

OFF

Linde Type T

Si(78), Al(22)

Contributed by Andrzej Cichocki

Verified by S. Khvoshchev, M. Shubaeva, J. Warzywoda, J. Parise, M. Kleinsorge, S. Park and C. Liu

Type Material $\text{Na}_{2.9}\text{K}_{5.4}[\text{Al}_{8.3}\text{Si}_{27.7}\text{O}_{72}] \cdot w\text{H}_2\text{O}$ (w ~7)

Method A. Cichocki, P. Koscielniak [1, 2]

Batch Composition 4.18 Na_2O : 1.25 K_2O : Al_2O_3 : 16.5 SiO_2 : 175 H_2O

Source Materials

distilled water

sodium hydroxide (97% NaOH)

potassium hydroxide (86% KOH)

silica sol (Rudniki Chemical Works, 29.5% SiO_2 , 0.2% Na_2O)

sodium aluminate solution (26.6% Al_2O_3 19.6% Na_2O) = NaAlO_2

Batch Preparation (for 14 g dry product)

- (1) [9.57 g water + 4.67 g sodium hydroxide + 3.24 g potassium hydroxide], dissolve and cool to room temperature
- (2) [67.0 g silica sol + (1)], mix in a porcelain mortar and stir for 2 minutes ^a
- (3) [(2) + 7.64 g sodium aluminate solution], add NaAlO_2 drop by drop to the stirred silicate over a 10 min. interval and continue stirring for 20 minutes

Crystallization

Vessel: stainless steel autoclave, 120 cm^3 capacity

Aging: 24 h at room temperature

Temperature: 140 °C

Time: 7 days

Agitation: none

Product Recovery

- (1) Cool; transfer the reaction mixture to a mortar and grind
- (2) Filter and wash in a Buechner funnel until pH of the filtrate is 10
- (3) Dry at 110 °C
- (4) Equilibrate in laboratory air for a few days
- (5) Yield: near 95% on Al_2O_3 and 41.5% on SiO_2 ^b

Product Characterization

XRD pure OFF ^c competing phases: PHI, CHA, LTL ^d

Elemental Analysis: 0.35 Na_2O : 0.65 K_2O : Al_2O_3 : 6.65 SiO_2

Crystal Size and Habit: rods and "bones" type crystals ~9 μm long on average (longest ~22 μm)

References

- [1] A. Cichocki, P. Koscielniak, M. Michalik, M. Bus, *Zeolites* 18 (1997) 25
- [2] A. Cichocki, P. Koscielniak, *Micropor. Mesopor. Mater.* 29 (1999) 369
- [3] A. Cichocki, *Zeolites* 11(1991) 758
- [4] R. Aiello, R. M. Barrer, *J. Chem. Soc.* 1970 (A), 1470

Notes

- a. The use of a porcelain mortar gives a good mix of the reagents, particularly aluminate and silica sol.
- b. 39.9% of the sum of Na₂O, K₂O, Al₂O₃ and SiO₂ masses introduced into the reaction mixture. Synthetic Erionite with SiO₂/Al₂O₃= 6.55 forms from a reaction mixture of composition 8.09 Na₂O : 2.38 K₂O : Al₂O₃ : 27.0 SiO₂ : 448 H₂O when crystallization is carried out in a stainless steel autoclave at the temperature 373 K and time 7 days, but the yield is reduced to 22.5%. This composition differs slightly from that given in ref. [3] where NaOH and KOH were assumed 100%.
- c. The Na/K-system synthetic product shows domains of erionite and offretite in a single crystal. The XRD pattern agrees with natural offretite (PDF), but the product shows adsorption properties erionite. Unfaulted erionite crystallized in the Na/Me₄N-system. [41
- d. Formation of PHI is favored by decreasing relative alkalinity (OH⁻/SiO₂). Decreasing the temperature of crystallization favors CHA. Increasing relative alkalinity leads to formation of LTL.