Supporting Information

Enhancing CO_2-facilitated transport of PVAm membrane by the synergistic effect of porous molybdenum disulfide and mobilizable sulfonic groups

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S2.1. Materials

Polyvinylamine (PVAm, solid content of 18 wt%, Mw=60 KDa) was bought from BASF (Germany), and polyether-block-amide (Pebax-1657) was supplied by Arkema Company (France). Ammonium molybdate tetrahydrate ($(NH_4)_6Mo_7O_{24}$⑩$4H_2O$, 99%) and L-cysteine (L-Cys, 99%) were supplied by Shanghai Macklin Biochemical Co., Ltd. Polydimethylsiloxane (PDMS, 50000 mPa s) and ethyl orthosilicate (TEOS) were supplied by Jinan Xingfeilong Co., Ltd. Hydrochloric acid (HCl, 36-38 wt%) was supplied by Sinopharm Chemical Reagent Co., Ltd. Sodium hydroxide (NaOH, 96%) was supplied from Tianjin Guangfu Fine Chemical Research Institute (Tianjin, China). Thiourea (99%) and ethanol (99.7%) were supplied from Tianjin Tianli Chemical Reagent Company Co., Ltd. Sodium 3-mercapto-1-propanesulfonate (MPS, 90%) was supplied by Aladdin. Dibutyltin dilaurate (DBD, tin content: 17-19%) was purchased from Chengdu Kelong Chemical Reagent Factory. Polyvinyl alcohol (PVA, 1799) and ethanol were purchased from Sinopharm Chemical Reagent Co., Ltd. N-hexane (98%) was purchased from Tianjin Tianli Chemical Reagent Co., Ltd. The polysulfone (PSf) ultrafiltration membrane (the cut-off molecular weight of 45000) was bought from Jiuzhang Membrane Technology Co., Ltd. N_2 and CO_2 with purities of 99.999%, and the mixed gas (CO_2/N_2 = 15/85 vol%) were provided by Taiyuan Anxuhongyun Technology Development Co., Ltd. All the reagents were chemical pure and used directly without purification.

S2.2. Modification of PSf ultrafiltration membrane (mPSf)

PDMS, TEOS as the cross-linking agent, and DBD as the catalyst were dissolved in n-hexane with the mass ratio of 5:4:4, and then magnetically stirred for 30 min at 30 °C, and as-prepared pre-crosslinked PDMS solution was coated on the PSf ultrafiltration membrane with a wet film thickness of 50 μm, and then placed at 30 °C and 40% RH for 4 h to prevent the leakage and blocking pore of casting solution in mixed matrix composite membranes.[1] For further hydrophilic modification, 0.1 wt% PVA solution was coated on above as-prepared support with a wet film thickness of 50 μm, and then placed at 30 °C and 40% RH for 4 h. The as-obtained membrane was referred to as modified PSf ultrafiltration membrane (mPSf). mPSf membrane not only functions as a porous support to improve the mechanical properties of MMCMs, but also has a CO_2 permeance of 400 GPU with a CO_2/N_2 selectivity of 5.5.

S2.3. Preparation of Pebax-based MMCMs

By contrast, Pebax-based MMMs were prepared by the similar method to that of PVAm-based MMCMs except for coating solution, and the preparation of this solution was as follows. Pebax pellets were first dissolved in mixed solvent (ethanol/water=70/30 wt%) under mechanical stirring at 80 °C for 4 h to acquire 8 wt% polymer solution. Subsequently, fillers were dispersed into mixed solvent (ethanol/water=70/30 wt%) under ultrasonication at 25 °C for 1.5 h to get a uniform suspension, which was then added dropwise to 8 wt% Pebax solution. Next, it was sonicated for 1 h and then magnetically stirred for 2 h, which was repeated with 3 times to get 2.5 wt% mixed solution. Moreover, the pristine Pebax membrane and Pebax-based MMMs incorporating with MoS_2, MoS_2(L), SO_3H-MoS_2(L), MPS/MoS_2(L) and S-MoS_2(L) were prepared by a solution-casting method, which was represented as Pebax, Pebax/MoS_2, Pebax/MoS_2(L), Pebax/SO_3H-MoS_2(L), Pebax/MPS/MoS_2(L) and Pebax/S-MoS_2(L) respectively. Besides, corresponding free-supporting membranes were prepared by putting 4 wt% of casting solution into a Teflon petri dish for 12 h at room temperature, which were dried in a vacuum oven at 50 °C for another 12 h to get MMMs.

