Supporting Information

Controllable synthesis of a chemical stable molecular sieving nanofilm for high efficient organic solvent nanofiltration

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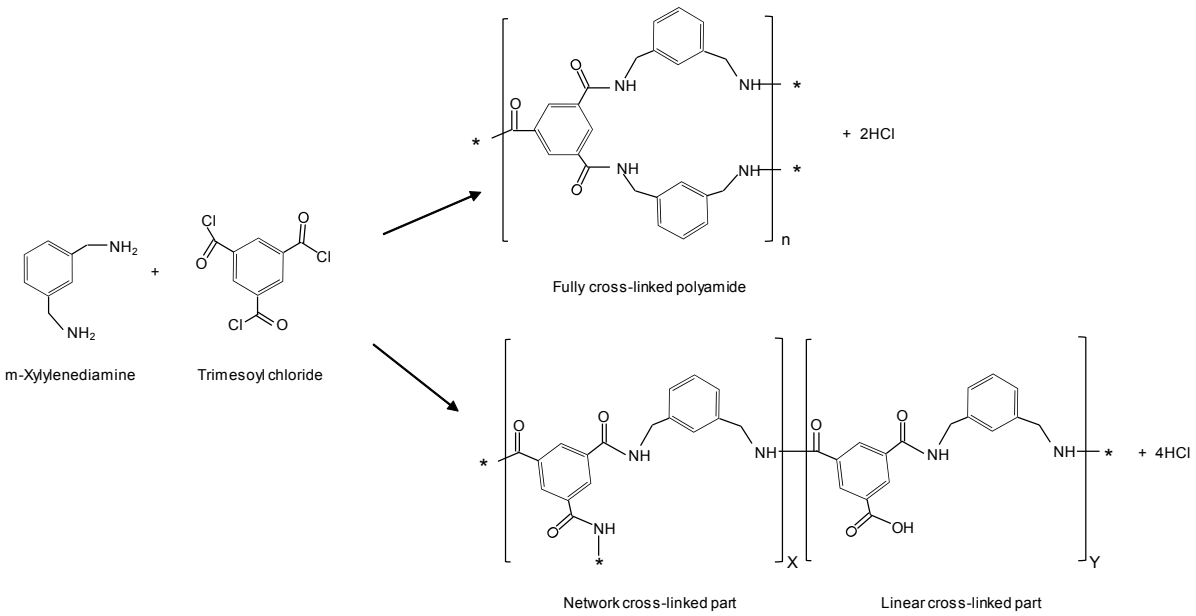

Figure S1 Interfacial reaction of m-xylylenediamine (m-XDA) with trimesoyl chloride (TMC) and formation of network and linear cross-linked polyamide thin film.

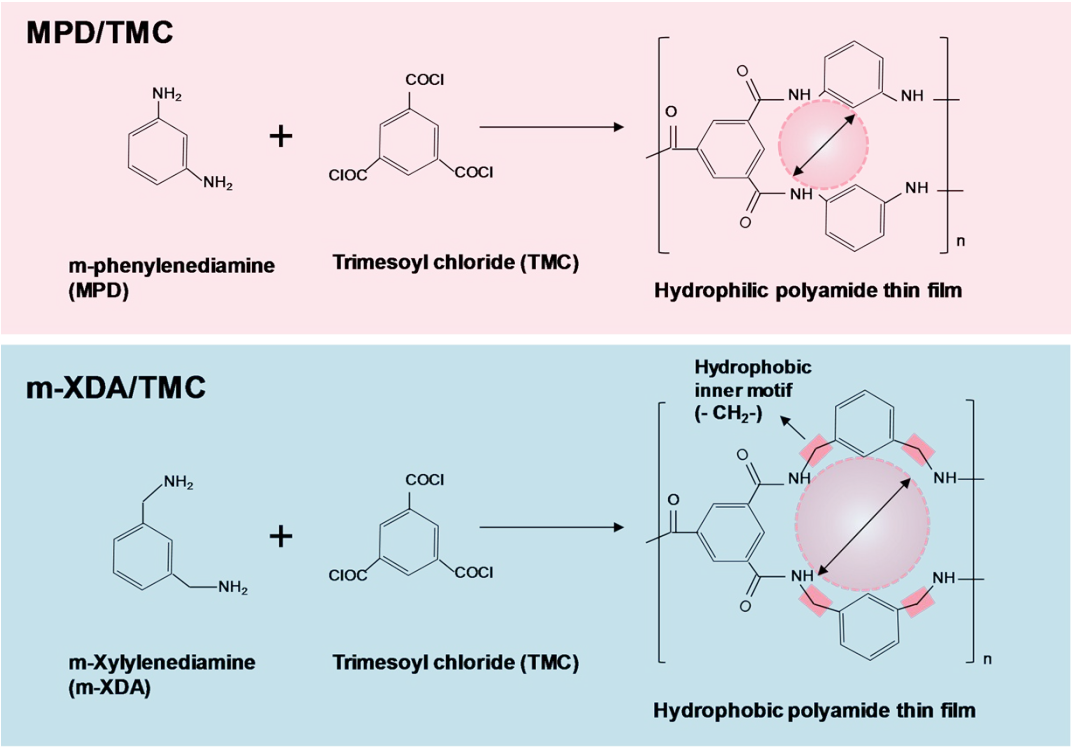

Figure S2 Comparison of the interfacial polymerization process for polyamide nanofilms preparation using different amine monomers.

