Ultra-dehydration of reactive epichlorohydrin-containing organic mixture using a defect-free thin carbon molecular sieve composite membrane

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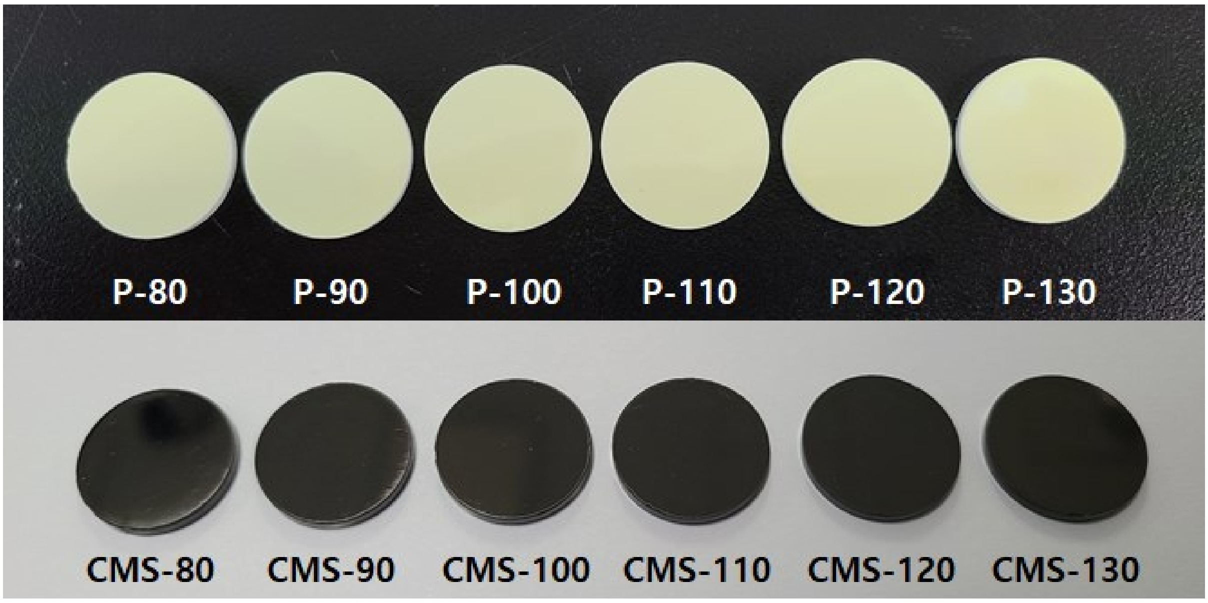
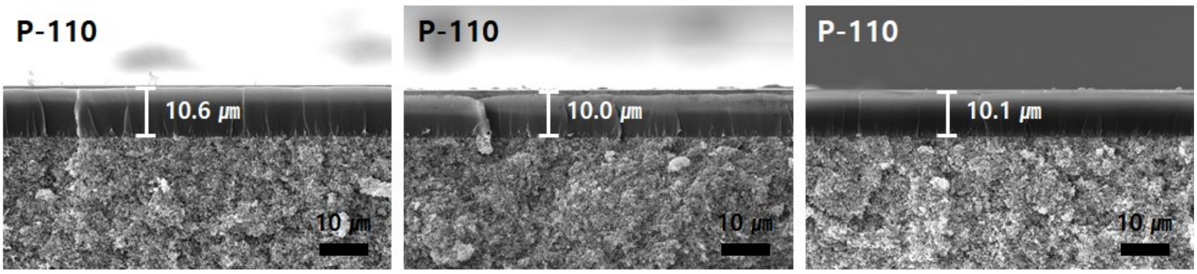
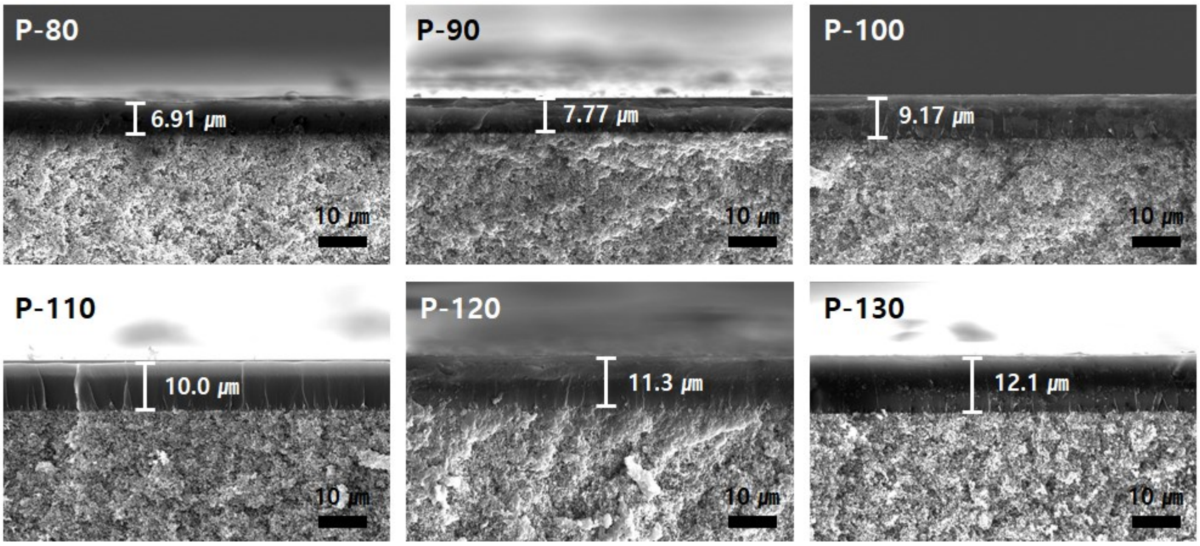


