Supporting Information

Pore Space Partition on Sulfonated Metal-organic Framework for

Purification of Methane from Natural Gas

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Experimental Section

Materials

Ferric chloride hexahydrate (FeCl₃· $6H_2O$, 99%), 4,8-disulfonaphthalene-2,6-dicarboxylic acid (H₂NDC(SO₃H)₂, 98%), sodium tricyanomethanide (NaC(CN)₃, 98%), 4-pyridylamidine hydrochloride (97%), N,N-dimethylformamide (DMF, >99.8%), acetone (99%), trifluoroacetic acid (TFA, 95%), deionized water (DI H₂O), methanol (99.9%). All reagents were obtained from commercial sources and used without further purification.

Synthesis procedure

Synthesis of TPH: The synthesis of TPH was carried out according to the reference.¹ NaC(CN)₃ (0.750g, ~6.6 mmol) and pyridine-4-amidine hydrochloride (4.50 g, ~28 mmol) were finely ground in an agate mortar to ensure thorough mixing. The resulting mixture was then transferred to a 23 mL Teflon-lined autoclave and heated at 200 °C overnight. After cooling to ambient temperature, a 10 wt% HCl solution was added to dissolve the crude product. Following the filtration of undissolved particulates, the solution was neutralized with acetone, and the isolation process was repeated twice. The final product was collected and dried overnight under vacuum at 60 °C, resulting in a light tan powder of TPH.

Synthesis of Fe-NDC(SO₃H)₂: FeCl₃·6H₂O (13.50 mg, ~0.05 mmol), H₂NDC(SO₃H)₂ (17.50 mg, ~0.047 mmol), DMF (3 mL), H₂O (1 mL) and TFA (500 μ L) were added in 15 mL glass vial and then ultrasonically treated for 10 min. The vial was placed in 150 °C oven for 8 h, and then yellow Fe-NDC(SO₃H)₂ crystals with hexagonal prism morphology were synthesized. To obtain the pure sample, the vial was taken out of the oven and quickly added with hot fresh DMF to avoid impurity generation. The sample was then washed with DMF for three times.

Synthesis of Fe-NDC(SO₃H)-TPH: FeCl₃·6H₂O (13.5 mg, ~0.05 mmol), H₂NDC(SO₃H)₂ (17.5 mg, ~0.047 mmol), HTPH (8.0 mg, 0.02 mmol), DMF (3 mL), H₂O (1 mL) and TFA (500 μ L) were added in 15 mL glass vial and then ultrasonically treated for 10 min. The vial was placed in 150 °C oven for 12 h, and then brown Fe-NDC(SO₃H)₂-TPH crystals with hexagonal prism or hexagonal plate morphology were synthesized. To obtain the pure sample, the vial was taken out of the oven and quickly added with hot fresh DMF to avoid impurity generation. The sample was then washed with DMF for three times.

Structural characterizations

Single-crystal X-ray diffraction (SCXRD) data for Fe-NDC(SO₃H) was collected using Bruker CCD diffractometer with graphite-monochromated Mo-K α ($\lambda = 0.71073$ Å) radiation at 273 K. The crystal data for Fe-NDC(SO₃H)-TPH was collected at the BL17B1 macromolecular crystallography beamline at Shanghai Synchrotron Radiation Facility. Absorption correction was performed using the multi-scan program in APEX3. The structure was solved by a direct method using SHELXS-2014, and refinement against all reflections of the compound was performed using SHELXL-2014. Solvent molecules were removed from the data set using the SQUEEZE routine of PLATON and refined further using the data generated. Powder X-ray Diffraction (PXRD) measurements on as-synthesized samples by using a Rigaku SmartLab X-ray powder diffractometer with Cu-K α ($\lambda = 1.5406$) radiation. Thermogravimetric (TGA) analysis was carried out on a Netzch STA449F3 analyzer heated from ambient temperature to 800 °C under a nitrogen gas atmosphere with a heating rate of 25 °C/min. SEM measurement was carried out by using a FEI NNS450 field emission scanning electron microscope equipped with 50 mm² X-Max50 SDD energy dispersive spectroscopy (EDS) detector. Data acquisition was performed with an accelerating voltage of 15 kV and 20 s accumulation time. Fourier transform infrared spectroscopy (FTIR) was recorded on a Brucker Tensor 27 in the range of 4000-400 cm⁻¹ using the KBr pellets.