S2.4. Measurement of total water, free water and bound water for Pebax-based MMMs

The total water, free water and bound water of the membranes were measured according to the literature.[2, 3] After gas permeation test, the mass of Pebax/S-MoS_2(L) MMMs was immediately weighted and recorded as m_2, mg. Then, in order to remove the free water in the membrane, the wet membrane was dried in a vacuum oven at 100 ºC for 6 h, and the membrane was weighed again and recorded as m_1, mg. Subsequently, the membrane continued to be dried in a vacuum oven at 150 ºC for 6 h to remove bound water, and the membrane was weighed immediately and recorded as m_0, mg. The total water content (W_t, %), the free water content (W_f, %), and the bound water content (W_b, %) can be calculated by following equation 1-3 respectively.

$$W_t = \frac{(m_2 - m_0)}{m_0} \times 100\% \#(1)$$

$$W_f = \frac{(m_2 - m_1)}{m_0} \times 100\% \#(2)$$

$$W_b = \frac{(m_1 - m_0)}{m_0} \times 100\% \#(3)$$

S2.5. Characterization of filler and the membranes

UV-vis adsorption spectra of hybrids were collected with a GENESYS 180 UV–Visible Spectrophotometer (Thermo Scientific, USA) in the wavelength range of 700–190 cm^{-1} with a resolution of 6° s^{-1}. Fourier-transform infrared spectra (FTIR) and attenuated total reflection Fourier-transform infrared spectra (ATR-FTIR) of filler and membranes were collected with an ALPHA II spectrometer (Bruker, Germany) in the wavelength range of 4000–400 cm^{-1} with a resolution of 4° cm^{-1}. X-ray diffraction (XRD) patterns of all samples were recorded using a D8 X-ray diffractometer (Bruker, Germany) at a scanning rate of 4° min^{-1} over the 2θ range of 5–60°. Particle size distribution of MoS_2(L) and S-MoS_2(L) was measured by a nano measurer software from SEM images. The BET surface area and porosity of all fillers were carried out by a QUADRASORB SI surface area and porosity analyzer (Quantachrome, USA) at 77 K. The CO_2 adsorption isotherms were measured using an NOVA 1000e sorption analyzer (Quantachrome, USA) at 298 K. Electron paramagnetic resonance (EPR) spectra was applied by Bruker A300 (Bruker, Germany) to detects S vacancies in materials. Zeta potentials patterns of samples were recorded using a Nexis GD-2030 (Malvern Instruments, UK). Thermogravimetric analysis (TGA) was performed by a STA 449 F3 thermogravimetric analyzer (Netzsch, Germany) with a heating rate of 10 °C min^{-1} in temperature range of 50–700 °C under N_2 atmosphere.

