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Method S.I Zones [1-5]

Batch Composition 0.02 K₂O : 0.0045 Al₂O₃ : 0.16 SiO₂ : 0.04 SDA : 6.07 H₂O
(SDA = isobutylamine)

Source Materials

deionized water
potassium hydroxide (98+% Aldrich)
aluminium hydroxide (r.g., Al(OH)₃ 77 %, Alfa Aesar)
colloidal silica solution (Ludox HS 40, Aldrich)
isobutylamine (99 %, Aldrich)

Batch Preparation

- (1) [1.87 g KOH + 33.3 ml of deionized water] stir until all the pellets are dissolved.^a
Amount of all reactants divided by 4:
- (2) [(1) + 45.94 ml of deionized water + 0.715 g Al(OH)₃] stir at 500 rpm until the mixture becomes homogenous.^a
- (3) [(2) + 20.425 g of colloidal silica] added dropwise after 10 min of stirring.^a
- (4) [(3) + 3.38 ml of isobutylamine] dropwise; leave the gel stirring for 6 h at rate of 500 rpm. The gel is not dense.

Crystallization

Vessel: Teflon-lined stainless steel autoclave
Temperature: 170 °C
Time: 6 days
Vertical rotation: 35 rpm

Product Recovery

- (1) Dilute crystalline gel with water.
- (2) Filter and wash with water.
- (3) Dry at 105 °C for 12 h.
- (4) Yield: 8 g.
- (5) Calcination: heating to 500 °C for 3h, then static heating 3h at 500 °C

Product Characterization

XRD : MTT; pure SSZ-32 phase. Competing phase: MFI
SEM : Crystal size and habit: rod-like shape crystals with less than 100 nm size.

References

- [1] S.I. Zones, Zeolite SSZ-32 Google Patents (1991)
- [2] S.I. Zones, Zeolite SSZ-32 Google Patents (1993)

- [3] D.L. Holtermann, R.A. Innes, D.S. Santilli, J.N. Ziemer, S.I. Zones, Hydrocarbon conversion process using zeolite SSZ-32 as catalyst Google Patents (1994)
- [4] Y. Nakagawa, S.I. Zones, Preparation of zeolites using organic template and amine, Google Patents (1998)
- [5] S.I. Zones, B. Lee, L.-T. Yuen, T.M. Davis, J.N. Ziemer, A. Ojo, Method for preparing small crystal SSZ-32 Google Patentes (2013)

Notes

- a. Clear solution is obtained.
- b. SDA used for synthesizing SSZ-32 is isobutylamine which gives small crystals size.
- c. The source of Si is colloidal silica instead of fumed silica that was used in previous works.
- d. The above procedure is the first time tested in a rotational oven.
- e. The $\text{SiO}_2/\text{Al}_2\text{O}_3$ in this procedure is 30, within the range which favors the producing SSZ-32 over ZSM-23.