AFI $AlPO_4$-5 Al(50), P(50)

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Type Material $Al_{12}P_{12}O_{48}$

Method I. Girnus, K. Jancke, R. Vetter, J. Richter-Mendau, J. Caro [1] [a]

Batch Composition Al_2O_3 : 1.3 P_2O_5 : 1.6 TEA : 1.3 HF : 425 H_2O : 6 C_3H_7OH [b]

Source Materials

deionized water
orthophosphoric acid (Merck, 85 wt% H_3PO_4)
triethylamine (TriEA), (Riedel de Haen, $(C_2H_5)_3N$) [c]
aluminum triisopropylate (Merck, $Al(C_3H_7O)_3$) [d]
hydrofluoric acid (Merck, 40 wt% HF in water)

Batch Preparation (for 3 g product)

(1) [7 g water + 3.84 g of phosphoric acid], mix
(2) [(1) + 2.07 g TriEA], add TriEA dropwise and mix
(3) [(2) + 5.23 g aluminum isopropylate], add in small amounts at 0˚C with intense stirring then stir the mixture at room temperature for 2 hours
(4) [(0.83 g hydrofluoric acid + 89.2 g water], mix
(5) [(3) + (4)], stir for 2 hours

Crystallization

Vessel: 150 mL Teflon-lined steel autoclavese
Temperature: 180˚C (preheated oven)
Time: 6 hours [f]
Agitation: none

Product Recovery

(1) Decant the supernatant liquid and discard
(2) Wash the precipitate four times with 100 mL deionized water
(3) Calcine in air at 600˚C until product is colorless (white) [g]
(4) Yield: near 100% on Al_2O_3

Product Characterization

XRD: Characteristic strong reflections at d = 11.90, 5.93, 4.48, 4.24, 3.96, and 3.42 Å; competing phases (if present): tridymite
Elemental Analysis: 42.9 wt% P_2O_5, 30.5 wt% Al_2O_3 (P/Al = 1.00)
Crystal size and Habit: Hexagonal columns up to 50 μm [h]

References

[1] I. Girnus, K. Jancke, R. Vetter, J. Richter-Mendau, J. Caro, Zeolites 15 (1995) 33

[2] S. T. Wilson, B. M. Lok, C. A. Messina, T. R Cannan, E M. Flanigen, J. Am. Chem. Soc. 104 (1982) 1146
[3] J. M. Bennett, J. P. Cohen, E M. Flanigen, J. J. Pluth, J. V. Smith, ACS Symp. Series 218, Am. Chem. Soc., Washington, D. C., 1983, p. 109
[4] S. Oju, W. Pang, H. Kessler, J.-L Guth, Zeolites 9 (1989) 440
[5] A. S. T. Chiang, C.-K. Lee, Z. H. Chang, Zeolites 11 (1991) 380
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[8] I. Girnus, K. Hoffmann, F. Marlow, G. Döring, J. Caro, Micropor. Mater. 2 (1994) 537

Notes

a. The decisive difference of this synthesis from that of Wilson and Flanigen [2,3] is the use of HF as proposed by Kessler and Guth [4]. However, crystallization proceeds also in the absence of HF, but less favorably.

b. Because Al triisopropylate is used as Al source, a fixed amount of 3 isopropyl alcohol molecules per Al is always present in the gel.

c. The $AlPO_4$-5 phase can be prepared with numerous templates. Good results are also reported using tripropylamine [3-5].

d. Other Al sources as pseudoboehmite [2, 3] and Al hydroxide [6] also give good and large crystals.

e. For microwave heating, full Teflon autoclaves.

f. For microwave oven (heating rate 4 grd/sec), 15 minutes at 180°C.

g If the product remains brown or gray after 4 hours at 600^0C, this can be taken as a hint that free diffusion in the one-dimensional pores is blocked (by stacking faults or by non-framework material) thus preventing oxygen from entering and oxidation products from leaving the pores. By going to 900°C, the material can be make "white," but the uptake capacity remains low.

h. Crystals up to 50 µm in length in the direction of the hexagonal columns are easily obtained; in optimized synthesis, Crystals up to 500 µm are observed. Incorporation of Si [7] or Co [8] gives larger and better crystals. Numerous compositional variants are known.