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Supporting Information

Two-Dimensional Cationic Covalent Organic Framework Membrane for Selective Molecular Sieving

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Experimental Section

1. Materials and reagents

General information: Unless otherwise noted, all chemical reagents and solvents from commercial sources were received and used without further purification. Twice-distilled water was used throughout all experiments. Solvents and reagents for synthesis were purchased from Sigma-Aldrich and Beijing chemical factory. It includes the following reagents, phloroglucinol, hexamethylenetetramine (HMTA), trifluoroacetic acid, ethidium bromide (EB) (3,8-diamino-5-ethyl-6-phenyl -phenanthridinium bromide), p-toluenesulfonic acid (PTSA), methyl orange (MO), fluorescein sodium salt (FSs), potassium permanganate (PP), nile red (NR), calcein (CA), p-nitroaniline (NA), rhodamine B (RB), methylene blue (MB), n, ndimethyl-p-phenylenediamin dihydrochloride (DMPD), Sodium bromide (NaBr). Analysis of pure: Iodine, Potassium iodide, Barium chloride. The average molecular weight is 200, 400, 600, 800, 1000 and 1500 Da Polyethylene glycol (PEG), analysis pure. N,N-dimethyl formamide, hydrochloric acid, dichloromethane, acetone, methanol, anhydrous ethanol.

2. Apparatus and conditions

X-ray diffraction (XRD) measurement was carried out on a Riguku D/MAX2550 diffractometer with Cu-K a radiation operating at a voltage of 50 kV and a current of 200 mA. FTIR spectra were performed using an IFS 66V/S Fourier trans-form infrared spectrometer. Scanning electronic microscopy (SEM) images were recorded by a Hitachi S-4800 microscope. Atomic force microscopy (AFM) images were captured on a Multimode 8 (Bruker Corporation, Santa Barbara, CA, USA) in tapping mode under ambient conditions. Transmission electron microscopy (TEM) images were measured on a JEM-2010 field-emission transmission election microscope made by Japanese JEOL Company. The UV-vis absorption spectra of different dye solutions were performed with a Shimadzu UV-3101 PC

spectrophotometer. The N_2 adsorption/desorption isotherms were measured on a Quantachrome Autosorb iQ₂ analyzer. N_2 adsorption/desorption measurements were carried out at 77 K. Ultra-high-purity grade N_2 gas was used for adsorption measurements. Liquid nitrogen bath was utilized to control the temperature at 77 K.

3. Methods

3.1. Synthesis of 1,3,5-triformylphloroglucinol (TFP).

1,3,5-triformylphloroglucinol (TFP) was synthesized as described in the literature.^[S1] To hexamethylenetetramine (HMTA) (15.098 g, 108 mmol) and dried phloroglucinol (6.014 g, 47.7 mmol) under N₂ was added 90 mL trifluoroacetic acid (TFA). The solution was heated at 100 °C for ca. 2.5 h. Approximately 150 mL of 3 M HCl was added and the solution was heated at 100 °C for 1 h. After cooling to room temperature, the solution was filtered through filter paper, and then extracted with ca. 150 mL dichloromethane, repeated the process three times. Finally, the solution of extraction was dried over magnesium sulfate and filtered further. Rotary evaporation of the solution, an off-white powder was obtained. Yield 1.12 g (5.3 mmol, 11.2%).



Scheme S1. Synthesis of 1,3,5-triformylphloroglucinol (TFP).

3.2 Synthesis of EB-COF:Br nanosheets.

EB-COF:Br nanosheets were prepared according to the previous literature reported methods with little modified.^[S2] In the typical route of interfacial synthesis, EB-COF:Br nanosheets were synthesized in a glass beaker. In the first place, 0.1 mmol of 1,3,5-triformylphloroglucinol (TFP) (21.0 mg) dissolved in 130 mL of dichloromethane was poured

into the beaker. Then a spacer layer of 80 mL of deionized water was added on surface of the Tp solution lightly. Finally, 0.15 mmol of ethidium bromide (EB) (3,8-diamino-5-ethyl-6-phenylphenanthridinium bromide, 59.2 mg) and PTSA (amine-p-toluene sulfonic acid) (0.3 mmol, 57.1 mg) were dissolved in 130 mL of water and added slowly on top of the spacer solution over a period of 50 min. The system was kept at 35°C for 10 days in undisturbed condition. The nanosheets formed at the interface were collected by removing the top aqueous layer with a dropper and washed with deionized water, N,N-Dimethylformamide (DMF), and acetone to purify the EB-COF:Br nanosheets. The yields of the EB-COF:Br nanosheets was 38%. A schematic of the fabrication steps is shown in Scheme 1d, and the photographs of interfacial synthesis of EB-COF:Br can see Figure S1 (Supporting Information).

