

Crystallization of High-silica Cubic and Hexagonal Faujasite Polymorphs in the presence of the Tetraethylammonium (TEA⁺) Cation

Corentin Chatelard^a, Raquel Martinez Franco^b, Mathias Dodin^b, Alain Tuel^{a*}

Electronic Supplementary Information

Table S1 Chemical composition of **FAU** zeolites obtained as intermediates in the crystallization of **CHA** with TEAOH at different temperatures

Crystallization temperature (°C)	Crystallization time (h)	Phases observed (XRD)	SiO ₂ /Al ₂ O ₃ (XRF)
130	9	FAU	8.8
110	24	FAU	8.2
90	30	FAU	9.2
80	48	FAU	8.6
70	72	FAU	9.0

Table S2 Chemical composition of **FAU** zeolites obtained as intermediates in the crystallization of **CHA** with TEAOH at different temperatures and using various amounts of CBV 720 as seeds

Crystallization temperature (°C)	Percentage of seeds (CBV 720)	Phases observed (XRD)	SiO ₂ /Al ₂ O ₃ (XRF)
130	10	FAU	8.8
130	2.5	FAU	7.2
130	0	FAU	6.6
110	10	FAU	8.2
110	2.5	FAU	7.6
90	10	FAU	9.2
90	5	FAU	7.6

Table S3 Chemical composition of FAU zeolites obtained as intermediates in the crystallization of CHA with TEAOH from gels with various alkalinities at 110 °C

X TEAOH (mol) ^a	y Na ₂ O (mol) ^a	Crystallization time (days)	Phases observed (XRD)	SiO ₂ /Al ₂ O ₃ (XRF)
11	4.8	1	FAU	8.2
11	4.4	2	FAU	9.0
11	4.0	3	FAU	9.2
9	4.8	1	FAU + Am ^b	9.2 ^c
9	4.4	4	FAU + Am ^b	10.2 ^c
9	4.0	5	FAU + Am ^b	-

^aThe gel composition was: 27.6 SiO₂ – 0.76 Al₂O₃ - x TEAOH - y Na₂O - 514 H₂O

^bAm = amorphous phase

^cValue determined in the presence of amorphous phase

Table S4 Position and relative area of the 4 components used to decompose the ²⁹Si NMR signal of sample 9 (FAU zeolite with SiO₂/Al₂O₃ = 13)

Line	Position (ppm)	% Integral
1	-105.6	41.84
2	-100.54	34.92
3	-98.21	7.83
4	-94.65	15.41

Table S5 SiO₂/Al₂O₃ ratios of zeolites obtained using HEMT seeds with various Al contents and corresponding proportions of FAU and EMT in the frameworks

Entry	Seeds	SiO ₂ /Al ₂ O ₃ Gel ^a	Product	FAU:EMT ratio
1	64	36	8.6	80:20
2	64	30	7.9	70:30
3	64	20	7.4	60:40
4	64	15	6.8	60:40
5	47.6	36	8.3	50:50
6	25.4	36	8.0	10:90
7	25.4	50	9.4	0:100 ^b
8	25.4	80	11.2	0:100 ^b
9	21.2	36	7.9	30:70
10	18.2	36	7.7	30:70
11	16	36	7.0	40:60
12	7.2	36	6.5	50:50
13	7.2	50	6.7	40:60
14	7.2	80	7.1	40:60

