Contributed by J. P. Verduijn

Verified by C. Williams, by M. Uguina, and by J. Warzywoda

Type Material K₉[Al₆Si₂₇O₇₂] : wH₂O (w = 0 to 36)

Method J. P. Verduijn [1]

Batch Composition 2.35 K₂O : Al₂O₃ : 10 SiO₂ : 160 H₂O : trace MgO a

Source Materials
deionized water
potassium hydroxide (Baker 0222 pellets, 86.8% KOH)
alumina (Baker 0005, 92.6% Al(OH)₃)
silica sol (Dupont Ludox HS-40, 40% SiO₂)
magnesium nitrate (Baker, Mg(NO₃)₂. 6 H₂O)

Batch Preparation (for 59 g product)
(1) [50.00 g water + 30.39 g potassium hydroxide + 15.82 g alumina], heat to boiling until clear. Cool to room temperature and correct water loss due to boiling
(2) [150.24 g silica sol + 99.0 g water + 14.5 g Mg(NO₃)₂ solution], b mix until homogeneous (~3 minutes)
(3) [(1) + (2) + 25.00 g water (rinse)], c mix until thickening starts (~ 3 minutes) d

Crystallization
Vessel: 300 mL stainless steel autoclave e
Temperature: 175°C
Time: 48 hours f
Agitation: none

Product Recovery
(1) Cool to room temperature
(2) Filter and wash (5 times) with 650 mL water. The pH of the last wash water should be ~10
(3) Dry at 150°C for 16 hours
(4) Yield: After drying at 150°C, ~ 15.3 wt% based on the weight of synthesis gel in the autoclave (nearly 100% on Al₂O₃)

Product Characterization
XRD: LTL (only crystalline product). Competing phases: MER (without MgO)
Elemental Analysis: 6.2 SiO₂/Al₂O₃, 1.0 K₂O/Al₂O₃
Crystal Size and Habit: cylindrical, 0.2 to 0.4 μm diameter, 0.4 to 0.7 μm long (L/D ~ 2)

Reference
Notes
a. The synthesis mixture contains 9 wt ppm of added Mg$^{2+}$ species (based on the weight of the synthesis mixture).
b. Mg(NO$_3$)$_2$ solution: dissolve 2.5645 g magnesium nitrate (Mg(NO$_3$)$_2$. 6 H$_2$O) in 997.4 g water. This solution contains 0.24 mg Mg$^{2+}$/g solution. The function of the Mg$^{2+}$ species is to avoid the formation of byproducts such as MER, and to control the particle size of the LTL product.
c. This water is used to quantitatively transfer the aluminate solution.
d. After 3 minutes mixing, the gel is still pourable. Longer mixing is permitted, but the gel then tends to stiffen and is difficult to transfer to the autoclave.
e. No Teflon liner was used.
f. Crystallization time is not critical (24 to 72 hours).