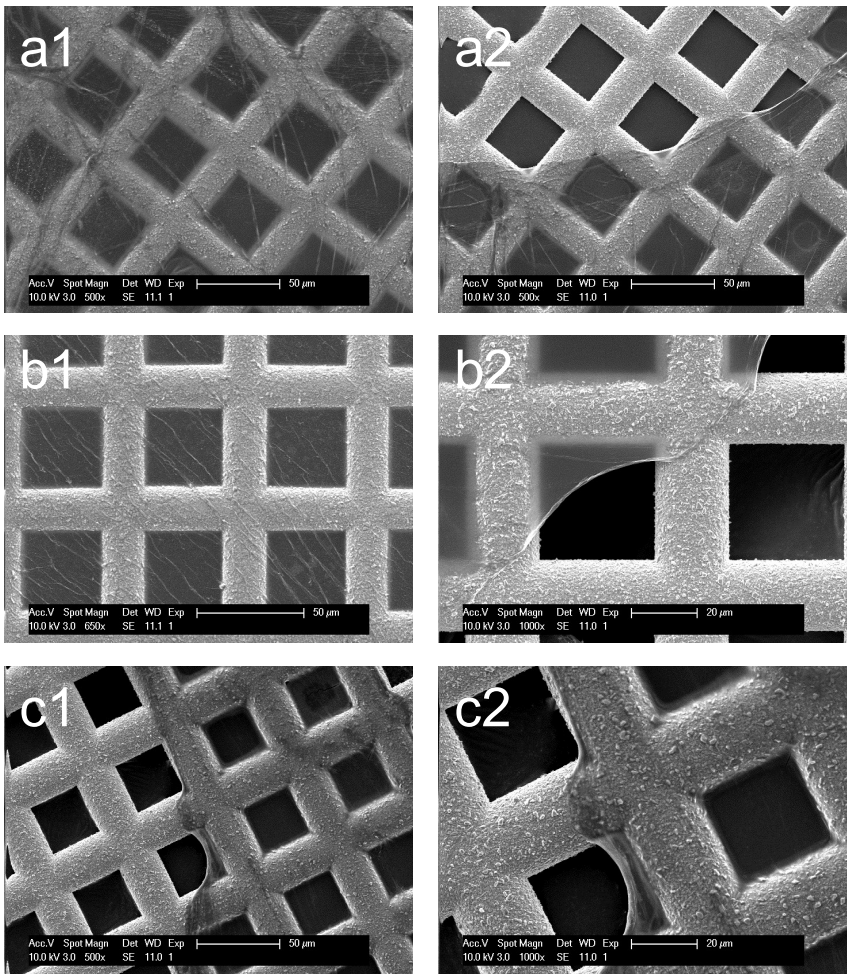

Figure S3 Molecular sieving nanofilms prepared from different concentrations of monomers. The nanofilms were supported by the copper mesh. (a1, a2: m-XDA/TMC F-0.5/0.025; b1, b2: m-XDA/TMC-1/0.05; c1, c2: m-XDA/TMC-3/0.15;)

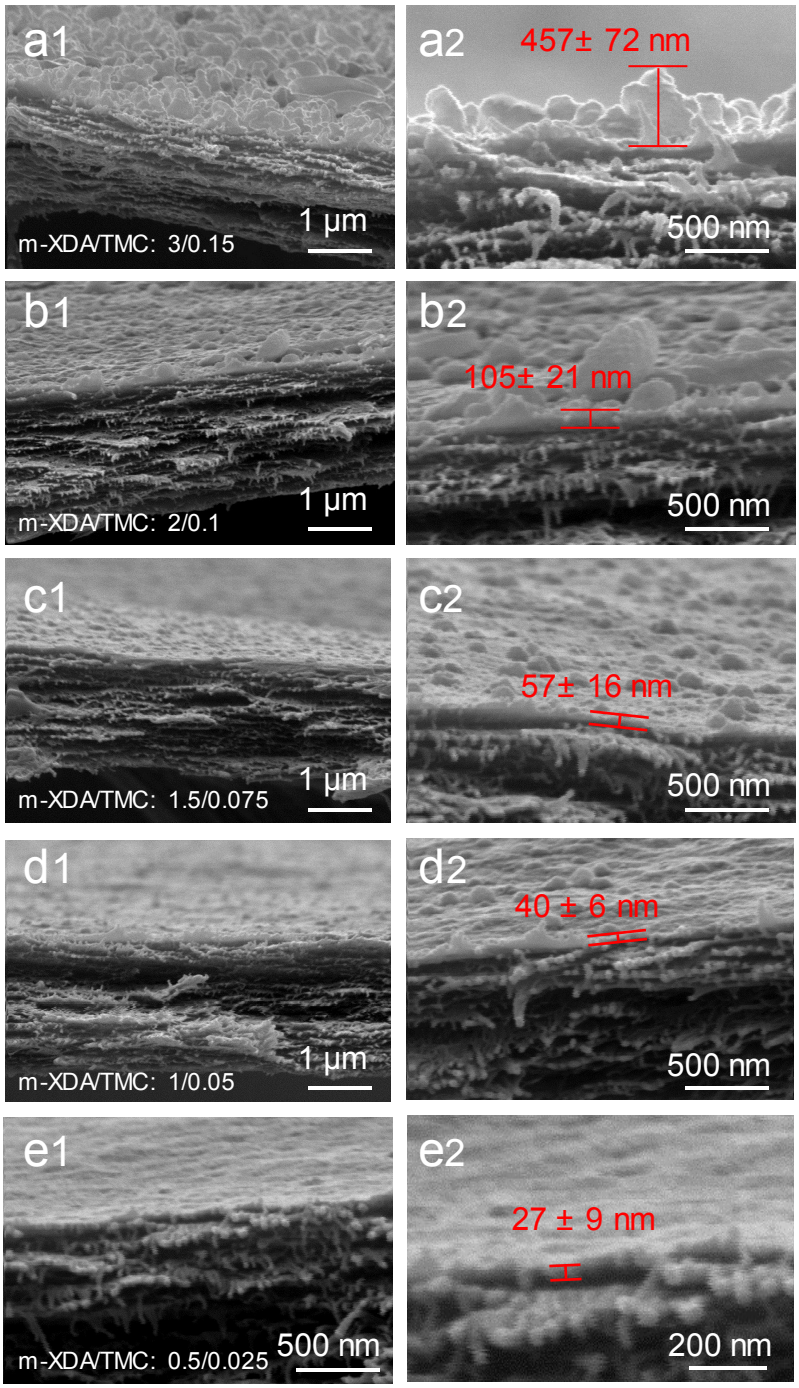

Figure S4 SEM observations of the cross-section and the thickness of the polyamide layer of PA/ANF composite membranes. (a1, a2: m-XDA/TMC F-3/0.15; b1, b2: m-XDA/TMC-2/0.1; c1, c2: m-XDA/TMC-1.5/0.075; d1, d2: m-XDA/TMC-1/0.05; e1, e2: m-XDA/TMC-0.5/0.025).