^aRatio before addition of seeds

^bNo contribution of the FAU topology detected by standard XRD

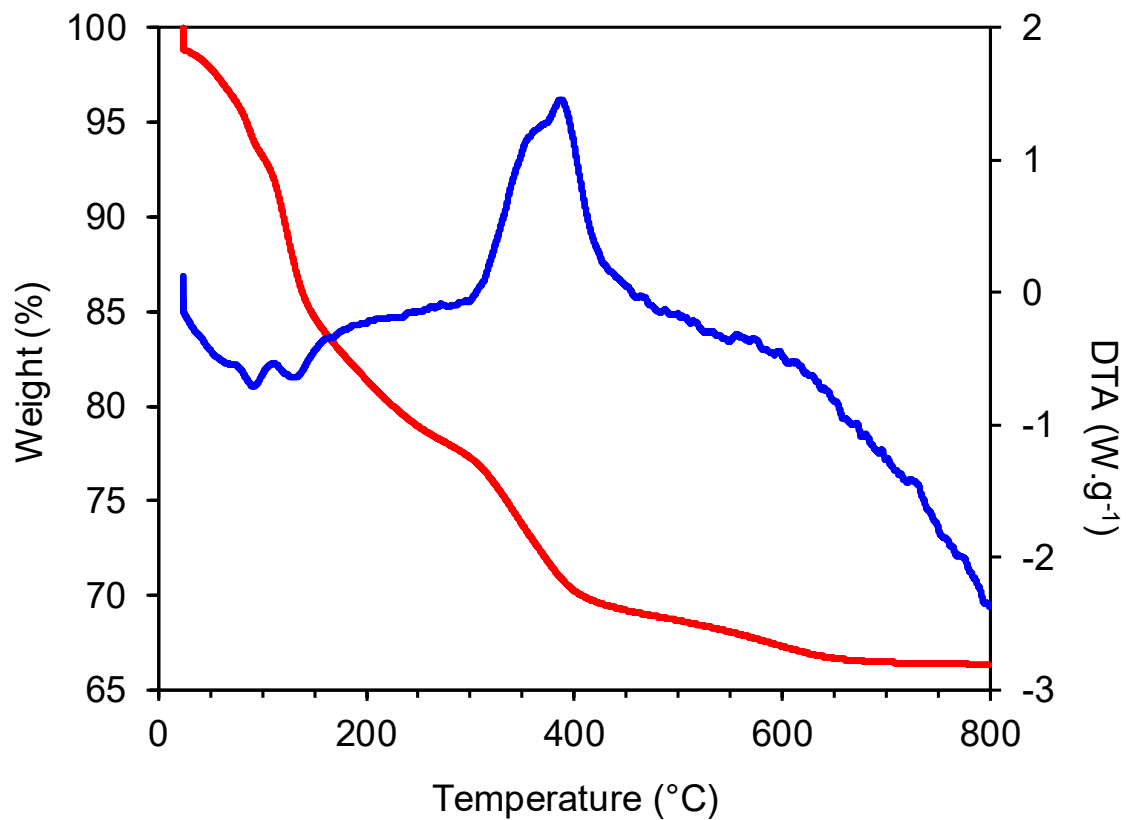


Figure S1 Thermal analysis data for sample 4 obtained with $\text{SiO}_2/\text{Al}_2\text{O}_3 = 60$ in the gel prior to addition of the seeds

