

JZO

ZEO-1 (F method)

Si(96.2), Al(3.8)

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Type Material [Si_{596.87}Al_{27.13}O₁₂₄₈]

Method Citation M. Fahda, [1]^a

Batch Composition 1 SiO₂ : 0.02 Al₂O₃ : 0.33 OSDAOH : 0.05 HF : 7 H₂O

Source Materials

distilled water

MilliQ water (Merck Millipore)

methyl iodide (MeI, Sigma-Aldrich, 99.5%)

tricyclohexylphosphine (TCy, BLD Pharmatech Ltd., 97%)

acetonitrile (MeCN, Fisher Scientific, 98%)

ambersep 900 hydroxide resin (ThermoScientific)

aluminumisopropoxide (Al(O-*i*Pr)₃, Fluka, 98%)

colloidal silica (SiO₂ Ludox ® HS-40, Sigma-Aldrich, 40% wt)

hydrofluoric acid solution (HF 40 wt%, Fisher Scientific, 98%)

OSDAOH Preparation

- (1) [10.g (35.6 mmol) TCy + 40 mL MeCN], in a Schlenk tube purged with argon
- (2) [(1) + 19.58 g (138 mmol) MeI], slow dropwise addition at room temperature for 48 hours
- (3) Evaporate the solvent and excess MeI under reduced pressure
- (4) Yield: 15 g of product tricyclohexylmethylphosphonium iodide, characterized for purity using liquid proton, carbon, and phosphorus NMR spectroscopy (35.5 mmol, 98% purity)
- (5) [10 g (4) + 250 g MilliQ water + 45 g dry Ambersep 900 hydroxide resin], in a 500 mL polypropylene bottle. This step will convert the tricyclohexylmethylphosphonium iodide to its hydroxide form
- (6) Overnight exchange. The resulting SDAOH solution was collected via filtration and concentrated to the desired volume using a rotary evaporator at 60 °C
- (7) The concentrated solution was titrated with potassium hydrogen phthalate using phenolphthalein as an indicator to determine the extent of exchange and the concentration of the hydroxide. The resultant basic solution exhibited an exchange ratio of 98% and contained 20 wt% OSDAOH^b

Batch Preparation (for 1 g product)

- (1) [6.21 g OSDAOH + 0.204 g of Al(O-*i*Pr)₃], stir until dissolved at room temperature for 2 hours in a capped polypropylene bottle
- (2) [(1) + 3.75 g Ludox ® HS-40], colloidal silica is added dropwise under vigorous stirring and the solution is left to homogenize at room temperature for 1 hour
- (3) [(2)+ 55.2 µl of 40 wt% HF], HF is carefully added under a well-ventilated hood to the gel mixture under vigorous stirring, after which the synthesis gel is left for aging overnight
- (4) Remove the excess water in the synthesis gel by a freeze drying. The weight of the polypropylene bottle containing the precursor gel was monitored periodically until the desired water content is reached

Crystallization

Vessel: Teflon-lined autoclave

Time: 22 ± 1 days

Temperature: 190 °C

Agitation: non

Product Recovery

- (1) The zeolite product is collected by centrifugation at 21000 rpm for 10 minutes.
- (2) The collected zeolite product is purified by washing with deionized water followed by centrifugation until a neutral pH is attained.
- (3) Yield: ~ 1g

Product Characterization

XRD: JZO

Competing phases: no

Elemental Analysis: [Si_{596.87}Al_{27.13}O₁₂₄₈]

Crystal Size and Habit: isometric shaped crystals (~450 nm)

References

- [1] Fahda, Mohammad, et al. "Investigating the physicochemical properties of an extra-large pore aluminosilicate ZEO-1." *Chemistry of Materials* 36.11 (2024): 5405-5421.

Notes

- a. The published article discusses in detail the influence of various synthesis parameters (such as aluminum source and loading, temperature, fluoride, and water content) on the kinetics of ZEO-1 synthesis. The protocol described herein ensures the synthesis of ZEO-1 material with a micropore volume of 0.3 cm³/g after calcination and removal of residual phosphorus species, as confirmed by N₂ analysis
- b. The weight percentage (wt%) of the OSDAOH solution does not need to strictly adhere to 25%; however, a more concentrated solution results in faster adjustment of water content

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