S2.6. Gas permeation experiments

The gas permeation properties of membranes were determined using the laboratory-made equipment described in the literature,[4] and measured using the constant pressure/variable volume method as reported in our article.[5] Test conditions and procedures can be detailed in our article.[6] The effective area of the membrane used in the test cell was about 12.56 cm^2. H_2 was used as the sweep gas and kept at atmospheric pressure. CO_2/N_2 mixed gas (CO_2/N_2=15/85 vol%) and pure CO_2 and N_2 (99.999%) were applied as a feed gas. To understand the gas separation performance of PVAm/S-MoS_2(L) in the acidic environment, we injected H_2SO_4 into the humidifier to simulate the flue gas environment, where sulfuric acid solution of different pH values was controlled by adding an amount of 0.01 mol L^{-1} H_2SO_4 and the pH was measured with pH paper. The flow rate of permeate gas was measured using a soap bubble flowmeter, of which the compositions were analyzed using a gas chromatograph (Shimadzu, GC-2014C, Japan) equipped with a thermal conductivity detector. Permeance is used to evaluate the permeance performance of mixed matrix composite membranes, which is equal to the quotient of volumetric flow rate divided by the transmembrane-pressure difference and membrane area. Permeance involves the effect of film thickness, which can directly reflect the actual permeability of the membrane [7]. The permeance of each gas is calculated using Eq. (4):

$$R_{\mathrm{i}} = \frac{Q_{\mathrm{i}}}{\Delta P_{\mathrm{i}} \cdot A} \#(4)$$

where, R_{i} is the permeance of the gas ‘i’ (GPU, 1 GPU = 10^{-6} $\mathrm{cm^3}$ (STP) $\mathrm{cm^{-2}}$ $\mathrm{s^{-1}}$ $\mathrm{cmHg^{-1}}$). Q_{i} is the volumetric flow rate of the gas ‘i’ ($\mathrm{cm^3}$ (STP) $\mathrm{s^{-1}}$); ΔP_{i} is the transmembrane-pressure difference (cmHg) of the gas ‘i’, and A is the effective membrane area ($\mathrm{cm^2}$). The ideal selectivity (α_{ij}) of the pure gas is calculated using Eq. (5):

$$\alpha_{\mathrm{ij}} = \frac{R_{\mathrm{i}}}{R_{\mathrm{j}}} \#(5)$$

The mixed-gas separation selectivity of gases ‘i and j’ (α_{ij}^{*}) is calculated using Eq. (6):

$$\alpha_{\mathrm{ij}}^{*} = \frac{y_{\mathrm{i}}/y_{\mathrm{j}}}{x_{\mathrm{i}}/x_{\mathrm{j}}} \#(6)$$

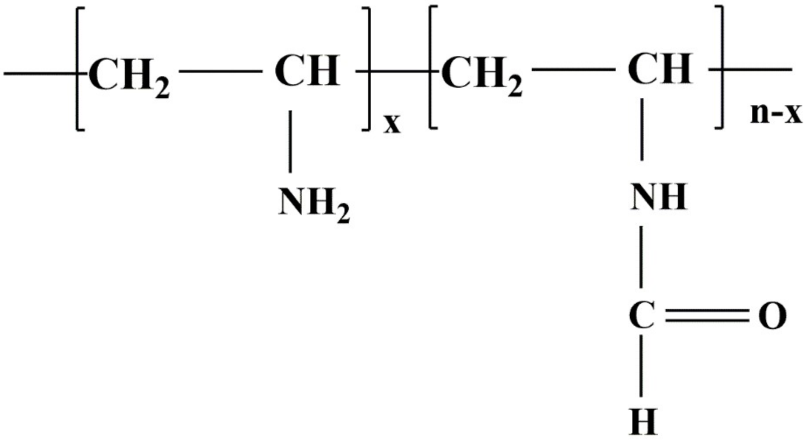

Fig. S1. Chemical structure of PVAm.

