AFO SAPO-41 Al(51), P(46), Si(3)

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Type material $[(Al_{20.4}P_{18.4}Si_{1.2})\ O_{80}] \cdot mR \cdot nH_2O$ (R = Di-n-propylamine)

Method A. M. Prakash, S. V. V. Chilukuri, R. P. Bagwe, S. Ashtekar, D. K. Chakrabarty [1]

Batch Composition 1.0 Al_2O_3 : 1.3 P_2O_5 : 0.1 SiO_2 [a] : 4.0 R [b] : 58.2 H_2O [c]

Source Materials

deionized water
orthophosphoric acid (Merck, 85%)
pseudoboehmite (Vista; Catapal-B, assumed 70 wt% Al_2O_3)
fumed silica (Degussa, Aerosil-200)
di-n-propylamine (Merck, 99%)

Batch Preparation (for 16 g product)

(1) [23.06 g phosphoric acid + 25 g water], mix together
(2) [(1) + 14.57 g pseudoboehmite], add slowly over a period of 3.5 hours and continue stirring for 1.5 hours
(3) [0.60 g silica + 25 g water], mix together to form a slurry
(4) [(2) + (3)], add silica slurry over a period of 30 minutes and continue stirring for 1 hour
(5) [(4) + 40 g water], mix together
(6) [(5) + 40.88 g di-n-propylamine], add dropwise to gel and continue stirring for 30 minutes
(7) Adjust pH of the gel to 7.7 by slowly adding 4 mL of phosphoric acid diluted in 6 g water and stir the final gel for 30 minutes to ensure homogeneity

Crystallization

Vessel: 500 mL stainless steel autoclave
Temperature: 180°C
Time: 11 days

Product Recovery

(1) Decant the mother liquor
(2) Slurry with deionized water. Allow the crystallites to settle and decant the water
(3) Repeat step (2) three times
(4) Filter off product and wash again with water
(5) Dry at 100°C overnight
(6) Yield: 65% based on alumina

Product characterization

XRD: SAPO-41 [1][d] Orthorhombic; a = 9.7 Å, b = 25.5 Å, c = 8.4 Å, competing phases: SAPO-11 and SAPO-31 at low template concentration and SAPO-46 at high silica concentration [1, 2]
Elemental Analysis (exclusive of R and H_2O): 1.00 Al_2O_3 : 0.90 P_2O_5 : 0.11 SiO_2
Crystal size and habit: 5-10 μm crystals of rectangular morphology

References

[1] A. M. Prakash, S. V. V. Chilukuri, R. B. Bagwe, S. Ashtekar, D. K. Chakrabarty, Micropor. Mater. 6 (1996), 89

[2] P. Mériaudeau, V. A. Tuan, V. T. Nghiem, S. Y. Lai, L. Hung, C. Naccache, J. Catal. 169 (1997) 55

Notes

a. In this synthesis pure phase SAPO-41 crystallizes only at low SiO_2 concentration in the gel. High concentration of silica generally leads to phases such as SAPO-11, SAPO-31, SAPO-46 depending on template concentration, temperature and period of crystallization.

b. Template concentration should be high (3 mol < R < 4 mol) for obtaining pure SAPO-41. Lower template concentration leads to SAPO-11 and SAPO-31 depending on silica concentration.

c. H_2O included water from pseudoboehmite, phosphoric acid and added water.

d. Extra low-intensity lines between 9-18 degrees (2ɵ) have not been identified. They may indicate a lower symmetry due to retained template. However, a competing phase cannot be ruled out.