3.3 Post-fabrication of EB-COF:Br membrane.

As a simple and feasible method, the layer-by-layer restacking approach of nanosheets was widely used in the process of membrane preparation. Here, EB-COF:Br membrane was obtained by layer-by-layer restacking of the EB-COF:Br nanosheets dispersion liquid onto a nylon 66 support via a vacuum filtration system. Then, the EB-COF:Br membrane was placed in acetone before the following characterizations and molecular sieving test. A schematic of the fabrication steps is shown in Scheme 1e.

4. Evaluation the selective sieving performance of the EB-COF:Br membrane

4.1. Solvent flux measurements.

In order to evaluate the solvent flux of EB-COF:Br membrane to pure solvents of different polarity and molecular weight. A series of EB-COF:Br membranes with a diameter of 2.5 cm were placed on sand core filter unit (1.77 cm² active area) and all solvents permeation were performed under 0.5 bar downstream negative pressure. Solvent flux values reported were the average of triplicate experiments conducted with three different samples.^[S2]

Solvent flux (J) was calculated by measuring permeate volume (V) per unit area (A) per unit time (t) according to the following equation:

 $J = V / A \bullet t$ in liters per square meter hour (L m⁻² h⁻¹)

Permeance (P) was calculated according to the following equation:

 $P = V/A \cdot t \cdot \Delta p$ in liters per square meter hour bar (L m⁻² h - 1bar-1).

Moreover, the effect of film thickness to flux was evaluated via measuring deionized water flux through EB-COF:Br membrane with different thickness.

4.2. Polyethylene glycol (PEG) based molecular weight cut-off (MWCO) analysis.

The MWCO rejection analysis of EB-COF:Br membrane was performed using PEG of different molecular weights. These solutes with the average molecular weight of 200, 400, 600, 800, 1000 and 1500 Da were used. Feed concentrations of these PEG solutions were 0.1%, as this concentration is commonly being used for the rejection analysis.

MWCO analysis was performed by filtering 0.1% solution of PEG through EB-COF:Br membrane. Typically, a 20 mL of PEG solution was charged to a sand core filter unit with EB-COF:Br membrane, and the filtrate was collected at 0.5 bar pressure while maintaining 300 r.p.m as the stirring speed. In the process of filtering, the initial 0.5 mL of the filtrate was discarded and 5 mL was collected for the rejection analysis.

In this work, spectrophotometry, as an indirect method, was taken to determine the concentration of PEG in the solution. First, the standard solutions of BaCl₂ with quality fraction of 5% and I₂ with molarity of 0.05 mol/L, as chromogenic agents for PEG, were prepared. When BaCl₂ and I₂ standard solutions were added in a PEG solution, there is a complex reaction between PEG and Ba²⁺ in aqueous solution, then the complex (PEG-Ba²⁺) further form a reddish-brown compound (PEG-Ba²⁺-I₃⁻) with I₃⁻, as clearly shown in Scheme S2. The wavelength of the compound maximum absorbance is 620 nm. And the absorbance

intensity of the compound is positively correlated with the concentration of PEG solution. Therefore, we can obtain a series of PEG standard curve of absorbance-concentration by measuring the absorbance of compound (PEG-Ba²⁺-I₃-) derived from different concentrations and average molecular weights of PEG. Finally, spectrophotometry was used to measure the concentrations of PEG (different average molecular weight) in the feed and filtrate sample by using a double-beam ultraviolet-visible spectrometer (Shimadzu UV-3101). Whereafter, the concentration of PEG solution was calculated via corresponding standard curve of absorbance-concentration.

It is important to note that when we used the same one membrane to do a series of MWCO rejection analysis, before charging the sand core filter unit by another PEG solution, the EB-COF:Br membrane were washed with 0.1 M NaOH solution in order to minimize the fouling. This was performed by charging the sand core filter unit with 30 mL of NaOH solution and stirring for 10 min at ambient pressure, followed by collecting 5 mL of the permeate at 0.5 bar downstream pressure; so that the PEG adsorbed on the membrane as well as on the pore surface can be removed. This was followed by water wash in a stirred filter unit itself, till the filtrate was neutral to pH.