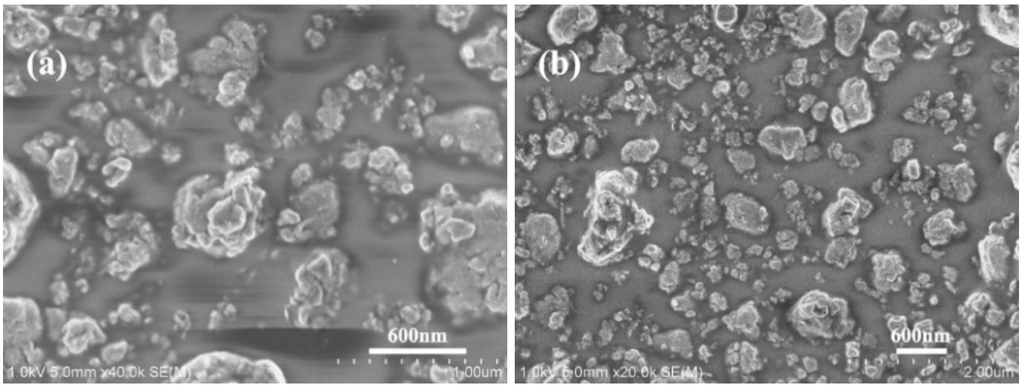

Fig. S2. SEM images of (a, b) S-MoS_2(L) with high magnification.

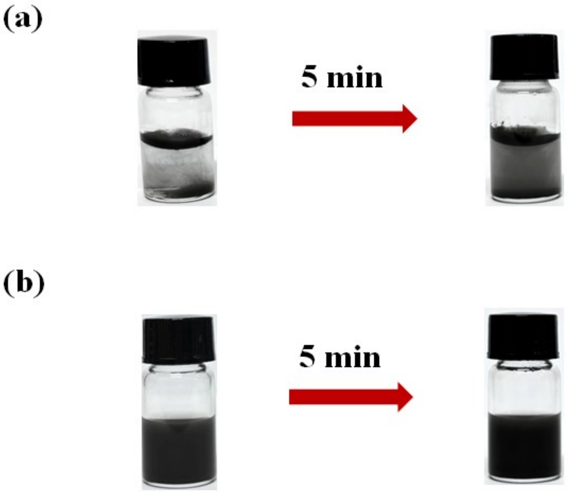

Fig. S3. The optical photograph of the dispersibility of (a) MoS_2(L) and (b) S-MoS_2(L).

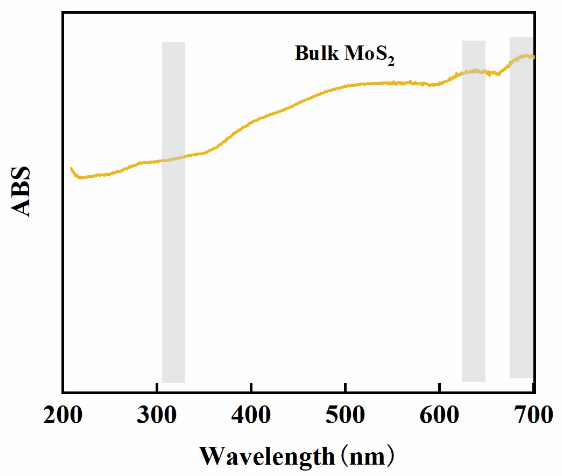

Fig. S4. UV-vis adsorption spectra of 2H-MoS_2.

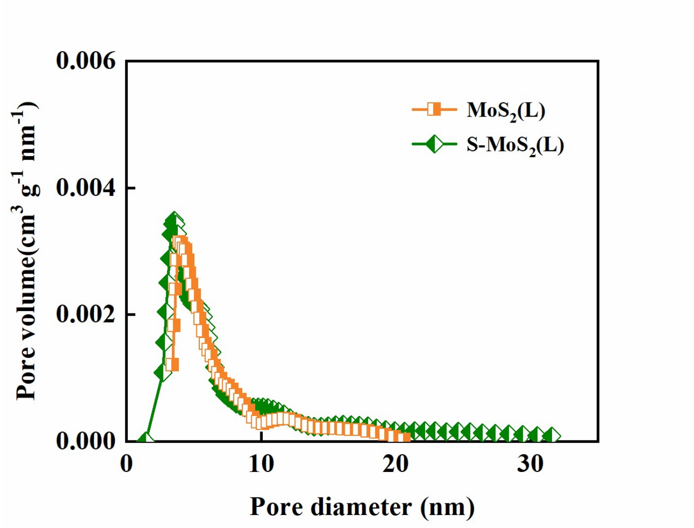

Fig. S5. Pore size distribution of MoS$_2$(L) and S-MoS$_2$(L).

