Novel method for incorporating hydrophobic silica nanoparticles on polyetherimide hollow fiber membranes for CO₂ absorption in gas-liquid membrane contactor

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Table 1. Spinning parameters for fabricating PEI hollow fiber substrates

Parameters	PEI
Dope composition (wt.%)	PEI/LiCl/NMP (14/3/83)
Dope flow rate (g/min)	3.78
Outer channel solvent	PEI A: - PEI B: NMP
Solvent flow rate (ml/min)	PEI A: - PEI B: 1
Bore fluid (wt.%)	DI water/NMP (20/80)
Bore fluid flow rate (ml/min)	3
Length of air gap (cm)	5
Take up speed	Free fall
External coagulant	Water
Spinning temperature	25°C

Table 2. Characteristics of the membrane contactor module

Module ID (mm)	6.4
Module length (cm)	25
Fiber OD (mm)	1.0
Fiber ID(mm)	0.7
Effective fiber length (cm)	16
Effective contact area (cm ²)	20
Number of fibers	4

Table 3. Characteristics of the PEI hollow fiber substrates

Properties	PEI A	PEI B
Fiber OD (mm)	1042	966
Fiber ID (mm)	736	737
Fiber wall thickness (μm)	153	115
Surface pore size (μm)	0.03	0.09
Overall porosity (%)	81.2	71.2
PWP ($\text{L}/\text{m}^2 \cdot \text{h} \cdot \text{bar}$)	662	665

Table 4. Surface roughness parameters of the PEI membranes before and after fSiO₂ incorporation

Membrane code	Roughness	
	R _a (nm)	R _q (nm)
PEI A	35.80	50.58
PEI B	79.12	99.87
PEI A-fSiO ₂	64.99	79.91
PEI B-fSiO ₂	72.92	92.08

Table 5. Dynamic contact angles of PEI membrane before and after fSiO₂ incorporation

Membrane code	Contact direction	First cycle (°)	Second cycle (°)	Third cycle (°)	Contact angle hysteresis (°)
PEI A	Advancing	86.2 ± 1.1	70.7 ± 2.5	70.4 ± 2.7	23.2 ± 4.1
	Receding	47.6 ± 1.5	47.5 ± 1.6	47.6 ± 1.7	
PEI B	Advancing	109.9 ± 1.4	60.8 ± 1.7	58.3 ± 3.1	12.7 ± 2.5
	Receding	48.0 ± 1.0	48.1 ± 0.8	48.2 ± 0.9	
PEI A-fSiO ₂	Advancing	121.0 ± 2.0	121.0 ± 2.1	120.8 ± 2.2	22.3 ± 3.3
	Receding	98.2 ± 1.0	98.7 ± 1.1	98.7 ± 1.5	
PEI B-fSiO ₂	Advancing	123.2 ± 1.3	123.2 ± 0.7	123.2 ± 0.7	15.9 ± 4.5
	Receding	106.1 ± 3.9	107.2 ± 3.9	107.1 ± 3.8	
PVDF	Advancing	92.3 ± 3.7	89.8 ± 5.1	89.2 ± 5.0	32.6 ± 7.4
	Receding	57.6 ± 2.6	57.2 ± 2.3	57.2 ± 2.4	

Table 6. Mechanical properties of PEI membrane before and after fSiO₂ incorporation

Membrane code	Young's modulus (MPa)	Stress at break (MPa)	Strain at break (%)
PEI A	116.9 ± 7.8	5.4 ± 0.1	21.1 ± 2.1
PEI B	233.7 ± 14.3	10.4 ± 0.2	49.4 ± 3.6
PEI A-fSiO ₂	125.6 ± 5.7	5.5 ± 0.1	11.3 ± 0.4
PEI B-fSiO ₂	274.9 ± 16.2	10.6 ± 0.1	19.2 ± 1.0

Table 7. Overall comparison of CO₂ flux of various membranes used in membrane contactors

Membrane	ID/OD (mm)	Pore size (μm)	Porosity (%)	CO ₂ flux ^a (mol / m ² s ⁻¹)	Shell side	Highlight	Manufacturer	Ref.
#1 PEI	0.7/1	0.03 ^b	81	1.38 x 10 ⁻³	Water	Composite/NPs incorporated	In-house made	Current work
#2 PEI	0.7/0.9	0.09 ^b	71	2.65 x 10 ⁻³	Water	Novel spinning technique Composite/NPs incorporated	In-house made	Current work
#3 PEI	0.8/1.1	0.04 ^b	-	8.68 x 10 ⁻⁴	Water	Composite/NPs incorporated	In-house made	[15]
#4 PEI	0.3/0.6	0.05	77	1.2 x 10 ⁻³	CO ₂	Pristine PEI	In-house made	[29]
#5 PEI	0.35/0.6	0.07	81	2.3 x 10 ⁻³	CO ₂	SMMs modified		
#6 PVDF	0.3/0.4	MWCO 290 kDa	76	1.0 x 10 ⁻³	Water	Phosphorous acid as the additive	In-house made	[7]
#7 PVDF	-/-	-	-	8.0 x 10 ⁻⁴	Water	Commercial	Tianjin Motian Membrane	
#8 PVDF	0.3/0.5	MWCO 40 kDa	-	8.0 x 10 ⁻⁴	Water	Pristine PVDF	In-house made	[30]
#9 PVDF	0.65/1	0.2	75	6.5 x 10 ⁻⁴ (extrapolated)	CO ₂	Commercial	Memcor Australia	[31]
#10 PAI	1.1/1.4	MWCO 20 kDa ^b	72	8.5 x 10 ⁻⁴	Water	APTMS & FS10 modified	In-house made	[32]
#11 PSf	0.5/1	0.01	72	7.5 x 10 ⁻⁴	CO ₂	Glycerol as the additive	In-house made	[4]
#12 PP	0.22/0.23	0.04	40	1.4 x 10 ⁻³	CO ₂	Commercial	Celgard Inc.	[28]

a. Achieved at liquid velocity of 0.3m/s

b. Pore size/porosity of the polymeric membrane substrate

Figure 1. Chemical structure of (a) Ultem[®]1000; (b) APTMS; (c) TEOS; (d) PFTS

Figure 2. Reaction route of forming the PEI-fSiO₂ organic-inorganic composite membranes

Figure 3. Mechanism of forming the PEI-fSiO₂ organic-inorganic composite membranes

Figure 4. SEM images of the surface and cross-section of the PEI A and PEI B substrates. Surface: left 10,000x, middle 30,000x and cross section: right 600x.

Figure 5. ATR-FTIR spectra of the PEI-SiO₂ and PEI-fSiO₂ composite hollow fiber membranes

Figure 6. SEM images of the PEI A and PEI B membranes after APTMS treatment (surface, enlarged at 30,000x) and fSiO₂ NPs incorporation (surface, enlarged at 10,000x and 30,000x & cross section, enlarged at 5000x and 20,000x)

Figure 7. Three-dimensional topography AFM images of (a1) PEI A; (b1) PEI B; (a2) PEI A-fSiO₂; (b2) PEI B-fSiO₂ membranes

Figure 8. CO₂ flux using pure water as the absorbent
(atmospheric pressure, absorbent temperature: 25 °C, gas velocity 0.12 m/s)

Figure 9. CO₂ flux using 2M taurinate sodium as the absorbent (atmospheric pressure, absorbent temperature: 25 °C, **gas velocity 0.8 m/s**)

Figure 10. Long term CO₂ flux using 2M taurinate sodium as the absorbent (atmospheric pressure, absorbent temperature: 25 °C, **gas velocity 0.8 m/s**)