

LTA

ZK-4

Si(59), Al(41)

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Type Material $\text{Na}_{9.2}(\text{TMA})_{0.8}[\text{Al}_{10}\text{Si}_{14}\text{O}_{48}]$ (TMA = tetramethylammonium)

Method G. T. Kerr [1]

Batch Composition 1.55 Na_2O : Al_2O_3 : 3.91 SiO_2 : 4.13 $(\text{TMA})_2\text{O}$: 320 H_2O

Source Materials

distilled water

sodium hydroxide (50% solution)

sodium aluminate (~ 46% Al_2O_3 , 31% Na_2O ; Fisher, MC&B, Nalco)

tetramethylammonium hydroxide (TMA-OH, Southwestern Analytical Chemical, 25% aqueous solution)

silica sol (Dupont HS-40 or AS-40, 40% SiO_2)

Batch Preparation (for 34 g product)

- (1) [290 g water + 6.0 g sodium hydroxide solution + 21.5 g sodium aluminate], stir until dissolved
- (2) [292 g TMA-OH (25% solution) + 57.0 g silica sol], stir for approximately 30 minutes
- (3) [(1) + (2)], stir vigorously; gel pH = 14.0 to 14.5

Crystallization

Vessel: Teflon bottle, 1000 mL

Incubation: 24 hours at 25°C (optional)

Temperature: 100°C (oven with efficient air circulation)

Time: 16-48 hours

Agitation: none

Product Recovery ^a

- (1) Filter and wash with 0.5 to 1 L water
- (2) Dry at 100°C
- (3) Yield: approximately 34 g (100% on Al_2O_3)

Product Characterization

XRD LTA, $a_0 = 24.38 \text{ \AA}$; competing phases: GIS (long reaction time) and EAB ^b

Elemental Analysis (dried at 100°C): 15.7% Al (29.7% Al_2O_3), 23.1% Si (49.4% SiO_2), 12.4% Na (16.7% Na_2O), 2.24% C (3.83% $(\text{TMA})_2\text{O}$) ^c

Crystal Size and Habit: cubes (some with penetration twinning) approximately 1.0-1.5 μm on an edge

References

- [1] G. T. Kerr, Inorg. Chem. 5 (1966) 1537

- [2] R. H. Jarman, M. T. Melchior, D. E. W. Vaughan, ACS Symposium Series 218, American Chemical Soc., Washington, D. C., 1983, p 267

Notes

- a. Although no decomposition of TMA-OH is expected, it is advisable to carry out the crystallization and product work-up in a fume hood. Temperature excursions can produce noxious and toxic by-products, *e.g.*, trimethylamine and methanol.
- b. EAB can co-precipitate with ZK-4 if the TMA-OH is added to solution (1) before the silica sol.
- c. The Si/Al of products by this recipe ranged from 1.39 to 1.43 (average 1.41). Higher and lower Si/Al products have been made using other recipes. [2]