FAU Silica Type X (LSX) Si(50.5), Al(49.5)

Contributed by Gunter Kühl

Verified by M. Ludvig and D. Millar

Type Material $Na_{73}K_{22}Al_{95}Si_{97}O_{384} : wH_2O$

Method G. H. Kühl [1]

Batch Composition 5.5 Na_2O : 1.65 K_2O : Al_2O_3 : 2.2 SiO_2 : 122 H_2O[a-c]

Source Materials

distilled water
sodium aluminate (Nalco 680, 45.6% Al_2O_3, 29.65% Na_2O)
potassium hydroxide, reagent grade (usually ~86% KOH)
sodium hydroxide, reagent grade (usually ~97% NaOH)
sodium silicate solution (PQ Corp. N-brand clarified, 28.7% SiO_2, 8.9% Na_2O)

Batch Preparation (for 29 g dry product)[d]

(1) [30 g water + 22.37 g sodium aluminate], stir until dissolved
(2) [70 g water + 21.53 g potassium hydroxide + 31.09 g sodium hydroxide], stir until dissolved
(3) [(1) + (2)], mix thoroughly [e]
(4) [(3) + 71.8 g water + 46.0 g sodium silicate solution], mix thoroughly [f]

Crystallization

Vessel: sealed polypropylene or Teflon jar
Incubation: 3 hours at 70°C without stirring
Temperature: 93-100°C
Time: 2 hours
Agitation: either with or without stirring

Product Recovery

(1) Dilute the reaction mixture with distilled water
(2) Filter and wash with 0.01 N NaOH [g]
(3) Dry at ambient temperature (drying at 110-125°C acceptable)
(4) Yield: 99+% based on alumina

Product Characterization

XRD: FAU (a_0 = 25.03 Å);[h] competing phases: LTA (when gel SiO_2/Al_2O_3 = 2.0), SOD (concentration is too high), P (extended aging or crystallization times)
Elemental Analysis: 0.77 Na_2O. 0.23 K_2O. Al_2O_3. 2.04 SiO_2

Crystal Size and Habit: multi-faceted spherulites of 2-6 μm dia. with 111 faces exposed [1]

Reference

[1] G. H. Kühl, Zeolites 7 (1987) 451

Notes

a. H_2O includes water from sodium aluminate, watergiass, free and bound water in NaOH + KOH, and added water.

b. $(K_2O + Na_2O)/SiO_2$ can be reduced to 2.25 without loss of product quality. However, at least for the initial crystallizations, a value of 3.25 is recommended.

c. The ratio: $Na_2O/(Na_2O + K_2O)$ is critical; it should be in the range 0.77 to 0.78.

d. Use plastic or stainless steel equipment throughout.

e. Solution (1) should be perfectly clear, but it is probably acceptable if a clear solution (3) is obtained after adding NaOH and KOH.

f. The mixture must not gel before it is well mixed. It usually takes several minutes before a gel if formed.

g. Wash or exchange in 0.01 N NaOH to prevent hydrolysis; low-silica X hydrolyzes as easily as NaA.

h. The lower SiO_2/Al_2O_3 ratio enhances the line intensities, the presence of K^+ attenuates them.