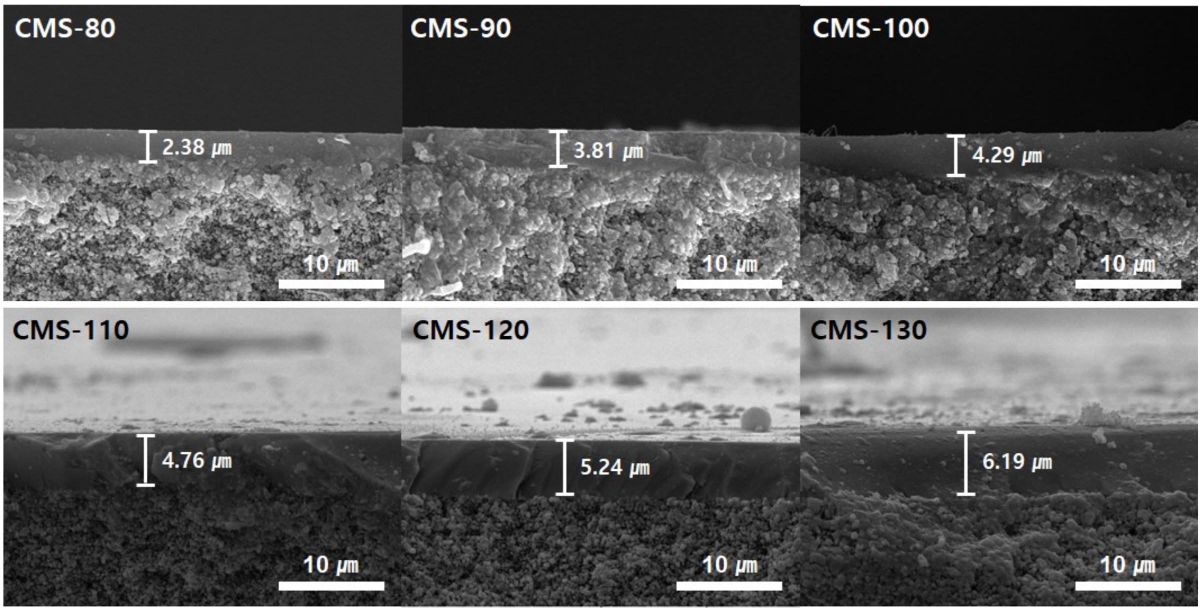
S1 Images of polymeric (upper) and CMS (bottom) composite membranes deposited on alumina discs. The number represents the drop-amount (μL) of polymer solution. P and CMS are abbreviations for polymer and CMS, respectively



S2 SEM images of the polymer layer coated on alumina disc with the polymer solution of 110 μL (standard deviation: ±0.26 μm)