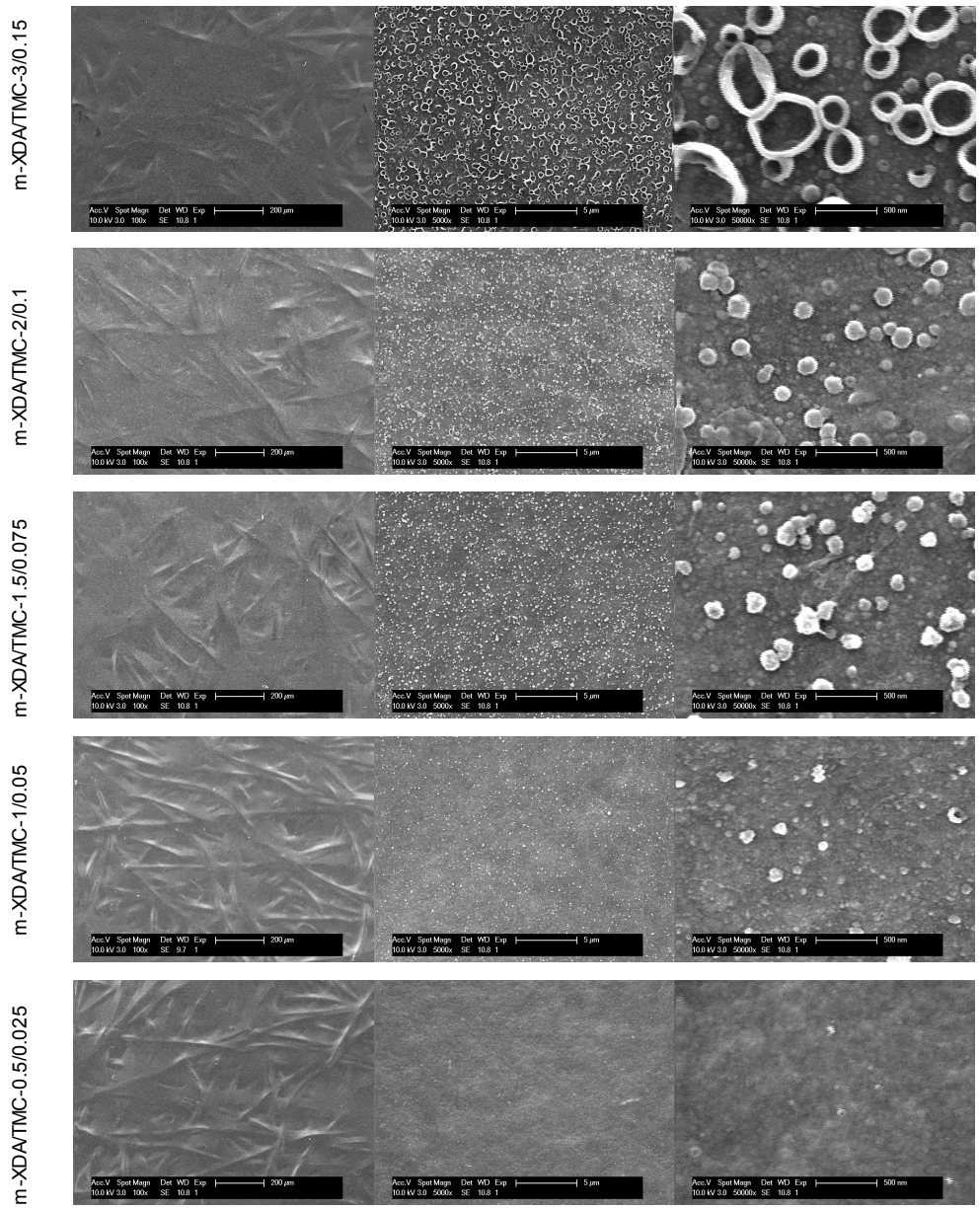

Figure S5 SEM observations at different magnifications (100×, 5000×, 50000×) of the membrane surface prepared from different concentration ratios of m-XDA/TMC.

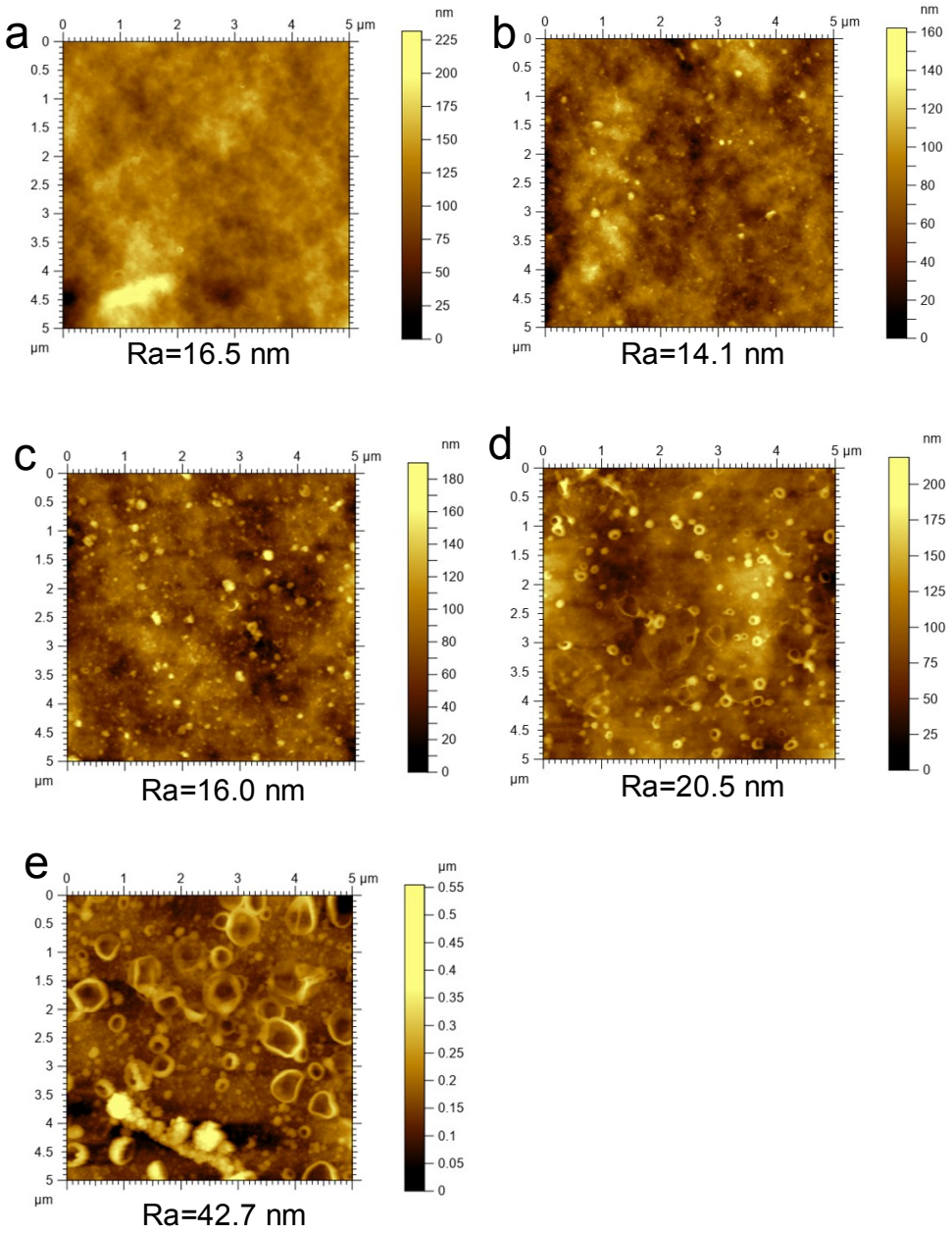

Figure S6 AFM observation and roughness of m-XDA/TMC membranes. (a. 0.5/0.025; b. 1/0.05; c. 1.5/0.075; d. 2/0.1; e. 3/0.15)

