

LTA

Zeolite Alpha

Si(75), Al(25)

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Type Material $\text{Na}_4(\text{TMA})_2[\text{Al}_6\text{Si}_{18}\text{O}_{48}]$ (TMA = tetramethylammonium)

Method G. H. Kühn [1]

Batch Composition 1.07 Na_2O : 2.37 $(\text{TMA})_2\text{O}$: Al_2O_3 : 10 SiO_2 : 120 H_2O

Source Materials

distilled water

sodium aluminate (MCB, 28.5% Na_2O , 42.75% Al_2O_3)

tetramethylammonium hydroxide solution (25% TMAOH) ^a

precipitated silica (PPG Corp. Hi-Sil 233, 88% SiO_2) ^b

Batch Preparation (for 36 g product)

- (1) (46.4 g water + 15.6 g sodium aluminate), stir at room temperature until dissolved ^c
- (2) [(1) + 111.7 g tetramethylammonium hydroxide solution], mix
- (3) (2) + 44.5 g precipitated silica, add silica to the aluminate solution gradually with stirring ^d
- (4) Stir or blend for 30 minutes ^d

Crystallization

Vessel polypropylene bottle

Incubation: 48 h at room temperature

Time: 24- 30 hours ^e

Temperature: 90°C

Agitation: none

Product Recovery

- (1) Dilute reaction mixture with water
- (2) Filter on a dense filter, such as Whatman #5, or separate by decantation, then reslurry sediment, flocculate, ^f and wash with water ^g
- (3) Dry at room temperature or at 110°C
- (4) Yield: 36 g (near 100% on Al_2O_3)

Product Characterization

XRD: LTA (contracted unit cell); competing phase: high-silica sodalite

Elemental Analyses: 0.6 Na_2O : 0.4 $(\text{TMA})_2\text{O}$: Al_2O_3 :6 SiO_2

Crystal size and habit: cubes, <1 μm on edge

Reference

- [1] G. H. Kühn, US Patent 4 191 663

Notes

- a. TMA salts cannot be used because the anions tend to cause nucleation of high-silica sodalite ($\text{SiO}_2/\text{Al}_2\text{O}_3 = 10$).
- b. Hi-Sil 233 has a median particle size of 18-19 μm ; precipitated silica of larger particle size tends to be insufficiently reactive. Ultrasil 320 is an acceptable substitute. If less reactive silica is to be used in this preparation, 10% of the silica should be slurried in the (TMA)OH solution prior to combining the (TMA)OH with the NaAlO_2 solution.
- c. Small amounts of iron may be removed by filtration although this iron does not affect the crystallization. If the sodium aluminate does not dissolve completely, it probably contains $\text{Al}(\text{OH})_3$ and cannot be used.
- d. Slow addition of Hi-Sil is recommended for proper dispersion. Silica-rich gel particles tend to cause nucleation of high-silica sodalite.
- e. The longer crystallization time improves the crystallinity, unless sodalite nuclei are present.
- f. Avoid flocculating in the presence of mother liquor because colloidal silica will coagulate.
- g. Alternatively, use repeated decantation and reslurrying sequences (settling may be accelerated by centrifuging), and optionally, flocculation^h after having removed the bulk of the alkalinity.
- h. See Introductory Article on "Product Recovery."