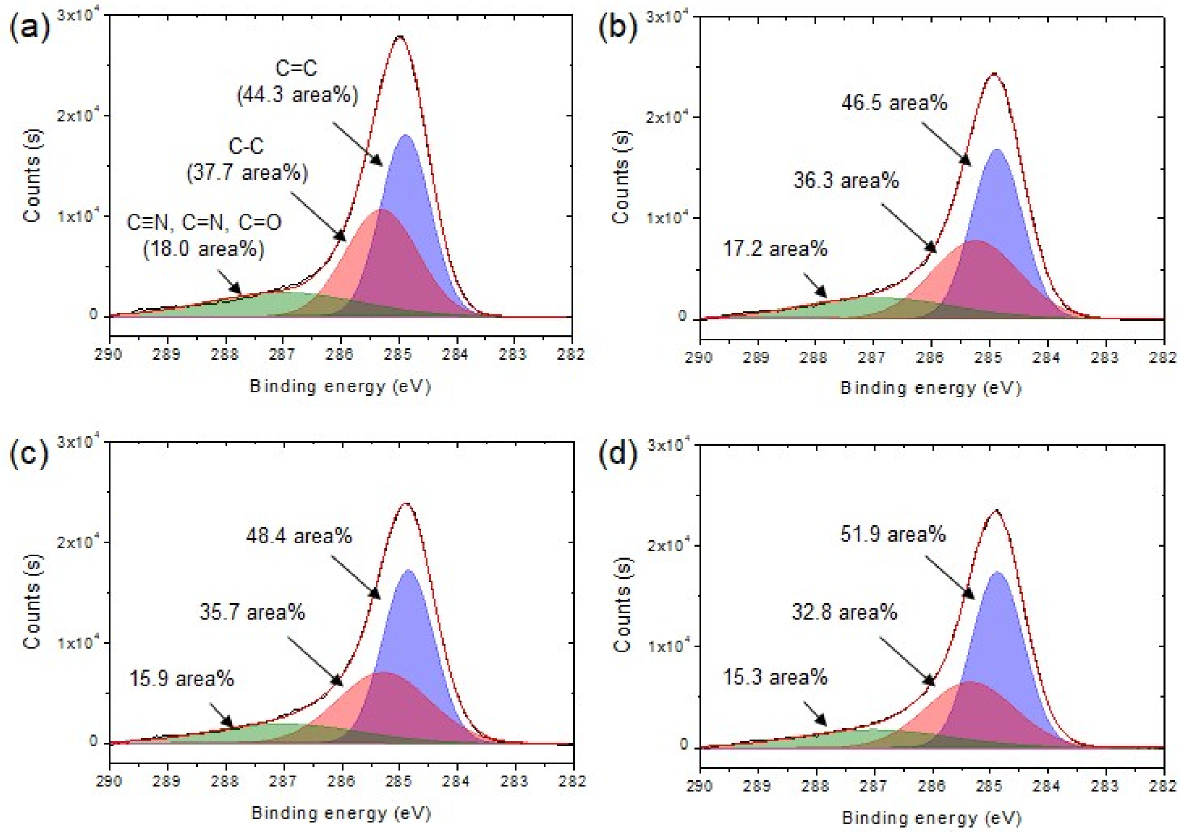
S3 SEM images and layer thickness of Matrimid polymer composite membranes deposited on alumina discs using various drop-amounts of polymer solution



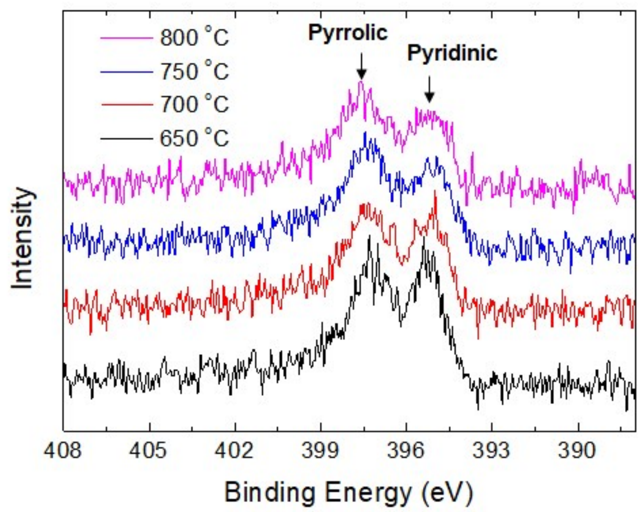
S4 SEM images and layer thickness of CMS composite membranes deposited on alumina discs using various drop-amounts of polymer solution