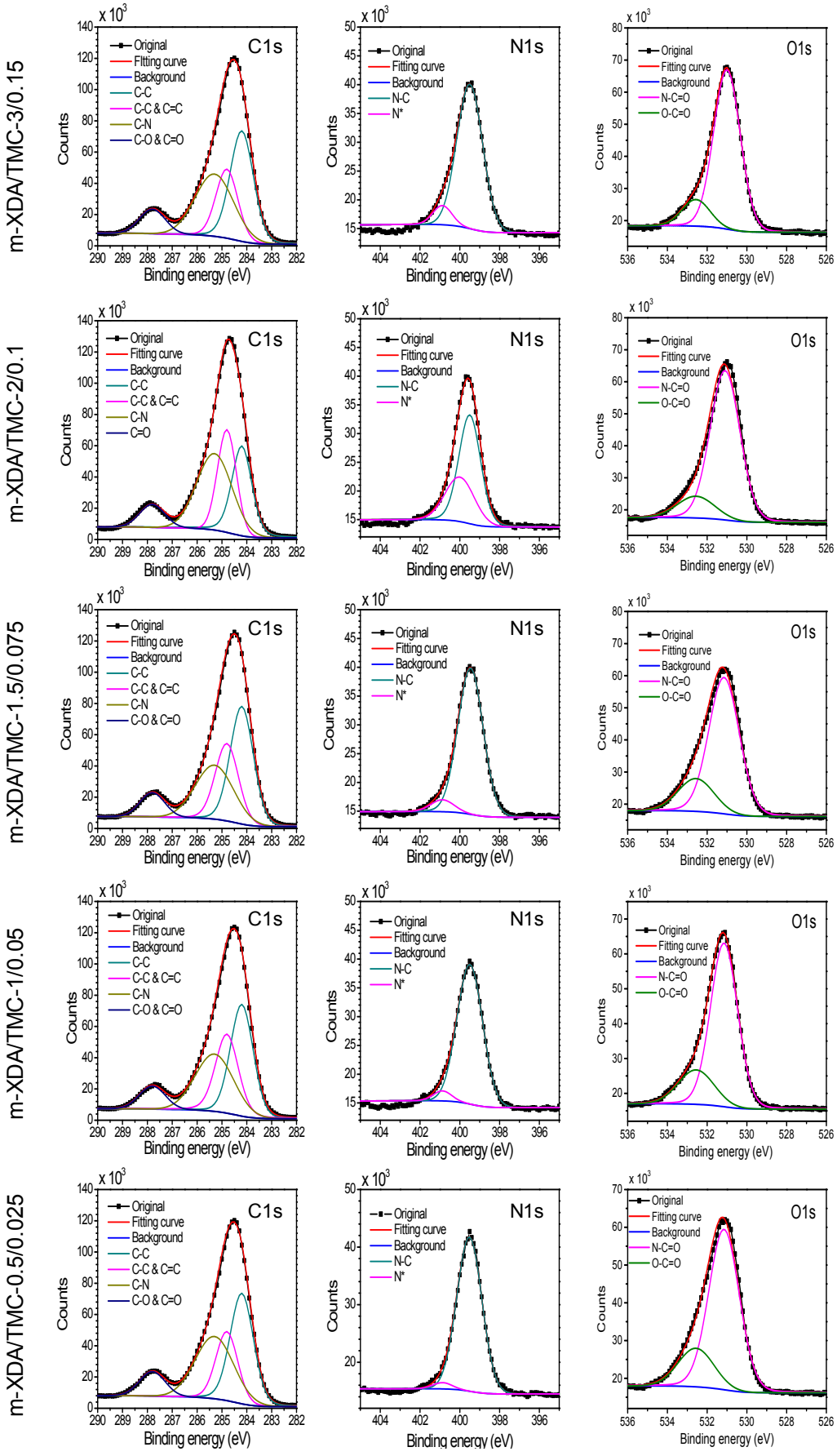

Figure S7 Narrow scan results of X-ray photoelectron spectra of composite membranes.

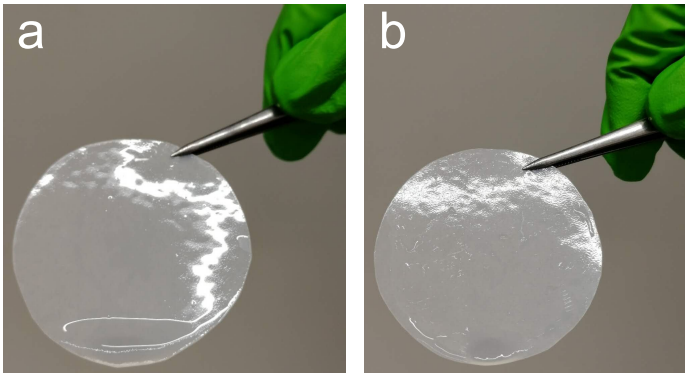

Figure S8 Photograph of the membrane surfaces for polyamide composite membrane prepared from MPD/TMC (a) and composite membrane prepared from m-XDA/TMC (b).

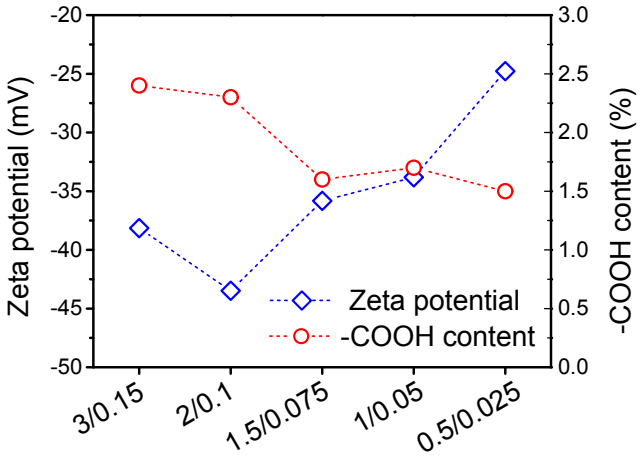

Figure S9 The relationship between the zeta potential and the carboxyl groups content in the nanofilms.

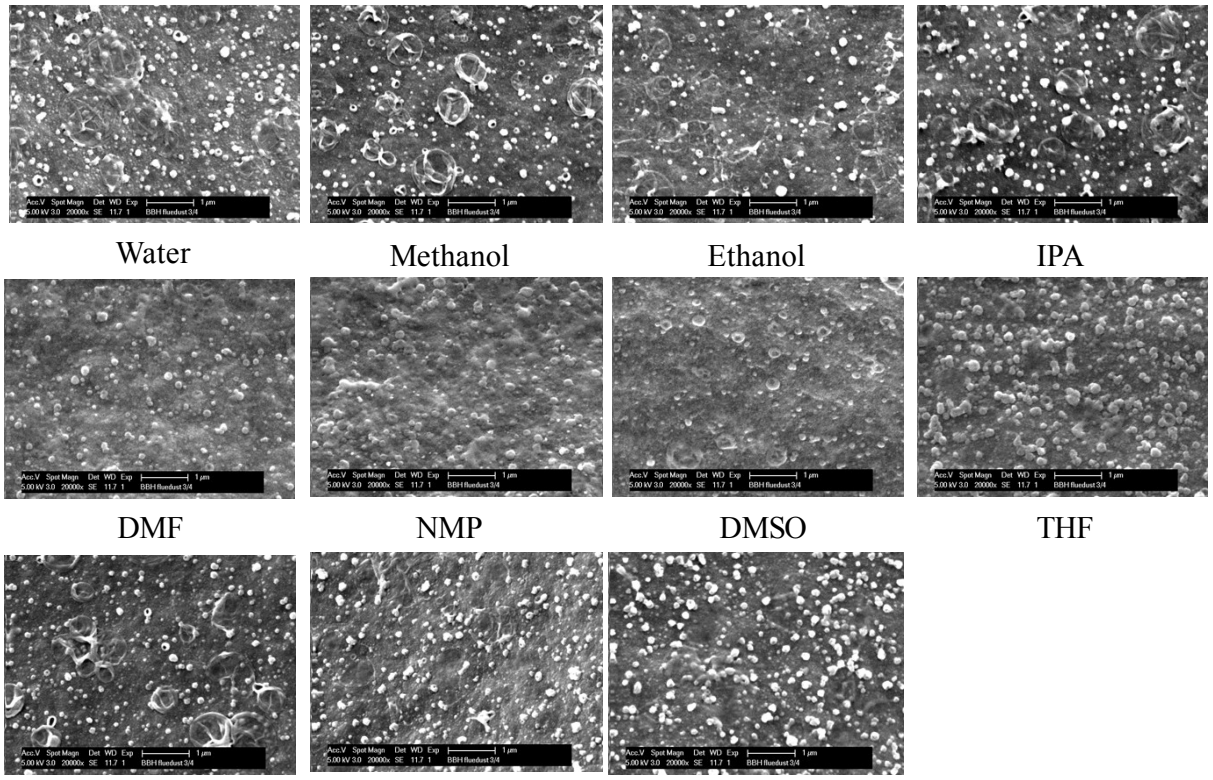

Figure S10 SEM observations of the membrane surface after treated with different solvents for one week. Membranes were prepared from m-XDA/TMC-2/0.1.