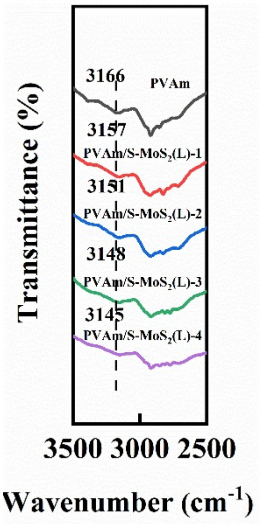

Fig. S6. The partially magnified ATR-FTIR spectra of the pristine PVAm membrane and PVAm/S-MoS$_2$(L).

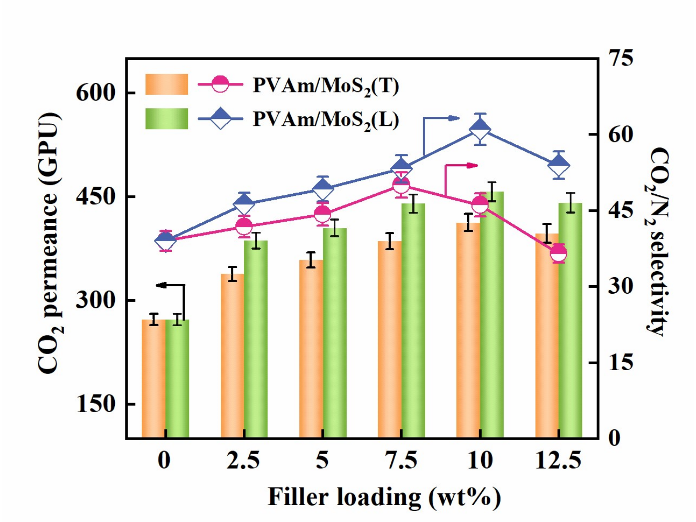

Fig. S7. Effects of MoS_2 synthesized by different sulfur source on CO_2 permeance and CO_2/N_2 selectivity of MMCMs (Temperature: 25 °C; feed gas pressure: 1 bar; relative humidity: 100%; pH value: 7).

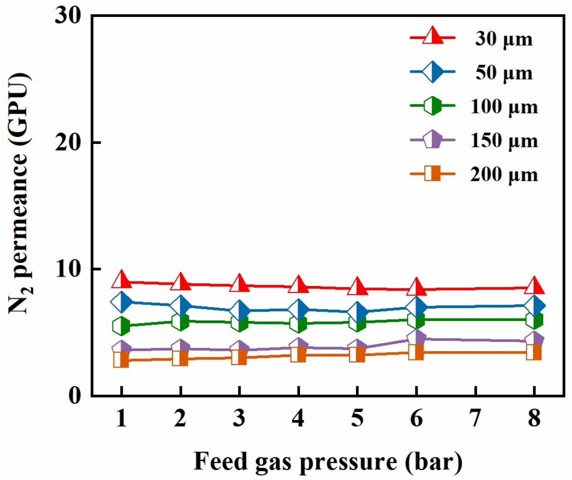

Fig. S8. The effect of the wet film thickness of the selective layer on N_2 permeance of MMCMs (Temperature: 25 °C; relative humidity: 100%; pH value: 7).

