 3. Comparison of current composite hollow fibers to various commercially available low-pressure NF membranes

Membrane	PWP (L/m ² h bar)	Solute rejection (%)				Operating pressure (bar)	Solute concentration (ppm)	Ref.
		MgCl ₂	MgSO ₄	NaCl	Na ₂ SO ₄			
PEI only	15.3	95.1	79.9	44.6	50.4	2	1000	Current work
PEI/PIP	18.2	96.3	93.8	53.6	77.4			
PIP only	6.8	70.6	98.5	13.6	99.3			
Dow-Filmtec NF90	6.7	-	97	90	-	4.8	2000	[37]
Dow-Filmtec NF270	13.2	60	97	51	98	4.8	2000	[38]
GE-Osmonics HL	6.9	-	97	33	-	7.6	1000	[39]
Hydranautics ESNA1	7.5	89	-	93	-	5.2	500	[39]
Koch TFC-SR2	14.5	-	92	20	-	5	600	[40]
Nitto-Denko NTR-7450	10.9	16	48	53	92	10	1000	[36]
Toray UTC20	10.2	98	-	55	93	10	1500	[36]

Table 4. Characteristics of simulated hard water feed solutions

Parameter	Feed 1	Feed 2	Feed 3
TDS (ppm)	1000	3000	5000
Conductivity ($\mu\text{S}/\text{cm}$)	2040	5840	8910
Hardness (mg/l as CaCO_3)	451	691	1923
Mg^{2+} (ppm)	-	102	204
Ca^{2+} (ppm)	180	108	433
Na^+ (ppm)	197	870	1104
Cl^- (ppm)	623	1582	2515
SO_4^{2-} (ppm)	-	338	744

Table 5. Water softening performance of newly developed composite hollow fiber membranes at 2 bar operating pressure

Hard water	Membrane	Permeability (l/m ² h bar)	Hardness removal (%)	Conductivity removal (%)	Ionic rejection (%)				
					Mg ²⁺	Ca ²⁺	Na ⁺	Cl ⁻	SO ₄ ²⁻
Feed 1	PEI only	9.8	94.0	69.5	-	94.0	42.1	68.7	-
	PEI/PIP	13.4	97.4	71.0	-	97.4	41.0	70.0	-
	PIP only	6.4	86.7	42.8	-	86.7	-6.6	41.2	-
Feed 2	PEI only	8.7	82.1	34.4	83.3	80.2	19.0	28.8	80.3
	PEI/PIP	12.8	90.2	34.3	92.9	86.0	17.8	28.2	94.0
	PIP only	5.8	82.3	25.2	85.7	76.9	7.6	16.3	98.5
Feed 3	PEI only	7.1	75.6	32.6	77.8	73.8	6.7	29.0	75.3
	PEI/PIP	10.0	86.9	36.7	91.4	83.4	6.1	31.7	89.6
	PIP only	4.7	77.9	30.2	83.2	73.8	1.0	21.5	97.8
Morocco groundwater*	NF90	4.1	96	-	84	97	85	96	-
	NF270	9.2	40	-	45	40	14	43	-

*Ref. [44] at 10 bar operating pressure.