S5 Gas separation performance of the Matrimid polymer composite membranes deposited with various drop-amounts of polymer solution

		Drop amount (μL)					
		80	90	100	110	120	130
Gas permeance (GPU)	H_2	4.95	3.65	2.48	2.15	1.90	1.74
	CO_2	1.34	0.92	0.61	0.52	0.46	0.41
Ideal selectivity	H_2/CO_2	3.69	3.97	4.06	4.13	4.13	4.23

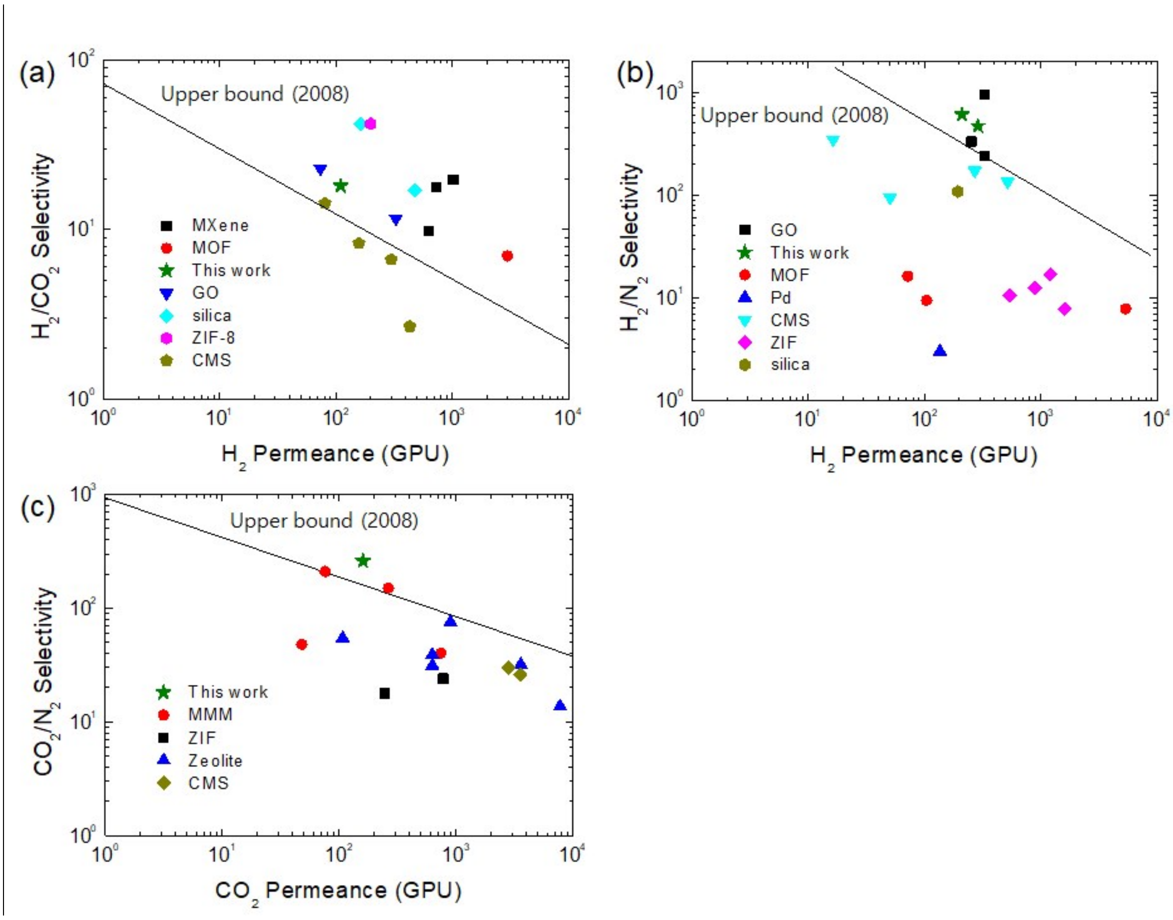


S6 C1s XPS spectra of CMS composite membranes carbonized at temperature of (a) 650, (b) 700, (c) 750, and (d) 800 °C



