MWW MCM-22 Si(94), Al(6)

Contributed by Avelino Corma

Verified by J. Weitkamp and N. Kumar

Type Material $Na_{0.08}[Al_4Si_{68}O_{144}] : (C_6H_{12}NH)_{10.8}$ [1][a]

Method A. Corma, C. Corell, J. Pérez-Pariente [1]

Batch Composition 2.7 Na_2O : Al_2O_3 : 30 SiO_2 : 1347 H_2O : 15 HMI [b] (HMI = hexamethyleneimine)

Source Materials

deionized water
sodium alummate (Carlo Erba, 56% Al_2O_3, 37% Na_2O)
sodium hydroxide (98% NaOH)
hexamethyleneimine $C_6H_{12}NH$ (Aldrich, 99% HMi)
silica (Degussa, Aerosil 200, or Cab-O-Sil M5)

Batch Preparation (for 12.8 g dry product)[c]

(1) [124.20 g water + 0.92 g sodium aluminate + 0.60 g sodium hydroxide], stir until dissolved [d]
(2) [(1) + 7.61 g hexamethyleneimine], mix thoroughly[e]
(3) [(2) + 9.23 g silica], mix thoroughly[f]

Crystallization

Vessel: PTFE-lined stainless steel autoclaves
Time: 7 days
Temperature: 150˚C
Agitation: 60 rpm

Product Recovery

(1) Dilute the reaction mixture with distilled water
(2) Filter and wash with distilled water
(3) Dry at 100˚C overnight
(4) Yield; 99% based on alumina

Product Characterization

XRD: MCM-22 [2]; competing phases: FER (when crystallized under static conditions), ZSM-5 (when gel $SiO_2/A1_2O_3$> 100), ZSM-12 (when gel SiO_2/Al_2O_3> 200)
Elemental Analysis: 0.02 Na_2O : Al_2O_3 : 34 SiO_2
Crystal Size and Habit: small thin platelets occasionally forming spherical aggregates of 6-8 μm [g]

References

[1] A. Corma, C. Corell, J. Pérez-Pariente, Zeolites 15 (1995) 2
[2] M. K. Rubin, P. Chu, US Patent 4 954 325 (1990)

Notes

a. Missing cations assumed to be protonated HMI.
b. H_2O includes water from sodium aluminate and added water.
c. Use polypropylene vessel and vigorous stirring for 30 minutes.
d. Clear solution
e. Slightly yellow clear solution
f. White and fluid gel
g. Toluene sorptive capacity at 0.1 KPa, 42°C : 10.07 mmol/g. The sample was heated in oxygen flow (30 cm^3/min^{-1}) up to 500°C and outgassed overnight at 500°C in a vacuum better than 1 mPa.