The percent rejection was calculated by using Equation 1.^[S3] The MWCO of a membrane was deduced from the characteristic rejection at 90 %.

$$HO(CH_2CH_2O)_nH \xrightarrow{Ba^{2+}} [HO(CH_2CH_2O)_nH]^{2+} \xrightarrow{I_3^-} [HO(CH_2CH_2O)_nH]^{2+} 2I_3^-$$

Scheme S2. Chromogenic mechanism of PEG (polyethylene glycol) with chromogenic agents.

Rejection efficiency (%) = $(C_0-C_1)/C_0 \times 100$ (1)

where C_0 and C_1 were the concentration of PEG at initial condition and in the filtrate, respectively.

4.3 Selective Dye Sieving Experiments.

All the batch adsorption experiments were conducted at room temperature. Initial dye concentrations were fixed to be 50 µM. Dye rejection was performed by filtering 50 µM solution of dye through EB-COF:Br membrane at 0.5 bar pressure. Typically, the initial 8 mL dye solution of the filtrate was collected for the rejection analysis. Dye concentration at initial condition and in the filtrate was monitored using a double-beam ultraviolet-visible spectrometer (Shimadzu UV-3101) at a wavelength of maximum absorbance [462 nm for Methyl Orange (MO); 487 nm for Fluorescein Sodium salt (FSs); 524 and 543 nm for Potassium Permanganate (PP); 263 and 552 nm for Nile Red (NR); 224 nm for Calcein (CA); 369 nm for p-Nitroaniline (NA); 553 nm for Rhodamine B (RB); 663 nm for Methylene Blue (MB); 237 nm for N,N-Dimethyl-p-phenylenediamin dihydrochloride (DMPD)]. The rejection efficiency of dye was calculated as follows:

Rejection efficiency (%) = $(C_0 - C_1)/C_0 \times 100$ (1)

Where C_0 and C_1 were the concentration of dye at initial condition and in the filtrate, respectively.

4.4. The time course of dyes rejection and permeate flux.

The time course of rejection and permeate flux experiment of EB-COF:Br membrane for dyes was carried out as follows.

In order to elucidate the influence of dye molecules on the permeation flux of solution in filtration process. The time course of dyes rejection and permeation flux analysis was performed by filtering 50 μ M solution of dye through EB-COF:Br membrane. Typically, a 16 mL of dye solution was charged to a sand core filter unit with EB-COF:Br membrane and the filtrate was collected at intervals of two minutes. Then the volume of each filtrate collected at different points in time was measured accurately, and the absorbance of each filtrate was also measured. Finally, the time course of dyes rejection and permeation flux was obtain.

All dye solutions filtration were performed under 0.5 bar downstream negative pressure while maintaining 300 r.p.m as the stirring speed. Solvent flux values reported were the average of triplicate experiments conducted with three different EB-COF:Br membrane samples.

4.5. Cycle study of EB-COF:Br membrane for dyes interception efficiency.

A cycling experiment of EB-COF:Br membrane for MO interception was carried out as follows.

In the beginning, a anionic MO solution was filtered through EB-COF:Br membrane and the filtrate was collected for rejection analysis. Then the membrane, after intercepting anionic MO, was rinsed thoroughly with aqueous NaBr solution (2.0 mol L⁻¹) at room temperature. Subsequently, the filtration experiment of EB-COF:Br membrane for MO solution was repeated several times using the same membrane under the same conditions as above. Finally, these filtrates were monitored using a double-beam ultraviolet-visible spectrometer to evaluate the rejection efficiency of cycle experiment.



Fig. S1 Optical images of progress of interfacial synthesis of EB-COF:Br nanosheets at different points in time. (a) 0 h, (b) 12 h, (c) 24 h, (d) 36 h, (e) 48 h, (f) 60 h.



Fig. S2 (a) FT-IR spectra of EB-COF:Br, triformylphloroglucinol (TFP), and ethidium bromide (EB); (b) Amplifying view of FT-IR spectra in the range of 1800 - 500 cm⁻¹.



Fig. S3 (a) Top view and (b) side view of the reversed slipped AA-stacking mode structure of EB-COF:Br.