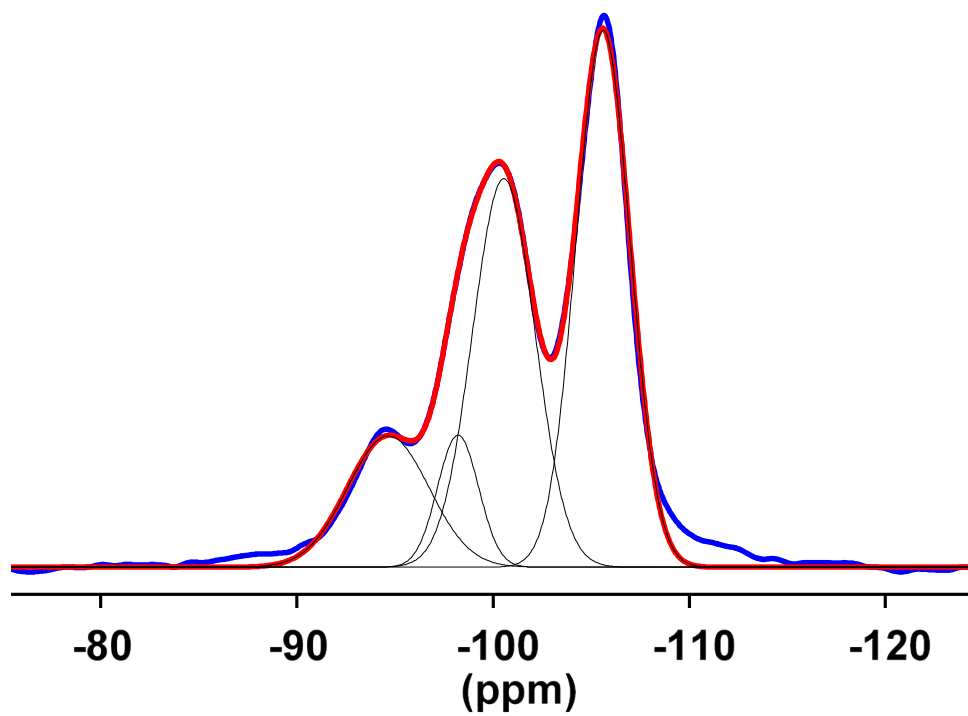


Figure S2 Decomposition of the ^{29}Si NMR signal of calcined sample 9 ($\text{SiO}_2/\text{Al}_2\text{O}_3 = 13$) into Gaussian lines. Blue: experimental spectrum, red: simulated spectrum, black: individual components

