FAU Linde Type X Si(55), Al(45)

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Type Material $Na_{86}Al_{86}Si_{106}O_{384}$: wH_2O (w ~ 260)

Method H. Lechert, H. Kacirek [1, 2]

Batch Composition $NaAlO_2$: 4 SiO_2 :16 NaOH : 325 H_2O[a,b]

Source Materials

distilled water
sodium hydroxide (99+% NaOH)
alumina trihydrate (Merck, 65% Al_2O_3) [c]
sodium silicate solution (27.35% SiO_2, 8.30% Na_2O, 1.37 g/mL)

Batch Preparation (for 42 g product)

(1) [100 g water + 100 g sodium hydroxide], stir until dissolved
(2) [(1) + 97.5 g alumina trihydrate], stir at 100˚C until dissolved, cool to 25˚C
(3) [(2) + 202.5 g water], mix
(4) [100 g of solution (3) + 612 g water + 59.12 g sodium hydroxide], mix until dissolved
(5) [219.7 g sodium silicate solution + 612 g water + 59.12 g sodium hydroxide], mix until dissolved
(6) [(4) + (5)], combine quickly and stir for 30 minutes[d]

Crystallization

Vessel: polyethylene bottles
Temperature: 90˚C
Time: 8 hours
Agitation: none

Product Recovery

(1) Filter and wash to pH < 10
(2) Dry at 100˚C, equilibrate over saturated aqueous NaCl
(3) Yield: near 100% on Al_2O_3

Product Characterization

XRD 100% FAU, a_0 = 24.92 Å, competing phases: LTA, GIS, ANA, SOD
Elemental Analysis: $NaAlO_2$. 1.24 SiO_2[f]
Crystal Size and Habit: spherical aggregates, 0.8 μm dia.

References

[1] H. Lechert, H. Kacirek, Zeolites 11(1991) 720
[2] H. Lechert, H. Kacirek, Zeolites 13 (1992) 192
[3] G. H. Kühl, Zeolites 7 (1987) 451

Notes

a. NaX zeolites are easily obtained with gel $SiO_2/NaAlO_2$ = 1.4-5.0, $NaOH/NaAlO_2$ = 3.8-20 and $H_2O/NaAlO_2$ = 150-400.

b. Crystallization at lower water contents suffers from the high initial viscosities of the batches, thus preventing sufficient homogenization. NaX can be obtained without precautions down to $H_2O/NaAlO_2$ = 80. The crystallizing zeolite and its composition depend strongly on the alkalinity being held in the solution phase during the nucleation and growth of the zeolite. For a given batch composition, if the water content is decreased appreciably, the alkalinity will increase. If there are reasons to decrease the water content, the NaOH content should be decreased. Good results were obtained by reducing the alkali content proportional to the water content.

c. For the batch preparation, the authors would always prefer sodium aluminate instead of alumina trihydrate. Problems often occurred with the solubiity of the $Al(OH)_3$ at the given NaOH concentration, depending on the alumina source. Sodium aluminate is usually available only in technical-grade quality. If only small quantities of NaX or pure substances are desired, it was preferable to use $AlCl_3$ as the alumina source and to increase the NaOH content of the batch by 3 NaOH on the given batch composition to: $AlCl_3$: 4 SiO_2 : 20 NaOH : 325 H_2O. The resulting NaCl does not disturb the crystallization.

d. Longer times of homogenization give narrower particle size distributions.

e. Preferable crystallization temperatures: 67° to 97°C. For safety the crystallization time should be increased to 12 to 14 hours. Experiments have shown that in the given batches up to 30 hours at 90°C no other zeolite impurities were observed.

f. The Si/Al ratio of the product depends strictly on the NaOH concentration in the batch. Below 2.0 NaOH/liter, the nucleation rate of NaX goes almost to zero and nucleation of GIS occurs, which grows faster than NaX. Pure FAU with Si/Al> 1.5 in the product cannot be obtained without seeding. Above about 3.0 NaOH/liter, analcime or sodalite is obtained. Below Si/Al = 12.4, NaA appears. [3]