Supporting Information

Crystallization pathway from highly viscous colloidal suspension to ultra-small FAU zeolite nanocrystals

Hussein Awala,[1] Shrikant M. Kunjir,[1] Aurélie · Vicente,[1] Jean-Pierre Gilson,[1] Valentin Valtchev,[1]Hussein Seblani,[1] Richard Retoux,[2] Louwanda Lakiss,[1] Christian Fernandez,[1] Robert Bedard,[3] Suheil Abdo,[3] Jeffery Bricker,[3] Svetlana Mintova[1*]

[1] Normandie Université, ENSICAEN, UNICAEN, CNRS, LCS, 14000 Caen, France

[2] Normandie Université, ENSICAEN, UNICAEN, CNRS, CRISMAT, 14000 Caen, France

· [3] UOP-Honeywell LLC, R&D, 25 East Algonquin Road, Des Plaines, IL 60016, USA

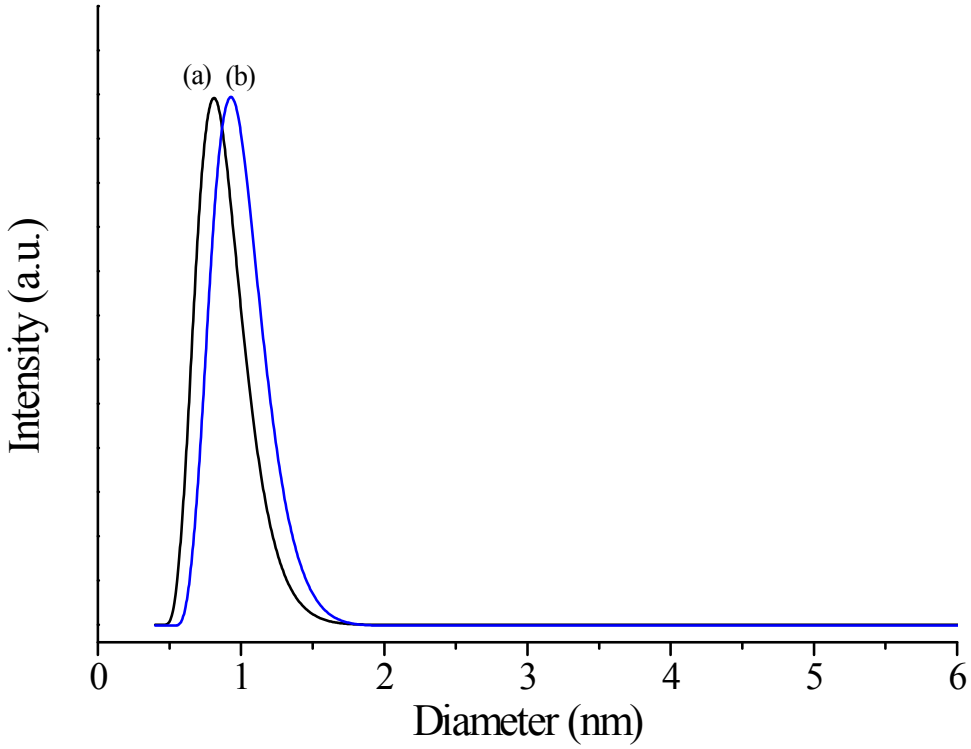

Figure S1. Dynamic light scattering (DLS) particle size distribution curves of (a) *solution A* (sodium aluminate) and (b) *solution B* (sodium silicate) used to prepare the nano FAU-type zeolite