Gas adsorption measurements

 CO_2 adsorption measurements were carried out on Micromeritics ASAP 2020Plus adsorption apparatus. Single-component CH_4 , C_2H_6 and C_3H_8 adsorption isotherms at 273 K and 298 K were performed by using an Automatic High Performance Surface Area and Aperture Analyzer (BSD-660 A3M). Ultrahigh purity grade CO_2 (99.999 %), CH_4 (99.999 %), C_2H_6 (99.999 %) and C_3H_8 (99.999 %) were applied for all measurements. Prior to the measurement, the sample was immersed in anhydrous DMF for one day, followed by washing with methanol for 3 times and soaked in methanol for three days. During each day, the solution was refreshed. The sample was first dried under N_2 flow gently and was subsequently transferred into the test tube. The degas program was set as heating to 100 °C, then keeping 100 °C for 6 h in situ degassing.

Breakthrough measurements

Experimental column breakthrough measurements were conducted by using a Multi-constituent Adsorption Breakthrough Curve Analyzer (BSD-MAB) on a 7.5 cm long and 0.4 cm diameter column packed with solve-exchanged samples (0.240 g). The sample was degassed at 100 °C for 6 hours. After flowing pure He gas for three minutes, the binary mixture of C_2H_6/CH_4 (50/50, v/v), C_3H_8/CH_4 (50/50, v/v) or ternary mixture of $C_2H_6/C_3H_8/CH_4$ (10/5/85, v/v/v) was introduced to the fixed bed column, respectively, with a total flowing rate of 2 mL min⁻¹ at 298 K and 1 bar.

Isosteric Heat of Adsorption (Q_{st}). The isosteric heats of adsorption for all the gases were calculated using the isotherms at 273 K and 298 K, following the Clausius-Clapeyron equation. It was done with the calculation program embedded in the software of Automatic High Performance Surface Area and Aperture Analyzer (ASAP 2020Plus). High accuracy of the Q_{st} was found in all the calculations as evidenced by the linearity in the isosters.

Selectivity by IAST. To evaluate the Xe/Kr separation performance, the selectivity was calculated by ideal adsorbed solution theory (IAST). Dual-Site Langmuir-Freundlich (DSLF) model was employed to fit the gas adsorption isotherms over the entire pressure range. DSLF model can be written as:

$$N = \frac{A_1 B_1 P^{1/n_1}}{1 + B_1 P^{1/n_1}} + \frac{A_2 B_2 P^{1/n_2}}{1 + B_2 P^{1/n_2}}$$
(1)

Where *N* is the quantity adsorbed, *p* is the pressure of bulk gas at equilibrium with adsorbed phase, A_i is the saturation loadings for adsorption site *i* (*i*=1 or 2), and B_i are the affinity parameters. $1/n_i$ is the index of heterogeneity. The R factors for all the fitting are close to or higher than 99.999%.

The detailed methodology for calculating the amount of A and B adsorption from a mixture by IAST is described elsewhere. The adsorption selectivity is finally defined as:

$$selectivity = \frac{q_A}{p_A} \frac{q_B}{p_A}$$
(2)

where q_i (i = A or B) is the uptake quantity in the mixture and p_i is the feeding partial pressure of component *i*.

