FAU SAPO-37 Al(47), P(36), Si(17)

Contributed by M. J. Franco and J. Pérez-Pariente

Verified by J. Patarin and M. Ribeiro

Type Material (7TMA, 13TPA)[$Al_{90}Si_{33}P_{69}O_{384}$]
(TMA = tetramethyl ammonium, TPA = tetra-n-propyl ammonium)

Method M. J. Franco, J. Pérez-Pariente, A. Mifsud, T. Blasco, J. Sanz [1]

Batch Composition $0.025(TMA)_2O : 1.0(TPA)_2O : 1.0Al_2O_3 : 1.0P_2O_5 : 1.0SiO_2 : 50H_2O$

Source Materials
deionized water
phosphoric acid (Riedel-de Haen, 85% H_3PO_4)
alumina (pseudoboehmite, Vista, 70% Al_2O_3) [a]
tetrapropylammonium hydroxide (TPAOH), Alfa, 40% aqueous solution) [b]
tetramethylammonium hydroxide (TMAOH. $5H_2O$, Aldrich, 99%)
fumed silica (Aerosil 200, Degussa)

Batch Preparation (for 6.2 g product) [c]
(1) [20.69 g water + 23.04 g phosphoric acid + 14.56 g alumina]. Mix water and phosphoric acid at 293K and add the alumina very slowly with vigorous stirring (1500 rpm). Continue stirring for 8 hours at 293K in a temperature-controlled bath
(2) [101.68 g tetrapropylammonium hydroxide (40% solution) + 0.90 g tetra-methylammonium hydroxide + 6.00 g silica]. Dissolve the TMAOH in the TPAOH solution in a separate glass beaker. To this solution add the silica under vigorous stirring. Continue stirring for one hour
(3) [(1) + (2)] Add silicate solution (2) slowly to aluminate slurry (1) under vigorous stirring. Continue stirring for 24 hours at 293 K. The final gel pH should be approxunately 7.5

Crystallization
Vessel(s): 60 mL Teflon-lined stainless steel autoclave(s)
Temperature: 200˚C [d]
Time: 13 hours
Agitation: none

Product Recovery
(1) Quench autoclave in cold water
(2) Recover solids by centrifugation (6000 rpm)
(2) Wash with cold distilled water
(4) Dry at 80˚C for 16 hours
(5) Yield: 3.7 g solid/100 g of gel (19% on Al_2O_3)

Product Characterization

XRD: FAU-type, pure. Competing phases: AFI and SOD when other compositions and/or crystallization treatments are used
Elemental Analysis: $(Si_{0.17}Al_{0.47}P_{0.36})_{192}O_{384}$. $3.5(TMA)_2O$. $6.5(TPA)_2O$ [e]
Crystal Size and Habit: Crystals are quite homogeneous in size (approximately 4.0 μm dia.) and show interpenetrating octahedra morphology [f]

Reference

[l] M. J. Franco, J. Pérez-Pariente, A. Mifsud, T. Blasco, J. Sanz, Zeolites 12 (1992) 386

Notes

a. B.E.T. surface area 271 m2/g; the alumina may be critical. Aluminum isopropoxide gave negative results.
b. Commercial 20% TPAOH can be vacuum stripped to 40%; however, it is critical to make sure that there has not been decomposition of the TPAOH. For this reason, it is necessary to make a OH^- titration before and after the evaporation process to check that the total amount of OH has not changed. (Na + K) < 340 ppm
c. During preparation of the gel, it is very important to use a closed vessel and a temperature controlled bath (293K) to ensure a constant water concentration in the gel. This vessel can be a polypropylene bottle with a hole in the cap to fit the rotating shaft of the stirring device. A stirring device with two movable Teflon blades is recommended.
d. Heat-up rate: 2.5°/min.; autogenous pressure.
e. Flow thermogravimetric analysis showed three different weight losses: 373 to 473K (water desorption), 473 to 688K (decomposition of TPA^+ in the alpha cages), 588 to 973K (decomposition of TMA^+ in the sodalite cages).
f. Increasing $(TPA)_2O/Al_2O_3$ in gel from 1.0 to 1.5 (keeping $TPA^+/TMA^+ = 40$) decreased average crystal diameter from 4 to 2 μm and increased product yield by 100%. [1]