LTA Linde Type A Si(50), Al(50)

Contributed by R. W. Thompson and K. C. Franklin

Verified by B. Subotic, A. Cizmek and K. Hashimoto

Type Material $Na_{12}[(AlO_2)_{12}(SiO_2)_{12}] : 27\ H_2O$

Method R W. Thompson, M. J. Huber [1]

Batch Composition $3.165\ Na_2O : Al_2O_3 : 1.926\ SiO_2 : 128\ H_2O$ [a]

Source Materials

deionized water
sodium hydroxide (Fisher Scientific, 99+% NaOH)
sodium aluminate (Fisher Scientific, NaO_2: Al_2O_3: 3 H_2O)[b]
sodium metasilicate (Fisher Scientific, Na_2SiO_3: 5 H_2O)

Batch Preparation (for 10 g dry product)

(1) [80 mL water **+** 0.723 g sodium hydroxide], mix gently until NaOH is completely dissolved. Divide into two equal volumes in polypropylene bottles
(2) [One-half of (1) **+** 8.2 58 g sodium aluminate], mix gently in capped bottle until clear [c]
(3) [Second half of (1) **+** 15.48 g sodium metasilicate], mix gently in capped bottle until clear[c]
(4) [(2) **+** (3)], pour silicate solution into aluminate solution quickly; a thick gel should form. Cap tightly and mix until homogenized [d]

Crystallization

Vessel: 100-150 mL polypropylene bottle (sealed)
Incubation: none required
Temperature: 99 ± 1°C
Time: 3-4 hours [e]
Agitation: stirred or unstirred

Product Recovery

(1) Remove from heat source and cool to below 30°C
(2) Filter to recover solids and wash with deionized water until filtrate pH is below 9 [f]
(3) Dry product on filter paper and watch glass at 80-110°C overnight
(4) Yield: 28.1 g (hydrated) or 10.4 g (dry)

Product Characterization

XRD LTA; characteristic strong reflections at d = 4.107, 3.714, 3.293 and 2.987 Å
Competing phases (if present): SOD (HS), GIS (Pc)
Elemental Analysis: Na_2O . Al_2O_3. 2 SiO_2
Crystal Size and Habit: cubic crystals, 2-3 μm [g]

References

[1] R W. Thompson, M. J. Huber, J. Cryst. Gr. 56 (1982) 711
[2] D. W. Breck, Zeolite Molecular Sieves, John Wiley, New York, 1974, p 270
[3] J. F. Charnell, J. Cryst. Gr. 3 (1971) 291
[4] H. Neels, W. Schmitz, E.-M. Berger, D. Lutz, Krist. Tech. 13 (1978)1345
[5] G. Scott, A. G. Dixon, A. Sacco, Jr., R W. Thompson, in Stud. Surf. Sci. Catal. 49, P. A. Jacobs, R. A. Van Santen (eds.), Elsevier, Amsterdam,1989, p 363

Notes

a. Zeolite NaA can be synthesized from a wide range of batch compositions as noted in Breck [2] and temperatures other than used in this example, *e.g.,* 60-110°C. Also, it can be made from a variety of alumina and silica source materials other than those used in this example, e.g., pure aluminum powder[h] or aluminum wire, fumed silica, sodium disilicate, Ludox, silica gels, etc. The actual weights of these other sources used must be compensated for the presence of water of hydration, Na^+ ions, etc.
b. Assumed 100%.
c. Could take 10-20 minutes.
d. May be done with laboratory mixer or vigorously by hand for 5-10 minutes.
e. The turbid gel phase will be observed to diminish in height as the reaction proceeds, accelerating rapidly in the final stages of the crystallization, leaving a clear supernatant above the precipitated crystalline phase.
f. One-half liter should be sufficient for this preparation.
g. Zeolite NaA crystals are typically cubic. Dodecahedral crystals have been observed frequently, but there is little fundamental understanding of why this habit forms. Additions of triethanolamine are known to result in larger crystals, but the particle size distribution becomes broader, synthesis times are extended, and the impurity zeolite phases appear with increased abundance. [3-5]
h. Special care must be taken if powdered aluminum is dissolved in a caustic solution to make the aluminate solution. Since its dissolution is exothermic, the solution can become quite warm and hydrogen evolves.