DFT Calculation method

We have employed the Vienna Ab Initio Simulation Package (VASP)^{2, 3} to perform all the density functional theory (DFT) calculations within the generalized gradient approximation (GGA) using the PBE⁴ formulation. We have chosen the projected augmented wave (PAW) potentials^{5, 6} to describe the

ionic cores and take valence electrons into account using a plane wave basis set with a kinetic energy cutoff of 400 eV. Partial occupancies of the Kohn-Sham orbitals were allowed using the Gaussian smearing method and a width of 0.05 eV. The electronic energy was considered self-consistent when the energy change was smaller than 10-5 eV. A geometry optimization was considered convergent when the force change was smaller than 0.02 eV/Å. Grimme's DFT-D3 methodology⁷ was used to describe the dispersion interactions.

The equilibrium lattice constants of hexagonal X-758 unit cell were optimized to be a = b = 17.686 Å, c = 18.143 Å. We then use it for adsorption. During structural optimizations, the Γ point in the Brillouin zone was used for k-point sampling, and all atoms were allowed to relax. The adsorption energy (Eads) of adsorbate A was defined as $E_{ads} = E_{A/surf} - E_{surf} - E_{A(g)}$, where $E_{A/surf}$, E_{surf} and $E_{A(g)}$ are the energy of adsorbate A adsorbed on the surface, the energy of clean surface, and the energy of isolated A molecule in cubic periodic box with side length of 20 Å, respectively. а а

Single-crystal structure analysis



Fe-NDC(SO₃H)-TPH

Fig. S1 Structural illustration of Fe-NDC(SO₃H) and Fe-NDC(SO₃H)-TPH. The disordered regions of the crystal structures are depicted in cyan.



Fig. S2 PXRD patterns for as-synthesized Fe-NDC(SO₃H) (a) and Fe-NDC(SO₃H)-TPH (b). (Insert: optical pictures for two compounds)

SEM image EDS analysis



Fig. S3 SEM images and EDS analysis for Fe-NDC(SO₃H) (a) and Fe-NDC(SO₃H)-TPH (b).



Fig. S4 Fourier transform infrared spectroscopy of as-synthesized Fe-NDC(SO₃H), Fe-NDC(SO₃H)-TPH, HTPH and H₂NDC(SO₃H)₂. The strong peak at wavenumber of 1038 cm⁻¹ is belong to the $-SO_3^$ group. No obvious peaks belong to the $-NH_2^+$ - can be observed.

TG analysis



Fig. S5 TGA curves of as-synthesized Fe-NDC(SO₃H) (a) and Fe-NDC(SO₃H)-TPH (b). The TGA curves for both of as-synthesized samples and activated samples show a two-step weight loss, corresponding to the guest loss and framework collapse respectively. For as-synthesized samples, a significant weight loss, more than 40% for Fe-NDC(SO₃H) and about 30 % for Fe-NDC(SO₃H)-TPH, can be observed before 300 °C, which means that there is a large amount of high-volatility species, such as DMF, water, in the cavities of the framework. After the solvent exchange process with methanol, The TGA curves for both structures reveal a distinct trend compared to pristine samples. The activated samples show an obvious plateau from 140 °C to 280 °C, and the solvent weight loss decreased to about 23 % for Fe-NDC(SO₃H) and 15 % for Fe-NDC(SO₃H)-TPH before 140 °C, corresponding to the release of trapped methanol molecules in the sulfonate group decorated cavities due to the strong H bonding and adsorbed solvents on the crystal surface. From the change in TGA curve, we can conclude that the high-volatility species have been successfully removed from the pores.



Fig. S6 PXRD patterns for Fe-NDC(SO₃H) (a) and Fe-NDC(SO₃H)-TPH (b) after different pH solution treatments.