Fig. S4 Pore size distribution of EB-COF:Br membrane. The pore size and its distribution of EB-COF:Br were evaluated by using the nonlocal density functional theory method based on the model N_2 at 77 K on carbon.

Membrane Name	Membrane Thickness	Protic solvents permeance (L m ⁻² h ⁻¹ bar ⁻¹)						
		Water	Methanol	Ethanol	n-Propanol	n-Butanol	1-Pentanol	n-Hexanol
EB-COF:Br	189 μm	546	1272	564	477	378	248	166

Table S1: The permeability of EB-COF:Br membrane to pure solvent (Protic).

Table S2: The permeability of EB-COF:Br membrane to pure solvent (Aprotic).

Membrane Name	Membrane Thickness	Aprotic solvents permeance (L m ⁻² h ⁻¹ bar ⁻¹)				
		Acetonitrile	Acetone	Tetrahydrofuran	1,4-Dioxane	N,N-Dimethylacetamide
EB-COF:Br	189 µm	2095	2640	1532	973	565

Membrane type	Water/ Acetonitrile flux	Value (L m ⁻² h ⁻¹)	References
PBI	Acetonitrile Water	4 4	Ref. S4
PMMA	Water	45	Ref. S5
NTR 7410	Water	31.5-60.0	Ref. S6
mLbL-PA	Water	21.5	Ref. S7
(PDDA/PAA)/PAN	Acetonitrile	2	Ref. S8
Graphene	Water	51	Ref. S9
MPF-50	Water	10	Ref. S10
PA	Acetonitrile	112	Ref. S11
PSF Composite	Water	86	Ref. S12
DBX cross-linked PBI	Acetonitrile Water	37 12	Ref. S13
M-TpTD	Acetonitrile Water	278 118	D 450
M-TpBD	Acetonitrile Water	180 92	Ref.S3
COF-LZU1 membrane	Water	760	Ref.S14
Graphene	Acetonitrile Water	13.1 4.1	Ref.S15
Tp-Bpy thin film	Acetonitrile Water	339.5 211.5	Ref.S2
Tp-Azo thin film	Acetonitrile Water	70.3 45.9	
EB-COF:Br membrane	Acetonitrile Water	2095 546	This work

 Table S3: Current State of Art: Water and Acetonitrile flux value against different NF

 membrane.

Table S4: Comparison of water flux of EB-COF:Br membrane of different thickness.

Thickness of wet membrane	Thickness of dry membrane (from SEM)	Water flux $(L m^{-2} h^{-1} bar^{-1})$
146 μm	98 μm	756
195 μm	127 μm	531
240 μm	156 μm	480



Fig. S5 Water permeation flux of GO membrane as a function of time. The membrane was prepared using filtration with thickness about 3.6 μm.



Fig. S6 Water permeation flux of EB-COF:Br membrane as a function of time. The membrane was prepared using filtration with thickness about $308 \mu m$.



Fig. S7 Dye molecules used in molecular sieving experiments based on sizes and charges. Molecular models were displayed in space-filling style (gray, carbon; red, oxygen; white, hydrogen; blue, nitrogen; purple, manganese; yellow, sulfur); (a) Methyl Orange (MO) with a size of 5.19×14.62 Å, (b) Fluorescein Sodium salt (FSs) with a size of 9.64×10.34 Å, (c) Potassium Permanganate (PP) with a size of 4.00 Å, (d) Nile Red (NR) with a size of 6.51×14.12 Å, (e) Calcein (CA) with a size of 8.82×17.55 Å, (f) p-Nitroaniline (NA) with a size of 4.32×6.89 Å, (g) Rhodamine B (RB) with a size of 11.81×15.31 Å, (h) Methylene Blue (MB) with a size of 5.63×14.21 Å, (i) N,N-Dimethyl-p-phenylenediamin dihydrochloride (DMPD) with a size of 4.35×7.93 Å.

Dye molecule	Charge	λmax	MW	Molecule size	
-	Ū	(nm)	(g/mol)	(Å)	
Methyl Orange (MO)	-	462	327.3	5.19 × 14.62	
Fluorescein Sodium salt (FSs)	-	488	376.3	9.64 × 10.34	
Potassium Permanganate (PP)	-	524	158.0	4.00×4.00	
Nile Red (NR)	neutral	553	318.4	6.51 × 14.12	
Calcein (CA)	neutral	225	622.5	8.82 × 17.55	
p-Nitroaniline (NA)	neutral	369	138.1	4.32 × 6.89	
Rhodamine B (RB)	+	553	479.0	11.81 × 15.31	
Methylene Blue (MB)	+	664	319.9	5.63 × 14.21	
N,N-Dimethyl-p- phenylenediamin dihydrochloride (DMPD)	+	234	209.1	4.35 × 7.93	

 Table S5. Water- and methanol-soluble dye molecules and their properties.

