PHI High-alumina Philipsite Si(67), Al(33)

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Type Material $Na_{6.3}K_{4.2}[Al_{10.5}Si_{21.5}O_{64}]$. 23 H_2O[a]

Method A. Cichocki [1,2]

Batch Composition 1.53 Na_2O : 0.44 K_2O : Al_2O_3 : 5.0 SiO_2 : 82.7 H_2O

Source Materials

distilled water
sodium hydroxide (reagent grade, 97% NaOH)
potassium hydroxide (reagent grade, 86% KOH)
silica sol (Rudnild Chemical Works, 29.5% SiO_2, 0.22% Na_2O)
sodium aluminate solution (26.6% Al_2O_3, 19.6% Na_2O)[b]

Batch Preparation (for 26 g product)

(1) [36.0 g water + 1.53 g sodium hydroxide + 3.78 g potassium hydroxide], dissolve and cool to room temperature
(2) [67.0 g silica sol + (1), mix in a porcelain mortar and stir for 2 minutes
(3) [(2) + 25.22 g sodium aluminate solution], add sodium aluminate drop by drop to the stirred silicate over a 10 minutes period and continue stirring for 20 minutes

Crystallization

Vessel: stainless steel autoclave, 120 cm^3 capacity
Aging: 24 hours at room temperature
Temperature: 100°C
Time: 7 days
Agitation: none

Product Recovery

(1) Cool to room temperature; transfer the reaction mixture to a porcelain mortar and grind
(2) Filter and wash in a Buechner funnel until pH of the filtrate is ~10
(3) Dry at 110°C
(4) Equilibrate in laboratory air for a few days
(5) Yield: 25.8 g (near 100% on Al_2O_3) [c]

Product Characterization

XRD: Pure phillipsite (ZK-19 [3] in ASTM Powder Diffraction File) competing phases: ANA, CHA, ERI, LTL
Elemental Analysis: 0.60 Na_2O : 0.41 K_2O : Al_2O_3 : 4.07 SiO_2 : 4.30 H_2O[d]
Crystal Size and Habit: ~0.5 to 3 μm diameter, round twinned polycrystals

References

[1] A. Cichocki, Zeolites 11(1991) 758
[2] A. Cichocki, PL Patent 161 557 (1993)
[3] G. H. Kuehl, Am. Mineral. 54 (1969) 1607
[4] A. Cichocki, PL Patent 100 912 (1979)
[5] A. Cichocki, J. Grochowski, L. Lebioda, Kristall. Technik 14 (1979) 9
[6] A. Cichocki, PL Patent 162 653 (1993)

Notes

a. Al content of the unit cell varies from 9.5 to 12.8. More siliceous synthetic-type phillipsite forms when (1) higher silica batch composition is used or (2) borosilicate glass corrodes in the alkaline reaction mixture. Phillipsite with SiO_2/Al_2O_3 = 6.27 crystallized in a stainless steel autoclave from a reaction mixture of composition 7.16 Na_2O : 2.81 K_2O : Al_2O_3 : 26.5 SiO_2 : 442 H_2O. [4,5] Phillipsite with SiO_2/Al_2O_3 = 4.34 forms from a reaction mixture of composition 1.29 Na_2O : 0.37 K_2O : Al_2O_3 : 4.20 SiO_2 : 69.5 H_2O when crystallization is carried out in a borosiicate glass vessel. These compositions differ slightly from those given in reference [41 and [6] where NaOH and KOH were assumed 100%.

b. Prepared by the reaction of aluminum shavings with concentrated sodium hydroxide solution (~30 wt% NaOH).

c. Dry product contains 72.0% of the sum of Na_2O, K_2O, Al_2O_3 and SiO_2 masses introduced into the reaction mixture.

d. Sorptive Capacity (g/g at p/p_0 = 0.2): 0.185 for H_2O at 298 K, 0.002 for Ar at 77 K.