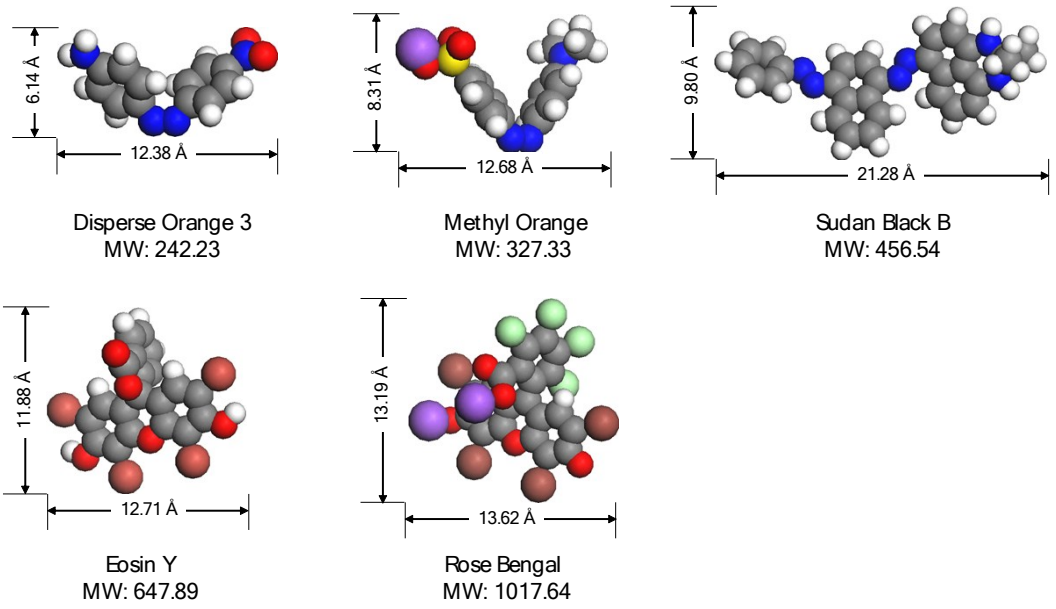

Figure S11 Molecular size of disperse orange 3, methyl orange, sudan black B, eosin Y, erythrosin B, rose Bengal. The molecular sizes were calculated using Materials Studio 8.0 after geometry optimization by modules of Forcite.

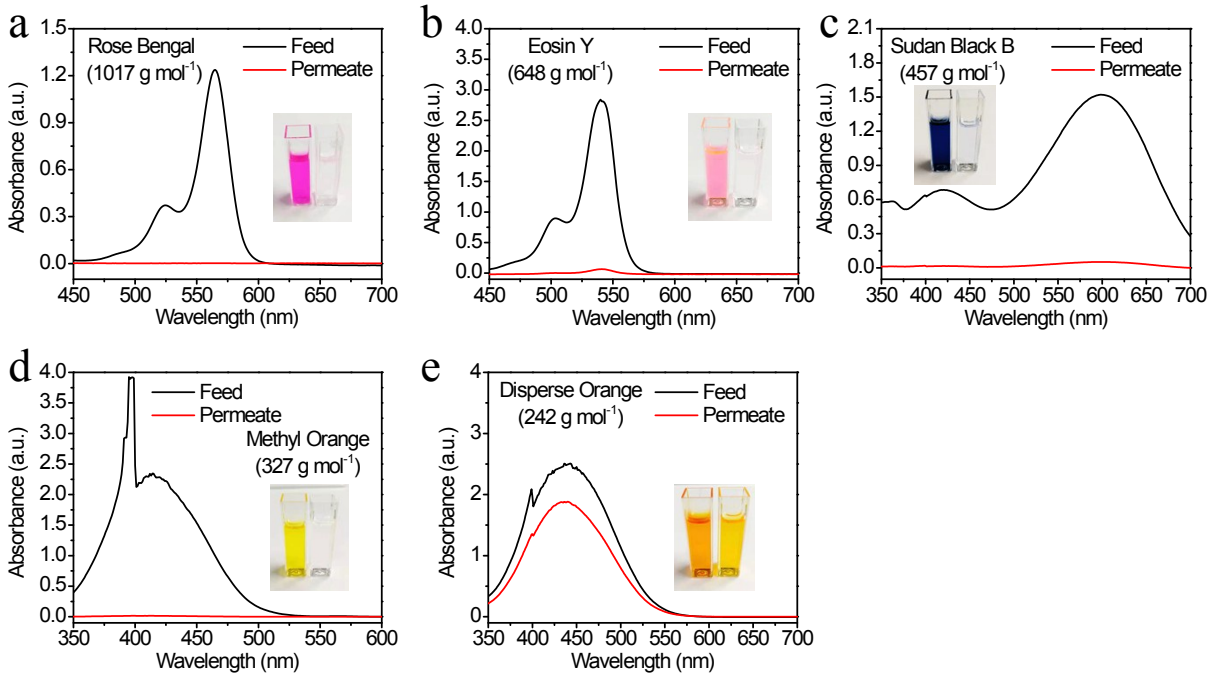

Figure S12 Rejection of dyes with different molecular weight (MW) in acetone. The molecular weight of dyes is 1017 g mol^{-1} for rose Bengal (RB), 648 g mol^{-1} for Eosin Y (EY), 457 g mol^{-1} for Sudan Black B (SBB), 327 g mol^{-1} for methyl orange (MO) and 242 g mol^{-1} for disperse orange 3 (DO3). RB and MO are negatively charged dyes. EY, SBB and DO3 are neutral charged dyes.

