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Type Material K₁₀ (Al₁₀Si₁₀O₄₀) · wH₂O (w ~ 8)

Method J. Warzywoda, R W. Thompson [1]

Batch Composition 5.26 K₂O : Al₂O₃ : 3 SiO₂ : 94.5 H₂O

Source Materials

deionized water

potassium hydroxide (pellets, 85% KOH min.)

aluminum (wire, 99.999% Al)

silica (Cab-O-Sil M5, amorphous fumed SiO₂)

Batch Preparation (for 4 g product)

- (1) [20.0 g water + 9.26 g potassium hydroxide]; dissolve KOH pellets in HDPE ^a bottle. Divide into two equal portions
- (2) [First half (1) + 0.719 g aluminum];^b dissolve the Al wire in half of the KOH solution by heating under reflux in a Teflon flask. Cool, filter and store in a HDPE bottle
- (3) [Second half (1) + 2.42 g silica]; dissolve the silica in the remaining KOH solution and heat at 80-95°C in a HDPE bottle. Cool, filter and store in a HDPE bottle^c
- (4) [(2) + (3)]; heat (2) and (3) in HDPE bottles to 80-95°C and mix together. Mix the resulting gel for 1-2 minutes at 1000 rpm with a mechanical stirrer to homogenize it ^e

Crystallization

Vessel: HDPE bottle

Temperature: 95°C

Time: 96 hours

Agitation: static conditions with occasional mixing

Product Recovery

- (1) Filter to recover solids
- (2) Wash with deionized water until the pH of wash water is neutral
- (3) Dry at 80°C
- (4) Yield: 4.4 to 4.7 g (near 100% based on Al₂O₃)

Product Characterization

XRD: Linde Type F (ref. [2], Table III, page 35)

Elemental Analyses: K₂O : Al₂O₃ : 2SiO₂ : 3H₂O [3]

Crystal size and habit: small submicron prismatic crystals forming 0.5 to 3 μm aggregates

References

- [1] J. Warzywoda, R W. Thompson, Zeolites 11(1991) 577
- [2] J. D. Sherman, in ACS Symp. Series 40, J. R Katzer (ed.), Am. Chem. Soc., Washington, D.C., 1977, p 30
- [3] R M. Barer, J. W. Baynham, J. Chem. Soc. (1956) 2882

Notes

- a. High density polyethylene.
- b. The dissolution of aluminum powder in these caustic solutions generates heat and hydrogen and can be somewhat violent.
- c. To avoid precipitation of solids from solutions (2) and (3), carry out crystallization immediately after solution preparations is complete.
- d. A viscous alumino silicate gel is formed instantaneously.
- e. If no mechanical stirring is used, the gel appears to be very viscous with no visible fluid phase, and homogenization may be difficult. Brief heating of the gel at 95°C will give a fluid phase and hand shaking can be used to homogenize it.