

JZO

ZEO-1 (OH method)

Si(95.3), Al(4.7)

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Type Material chemical composition not analysed

Method Citation Bieseki, L. et al. [1], Lin, Q.-F et al. [2]

Batch Composition 1 SiO₂ : 0.0125 Al₂O₃ : 0.5 OSDAOH : 5 H₂O

Source Materials

Deionised water

LC-MS grade water (FisherBrand, 99.99 %, pH ≈ 5.7)

Methyliodide (CH₃I, Sigma-Aldrich, 99%)

Tricyclohexylphosphine (Sigma-Aldrich, 98%)

Acetonitrile (CH₃CN, Fisher Scientific, 99.8%)

Ethanol (Carlo Erba, 96%)

Anionic exchange resin (Amberlite HPR-550 OH, 1.1 meq/g, ThermoScientific)

Al₂O₃ (Condea, 89% purity determined from thermogravimetric analysis)

TEOS (Si(OEt)₄) (Sigma-Aldrich, 98%)

OSDAOH Preparation

- (1) Tricyclohexylphosphine (10.0 g, 35.6 mmol, Sigma-Aldrich) was suspended in acetonitrile (100 mL) and cooled down in an ice-bath. Iodomethane (8.60 mL, 19.61 g, 138.1 mmol, Sigma-Aldrich) was added dropwise and the solution was stirred at room temperature for 2 days forming a slightly yellow solution.
- (2) Ethanol (40 mL) was added to (1) in order to quench iodomethane present in excess and the solution was then concentrated in vacuo to obtain a white powder (13.1 g, 87 % yield).^a
- (3) For the anionic exchange, the ground iodate powder (6.79 g, 16.1 mmol) was suspended in HPLC grade water (50 mL) and the anionic exchange resin (32 g) was added. The suspension was stirred for 2 days, during which the white powder dissolves in the media, then it was filtered and the resin was washed with HPLC grade water (2 × 50 mL). The obtained solution was partly concentrated in vacuo with moderate heating (max. 50 °C) to obtain ~40 mL of SDA-OH solution used as such for the zeolite synthesis. The determined concentration and the exchange rate were 0.31 M and 79 %, respectively.^b

Batch Preparation (for about 1 g product)

- (1) [33.7 mL OSDAOH solution (0.31 M, 10.44 mmol) + 30 mg of Al₂O₃ (0.261 mmol)], the mixture was stirred until dissolution at room temperature in a becker for 3 hours.^c
- (2) [(1) + 4.35 g TEOS (20.88 mmol)], TEOS was added and the suspension was stirred overnight at room temperature
- (3) To reach the desired water content, the gel was heated at 70° C in a water bath under stirring (the weight of the (vessel + gel) was controlled periodically). The gel obtained display a vitreous aspect at 70 °C and forms a thick white solid upon cooling to room temperature.^d

Crystallization

Vessel: 10 mL Teflon-lined autoclave

Time: 31 ± 2 days

Temperature: 190 °C

Agitation: none

Product Recovery

- (1) The recovered material was dispersed in 100 mL deionized water and 20 mL acetone, then sonicated in ultra-wave bath for 5 min
- (2) The suspension was centrifuged at 10,000 RPM for 20 min, further washed twice with water
- (3) The white powder was dried at 80 °C overnight
- (4) Yield: 1.11 g (62 % determined based on the silica source and considering a SDA content of 30 wt. %)

Product Characterization

XRD: **JZO**

Competing phases: no

Elemental Analysis: Si/Al = 20.3, Si/P = 16.2, Al/P = 0.80^e

Crystal Size and Habit: isometric shaped crystals (~300 ±150 nm)

References

- [1] Bieseki, L.; Habrioux, A.; Paillaud, J.-L.; Pergher, S.; Rousseau, J.; Batonneau-Gener, I.; Oheix, E.; Sachse, A. A Zeolite-Templated Carbon Synthesized from Extralarge-Pore Zeolite ZEO-1. *Inorganic Chemistry* **2024**, *63* (42), 19509–19512.
- [2] Lin, Q.-F.; Gao, Z. R.; Lin, C.; Zhang, S.; Chen, J.; Li, Z.; Liu, X.; Fan, W.; Li, J.; Chen, X.; Cambor, M. A.; Chen, F.-J. A Stable Aluminosilicate Zeolite with Intersecting Three-Dimensional Extra-Large Pores. *Science* **2021**, *374* (6575), 1605–1608.

Notes

- a. ^{31}P NMR (162 MHz, CDCl_3): s 33.8 ppm, (the yield can reach 98 %)
- b. The concentration of SDA was measured by ^1H NMR measurements using dioxane as an internal standard and the concentration of OH^- was determined by titration with an HCl solution
- c. It is possible that a very small amount of the Al_2O_3 source remains undissolved, but this does not affect the final result
- d. At this reaction scale, significant weight loss are associated with the transfer of the gel into the liner (19 wt. % in this case).
- e. Semi-quantitative analysis obtained from EDX spectra

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