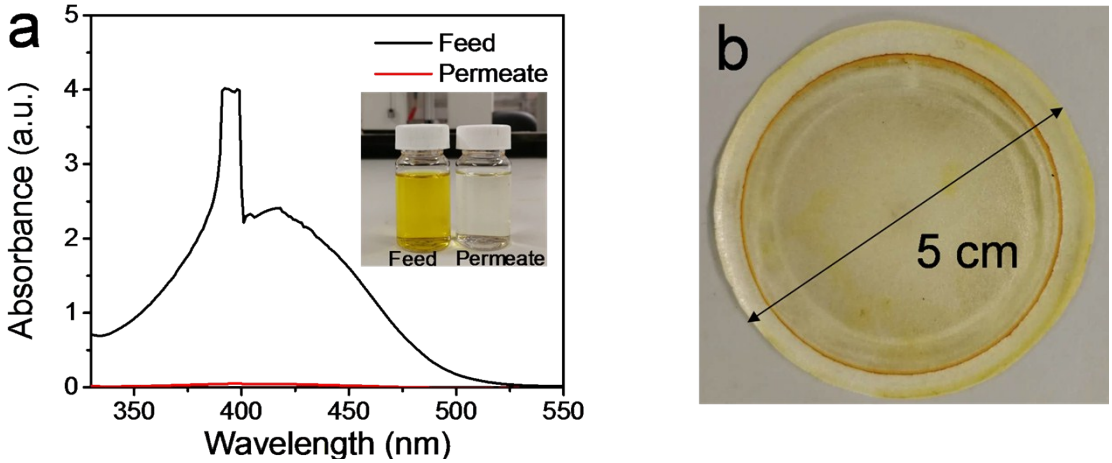

Figure S 13 (a) UV-vis absorbance spectrums of the feed and permeate of acetone/MO solution. Inset photo of the feed and permeate. The feed solution was diluted five times before UV-vis measurement. (b) Surface observation of the m-XDA/TMC-3/0.15 membrane after long-term filtration in acetone/MO.

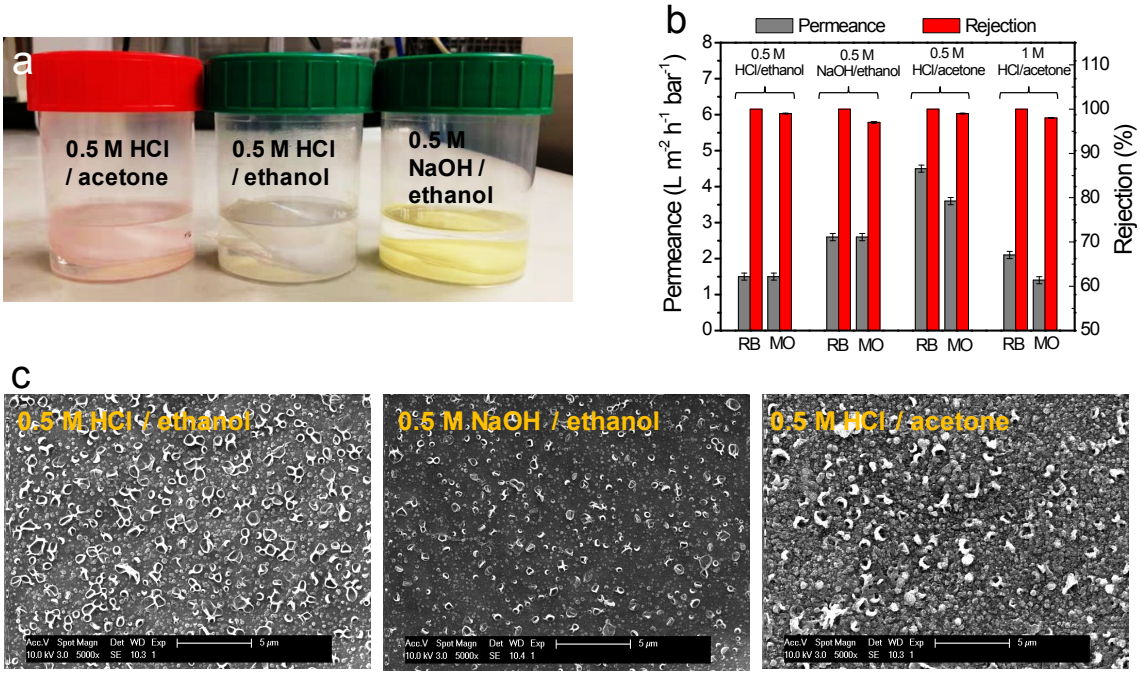

Figure S14 Photographs of the m-XDA/TMC-0.3/0.15 composite membrane in harsh organic liquids. (b) The molecular separation performance of treated composite membrane. The membranes were soaked in 0.5 M HCl water/ethanol and 0.5 M NaOH water/ethanol, 0.5 M HCl water/acetone and in 1.0 M HCl water/acetone, respectively for 15 days. The membranes after treatment were tested using dye/organic solution in ethanol and acetone, respectively. (c) The SEM images of the treated composite membranes after filtration in different solvent.

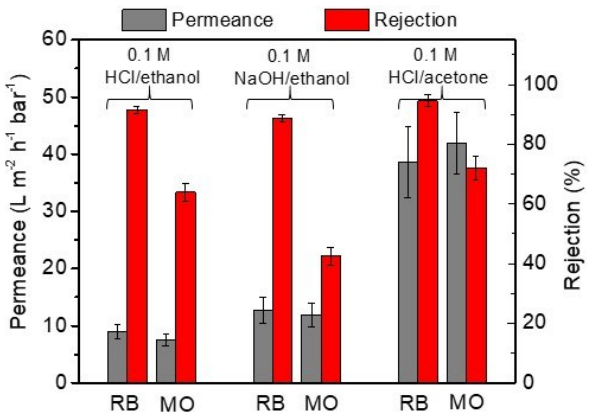

Fig. S15 The chemical stability of the m-XDA/TMC-0.5/0.25 composite membrane in harsh acid and base environments. The membranes were soaked in 0.1 M HCl water/ethanol, 0.1 M NaOH water/ethanol and in 0.1 M HCl water/acetone, respectively for 10 days. The membranes after treatment were tested using dye/organic solution in ethanol and acetone, respectively.

Table S1
The compositions of the acid/solvent and base/solvent solutions. (As an example of preparing 30 ml of solution)

Organic solution	NaOH (50%) aqueous solution (ml)	HCl (37%) aqueous solution (ml)	Ethanol (ml)	Acetone (ml)	Total (ml)
0.1 M HCl water/ethanol	-	0.25	29.75	-	30
0.5 M HCl water/ethanol	-	1.24	28.76	-	30
0.1 M NaOH water/ethanol	0.16	-	29.84	-	30
0.5 M NaOH water/ethanol	0.79	-	29.21	-	30
0.1 M HCl water/acetone	-	0.25	-	29.75	30
0.5 M HCl water/acetone	-	1.24	-	28.76	30
1.0 M HCl water/acetone	-	2.48	-	27.52	30

Table S2

The degree of crystallinity of the free-standing m-XDA/TMC nanofilms.

Free standing nanofilm	Crystallinity (%) [a]
m-XDA/TMC-3/0.15	81.67
m-XDA/TMC-2/0.1	98.09
m-XDA/TMC-1.5/0.075	95.77
m-XDA/TMC-1/0.05	92.21
m-XDA/TMC-0.5/0.025	86.39

[a] The degrees of crystallinity of the free-standing nanofilms were analyzed by MDI Jade 6.0 software. The XRD raw data was fitted after fitting the background and strip Kα2. Crystallinity was calculated by the ratio of the areas of the crystalline peaks to the total area.

Table S3

XPS results from polyamide composite membranes. Plausible species were determined from the deconvolution of C1s, O1s and N1s core level XPS spectra. By considering the C-O is only originating from the carboxylic acid group, the COOH content was calculated by the product of the C-O content in core level O1s XPS spectra multiply the atomic composition of O1s in the core level XPS spectra.