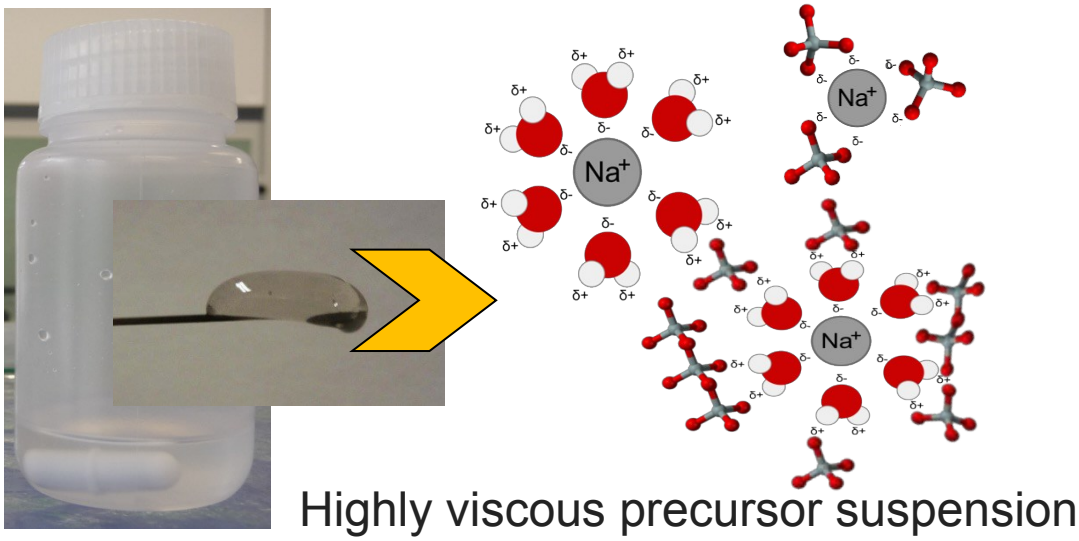

Figure S2. Highly viscous and transparent suspension at step 3 (P-3).

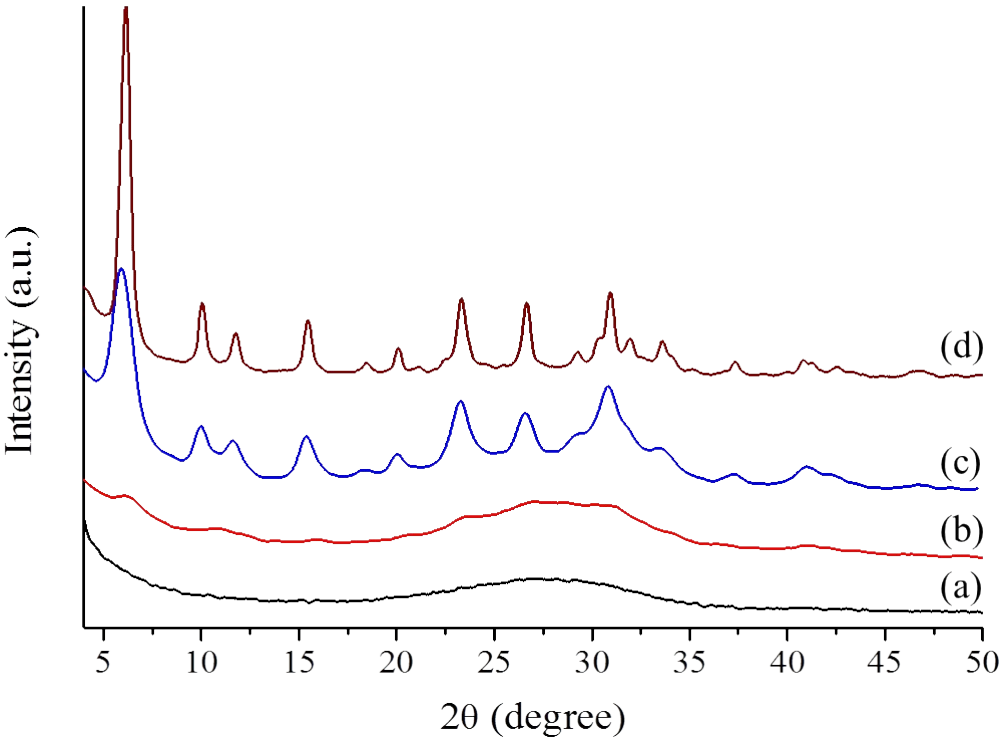

Figure S3. XRD patterns of (a) P-3, (b) FAU -45 min, (c) FAU-3 h and (d) FAU-24 h.