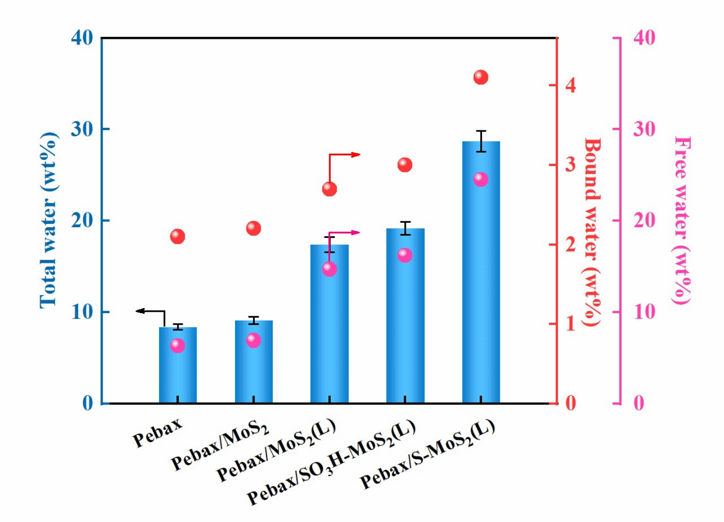

Fig. S9. The content of total water, free water and bound water of the pristine Pebax membrane and Pebax-based MMMs doped with different fillers.

Table S1. Porous structure parameters of MoS_2(L) and S-MoS_2(L).

Samples	BET surface area (m^2 g^{-1})	Total pore volume (cm^3 g^{-1})	Average pore diameter (nm)
MoS_2(L)	10.6	0.011	4.57
S-MoS_2(L)	13.9	0.016	5.53

Table S2. Thermogravimetric characteristic parameters of the membranes.

Membranes	First weight loss stage (°C)	Second weight loss stage (°C)	T_{onset} (°C)	$T_{max}{}^{a}$ (°C)	$T_{max}{}^{b}$ (°C)	T_{d5} (°C)
PVAm	164–290	290–532	181	235	337	169
PVAm/S-MoS_2(L)-1	166–293	293–536	180	232	383	182
PVAm/S-MoS_2(L)-3	168–289	289–541	179	231	340	193
PVAm/S-MoS_2(L)-4	163–280	280–524	164	229	350	185

Note: T_{onset} is the temperature of intersection point between tangent and baseline, $T_{max}{}^{a}$ is the temperature at which the rate of weight loss reaches a maximum in the first weight loss stage, $T_{max}{}^{b}$ is the temperature at which the rate of weight loss reaches a maximum in the second weight loss stage; T_{d5} is the temperature, when the weight loss of the sample reaches 5%.

Table S3. The content of total water, free water and bound water of the pristine Pebax membrane and MMMs doped with different fillers.

Membranes	Total water (W_t, %)	Free water (W_f, %)	Bound water (W_b, %)
Pebax	8.4	6.3	2.1
Pebax/MoS_2	9.1	6.9	2.2
Pebax/MoS_2(L)	17.4	14.7	2.7
Pebax/SO_3H-MoS_2(L)	19.2	16.2	3.0
Pebax/S-MoS_2(L)	28.7	24.6	4.1

Table S4. Comparison of CO_2/N_2 separation performance with previously reported PVAm-based MMCMs.