Isosteric heats of adsorption (Q_{st})



 $\label{eq:Fig.solution} \textbf{Fig.} \quad \textbf{S7} \quad -\textbf{Q}_{st} \quad \text{plots} \quad \text{of} \quad \text{Fe-NDC}(\text{SO}_3\text{H})\text{-}\text{TPH} \quad \text{for} \quad \text{CH}_4, \quad \text{C}_2\text{H}_6 \quad \text{and} \quad \text{C}_3\text{H}_8.$

Fitting graphs of CH₄, C₂H₆ and C₃H₈ adsorption isotherms



Fig. S8 Fitting graphs of Xe/Kr adsorption isotherms for Fe-NDC(SO₃H)-TPH at 298 K (a) and 273 K (b).

Aperture size analysis



Fig. S9 Size analysis of the obligatory gas permeation aperture into the cavity. The shorter breakthrough time of C_3H_8 compared to C_2H_6 can be attributed to the kinetic constraint of C_3H_8 on gas mass transfer due to the small apertures of the framework as well as strong interaction sites derived from the sulfonate group. This may increase the difficulty in achieving adsorption equilibrium and decrease the dynamic adsorption capacity, especially for the gas molecules that with larger size, more interaction sites and in a high concentration. The kinetic diameter of C_3H_8 (4.3-5.1 Å) is close to or even larger than the aperture of the framework, which will dramatically increase the adverse effects for C_3H_8 penetrating through the channels of framework compared to C_2H_6 , resulting in shorter breakthrough time.

Cycle breakthrough experiments



Fig. S10 Cycle experiments on Fe-NDC(SO₃H)-TPH measured at gas flow rate of 2 mL min⁻¹ and 298 K.

PXRD pattern after breakthrough experiments.



Fig. S11 Comparison of PXRD patterns of Fe-NDC(SO₃H)-TPH between the samples of as-synthesized and after breakthrough experiments.

Tables

 Table S1.
 Crystal data and structure refinement for Fe_NDC(SO₃H).

Identification code	Fe_NDC(SO ₃ H)		
Empirical formula	C36 H25 Fe3 O34 S6		
Formula weight	1361.47		
Temperature	273(2) K		
Wavelength	1.54178 Å		
Crystal system	Hexagonal		
Space group	P63		
Unit cell dimensions	$a = 19.6053(8) \text{ Å}$ $\alpha = 90^{\circ}$		
	b = 19.6053(8) Å	β= 90°	
	c = 17.9477(11) Å	$\gamma = 120^{\circ}$	
Volume	5983.2(6) Å ³		
Z	2		
Density (calculated)	0.756 mg/m ³		
Absorption coefficient	4.259 mm ⁻¹		
F(000)	1374.0		
Crystal size	0.120 x 0.110 x 0.080 mm ³		
2Theta range for data collection	4.916 to 128.614°.		
Index ranges	-23<=h<=20, -17<=k<=22, -16<=l<=21		
Reflections collected	26574		
Independent reflections	5679 [R(int) = 0.1160]		
Refinement method	Full-matrix least-squares on F ²		
Data / restraints / parameters	5679 / 863 / 455		
Goodness-of-fit on F ²	0.951		
Final R indices [I>2sigma(I)]	R1 = 0.0597, wR2 = 0.1577		
R indices (all data)	R1 = 0.1195, wR2 = 0.1966		
Largest diff. peak and hole	0.39 and -0.37 e.Å ⁻³		