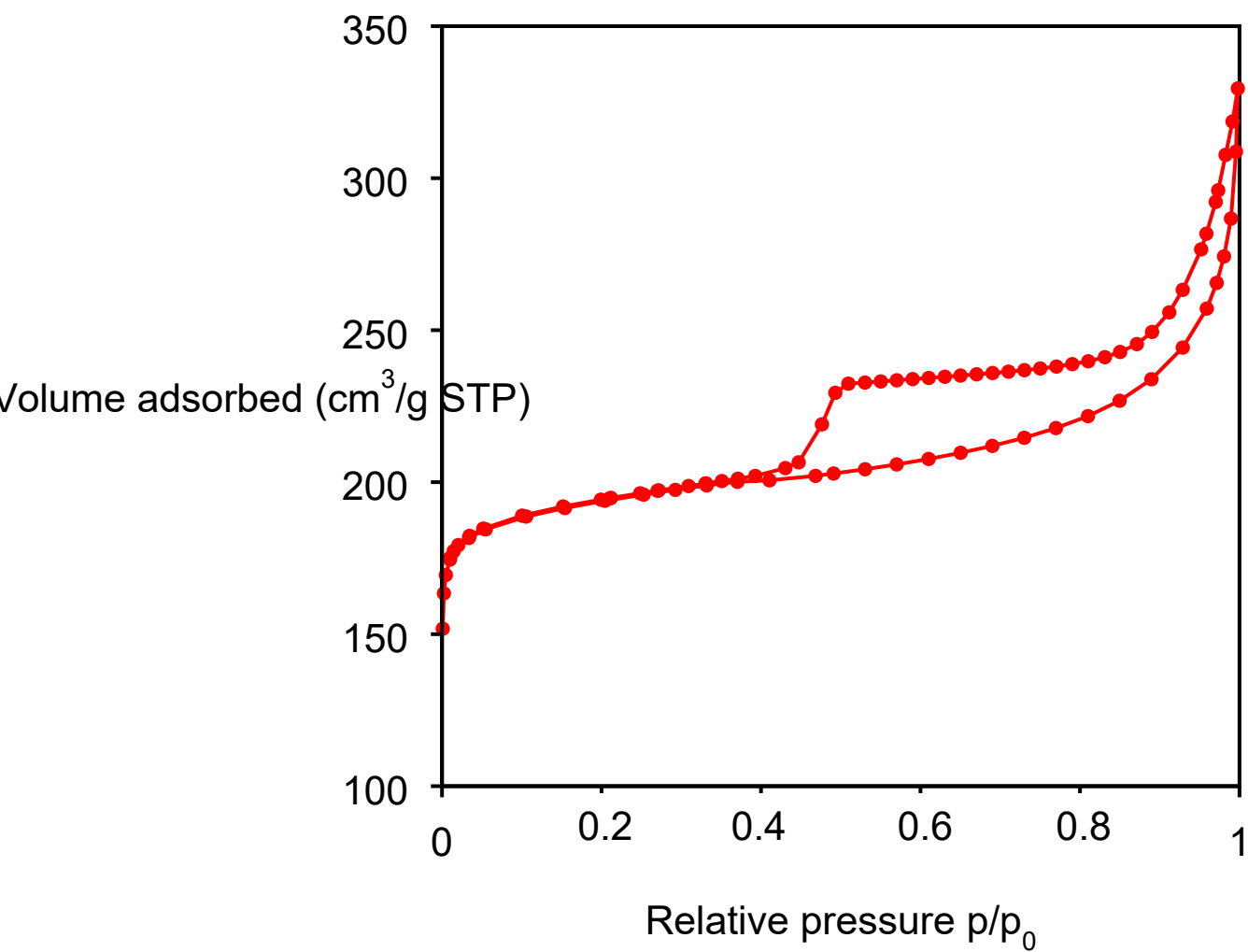


Figure S3 Nitrogen adsorption-desorption isotherm on a calcined faujasite with $\text{SiO}_2/\text{Al}_2\text{O}_3 = 11.4$ (sample 7 in Table 1)

