BEA Beta Si(93), Al(7)

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Type Material $Na_{0.92}K_{0.62}(TEA)_{7.6}[Al_{4.53}Si_{59.47}O_{128}]$ [a]

Method M. A. Camblor, J. Pérez-Pariente [1]

Batch Composition 1.97 Na_2O : 1.00 K_2O : 12.5 $(TEA)_20$: Al_2O_3 : 50 SiO_2 : 750 H_2O: 2.9 HCl [b]

Source Materials

deionized water
tetraethylammonium hydroxide (Alfa 40 wt% TEAOH, K < 1 ppm, Na <3 ppm)
sodium chloride (reagent grade)
potassium chloride (reagent grade)
silica (Degussa Aerosil 200, 99+% SiO_2)
sodium hydroxide (Prolabo reagent grade, 98%)
sodium aluminate (Carlo Erba, 56 wt% Al_2O_3, 37 wt% Na_2O)

Batch Preparation (for 20 g product)

(1) [59.4 g water + 89.6 g TEAOH (40%) + 0.53 g sodium chloride + 1.44 g potassium chloride], stir until dissolved
(2) [(1) + 29.54 g silica], stir until homogenized (10 minutes minimum)
(3) [20.0 g water + 0.33 g sodium hydroxide + 1.79 g sodium aluminate], stir until dissolved
(4) [(2) + (3)], stir for 10 minutes, (gives a thick gel)

Crystallization

Vessel: 60 mL stainless steel autoclaves with Teflon liners
Temperature: 135 ± 1°C
Time: 15 to 20 hours
Agitation: autoclaves are rotated (60 rpm) [c]

Product Recovery

(1) Quench autoclaves in cold water, product pH = 12.8 ± 0.1
(2) Centrifuge (10,000 rpm), wash until pH ~9 and dry overnight (77"C)
(3) Yield: 9.9 ± 0.2 g of solid / 100 g gel (~ 90% on Al)

Product Characterization

XRD: zeolite beta (no other phases)
Elemental Analysis (content per unit cell): $Na_{0.9}K_{0.62}(TEA)_{7.6}[Al_{4.53}Si_{59.47}O_{128}]$ [d] (Si/Al = 13.1±0.1)
Crystal Size and Habit: The crystals are round-shaped. They do not show any particular crystal habit. Average crystal size is 0.20 μm, and the crystal size distribution is very narrow. (The size of ~90% of crystals is between 0.10-0.30 μm)

References

[1] M. A. Camblor, J. Pérez-Pariente, Zeolites 11(1991) 202
[2] M. A. Camblor, A. Mifsud, J. Pérez-Pariente, Zeolites 11(1991) 792

Notes

a. Highly siliceous beta (Si/Al ~ 100 can be obtained by using tetraethyl-orthosilicate as silica source [2].
b. $OH^-/SiO_2 = 0.56$
c. In the specific synthesis conditions given in the recipe, the agitation has practically no influence on the properties of the product. However, by using different synthesis conditions, large differences in total crystallization time, average crystal size and crystal size distribution can be found between static and agitated synthesis.
d. Excess cations assumed to be occluded TEAOH or TEA^+ compensating SiO- structure defects.