Supporting information

Fabrication of a COF-5 membrane on functionalized α-Al₂O₃ ceramic support using microwave irradiation method

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1. Experimental Section

1.1 Materials:

All chemicals were purchased from commercial sources and used without any further purification: ammonium persulfate (AR, Kermel, China), catechol (99.0%, Aladdin, China), benzene-1.4-diboronic acid (\geq 95.0%, Sigma-Aldrich), 4-formylphenylboronic acid (97%, Aladdin, China), (3-aminopropyl)triethoxysilane(APTES, 99%, Aladdin, China), 1,4-dioxane(\geq 99.5%, Aladdin, China), mesitylene (98%, Sigma-Aldrich). Porous α -Al₂O₃ ceramic supports (obtained from Singapore Hyflux Company, with a pore size of 40 nm in the surface modification layer of the asymmetrical support) were used as supports.

1.2 Synthesis of HHTP

17.20 g of ammonium persulfate (0.075mol) was added to 8.25 g of catechol (0.075 mol) dispersed in 25ml of a 70 wt.% aqueous solution of sulfuric acid. The mixture was stirred for 7 h at room temperature, and the resultant precipitate was then filtered and washed with water. 300ml of acetone and 0.75 g of activated carbon were added to the precipitate, the mixture was stirred for 30 min at room temperature, and insoluble matter was subsequently filtered off from the mixture. 300 ml of deionized water was added to the filter, and then acetone was distilled off under atmospheric pressure. The resultant precipitate was filtered and dried under reduced pressure to afford 3.457 g of HHTP crystals (yield: 41.9%; purity > 99%).The reaction equation is as follow:



1.3 Synthesis of COF-5 and COF-5 membrane by using microwave

irradiation

Sealed the mixture of 3:2 molar ratio solution a of 1,4-benzenediboronic acid (BDBA; 185.0 mg, 1.11 mmol) and 2,3,6,7,10,11-hexahydroxytriphenylene (HHTP; 241.5 mg, 0.745 mmol) immersed in a 1:1 v:v mixture of mesitylene: 1,4-dioxane (20 mL) under nitrogen in a 35 ml glass microwave tube and then heated the tube by microwave irradiation at 100°C with stirring for 60 mins at a power of 300 W using a Microsynth Plus-position microwave reactor(Milestone Germany). A gray precipitate was observed to form in the reaction vessel. The resulting gray-purple powder was filtered and washed with anhydrous acetone for several times under nitrogen and then dried in a vacuum desiccator at 70°C for 24 h. The finally purified COF-5 was isolated as a gray solid, and then the COF-5 membrane were fabricated in the same reaction vessel. The reaction equation is as follows:

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1.4 Modification of α-Al₂O₃ ceramic supports

Porous ceramic α -Al₂O₃ supports were immersed in 50 ml of hydrochloric acid solution (1 mol/L) for 5 h, then washed and dried, and then treated with APTES (98.29 mg in 20 ml toluene) at 100°C for 3 h under argon, leading to APTES monolayers deposited on the surface of α -Al₂O₃ ceramic supports. Washed the supports with ethanol for several times, then immersed the tubes in a solution of 20 ml ethanol and 4-Formylphenylboronic acid (29.6 mg) for 1-15 h, and then the ceramic supports were washed by ethanol for several times, and then dried in a vacuum desiccator at room temperature for 24 h.

2 Characterizations

Scanning electron microscopy (SEM) micrographs were taken on a Netherlands QUANTA 200 FEG with a cold field emission gun operating at 20 kV. The X-ray diffraction (XRD) patterns were recorded at room temperature with a Japan D/MAX-2500/PC X-ray diffractometer with CuKa radiation at 20 kV and 40 mA. Samples were prepared as a compressed IR-transparent KBr disc for analysis and scanned intransmission mode by a Tensor 27 FTIR spectrophotometer.