Membranes	Thickness（μm）	Mixed matrix membranes			Pristine membrane			Enhancement		Test conditions	Ref.
		CO_2 permeance (GPU)	CO_2 permeability (Barrer)	CO_2/N_2 selectivity	CO_2 permeance (GPU)	CO_2 permeability (Barrer)	CO_2/N_2 selectivity	CO_2 permeance (%)	CO_2/N_2 selectivity (%)		
PVAm/MK	2.37	188	443	78	74	196	42	154	85.7	Pure gas 1 bar, 25 ºC	4
CS-PVAm/HPEI-GO	3.5	36	108.5	107	14	31.5	76	157.1	40.7	CO_2/N_2(10/90 vol%) 1 bar, 25 ºC	8
PVAm/PANI@CNTs-GO	0.73	170	124.1	122.4	73	53.3	45.3	132.9	170.2	Pure gas 1 bar, 25 ºC	5
PVAm/COF$_P$	0.15	1789	268.3	89	1400	210	54	27.8	64.8	Pure gas 5 bar, 25 ºC	1
PVAm/MWCNTs	~0.25	195	49	39	132	33	37.7	47.7	3.4	Pure gas 1.1 bar, 25 ºC	9
PVAm/SiO_2-NH_2/PS	~0.25	229	57.2	45.8	132	33	37.7	73.5	21.5	Pure gas 1.1 bar, 25 ºC	9
PVAm/MWCNT-NH_2	~0.28	174	48.7	74	89	40	60	95.5	23.3	Pure gas 1.1 bar, 25 ºC	9
PVAm/ZSM-5-NH_2/PS	~0.25	205	51.1	42.7	132	33	37.7	55.3	13.4	Pure gas 1.1bar, 25 ºC	9
PVAm/ZIF-8-d-MK	1.85	169	312	86.7	76	144.4	42	122	106	Pure gas 1 bar, 25 ºC	10
PVAm/HT	0.25	3187	796.75	296	419	104.7	68.8	660.0	330.2	CO_2/N_2(15/85 vol%) 1 bar, 25 ºC	11

Membranes	Thickness（μm)	Mixed matrix membranes			Pristine membrane			Enhancement		Test condition	Ref.
		CO_2 permeance (GPU)	CO_2 permeability (Barrer)	CO_2/N_2 selectivity	CO_2 permeance (GPU)	CO_2 permeability (Barrer)	CO_2/N_2 selectivity	CO_2 permeance (%)	CO_2/N_2 selectivity (%)		
PVAm/PANI NS	0.28	1200	336	120	26	35.3	50.6	852	137	CO_2/N_2(20/80 vol%) 1.1 bar, 25 ºC	12
PVAm/ASU-1	1.2	721	865.2	71.33	380	456	20	89.7	256.6	CO_2/N_2(15/85 vol%) 1.5bar, 25 ºC	13
PVA/PE/ZIF-8	4	82	328	370	45	180	210	82.2	76.2	CO_2/N_2(20/80 vol%) 2.5 bar, 95 ºC	14
PVAm/S-MoS_2(L)	0.326	554	181	74	272	64	39	104	90	Pure gas 1 bar, 25 ºC	This work

Table S5. Comparison of CO_2/N_2 separation performance with previously reported MMMs embedded with lamellar fillers.

Membranes	Thickness（μm）	Mixed matrix membranes			Pristine membrane			Enhancement		Test conditions	Ref.
		CO_2 permeance (GPU)	CO_2 permeability (Barrer)	CO_2/N_2 selectivity	CO_2 permeance (GPU)	CO_2 permeability (Barrer)	CO_2/N_2 selectivity	CO_2 permeance (%)	CO_2/N_2 selectivity (%)		
Pebax/COF-5	60	---	493	49.3	---	98	31.3	403	57.5	Pure gas 1 bar, 30 ºC	15
Pebax/ZIF-67 NS	77	---	139.4	73.2	---	92.5	41.6	50.7	75.9	Pure gas 1 bar, 25 ºC	16
Pebax/Cys-MoS_2	40	---	297	120	---	97	53.4	206	124. 7	Pure gas 5 bar, 25 ºC	17
Pebax-2.5-CuBDC-ns@10MoS_2		---	123	69	---	86.3	38.3	42.5	80.1	Pure gas 4 bar, 35 ºC	18
Pebax/0.25DCN-4/PAN	1.84	33.3	61.2	67.2	29	53.4	35	14.6	92	Pure gas 3 bar, 25 ºC	19
Pebax/Mxene-10	2	---	584	59	---	210	38	178	55.3	Pure gas 2bar, 30 ºC	20
Pebax/PRG	5.25	---	119	104	---	60	56	98.3	85.7	Pure gas 2bar, 30 ºC	21
Pebax-PCN-10	65	---	613.4	60.89	---	301.9	53.8	103	13	CO_2/N_2(10/90 vol%) 1bar, 25 ºC	22
0.5wt%MCN/Pebax	50	---	123	76	---	80.3	59	53	28.8	Pure gas 4 bar, 25 ºC	23
Pebax/ZIF-8@BNNS	80	---	106.5	83.2	---	80.97	47.9	31.5	73.7	Pure gas 3 bar, 30 ºC	24