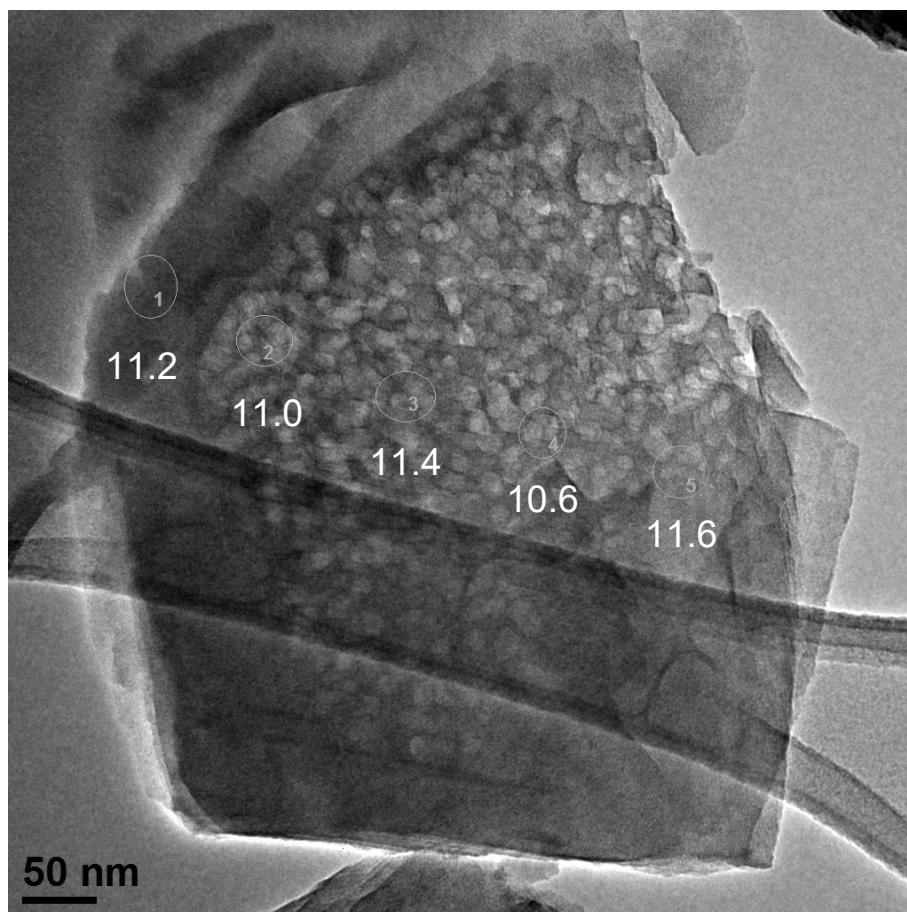


Figure S4 TEM image of a 60 nm thin slice of FAU zeolite with $\text{SiO}_2/\text{Al}_2\text{O}_3 = 11.4$ (determined by XRF). Data on the picture correspond to $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios calculated by EDX at different spots of the crystal.