S7 N1s XPS spectra of CMS composite membranes carbonized at temperature of 650–800 °C

The gas separation performance of the CMS membranes studied in this work surpassed the Robeson upper bound trade-off relationship, as shown in S8–11. In particular, H_2 and CO_2 selectivity against N_2 was placed near the highest position. Moreover, these are the most selective performances when compared with other CMS membranes reported in the literature, even though the gas permeance in this work was slightly lower than in those studies. In particular, our CMS membrane showed higher H_2/CO_2 selectivity than other Matrimid-based CMS membrane reported in the literature. This is attributed to the formation of a defect-free pore structure from using the drop-coating process with a carbon layer of moderate thickness.



S8 Comparison of gas separation performance of the CMS membranes studied in this work and of other membranes reported in the literature for: (a) H_2/CO_2, (b) H_2/N_2, and (c) CO_2/N_2 separation

S9 H_2 permeance and H_2/CO_2 selectivity of various membranes reported in other literature

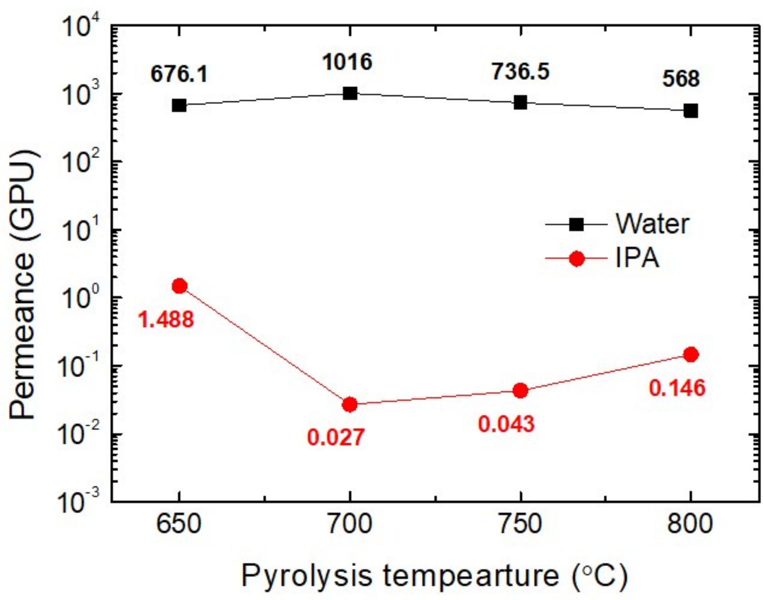
Materials		Operating tem. (°C)	H_2 permeance (GPU)	H_2/CO_2 selectivity	Ref.
Mxene		25	733.4	17.8	1
		25	1030.6	19.7	1
		25	628.7	9.8	1
MOF		25	2985	7	2
GO		RT	328	11.63	3
		RT	73.42	22.93	3
silica		20	477.6	17	4
ZIF-8		25	200	42	5
MFI zeolite		25	164	42.2	6
CMS	Matrimid	50	298.5	6.67	7
	PF resin	20	5.1	9.7	8
	Phenol resin	27	80.56	14.28	9
	PEI	25	158.2	8.3	10
	PEI	25	432.8	2.7	11
	Matrimid	25	109	18.1	This work

S10 H_2 permeance and H_2/N_2 selectivity of various membranes reported in other literature

Materials		Operating tem. (°C)	H_2 permeance (GPU)	H_2/N_2 selectivity	Ref.
MOF		25	104.3	9.4	12
		25	71.9	16.2	12
		25	5373	7.8	13
GO		20	328	240	14
		20	328	940	14
		20	253	330	14
silica		50	194	108	15
ZIF-8		25	537	10.58	16
ZIF-7		25	1614	7.84	16
		25	1211	16.9	16
		25	895	12.5	16
Pd		35	136	3	17
CMS	PEI+PPO	25	519	134	18
	PI+PPO	25	271	172	18
	PFA	RT	50.74	94	19
	PFA	25	16.4	347	20
	Matrimid	25	288	468	This work
	Matrimid	25	210	605	

S11 CO_2 permeance and CO_2/N_2 selectivity of various membranes reported in other literature