Membrane	C1s		N1s		O1s		COOH
	Species	(%)	Species	(%)	Species	(%)	(%)
PA/ANF-3/0.15	C-C	12.3	N-C	66.0	N-C=O	78.7	2.4
	C-C & C=C	45.8	-N<	34.0	O-C=O	21.3	
	C-N	30.9					
	C-O & C=O	11.0					
PA/ANF-2/0.1	C-C	44.6	N-C	79.7	N-C=O	79.1	2.3
	C-C & C=C	23.8	-N<	20.3	O-C=O	20.9	
	C-N	21.6					
	C-O & C=O	10.0					
PA/ANF-1.5/0.075	C-C	22.7	N-C	90.5	N-C=O	85.5	1.6
	C-C & C=C	41.8	-N<	9.5	O-C=O	14.5	
	C-N	25.1					
	C-O & C=O	10.4					
PA/ANF-1/0.05	C-C	54.2	N-C	81.2	N-C=O	85.1	1.7
	C-C & C=C	17.5	-N<	18.8	O-C=O	14.9	
	C-N	17.8					
	C-O & C=O	10.5					
PA/ANF-0.5/0.025	C-C	36.8	N-C	84.1	N-C=O	85.6	1.5
	C-C & C=C	23.9	-N<	15.9	O-C=O	14.4	
	C-N	29.4					
	C-O & C=O	9.9					
PA/ANF-3/0.15 (without TEA)	C-C	20.0	N-C	100	N-C=O	83.0	2.4
	C-C & C=C	48.9			O-C=O	17.0	
	C-N	20.1					
	C-O & C=O	11.0					

Table S4
Comparison of non-polar solvent permeances of the m-XDA/TMC TFC membrane with other commercial OSN membranes and reported composite membranes in literatures.

Membrane type	Membrane material	Solvent	Permeance (L m^{-2} h^{-1} bar^{-1})	Reference
Commercial membrane	Starmem 122 (PI)	Toluene	0.6	[1]
	Starmem 240 (PI)	Toluene	0.7	[2]
	SOLSEP NF030306	n-hexane	0.55	[3]
	Duramem 150	n-hexane	0	[4]
	Duramem 300	n-hexane	0	
	Duramem 500	n-hexane	0	
	Duramem 900	n-hexane	0	
	Puramem 280	n-hexane	0	
	Puramem 600	n-hexane	0	
ISA membrane	P84 PI	Toluene	3.6	[5]
	XP84 PI	Toluene	0.2	[6]
	ANF/ZIF-8 nanocomposite	n-hexane	1.8	[7]
	ANF/PEI	n-hexane	0.8	[8]
TFC membrane	MPD/TMC/PI	Toluene	0.14	[1]
	Hydrophobic PA/XP84 PI	Toluene	1.7	[9]
	PA/PDMS/PAN	n-hexane	9.0	[10]
	MPD/TMC/GO-PI	n-hexane	0.01 [a]	[11]
	MPD/TMC/GQDs-PI	n-hexane	0.01 [a]	[12]
	CDs/TMC/PI	n-hexane	6.3	[13]
	PEI/TMC/PAN	Toluene	0.06	[14]
	PEI-SiO_2-C_6H_6/TMC/PAN	Toluene	1.28	
	PEI-CD/TMC/PAN	Toluene	0.56	[15]
	PEI-CDs/TMC/PAN	Toluene	0.88	[16]
	PEI/IPC/PSf	Toluene	1.2 [a]	[17]
TFC via coating	PIM-1/PEI/PAN	Toluene	1.1	[2]
	PIM1-CO1-50/PEI/PAN	Toluene	0.9	
Mixed matrix membrane	AlO_x/PIM-1	Toluene	0.5 [a]	[18]
	PDMS-zeolites/PI	Toluene	0.6	[19]
	PDMS-silicalite/PI	Toluene	0.9	[20]
	PDMS-silicalite hollow spheres/PI	Toluene	1.3	
	Polymer nanoparticles/PI	Toluene	0.6	[21]
	TiO_2/alumina	n-hexane	0.36	[22]
	PVDF-silica	n-hexane	1.0	[23]
TFC membrane	m-XDA/TMC/ANF	Toluene	4.2	This work
		n-hexane	2.6	This work

[a] Those permeabilities are estimated from the literatures.

Table S5

The values of the group contributions for the crosslinked polyamide. Data are obtained from Hoy's system (1985).[24]

Groups	Numbers				F_t ((MJ/m^3)$^{1/2}$/mol)	F_p ((MJ/m^3)$^{1/2}$/mol)	V (cm^3/mol)	ΔT (p)
$CH_{(aromatic)}$	9	7	9	7	241	62.5	13.42	0.018
$C_{(aromatic)}$	6	5	6	5	201	65	7.42	0.015
-CONH-	3	2	3	2	1131	895	28.3	0.073
$-CH_2-$	3	2	-	-	269	0	15.55	0.02
-COOH	-	1	-	1	241	62.5	13.42	0.018
Sum	A[a]				7575	3637.5	296.85	0.531
		B[a]			6057	2967.5	244.84	0.426
			C[a]		6768	3637.5	250.2	0.471
				D[a]	5519	2967.5	213.74	0.386

[a] A- D are the fully cross-linked (A), linear cross-linked (B) m-XDA/TMC polyamide, fully cross-linked (C) and linear cross-linked (D) MPD/TMC polyamide, respectively;

Table S6

The solubility parameters of solvents and polymers.

Solvent	Solubility parameters [a]			Fully cross-linked		Linear cross-linked	
	δ_d (MPa$^{1/2}$)	δ_p (MPa$^{1/2}$)	δ_h (MPa$^{1/2}$)	$\Delta\delta_{s\text{-}p}$ (MPD/TMC) (MPa$^{1/2}$) [b]	$\Delta\delta_{s\text{-}p}$ (m-XDA/TMC) (MPa$^{1/2}$) [b]	$\Delta\delta_{s\text{-}p}$ (MPD/TMC) (MPa$^{1/2}$) [b]	$\Delta\delta_{s\text{-}p}$ (m-XDA/TMC) (MPa$^{1/2}$) [b]
Methanol [c]	15.1	12.3	22.3	7.9	8.9	8.9	9.7
Ethanol [c]	15.8	8.8	19.4	8.7	8.5	9.0	9.0
Acetone [c]	15.5	10.4	7.0	10.8	8.6	9.5	7.8
n-hexane [c]	14.9	0	0	23.0	20.8	21.8	20.1
Toluene [c]	18.0	1.4	2.0	20.7	18.4	19.5	17.8
MPD/TMC (Fully) [d]	16.1	16.7	15.8	-	-	-	-
MPD/TMC (Linear) [d]	15.7	16.3	14.4	-	-	-	-
m-XDA/TMC (Fully) [d]	16.5	15.3	14.0	-	-	-	-
m-XDA/TMC (Linear) [d]	16.1	15.2	13.1	-	-	-	-

[a] δ_d, δ_p and δ_h are the Hansen Solubility Parameters (HSP) for the dispersion, polarity and hydrogen bonding interactions.[25]