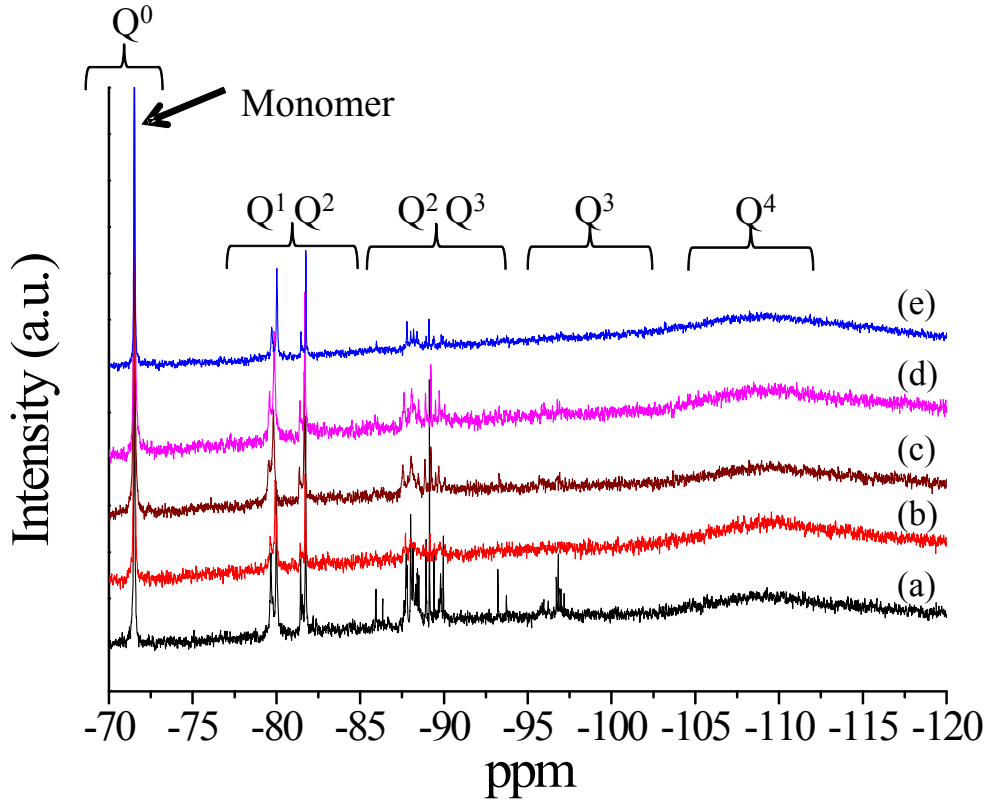

Figure S4. ^{29}Si liquid NMR spectra of (a) solution B, (b) P-1, (c) P-2, (d) P-3, and (e) FAU-45 min.