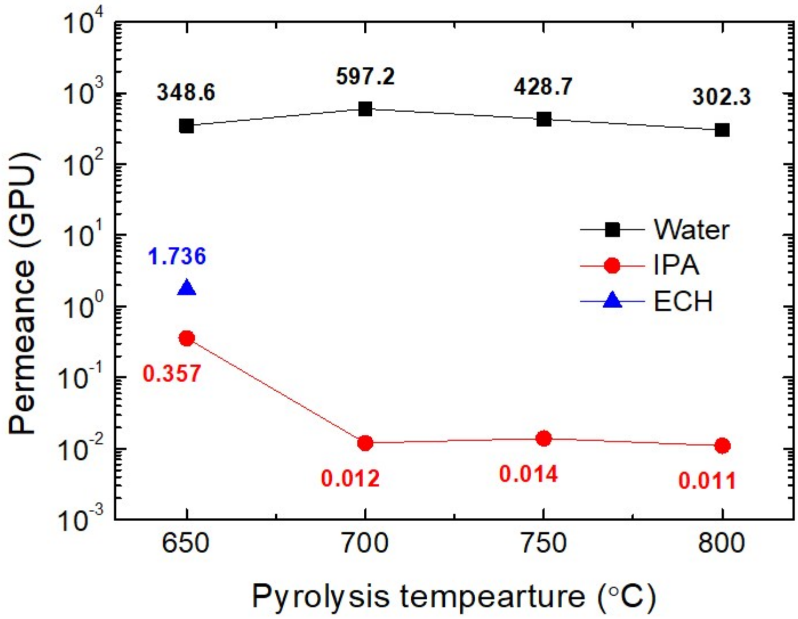
Materials		Operating tem. (°C)	CO_2 permeance (GPU)	CO_2/N_2 selectivity	Ref.
GO-MMM		24	76	210	21
		24	48	48	21
GO-CNT-MMM		25	264	149.8	22
Co-ZIF		RT	244.9	17.8	23
ZIF-8		25	776	24	24
MWCNT-MMM		RT	741.67	40.17	25
zeolite		22	3582	32	26
		22	626	39	27
		25	107	54.3	28
		27	7761	13.7	29
		RT	626	31.2	30
		30	895	75	31
CMS	6FDA/BPDA-DAM + Matrimid	35	2800	30	32
		35	3522	26	32
	Mtrimid	25	160	259	This work



S12 Water and IPA permeance of CMS composite membranes carbonized at temperatures of 650–800 °C for dehydration of a water/IPA binary mixture

S13 Water/IPA flux, purity, and separation factor of CMS composite membranes carbonized at temperatures of 650–800 °C for dehydration of a water/IPA binary mixture with various content (at the feed temperature of 60 °C)

Pyrolysis tem. (^oC)	Feed con.wt% H_2O	Feed tem. (^oC)	IPA Flux ($kg/m^2.hr$)	Water Flux ($kg/m^2.hr$)	S.F.	IPA Purity (%)	Water Purity (%)
650	5	60	0.0025360	0.3956465	3494	0.54087	99.45913
	10	60	0.0026647	0.6130840	2147	0.41743	99.58257
	15	60	0.0023206	0.6513416	1841	0.30694	99.69306
	20	60	0.0026143	1.0715845	1640	0.24337	99.75663
700	5	60	0.0000627	0.6449372	195253	0.00973	99.99027
	10	60	0.0000509	1.0709490	189066	0.00476	99.99524
	15	60	0.0000440	1.2612345	162363	0.00349	99.99651
	20	60	0.0000480	1.7849519	148695	0.00269	99.99731
750	5	60	0.0002065	0.4987935	98172	0.01935	99.98065
	10	60	0.0003883	0.8686117	81439	0.01105	99.98895
	15	60	0.0002954	0.9672708	86641	0.00654	99.99346
	20	60	0.0000752	1.2936773	76042	0.00526	99.99474
800	5	60	0.0002327	0.3647672	39156	0.04850	99.95150
	10	60	0.0002917	0.5977082	32202	0.02794	99.97206
	15	60	0.0002357	0.6827553	28117	0.02015	99.97985
	20	60	0.0002567	0.9977433	21525	0.01858	99.98142



