MAZ Mazzite Si(78), Al(22)

Contributed by David Vaughan and Karl Strohmaier

Verified by S. Yang and A. Navrotsky, C. Williams and C. Round, Hyun-ku Rhee and J. Kornatowski

Type Material $Na_{73}(TMA^+)_{0.7}A1_8Si_{28}O_{72}$: wH_2O (TMA = tetramethylammonium)

Method D. E. W. Vaughan [1]

Batch Composition 3.35 Na_2O : 1.24 (TMA)Br : Al_2O_3 : 9.17 SiO_2 : 125 H_2O : 0.66 Na_2SO_4

Source Materials

deionized water
sodium hydroxide (J. T. Baker, ~ 99% NaOH)
alumina (Alcoa C-31, assumed 100% Al_2O_3. 3 H_2O)
sodium silicate (PQ Corp. N brand, 8.9% Na_2O, 28.7% SiO_2)
tetramethylammonium bromide (RSA)
aluminum sulfate (J. T. Baker, $Al_2(SO_4)_3$: 17 H_2O)

Batch Preparation (for 112 g dry product)

Preparation of Seed Solution (2)

(1) [30 g water + 16 g NaOH + 3.25 g alumina] reflux until a clear solution forms, then cool to room temperature and add water back to the original weight if necessary
(2) [54.4 g sodium silicate + 31.3 g water + (1)], add sodium aluminate solution slowly with mixing in a 200 mL Waring blender
(3) Age for 24 hours at room temperature [a]

Preparation of Crystallization Batch

(4) [50 g water + 19.6 g NaOH + 25.1 g alumina], reflux until clear. Cool to room temperature and add water to attain the original weight
(5) [50 g water + 40 g tetramethylammonium bromide], mix until dissolved.
(6) [396.4 g sodium silicate + 35 g water + 13.9 g (3) + (5)]. Add components sequentially with mixing in a Pyrex one-L reaction kettle with mixing. Heat mixture to 80°C
(7) [50 g water + 30 g aluminum sulfate], mix until dissolved
(8) [(6) + (4) + (7)], add sodium alumina to solution followed by alum solution with stirring at 80°C [b]
(9) Increase temperature to 100°C and stir until homogeneous

Crystallization

Vessel: one-L Pyrex reaction kettle with reflux condenser and stirrer[c]
Time: 40+ hours [d]
Temperature: 100°C
Agitation: None, except just prior to sampling

Product Recovery

(1) Vacuum filter on a Buchner funnel
(2) Wash to pH < 10
(3) Dry at 110°C
(4) Yield near quantitative on Al_2O_3

Characterization

XRD excellent MAZ
Elemental analysis: 0.92 Na_2O : 0.1 $(TMA)_2O$: Al_2O_3 : 7.14 SiO_2
Crystal size and habit: barrel-shaped aggregates of needle-like crystals (2 to 3 μ long and 0.1 μm dia.) [e]

References

[1] D. E. W. Vaughan, Mater. Res. Soc. Symp. Proc. 111 (1988) 89
[2] D. E. W. vaughan, US Patent 4 178 352 (1979)

Notes

a. Stored at room temperature, this seed solution will be stable and usable for several months.
b. This formulation produces a hard gel when the alum is added making it difficult to fully homogenize. A Hobart or Kitchenaid mixer is better than a blender if available. The gel breaks up at about 80°C.
c. Alternatively, a one-L Teflon jar (Nalgene) or subdividing the gel between smaller vessels is appropriate.
d. After about 18 hours, faujasite is fully crystallized (Si/Al = 2.4). Continuing the crystallization for more than 40 hours produces fully crystalline MAZ. It was still pure MAZ after 7 days.
e. ^{13}C NMR shows one site in the GME cage.