LTL

Linde Type L

Si(76), Al(24)

Contributed by J. P. Verduijn

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Type Material $K_9[Al_9Si_{27}O_{72}]$: wH_2O (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)], 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
- Yield: After drying at 150°C, ~ 15.3 wt% based on the weight of synthesis gel in the arnoclave (nearly 100% on Al₂O₃)

Product Characterization

XRD: LTL (only crystalline product). Competing phases: MER (without MgO) Elemental Analysis: $6.2 \, \text{SiO}_2/\text{Al}_2\text{O}_3$, $1.0 \, \text{K}_2\text{O}/\text{Al}_2\text{O}_3$ Crystal Size and Habit: cylindrical, $0.2 \, \text{to} \, 0.4 \, \mu\text{m}$ diameter, $0.4 \, \text{to} \, 0.7 \, \mu\text{m}$ long (L/D ~ 2)

Reference

[1] J. P. Verduijn, US Patent 5 242 675 (1993)

Notes

- a. The synthesis mixture contains 9 wt ppm of added Mg²⁺ 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_2O) in 997.4 g water.
 - This solution contains 0.24 mg Mg²⁺/g solution. The function of the Mg²⁺ 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).