S14 Water, IPA, and ECH permeance of CMS composite membranes carbonized at temperatures of 650–800 °C for dehydration of a water/IPA/ECH ternary mixture

S15 Water/IPA/ECH flux, purity and separation factor of CMS composite membranes carbonized at temperature of 650–800 °C with various feed content and temperature for dehydration of a water/IPA/ECH ternary mixture

Pyrolysis tem. (^oC)	Feed con.% H_2O	Feed temp. (^oC)	IPA flux ($kg/m^2.hr$)	ECH flux ($kg/m^2.hr$)	Water flux ($kg/m^2.hr$)	S.F.	IPA Purity (%)	ECH Purity (%)	Water Purity (%)
650	5	60	0.0001882	0.0009172	0.1838945	3161	0.10174	0.49581	99.40245
	10	60	0.0004439	0.0036478	0.3709083	816	0.11838	0.97274	98.90888
	15	60	0.0007905	0.0031042	0.5178868	752	0.15174	0.59581	99.25245
	20	60	0.0006267	0.0030542	0.6123191	665	0.10174	0.49581	99.40245
	20	50	0.0023563	0.0035645	0.4929352	339	0.46384	0.70167	98.83449
	20	40	0.0002312	0.0029195	0.1838492	233	0.12365	1.56124	98.31511
	20	30	0.0002016	0.0008115	0.0589869	233	0.33604	1.35242	98.31154
700	5	60	0.0000318	0.0000078	0.5851509	280631	0.00544	0.00133	99.99323
	10	60	0.0000264	0.0000163	0.8128259	171420	0.00325	0.00200	99.99475
	15	60	0.0000244	0.0000080	0.8369432	146042	0.00292	0.00096	99.99612
	20	60	0.0000212	-	1.0489788	158726	0.00202	<0.0005	99.99748
	20	50	0.0000242	-	0.8030074	113956	0.00301	<0.0005	99.99649
	20	40	0.0000240	-	0.5956300	88492	0.00402	<0.0005	99.99548
	20	30	0.0000210	-	0.3016233	53472	0.00698	<0.0005	99.99252
750	5	60	0.0000335	0.0000055	0.4094637	199351	0.00819	0.00134	99.99047
	10	60	0.0000393	0.0000098	0.6729400	123110	0.00585	0.00146	99.99269
	15	60	0.0000367	0.0000086	0.7439495	93043	0.00494	0.00115	99.99391
	20	60	0.0000247	-	0.7529838	91112	0.00389	<0.0005	99.99561
	20	50	0.0000340	-	0.6691679	71681	0.00508	<0.0005	99.99442
	20	40	0.0000197	-	0.3103751	58305	0.00636	<0.0005	99.99314
	20	30	0.0000371	-	0.2416600	25249	0.01534	<0.0005	99.98416
800	5	60	0.0000288	0.0000116	0.3752614	176397	0.00768	0.00309	99.98923
	10	60	0.0000323	0.0000115	0.4609562	94728	0.00701	0.00249	99.99050
	15	60	0.0000261	0.0000065	0.5159650	89799	0.00505	0.00126	99.99369
	20	60	0.0000196	-	0.5309564	86576	0.00412	<0.0005	99.99538
	20	50	0.0000257	-	0.4749534	67678	0.00541	<0.0005	99.99409
	20	40	0.0000168	-	0.2119811	47558	0.00791	<0.0005	99.99159
	20	30	0.0000246	-	0.1571360	24795	0.01563	<0.0005	99.98387

S16 Dehydration performance of various membranes reported in other literature for a water/IPA binary mixture