 Table S6. Performance comparison among various membranes/materials towards anionic

 dyes rejection.

Membranes/materials	Dye Molecules	Charge	Molecule	Rejectio	Referenc	
type			size (nm)	П %о	es	
	Rose bengal (RB)	-	1.2 × 1.54	99		
M-TpBD	Congo red (CR)	-	0.75 × 1.9	96	Ref. S3	
M-TpTD	Rose bengal (RB)	-	1.2 × 1.54	84		
	Congo red (CR)	-	0.75 × 1.9	81	-	
TnBny thin-film	Brilliant Blue-G (BB)	-	_	94		
	Congo Red (CR)	-	0.75 × 1.9	80		
	Acid Fuchsin (AF)	-	1.19 × 1.14	97	Ref. S2	
TpAzo thin-film	Brilliant Blue-G (BB)	-	_	90	-	
	Congo Red (CR)	-	0.75 × 1.9	79		
	Acid Fuchsin (AF)	-	1.19 × 1.14	99		
PyTTA-BFBIm-iCOF	Methyl Orange (MO)	-	0.52 × 1.46	99		
	indigo carmine acid blue 74 (IC-74)	-	_	98.5	Ref. S16	
Alumina tube	Chrome black T	-	1.55 × 0.88	98.2	Dof S14	
supported COP-LZOT	Congo red	-	0.75 × 1.9	98.6		
	Acid Fuchsin	-	1.19×1.14	91.4	-	
	Rose Bengal	-	1.2×1.54	99.1		
PEI/CMCNa/PP	Congo red	-	0.75×1.9	99.4	Ref. S17	
DEA-Modified PA-TFC	Congo red	-	0.75 × 1.9	99.6	Ref. S18	
PAA/PVA/GA	Congo red	-	0.75 × 1.9	96	Ref. S19	
ZIF-8/PA	Congo red	-	0.75 × 1.9	99.98	Ref. S20	
PVDF-SAN-60	Congo red	-	0.75 × 1.9	97.7	Ref. S21	
Ceramic NF	Chrome black T	-	1.55×0.88	>96.8	Ref. S22	
PSF-PEG	Acid blue	-	1.02×1.0	98	Ref. S23	
ZIF-8/PES	Rose Bengal	-	1.2×1.54	98.95	Ref. S24	
F127/PES	Alcian blue	-	_	95.7	Ref. S25	
PDDA/PSS	Methyl blue	-	2.36 × 1.74	92	Ref. S26	
CMCNa/PP	Methyl blue	-	2.36 × 1.74	99.75	Ref. S27	
ZIF-12/PAN	Methyl blue	-	2.36 × 1.74	99.4	Ref. S28	
PEI-GO/PAA/PVA/GA	Methyl blue	-	2.36 × 1.74	99.3	Ref. S29	
ZIF-8/PSS	Methyl blue	-	2.36 × 1.74	98.6	Ref. S30	
PVDF/nanoclay/chito	Methyl blue	-	2.36 × 1.74	75	Ref. S31	

	Methyl Orange (MO)	-	0.52 × 1.46	99.6	
EB-COF:Br membrane	Fluorescein Sodium salt (FSs)	-	0.96 × 1.03	99.2	This work
	Potassium Permanganate (PP)	-	0.40 × 0.40	98.1	



Fig. S8 The time course of permeation flux for Mixed dye (FSs + CA + MB).



Fig. S9 (a) Photograph schematic of the selective molecular sieving of Fluorescein Sodium salt (FSs) from a mixture of Fluorescein Sodium salt (FSs), Calcein (CA) and Methylene Blue (MB); (b) UV-vis absorption spectra of 2D cationic COFs membrane can remove completely the dye FSs from the mixture (FSs + CA + MB).



Fig. S10 (a)-(f) UV-vis absorption spectra of methyl orange (MO) before sieving and after sieving through EB-COF:Br membrane after six cycles, respectively.



Fig. S11 The time course of rejection efficiency of EB-COF:Br membrane for anionic dye (MO).

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