[b] $\Delta\delta_{S\text{-}P}$ is the solubility parameter differences between a solvent and polymer. "S" represents the solvent. "P" represents the polymer prepared from MPD/TMC or m-XDA/TMC. It is calculated by the equation of $\Delta\delta_{S-P} = \left[\left(\delta_{d,P} - \delta_{d,S}\right)^2 + \left(\delta_{p,P} - \delta_{p,S}\right)^2 + \left(\delta_{h,P} - \delta_{h,S}\right)^2\right]^{1/2}$.[24]

[c] Data were obtained from the book "Hansen Solubility Parameters: A User's Handbook (second edition)".[25]

[d] Solubility parameters are estimated based on Hoy's system (1985).[24]

To validate the interactions between the cross-linked polyamide polymer with the solvent molecules, the solubility parameter differences were calculated and compared. Four

representative solvents were selected: polar solvents (ethanol, methanol), non-polar solvents (n-hexane, toluene) and a polar aprotic solvent (acetone). m-XDA/TMC and MPD/TMC polyamide polymer were choosed to demonstrate the introducing of the methylene moieties into the cross-linked structure could promote the non-polar solvent permeances. By considering the different cross-linking degrees, both fully cross-linked and linear cross-linked polyamide network were investigated as representatives. The solubility parameters of solvent were obtained from Hansen solubility parameters.[25] While, the solubility parameters of different polyamide polymers are estimated from the Hoy's method[24] based on group contributions as shown in Table S6.

Based on those data, the solubility parameter differences between a solvent and polymer ($\Delta\delta_{s\text{-}p}$) were calculated, as shown in Table S7. The smaller the $\Delta\delta_{s\text{-}p}$, the better is the solvent for the polymer, in other words, a higher interaction exists between the solvent and the polymer. It could be found that both fully cross-linked and linear cross-linked MPD/TMC polyamide show high values of $\Delta\delta_{s\text{-}p}$ (~20 $MP^{1/2}$) for non-polar solvents and low values of $\Delta\delta_{s\text{-}p}$ (<11 $MP^{1/2}$) for polar solvents. This explains why conventional MPD/TMC membrane always has high solvent permeances for polar solvents, such as methanol, but has extremely low non-polar solvent permeances. It could be also observed in m-XDA/TMC polyamide membrane because of the low $\Delta\delta_{s\text{-}p}$ (<10 $MP^{1/2}$) for methanol, ethanol as well as acetone. However, the $\Delta\delta_{s\text{-}p}$ for non-polar solvents are reduced. e.g. the $\Delta\delta_{s\text{-}p}$ for n-hexane and toluene decreased from 23.0 to 20.8 $MP^{1/2}$ and 20.7 to 18.4 $MP^{1/2}$, respectively. It indicates that higher interactions exist between the m-XDA/TMC polyamide membrane and non-polar solvents. It could also be observed for the linear cross-linked m-XDA/TMC polyamide membrane. This is mainly originated from the introduced -CH_2- groups in the polyamide network, giving rise to a Janus pore structure. The alternate hydrophilic part (-COOH, -CONH-) and hydrophobic part (aromatic rings, -CH_2-) imparts the membrane with Janus interactive properties for polar solvent and non-polar solvent.

Table S7

Summary of molecular weight, molecular size, permeance and rejection of m-XDA/TMC-

3/0.15 composite membrane in acetone.

Organic molecules	Molecular weight (g mol^{-1})	Molecular dimension (Å × Å)	Flux (L m^{-2} h^{-1} bar^{-1})	Rejection (%)
Disperse orange 3 (DO3)	242.23	12.38 × 6.14	5.65	75.03
Methyl orange (MO)	327.22	12.68 × 8.31	5.34	99.57
Sudan Black B (SBB)	456.54	21.28 × 9.80	6.16	96.71
Eosin Y (EY)	647.89	12.71 × 11.88	5.96	97.89
Rose Bengal (RB)	1017.64	13.62 × 13.19	5.34	99.99

Table S8
Summary of molecular weight, molecular size, permeance and rejection of m-XDA/TMC-

3/0.15 composite membrane in methanol.

Organic molecules	Molecular weight (g mol^{-1})	Molecular dimension (Å × Å)	Flux (L m^{-2} h^{-1} bar^{-1})	Rejection (%)
Disperse orange 3 (DO3)	242.23	12.38 × 6.14	2.40	66.81
Methyl orange (MO)	327.22	12.68 × 8.31	2.45	97.45
Sudan Black B (SBB)	456.54	21.28 × 9.80	2.50	98.00
Eosin Y (EY)	647.89	12.71 × 11.88	2.37	98.40
Rose Bengal (RB)	1017.64	13.62 × 13.19	2.05	98.98

Table S9
Comparison of OSN performances with other reported TFC membranes.

Membrane	Support layer	Solvent	Permeance	Maker (g mol^{-1})	Rejection	Ref.
MPD/TMC	PI	Methanol	1.5	Styrene oligomers (236)	98.0	9
		Acetone	2.4	Styrene oligomers (236)	95.0	
MPD/TMC	PI	Methanol	19.1	HNSA (246)	91.0	26
			19.1	Methyl orange (327)	96.0	
MPD/TMC	ANF	Methanol	25.0	Rose Bengal (1017)	99.8	27
		Acetone	19.0		100	
MPD/TMC	PI	Methanol	5.1	Tetracycline (444)	95.0	28
MPD/TMC	PEEK	Methanol	0.9	Styrene oligomers (236)	92.5	29
MPD-TMC (0.4% NaOH)	Ceramic support	Methanol	26.3	Acid fuchsin (586)	90.2	30
MPD/MOF/TMC	PI	Methanol	3.9	Styrene dimer (230)	96.0	31
MPD/TMC/MOFs	PI	Methanol	20.0	Tetracycline (444)	99.0	32
MPD/PIP/TMC	PAN	Methanol	6.0	Oleic acid (282)	93.0	33
		Acetone	6.0	Oleic acid (282)	92.0	
PAR-BHPF/TMC	PI	Methanol	8.0	Direct Red (314)	90.0	34
		Acetone	8.4	Styrene oligomers (236)	97.0	
CD/TMC [a]	PI	Methanol	4.9	Remazol Brilliant Blue (626)	95.7	13
CD/AMA/TMC	PMIA/PI	Methanol	29.0	Rose Bengal (1017)	98.2	35
CD/TCL	PAN	Methanol	9.4	Methyl orange (327)	91.0	36
Triazine/PIP/TMC	PAN	Methanol	10.6	Reactive Black 5 (992)	96.7	37
PEI/IPD	PP	Methanol	0.15	Brilliant Blue R (826)	88.0	38

EDA/TiO_2/IPC	PI	Methanol	27.0	BEB (624)	89.0	[39]
PEI/GO/TMC	PAN	Acetone	0.8	PEG (1000)	99.0	[40]
m-XDA/TMC-3/0.15	ANF	Methanol	5.3	Methyl orange (327)	99.6	This work
			6.8	Sudan Black B (457)	96.7	This work
		Acetone	5.2	Methyl orange (327)	99.0	This work

[a] The solvent permeance is the pure methanol permeance and the provide dye rejection is tested in ethanol.

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