Electronic Supplementary Information

Encapsulation of an Ionic Liquid into the Nanopores of a 3D Covalent

Organic Framework

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Experimental Details Reagents: All the bought reagents were used as received without further purification. 4,4'-biphenyldicarboxaldehyde (BPDA) was purchased from TCI Chemicals. Tetra-(4-anilyl)-methane (TAM) was synthesized based on our previous report.^[1] 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([Emim][Tf₂N]) was purchased from Lanzhou Greenchem ILS, LICP. CAS. China.

Synthesis of COF-320: 4,4'-biphenyldicarboxaldehyde (BPDA, 20 mg, 0.095 mmol) and tetra-(4anilyl)-methane (TAM, 20 mg, 0.053 mmol) were dissolved in 1.0 mL of anhydrous dioxane within a 5 mL ampoule bottle under sonication. Then, 0.2 mL of aqueous acetic acid (3 mol/L) was added into the bottle. After quick freezing the mixture with liquid N₂, the reaction system was degassed by freeze-pump-thaw cycle for 3 times. The frozen bottle was vacuumed under 10 Pa and then quickly sealed with a torch. After heating at 120 °C for 72 h, the yellow COF-320 powder was isolated by filtration, washed with anhydrous THF thoroughly and immersed in anhydrous THF overnight. The solvent was then exchanged with fresh THF for several times. The COF-320 product was dried under vacuum at 100 °C for 24 hours.

Characterizations: The X-ray diffraction (XRD) patterns were recorded at room temperature with a D/MAX-2500/PC X-ray diffractometer with Cu Ka radiation at 20 kV and 40 mA. FT-IR spectra were measured by a transmission mode by FT-IR spectrophotometer using a compressed KBr disc. TG experiments were carried out on a TA Instruments Netzsch STA 449 F3 series thermal gravimetric analyzer under nitrogen atmosphere. The nitrogen sorption isotherms were measured at 77 K with a Quantachrome USA Inc. model ASIQMU00U-6 analyzer. Before measurement, the samples were degassed in vacuum at 100 °C for 12 h. The Brunauer-Emmett-Teller (BET) method was utilized to calculate the specific surface areas. The pore size distribution and total volume was calculated by the non-local density functional theory (NLDFT) model, respectively. ¹⁹F CP/MAS NMR spectra were measured on a Bruker Advance 400 MHz spectrometer.



Fig. S2: TGA curve of 100%IL@COF-320.

References:

[S1] Hui Lu, Chang Wang, Juanjuan Chen, Rile Ge, Wenguang Leng, Bin Dong, Jun Huang and Yanan Gao, *Chem. Commun.*, 2015, **51**, 15562-15565.