

ANA

Analcime

Si(68), Al(32)

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Type Material $\text{Na}_x[\text{Al}_x\text{Si}_{48-x}\text{O}_{96} : 16 \text{ H}_2\text{O}$ ($x = 15$ to 17)

Method Developed from J. F. Charnell [1], and A. Dyer, A. M. Yusof [2]

Batch Composition 4.5 Na_2O : Al_2O_3 : 4.5 SiO_2 : 3.0 H_2SO_4 : 380 H_2O : 6.1 triethanolamine ^a

Source Materials

demineralized water

aluminum sulfate [General Purpose Reagent, $\text{Al}_2(\text{SO}_4)_3 \cdot 16 \text{ H}_2\text{O}$]

sodium metasilicate (Technical Grade, $\text{Na}_2\text{SiO}_3 \cdot 5 \text{ H}_2\text{O}$)

triethanolamine [General Purpose Reagent, $\text{N}(\text{C}_2\text{H}_4\text{OH})_3$]

Batch Preparation (for approximately 7 g product) [3,4]

- (1) [55 g water + 9.8 g aluminum sulfate], stir until dissolved; filter through 0.7 micron glass microfibre filter
- (2) [36.5 g water + 14.9 g sodium metasilicate + 12.2 g triethanolamine], stir until dissolved; filter through 0.7 micron glass microfibre filter
- (3) [(1) + (2)], stir gently until gel thickens (do not over-stir)

Crystallization

Vessel: 150 mL Teflon-lined autoclave ^b

Time: 24 hours

Temperature: 200 °C (autoclave heated in the oven)

Agitation: none

Product Recovery

- (1) Cool to room temperature and filter to recover solids
- (2) Wash with distilled water until pH of filtrate < 10
- (3) De-agglomerate by adding 10 mL of 10% ethanol in water and immerse in a 150 watt ultrasonic bath for approximately 1 hour
- (4) Dry at 100 °C
- (5) Yield: approximately 90%

Product Characterization

XRD: ANA (only crystalline phase)

Elemental Analysis: 1.06 Na_2O : Al_2O_3 : 4.3 SiO_2 : 2 H_2O

H_2O (Wt. loss at 500 °C): 8.29% (± 0.20)

Na_2O : 14.6% (± 1.5)

Al_2O_3 : 22.7% (± 1.5)

SiO_2 : 57.3% (± 0.5)

Crystal Size and Habit: polycrystalline non-porous spherulites $\leq 180 \mu\text{m}$ dia. [2]

References

- [1] J. F. Charnell, J. Cryst. Growth 8 (1971) 291
- [2] A. Dyer, A. M. Yusof, Zeolites 7 (1987) 191
- [3] B. W. Garney, Fusion Technology 21 (1992) 604
- [4] B. W. Garney, UK Patent Application 9011151.9

Notes

- a. This preparation gives the non-porous form of analcime. Replacing aluminum sulfate with an equivalent weight of sodium aluminate gives the porous form of analcime. Gel composition: 6.5 Na₂O : Al₂O₃ : 4.5 SiO₂ : 380 H₂O : 6.1 triethanolamine.
- b. The method of heating the gel is very important if large crystals are required. The largest crystals were obtained when the autoclave was placed in a laboratory oven where the heat flow was uniform all around the pot. Experiments using autoclaves which were heated by electrical jackets around the sides were not so successful.