Category	Membrane materials	Feed Con.% H_2O	Feed temp. (^oC)	Total flux ($g/m^2.hr$)	Separation factor (α)	Ref.
Polymers	PBI (modified with chitosan)	70	25	247	21	33
	PBOZ (Poly (benz-3,1-oxazinone-4)	10	50	3	9000	34
	PVA-APTEOS blend (cross-linked with TEOS)	10	30	60	891	35
	PVA (cross-linked with 4-Sulfophthalic acid)	10	40	46	2638	36
	CMS-3(Commercialized dense membrane) supported on Electrospun PTFE	12.4	70	111	81	37
	PBO (Polybenzoxazole)	10	80	135	140	38
	PVA-PVAm blend (cross-linked with Glutaraldehyde)	15	60	160	2085	39
	Chitosan (cross-linked with Glutaraldehyde and Maleic anhydride)	10	60	300	634	40
	PVA (cross-linked with TMC)	10	60	320	400	41
	PVA-Na Alginate blend (cross-linked with Glutaraldehyde)	10	30	24	196	42
	PVA-Na Alginate blend (cross-linked with Maleic acid) supported on PSf	10	45	414	1727	43
MMMs	Alginate (RGO as filler) supported on PES	30	25	1700	23000	44
	PVA/Glutaraldehyde (GOQD as filler) supported on PSf	30	25	464	464	45
		30	70	1800	120	
	PVA-PVAm blend/Glutaraldehyde (modified HNT as filler)	10	40	31	93313	46
	PVA/PAA (Ag exchanged NaY zeolite as filler)	20	60	150	1100	47
		20	40	84	2718	
	P84_SPES Primer (30wt.% ZIF-90 as filler)	15	60	109	5668	48
	(MWNT modified with PSS) supported on PAN	10	30	168	882	49
	PVA/Glutaraldehyde (5wt.% ZIF-8 as filler)	10	30	868	132	50
	PVA/Glutaraldehyde (CNT as filler)	10	30	96	79	51
	Matrimid (β-cyclodextrin as filler)	14	22	52	5000	52
	PBI	15	60	9	5000	53
	PBI (ZIF-8 as filler)	15	60	86	3417	
TFCs	MPD-TMC supported on alumina tube	15	80	6050	1396	54
	Chitosan-TMC supported on alumina tube	10	60	640	9634	55

		10	70	908	8993	
	DAPE-TMC with TiO_2 interlayer supported on alumina tube	10	60	6440	12000	56
	HPEI-TMC supported on Torolon	15	50	1282	624	57
Ceramics	Ti_2CT_x Mxene (functionalized with PDDA) supported on PAN	10	50	1237	1932	58
	Vacuum assisted grown NaA Zeolite	05	70	1670	10000	59
	A-type Zeolite (Mitsui)	10.4	75	1580	30000	60
	T-type Zeolite (Mitsui)	10.2	75	2100	9000	
	ECN Silica	10.2	75	2760	90	
	Silica	N/A	80	650	75	61
	Silica with 10mol.% Zirconium		80	860	300	
	Silica with 10mol.% Titanium		80	780	400	
	Sulfonated PPO based CMS (Carbon molecular Sieve, pyrolysis at 600℃)	10	70	1400	99900	62
		10	80	2000	58100	
	Resorcinol based CMS (pyrolysis at 400℃)	10	70	233	10000	63
	Matrimid based CMS (pyrolysis at 700℃) supported on alumina disc	10	60	1071	189000	This work
		20	60	1785	148468	

S17 Dehydration performance of various membranes reported in other literature for a water/IPA/ECH ternary mixture

Membrane materials	Feed comp. wt.% (ECH/IIPA/Water)	Feed temp. (°C)	Total flux (g/m^2.hr)	Separation factor (α)	Ref.
PVA cross-linked with Glutaraldehyde	50/30/20	30	25	1908	64
PVA-PVAm blend cross-linked with Glutaraldehyde (PVAm_0.5)			110	149	
PVA-PVAm blend cross-linked with Glutaraldehyde (PVAm_1.0)			205	97	
PVA-PVAm blend cross-linked with Glutaraldehyde (PVAm_1.5)			320	60	
PVA crosslinked with TEOS and PVAm (STA as filler) layer by layer coating (0L-L)	50/30/20	30	140	2099	65
PVA crosslinked with TEOS and PVAm (STA as filler) layer by layer coating (5L-L)			220	1061	
PVA crosslinked with TEOS and PVAm (STA as filler) layer by layer coating (10L-L)			280	416	
PVA crosslinked with TEOS and PVAm (STA as filler) layer by layer coating (5L-L)	50/35/15	30	70	8033	
		40	150	4617	
		50	190	1729	
Matrimid based CMS (pyrolysis at 700°C) supported on alumina disc	50/30/20	30	285	53484	This work
		40	496	88448	
		50	869	113834	
		60	1049	158692	
	53.125/31.875/15	60	837	145995	
	56.25/3.75/10	60	813	171385	
	58.75/36.25/5	60	585	280729	

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