MFI Template-free ZSM-5 Si(96), Al(4)

Contributed by Wha Jung Kim

Verified by S. B. Hong, E.-P. Ng, J. Shin

Type Material: $Na_4[Al_4SiO_{92}]$: wH_2O

Method: W.J Kim and S.D. Kim [1]

Batch Composition: 10 Na_2O : 100 SiO_2 : 2 Al_2O_3 : 2250 H_2O

Source Materials

deionized water
10 wt.% NaOH solution prepared from 96 wt.% sodium hydroxide (SAMCHUN Chem. Co.)
sodium aluminate (Junsei Chem. Co., 37 wt.% Al_2O_3 and 31 wt.% Na_2O)
colloidal silica (Dupont Chem. Co., Ludox AS-40)

Batch Preparation

(1) [60g of silica sol + 21.4g of 10 wt.% NaOH solutions[a]], stir in a beaker
(2) [30g of water + (1)], stir at 200 rpm for 3h
(3) [57g of water + 1.8 g of 10 wt.% NaOH solutions + 2.2g of $NaAlO_2$], stir in a beaker for 3h
(4) [(3) +27g of water]
(5) [add (4) to (2)], stir for 1h to get a homogeneous solution

Crystallization

Vessel: Teflon-lined stainless steel autoclave equipped with a doubled-stirrer of pitched blade turbine and sampling port.
Temperature: 190° C
Time: 2h
Temperature: 150° C
Time: 35h[b]
Agitation: 200 rpm

Product Recovery

(1) Collect through the sampling port
(2) Filter and wash with water using membrane filter (Advantec MFS, Inc., pore size of 0.2 μm)
(3) Dry at ambient temperature or at 100°C

Product Characterization

XRD: fully crystalline MFI; Competing phase: mordenite at lower Si/Al ratios in the gel
Elemental Analysis: Si/Al = 25
Crystal Size and Habit: 2 μm crystals

Reference

[1] W.J. Kim and S.D. Kim, U.S. Pat. 7,361,328 (2008)
[2] S.D. Kim, S.H. Noh, K.H. Seong and W.J. Kim, Micropor. Mesopor. Mater. 72 (2004) 185
[3] S.D. Kim, Noh, J.W. Park and W.J. Kim, Micropor. Mesopor. Mater. 92 (2006) 181

Notes

a. Slowly added.
b. The reaction temperature of the resultant mixture was increased up to 190° C while performing stirring at 200 rpm, and maintained for 2 hours. Then, the reaction temperature was decreased to 150° C and maintained for 35 hours.