Membranes	Thickness（μm)	Mixed matrix membranes			Pristine membrane			Enhancement		Test condition	Ref.
		CO_2 permeance (GPU)	CO_2 permeability (Barrer)	CO_2/N_2 selectivity	CO_2 permeance (GPU)	CO_2 permeability (Barrer)	CO_2/N_2 selectivity	CO_2 permeance (%)	CO_2/N_2 selectivity (%)		
SHPAA/PVA/PGO/PVDF	0.185	607	112.3	36	383	70.8	55	58.5	-34.5	CO_2/N_2(10/90 vol%) 1 bar,35 ºC	25
Pebax/TpPa-1-nc/PVDF	2.6	7.4	19.2	64	4.5	11.7	41	64.1	56.1	Pure gas 3 bar, 25 ºC	26
MoS2/Pebax/PDMS/PSf	3.71	---	67	90	---	38.8	44	72	104	Pure gas 2bar, 30 ºC	27
Pebax/5MoS_2	3.71	---	67	91	---	46	45	45	102	Pure gas 2bar, 30 ºC	28
Pebax/A-rGO	2.98	---	65.2	95.9	---	58	41.1	12.4	133	Pure gas 4bar, 25 ºC	29
AMT/PSf	0.098	830	81.2	120	---	---	---	---	---	CO_2/N_2(15/85 vol%) 3 bar,50 ºC	30
PVAm/S-MoS_2(L)	0.326	554	181	74	272	64	39	104	90	Pure gas 1 bar, 25 ºC	This work

Table S6. Comparison of the long-term stability of PVAm/S-MoS_2(L) with previously reported MMMs.

Membranes	Test conditions	CO_2 permeance（GPU）	CO_2/N_2 selectivity	Testing time (hour)	Ref.
PVAm/PANI-c	CO_2/N_2 (20/80 vol%) 3 bar, 25 °C	492	71	300	12
VA-co-VAm/MWCNTs-NH_2	CO_2/N_2 (20/80 vol%) 5.4 bar, 28 °C	110	70	110	31
PVAm/PEG-UIO-66-NH_2	CO_2/N_2 (15/85 vol%) 3 bar, 25 °C	1263	91	100	32
SHPAA-PVA/MFC-PEG4	CO_2/N_2 (10/90 vol%) 1.7 bar, 25 °C	652	41	175	33
PVAm/EDA	CO_2/N_2 (20/80 vol%) 1.5 bar, 22 °C	530	110	300	34
PVAm/ZIF-8-d-MK	CO_2/N_2 (15/85 vol%) 1 bar, 25 °C	168.6	86.7	360	10
PVAm/PANI@CNTs-GO	CO_2/N_2 (15/85 vol%) 1 bar, 25 °C	172.1	122.4	360	5
PVAm/MK	CO_2/N_2 (15/85 vol%) 1 bar, 25 °C	150	78	360	4
PVA/ProK-40	CO_2/N_2 (10/90 vol%) 2 bar, 25 °C	790	39	18.5	35
Cs-PVAm/HPEI-GO	CO_2/N_2 (10/90 vol%) 1 bar, 25 °C	31.5	106	100	8
SHPAA/PVA/PGO	CO_2/N_2 (10/90 vol%) 1 bar, 35 °C	607	36	14	25
PVAm/ZIF-8@NENP-NH_2	CO_2/N_2 (15/85 vol%) 1 bar, 25 °C	301	91	360	36
PVAm/S-MoS_2(L)	CO_2/N_2 (15/85 vol%) 1 bar, 25 °C	680	82	360	This work

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