Table S2. Crystal data and structure refinement for	$r Fe_NDC(SO_3H)_TPH.$			
Identification code	Fe_NDC(SO ₃ H)_TPH			
Empirical formula	C58 H30 Fe3 N9 O31 S6			
Formula weight	1708.82			
Temperature	100 K			
Wavelength	0.68887 Å			
Crystal system	Hexagonal			
Space group	P63			
Unit cell dimensions	a = 19.2686(13) Å	$\alpha = 90^{\circ}$		
	b = 19.2686(13) Å	β=90°		
	c = 18.143(2) Å	$\gamma = 120^{\circ}$		
Volume	5833.7(10) Å ³			
Z	2			
Density (calculated)	0.973 mg/m ³			
Absorption coefficient	0.492 mm ⁻¹			
F(000)	1726.0			
Crystal size	0.060 x 0.060 x 0.030 mm ³			
2Theta range for data collection	2.176 to 49.112°.			
Index ranges	-21<=h<=21, -23<=k<=23, -21<=l<=21			
Reflections collected	60999			
Independent reflections	7143 [R(int) = 0.0719]			
Completeness to theta = 24.414°	99.9 %			
Refinement method	Full-matrix least-squares on F ²			
Data / restraints / parameters	7143 / 1055 / 481			
Goodness-of-fit on F ²	1.285			
Final R indices [I>2sigma(I)]	R1 = 0.0985, $wR2 = 0.2699$			
R indices (all data)	R1 = 0.1198, wR2 = 0.2970			
Largest diff. peak and hole	1.58 and -0.63 e.Å ⁻³			

e S2 Crystal data and structure refinement for Fe NDC(SO₂H) T

MOFs	C ₂ H ₆ /CH ₄	C ₃ H ₈ /CH ₄	C ₂ H ₆	C ₃ H ₈	Temp.	Ref.
	selectivity	selectivity	mmol g ⁻¹	mmol g ⁻¹	K	
NPC-700	65.7	501.9	7.59	11.56	298	8
BSF-2	53	2609	1.05	1.76	298	9
JLU-Liu7	50.4	128.5	4.78	5.04	298	10
FJI-C4	39.7	293.4	2.96	3.19	298	11
γ-CDMOF-1	29.7	998.1	2.35	4.24	298	12
JLU-Liu15	28.7	461.5	3.47	3.88	298	13
Ni(HBTC)(bipy)	27.5	1857	5.85	6.18	298	14
BSF-1	23	353	1.6	1.9	298	15
γ-CDMOF-2	22.5	190.1	2.15	3.58	298	12
FJI-C1	22	471	3.72	6.33	298	16
JLU-Liu6	20.4	274.6	2.19	2.54	298	17
UTSA-35a	20	80	2.43	2.97	296	18
Fe-NDC(SO ₃ H)-TPH	17	368	2.06	2.69	298	This work
InOF-1	17	90	4.14	4.25	298	19
JLU-Liu47	17	168	5.58	8.12	298	20
SNNU-186	15.9	132.5	3.32	4.33	298	21
UPC-21	15.3	67	4.65	4.60	298	22
FIR-7a-ht	14.6	78.8	4.06	7.24	298	23
MIL-142A	14.5	>1300	3.82	5.30	298	24
FJI-H23	14.2	224.5	6.26	14.63	298	25
SNNU-185	13.1	126.0	3.12	4.20	298	21
JLU-Liu18	13.1	108.2	4.11	5.18	298	26
JUC-106	13	75	3.49	5.09	298	27
MIL-126(Fe/Co)	13	784	4.06	7.00	298	28
BSF-3	13	138	2.35	2.98	298	29
PCN-224	12	609	2.93	8.25	298	30
MIL-126(Fe/Ni)	11.8	853	4.42	7.90	298	28
JLU-Liu37	11	206	4.42	7.95	298	31
JUC-100	11	80	4.11	6.07	298	27
Zn-BPZ-SA	10.5	40.6	2.97	2.73	298	32
MFM-202a	10	87	4.21	6.76	293	33
UiO-67	8.1	73.3	3.00	8.20	298	34
UiO-66	8	65	1.67	1.7	298	35
JLU-Liu21	7.0	99.2	5.8	8.88	298	36
1-CA·ClO ₄ ^a	6	18	1.25	1.81	298	37
CTGU-15	5.2	170.2	2.13	12.13	298	38
UPC-33	4.8	151.5	1.56	4.18	298	39

Table S3. Comparison of IAST (50/50, v/v) and capture capacity for C_2H_6 and C_3H_8 among reported MOFs.

^a IAST calculated in a ratio of 15/85 (v/v).

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