CDO CDS-1 Si(100)

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Type Material: CDS-1, $Si_{36}O_{72}$
PLS-1, $Si_{18}O_{34}(OH)_4K_{1.3}\cdot 1.7(CH_3)_4NOH$ (precursor material of CDS-1)

Method: T. Ikeda, Y. Akiyama, Y. Oumi, A. Kawai, and F. Mizukami [1]

Batch Composition: 1.0 SiO_2 : 0.015 KOH : 0.22 $(CH_3)_4NOH$: 16.2 H_2O : 3.41 1,4-dioxane

Source Materials
deionized water (DI)
potassium hydroxide (0.5 mol/L aqueous solution, KOH)
tetramethylammonium hydroxide (15 wt.% aqueous solution, $(CH_3)_4NOH$)
1,4-dioxane (Sigma Aldrich)
silica (Cab-O-Sil M5)

Batch Preparation (for *4 g* dry product as PLS-1)
(1) [25.0 g water + 10.0 g silica + 22.0 g $(CH_3)_4NOH$ aq.[a] + 0.5 g KOH aq.[b]], stir in a vessel
(2) [(1) + 50.0 g 1,4-dioxnane], vigorously stir for 1 h at room temperature

Crystallization
Vessel: Teflon-lined stainless steel autoclave (300 mL)[c]
Temperature: 150 °C
Time: 10 days
Agitation: no

Product Recovery
(3) Filter and wash with acetone and water
(4) Dry at 70 °C in a convection oven for 12 h
(5) Yield: *3.5* g

Calcination[d]
From 400 to 900 ºC; under vacuum at 10^{-3} to 10^{-8} Torr[e]

Product Characterization
XRD: CDO
Crystal size and habit: thin platelet-like crystals with a length < 2 □m and a thick ness of > 100 nm (approximately).

Reference
[1] T. Ikeda, Y. Akiyama, Y. Oumi, A. Kawai, F. Mizukami, Angew. Chem. Int. Ed. 43 (2004) 4892.
[2] S. Inagaki, T. Yokoi, Y. Kubota, T. Tatsumi, Chem. Commun. (2007) 5188.

[3] A. Kawai, T. Ikeda, Y. Kiyozumi, H. Chiku, F. Mizukami, Mater. Chem. Phys. 99 (2006) 470
[4] K. Komura, T. Kawamura, Y. Sugi, Catal. Commun. 8 (2007) 644

Notes

a. 15 wt.% aqueous solution
b. 0.5 mol/L aqueous solution
c. 150 mL autoclave is also utilized
d. The topotactic conversion from PLS-1 to CDS-1 requires the calcination treatment.
e. Typically, 500 ºC, 12 h, ambient pressure (evacuation not required), according to Ref. [2]