OFF [Ga] Offretite Si(65), Ga(35)

Contributed by Mario Occelli

Verified by F. Machado and S. Iwamoto

Type Material $K_{2.2}Na_{0.1}(TMA)_{4.1}Ga_{6.4}Si_{11.6}O_{36}$: wH_2O (TMA = tetramethylaminoiiium)

Method M. L Occelli [1]

Batch Composition 2.25 K_2O : 1.61 Na_2O : 0.5 $(TMA)_2O$: Ga_2O_3 : 11.7 SiO_2 : 250 H_2O : 1.0 HCl

Source Materials

deionized water
sodium hydroxide, reagent grade (~97% NaOH)
potassium hydroxide, reagent grade (~87% KOH)
gallium oxide (99.99%)
silica sol (Dupont HS-40, 39% SiO_2, 0.5% Na_2O)
tetramethylammonium chloride [99+% (TMA)Cl]

Batch Preparation

(1) [50 g water + 12.0 g sodium hydroxide + 29.0 g potassium hydroxide], mix until dissolved; heat to boiling
(2) [(1) + 18.7 g gallium oxide], stir at boiling point until a clear solution is obtained
(3) [(2) + 30 g water], mix and cool to ambient temperature [a]
(4) [20 g water + 11.0 g (TMA)Cl], mix until dissolved
(5) [(3) + (4)], mix thoroughly
(6) [180.3 g silica sol + 234.8 g water], mix for 4 hours
(7) [(6) + (5)], add gallate mixture dropwise to the vigorously-stirred diluted silica sol. Continue stirring in a round-bottomed flask for 10 hours

Crystallization

Vessel: 1000 mL round bottomed flask with agitator and reflux condenser
Incubation: 10 h at ambient temperature with stirring
Temperature: 98˚C [b]
Time: 3 days [c]
Agitation: Continue stirring until temperature reaches 98°C, then discontinue stirring for balance of crystallization period

Product Recovery

(1) Filter and wash with deionized water until pH < 10
(2) Dry at 110˚C
(3) Yield: near 100% on Ga_2O_3

Product Characterization

XRD: OFF, no other crystalline products or amorphous material detected
Elemental Analyses: 49.5% SiO_2, 42.2% Ga_2O_3, 7.2% K_2O, 1.1% Na_2O [d]

References

[1] M. L Occelli, US Patent 5 133 951 (1992)
[2] M. L Occelli, H. Eckert, C. Hudalla, A. Auroux, P. Ritz, P. S. Iyer, Micropor. Mater. 10 (1997) 123-135
[3] M. L Occelli H. Eckert, C. Hudalla, A. Auroux, P. Ritz, P. S. Iyer, in Proceedings of the 11th International Zeolite Conference, Seoul, Korea, Hakze Chon, Son-Ki Ihm (eds.), Elsevier, Amsterdam, 1997, p 1981
[4] M. L Occelli, H. Eckert, P. S. Iyer, P. Ritz, in Synthesis of Porous Materials, M. L Occelli, H. Kessler (eds.), Marcel-Dekker (1997), p. 283

Notes

a. Diluted Na,K gallate solution must be clear.
b. Stirring is continued as temperature is increased to 98°C, then discontinued for the remainder of the crystallization treatment.
c. Methods to reduce crystallization time are given in ref. [1].
d. After calcination in nitrogen for 2 hours at 500°C followed by calcination in air for 10 hours at 550°C; the BET surface area was 404 m^2/g. Characterizations of similar gallium offretites are given in references [2-4].
e. XRD: OFF, no other crystalline products or amorphous material detected
Elemental Analyses: 49.5% SiO_2, 42.2% Ga_2O_3, 7.2% K_2O, 1.1% Na_2O [d]