Dual layer composite nanofiltration hollow fiber membranes for low-pressure water softening

Setiawan, Laurentia; Shi, Lei; Wang, Rong

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Fig. 1. Cross-section morphology of the dual layer hollow fibers (a,b) before cross-linking; (c) after cross-linking.

Fig. 2. Outer/inner surface morphology of the dual layer hollow fibers at 100K magnification:
(a) outer PAI layer, (b) inner PES layer before cross-linking;
(c) outer PAI layer, (d) inner PES layer after cross-linking.

Fig. 3. Possible mechanism of chemical cross-linking between (a) PAI [37] and (b) PAAm;
(c) Cross-linked PAI-PAAm

Fig. 4. FTIR-ATR spectra of the dual layer hollow fiber membrane before (top) and after (bottom) cross-linking.

Fig. 5. Zeta-potential on the outer layer of original and cross-linking treated dual layer hollow fibers (—■— before cross-linking; —▲— after cross-linking)

Fig. 6. Static chlorine resistance test of cross-linking treated dual layer hollow fibers
(—▲— salt water permeability (SWP); —■— Rejection)

Table 1

Dope composition and spinning conditions

Outer dope composition (wt.%)	PAI/LiCl/PEG400/H ₂ O/NMP 16/3/3/3/75
Inner dope composition (wt.%)	PES/LiCl/PEG400/NMP 15/5/10/70
Outer dope flow rate (g/min)	2
Inner dope flow rate (g/min)	6
Coagulant	water
Coagulant temperature (°C)	23
Bore fluid composition	water
Bore fluid flow rate (mL/min)	8
Take up speed (m/min)	5.68
Air gap (cm)	0.5
Ambient temperature (°C)	23
Room humidity (%)	80

Table 2

Conditions of the cross-linking reactions

Code*	Molecular weight (kDa)	Reaction time, min
PA15-1	15	10
PA15-4	15	40
PA15-6	15	60
PA17-4	17	40
PA65-4	65	40

* PA-XX-Y: XX stands for molecular weight of PAAm used and Y stands for the reaction time; for example PA15-4 means PAAm of 15 kDa for 40 minutes

Table 3
Comparison of the membranes contact angles

Membrane	Contact angle (°)	Standard deviation
PES single layer	70.3	0.51
PAI single layer	61.5	1.80
PAI-PES dual layer before cross-linking	61.4	1.35
PAI-PES dual layer after cross-linking	52.2	1.26

Table 4

Comparison of the membrane mechanical strength

Membrane	Young's modulus (MPa)	Tensile stress (MPa)	Tensile strain (%)
PES single layer	69.7	3.3	62.5
PAI single layer	128	6.1	34.4
PAI-PES dual layer before cross-linking	96	3.7	27.2
PAI-PES dual layer after cross-linking	98	3.7	25.4

Table 5

Effect of PAAm molecular weight and cross-linking reaction time on membrane properties*

Code	Permeation of salt solution [#] (L/m ² .h.bar)	Rejection of MgCl ₂ (%)
PA15-1	25.3	66
PA15-4	15.9	96
PA15-6	1.4	98
PA17-4	13.6	95
PA65-4	8.3	96

* Cross-linking condition: 1 wt.% PAAm solution at temperature of 70 °C

[#] 500 ppm MgCl₂ solution as the feed at cross-flow pressure and flowrate of 1 bar and 120 mL/min, respectively

Table 6

Pure and mixed salt solution testing results of dual layer NF hollow fiber membranes*

Composition of feed solution	TDS (ppm)	SWP (L/m ² .h.bar)	Mg ²⁺ rejection (%)	Ca ²⁺ rejection (%)	Na ⁺ rejection (%)
1000 ppm MgCl ₂	1000	15.6 ± 1.1	95.7 ± 1.6	-	-
500 ppm MgCl ₂ + 900 ppm CaCl ₂ + 1600 ppm NaCl	3000	15.8 ± 0.4	94.2 ± 1.5	92.3 ± 1.6	11.7 ± 0.9
833 ppm MgCl ₂ + 1500 ppm CaCl ₂ + 2667 ppm NaCl	5000	12.9 ± 0.7	89.1 ± 2.0	84.0 ± 2.4	-10.6 ± 0.8

* PA15-4 membranes were used. Experiments were performed at constant operating pressure of 2 bar, cross-flow rate of 800 mL/min (velocity of 0.5 m/s).

Table 7

Comparison of the performance of various NF membranes for low-pressure water softening

Membranes	Salt water flux (L/m ² .h)	Solute rejection (%)			Operating pressure (bar)	Solute composition (ppm)	Ref.
		Mg ²⁺	Ca ²⁺	Na ⁺			
Dual layer NF hollow fiber	31.2 ^a	95.7	-	-	2	1000 MgCl ₂	Current work
	31.6 ^b	94.2	92.3	11.7	2	500 MgCl ₂ + 900 CaCl ₂ + 1600 NaCl	
	25.8 ^b	89.1	84.0	-10.6	2	833 MgCl ₂ + 1500 CaCl ₂ + 2667 NaCl	
		MgCl ₂ /CaCl ₂	NaCl	Na ₂ SO ₄			
NF-40 ^c	43	70	45	95	20	2000	[41]
NF-270 ^d	53	-	50	98	4.8	2000	[42]
NTR-7450 ^c	92	13	51	92	10	5000	[41]
SU-600 ^c	28	-	55	-	3.5	500	[43]
UTC-20 ^a	97	98	55	93	10	1500	[44]

^a Permeation flux were obtained using MgCl₂ feed solution.^b Permeation flux were obtained using mixed salt feed solution.^c Permeation flux were obtained using NaCl feed solution.^d Permeation flux were obtained using Na₂SO₄ feed solution