AFI CoAPO-5 P(51), Al(45), Co(4)

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Type Material [$CoAl_{11}P_{12}O_{48}$]: wH_2O (w = 0.1 to 0.2)

Method M. G. Uytterhoeven, R A. Schoonheydt [1, 2]

Batch Composition 0.7 TriEA : $(Co_{0.08}Al_{0.92}P)O_4$: 20 H_2O

Source Materials

water (doubly distilled)
phosphoric acid (Janssen Chimica, 85% H_3PO_4)
cobalt nitrate, $Co(NO_3)_2$. 6 H_2O (Janssen Chimica)
pseudoboebmite (Catapal, Vista, 70% Al_2O_3
triethylamine (TriEA) (Janssen Chimica 99%)

Batch Preparation (for 27 g product)
(1) [62.6 g water + 23.06 g phosphoric acid], mix; chill to 0¡C
(2) [(1) + 4.66 g cobalt nitrate], stir until dissolved
(3) [(2) + 13.4 g pseudoboehmite], add alumina under continuous stirring
(4) [(3) + 14.3 g TriEA], add amine under continuous stirring;[a] stir for one additional hour. Initial pH ~3

Crystallization
Vessel: stirred, Teflon-lined autoclave
Temperature: 200˚C
Time: 24 hours
Agitation: Stirring is essential.[b] Autoclaves were "tumbled" (end-over-end) in the oven

Product Recovery
(1) Filter and wash with distilled water; dry at room temperature
(2) Yield: near 100% on T-atom basis

Product Characterization
XRD: pure AFI, CHA impurity when present is evidenced by a line at 9.5¡(2q)[c]
Elemental Analysis: 0.10 TriEA. $(Co_{0.086}Al_{0.892}P_{1.000}O_4)$ .0.13 H_2O[d]
Crystal Size and Habit: agglomerates formed by hexagonal platelets, regularly shaped (spherical or diabolo-shaped) with dia. = 55 µm [e,f]

References
[1] M. G. Uytterhoeven, R. A. Schoonheydt, Micropor. Mater. 3 (1994) 265
[2] M. G. Uytterhoeven, R. A. Schoonheydt, Proc. Ninth Int. Zeo. Conf., R von Ballmoos, J. B. Higgins, M. M. J. Treacy (eds.), Butterworth-Heinemann, Boston, 1993, p. 329

Notes

a. TriEA addition causes an exothermic reaction; the preparation should be performed at 0˚C and TriEA added drop by drop under careful temperature control.

b. Under static conditions, co-crystallization of CHA is more probable.

c. The co-crystallization of CHA can be suppressed by reducing the amine content and/or decreasing the cobalt content. The latter requires an increase of the Al content so that [Al + Co = P].

d. On T-atom basis, the product composition is almost equal to the gel composition. Al + Co = P should be approximately achieved.

e. SEM is suited to distinguish between AFT and much smaller CHA crystals.

f. At low cobalt content (e.g., $Co_{0.02}Al_{0.98}P_{1.00}$), single crystals are formed, shaped as hexagonal bars. Twinning can occur. Increasing cobalt content causes agglomeration giving larger, regular agglomerates. At high cobalt content (for example, $Co_{0.08}Al_{0.92}P_{1.00}$) single crystal formation can be achieved by increasing dilution and increasing template content, although the latter favors the co-crystallization of CHA.