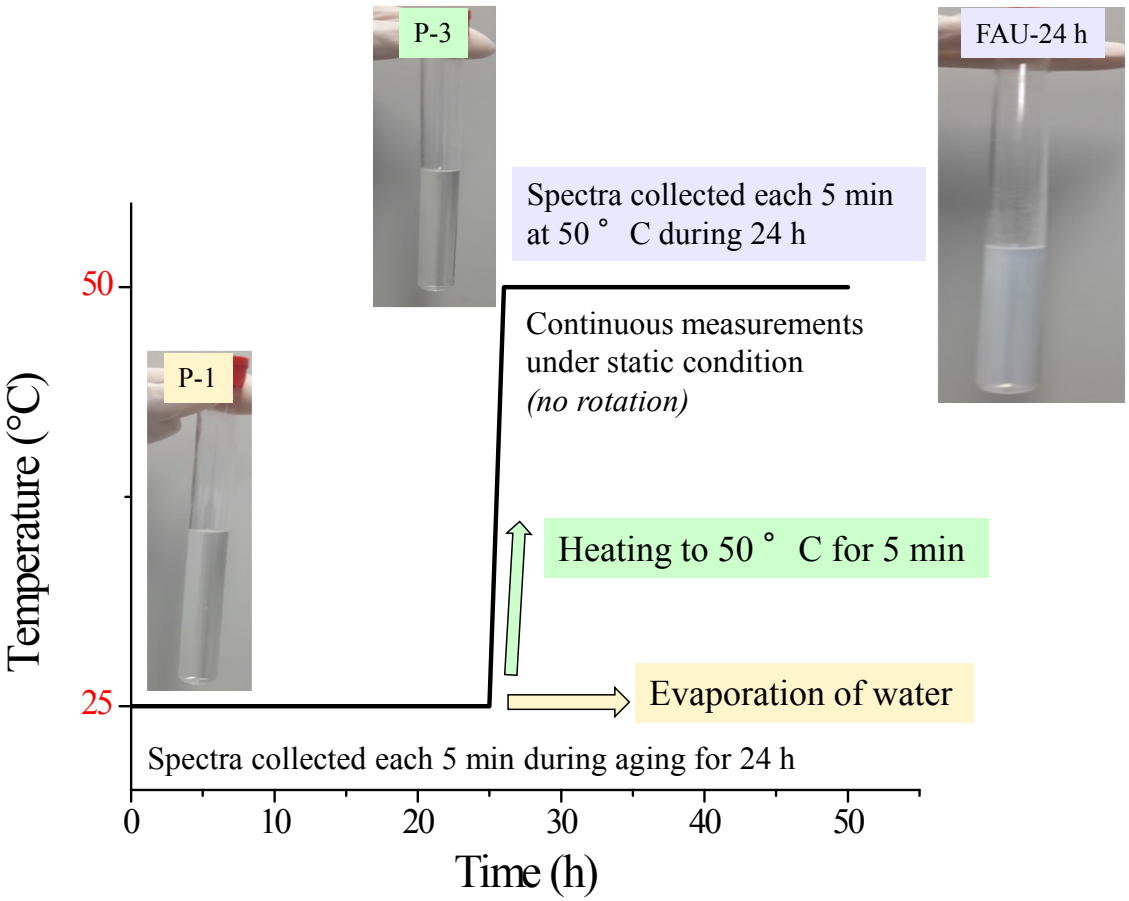

Figure S5. *In-situ* ^{29}Si NMR experiments as a function of time and temperature (10 mm glass NMR tube used) during FAU crystallization.

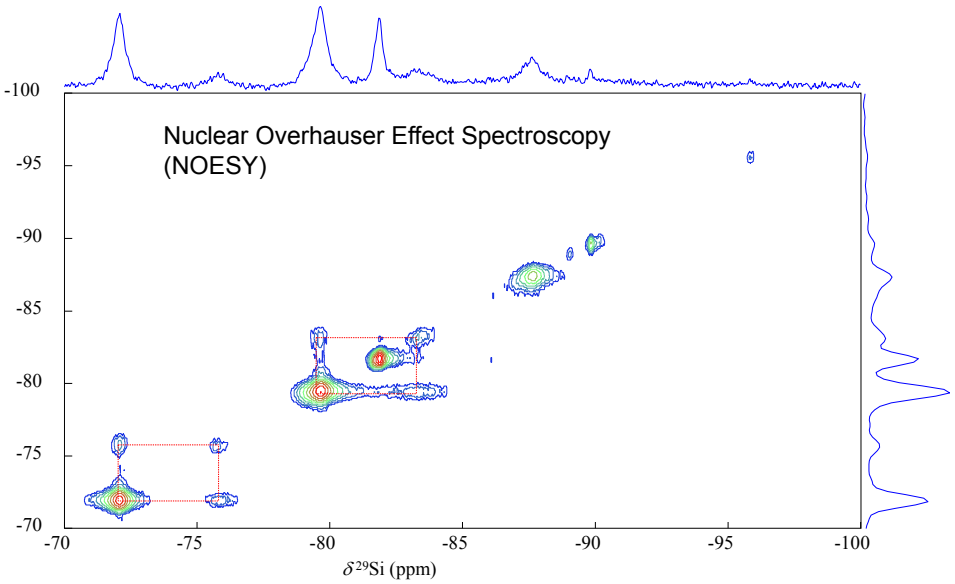

Figure S6. Nuclear Overhauser Effect SpectroscopY (NOESY) spectra on P-2 (NOE mixing time: 20 msec, Scans: 64, and Total number of points TD1: 256)

Table S1. Silicate species identified by liquid-state ^{29}Si MAS NMR spectroscopy in P-1.

Species	Chemical Shift (ppm)
Monomer	-71.05
Dimer	-79.80
Linear Trimer	-89.50
Cyclic Trimer	-81.20
Substituted Cyclic Trimer (Q^1), Substituted Cyclic Tetramer (Q^1)	-79.30
Substituted Cyclic Trimer (Q^2)	-80.90
Substituted Cyclic Trimer (Q^3), Substituted Cyclic Tetramer (Q^3), Tricyclic Hexamer (Q^2/Q^3)	-89.50
Cyclic Tetramer	-87.70
Bicyclic Pentamer (Q^2)	81.30
Bicyclic Pentamer (Q^3)	88.10; 88.60
Prismatic Hexamer	-88.0
Tricyclic Hexamer, Substituted Cyclic Tetramer (Q^3)	-97.0
Tetrahedral Tetramer	-97.70
Prismatic Tetramer	-98.11
Doubly Bridged Cyclic Tetramer (Q^2)	86.10
Doubly Bridged Cyclic Tetramer (Q^3)	92.8
Double 4-ring	-98.99
Double 5-ring	98.40
Double 6-ring	-97.80
Pentacyclic Dodecamer (Q^2)	89.0
Pentacyclic Dodecamer (Q^3)	96.20; 96.60

Table S2. Degree of crystallinity and Si/Al ratio calculated from solid-state ^{29}Si NMR.

Sample	**Degree of crystallinity (%)**	**Si/Al ratio**
P-1	0	----------
P-2	10-16	----------
P-3	23	2.8
FAU-45 min	36	1.7
FAU-3 h	50	1.41
FAU-24 h	100	1.06