OFF Offretite Si(79), Al(21)

Contributed by Hans Lechert

Verified by M. Mertens and R Russu

Type Material $Na_{0.2}K_{0.9}[Al_4Si_{14}O_{36}] : wH_2O$[a]

Method H. Lechert, H. Weyda [1]

Batch Composition 3.0 Na_2O : 1.0 K_2O : Al_2O_3 : 20.8 SiO_2 : 1.73 (TMA)Cl : 324 H_2O[b]
(TMA tetramethylammonium)

Source Materials

distilled water
sodium hydroxide, reagent grade (99+% NaOH)
aluminum tri-isopropylate [$Al(OC_3H_7)_3$]
potassium hydroxide, reagent grade (87% KOH)
silica (Merck, precipitated and dried, 87% SiO_2)
tetramethylammopaum chloride [Merck, $N(CH_3)_4Cl$]

Batch Preparation (for 64 g dry product)

(1) [28.1 g water + 16.8 g sodium hydroxide], mix until dissolved
(2) [(1) + 42.5 g aluminum tri-isopropylate], stir at 100˚C and evaporate to reduce to 42.0 g
(3) [(2) + 58 g water], stir and cool to ambient temperature; dilute to 100 g total
(4) [379.3 g water + 8.4 g sodium hydroxide + 13.4 g potassium hydroxide], mix until dissolved
(5) [(4) + 149.4 g silica], mix for 30 minutes
(6) [(5) + (3)], mix for 30 minutes
(7) [(6) + 126.4 g water], mix for 30 minutes
(8) [(7) + 19.7 g tetrametylammonium chloride], mix for 30 minutes

Crystallization

Vessel: Teflon-lined autoclave
Temperature: 160˚C
Time: 20 hours
Agitation: none

Product Recovery

(1) Centrifuge
(2) Wash to near neutrality
(3) Dry at 100˚C
(4) Yield: near 100% on Al_2O_3

Product Characterization

XRD: Strong OFF, no extraneous phases; competing phases: erionite,[c] Zeolite P (GIS), analcime Elemental Analysis: 0.10 Na_2O : 0.46 K_2O : Al_2O_3 : 7.72 SiO_2[d] Crystal Size and Habit: "rice grains", length 5-10 μm[e]

Reference

[1] H. Lechert, H. Weyda, in Synthesis of Microporous Materials, Vol. I, M. L Occelli, H. Robson (eds.), Van Nostrand Reinhold, New York, 1992, P. 77

Notes

a. Missing cations assumed to be TMA^+ or (after calcination) H^+.

b. OFF can be obtained without template in the temperature range 87-107°C with a gel (6.2 Na_2O 3.5 K_2O : $A1_2O_3$: 25 SiO_2 : 390 H_2O), but slight deviations from this composition give appreciable amounts of erionite or gismondine. At higher temperatures Zeolite P (GIS) and analcime co-crystallize with offretite.

c. ERI can be distinguished from OFF in the XRD pattern by lines at d = 9.13, 5.37, 4.60, 4.17, 3.28, and 2.82 Å.

d. The template content of the product is quite low (less than one TMA^+ per UC).

e. At 190°C, pure erionite can be obtained from a batch composition: 8.0 Na_2O : 1.7 K_2O : $A1_2O_3$: 24 SiO_2 : 1.7 TMACl : 410 H_2O. At this temperature the offretite is needles of about 1 μm diameter x 25 μm long. At crystallization times longer than 4 hours at 190°C, an increasing amount of analcime is observed.