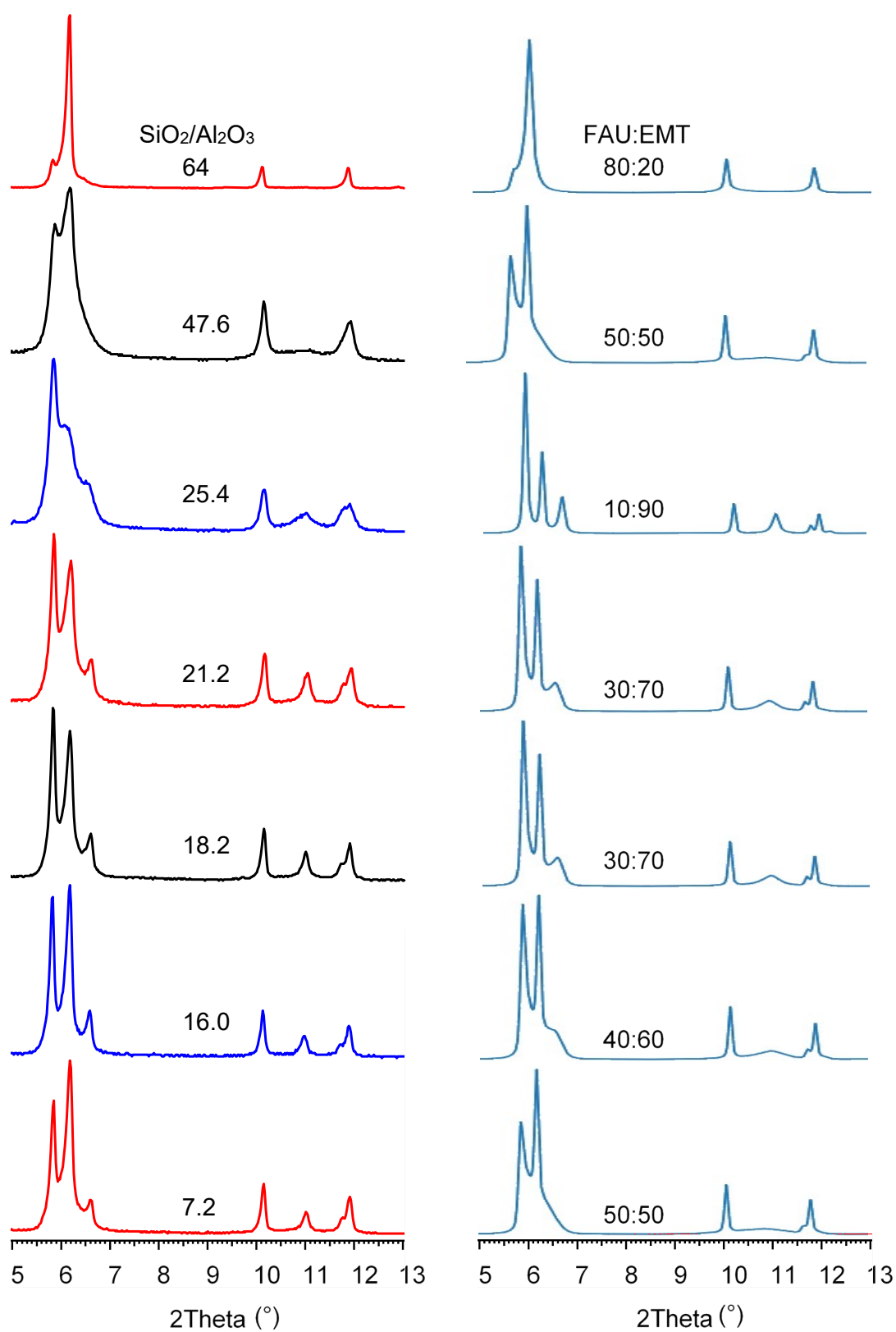


Figure S5 XRD patterns of calcined zeolites obtained using HEMT seeds with various $\text{SiO}_2/\text{Al}_2\text{O}_3$ (left) and the corresponding simulations using the DIFFaX program developed by J.M.M. Treacy [10] (right) with the relative percentages of **FAU** and **EMT**.

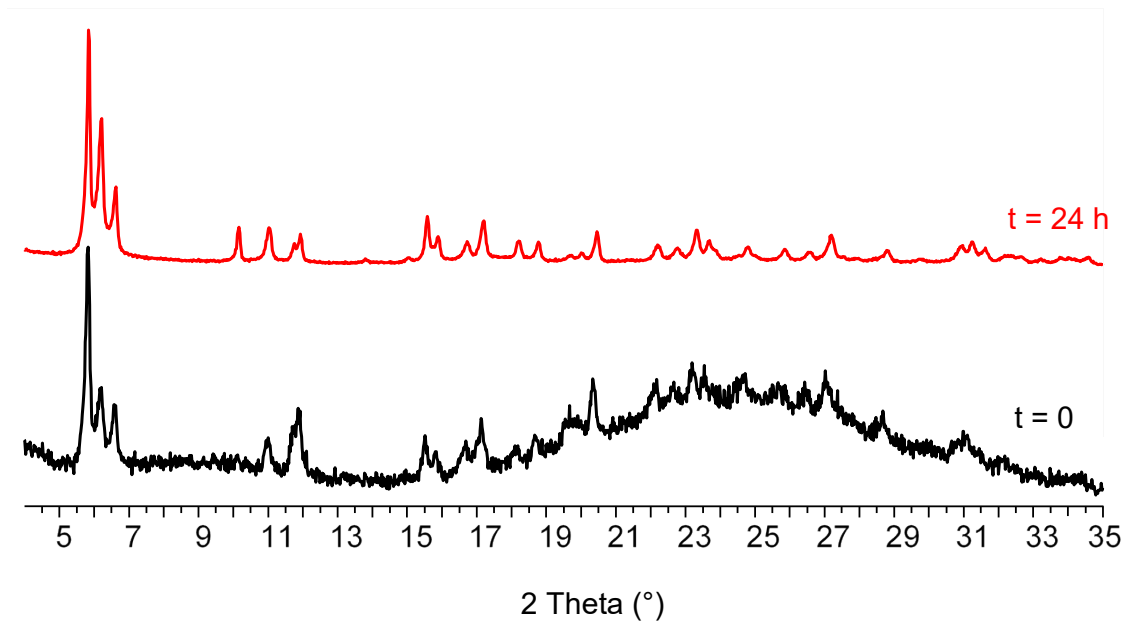


Figure S6 XRD patterns of as-made solids recovered during the crystallization of a gel with $\text{SiO}_2/\text{Al}_2\text{O}_3 = 80$ and HEMT seeds ($\text{SiO}_2/\text{Al}_2\text{O}_3 = 25.4$) before the hydrothermal treatment (bottom, $t = 0$) and after 24 h at 110°C (top, $t = 24$ h).

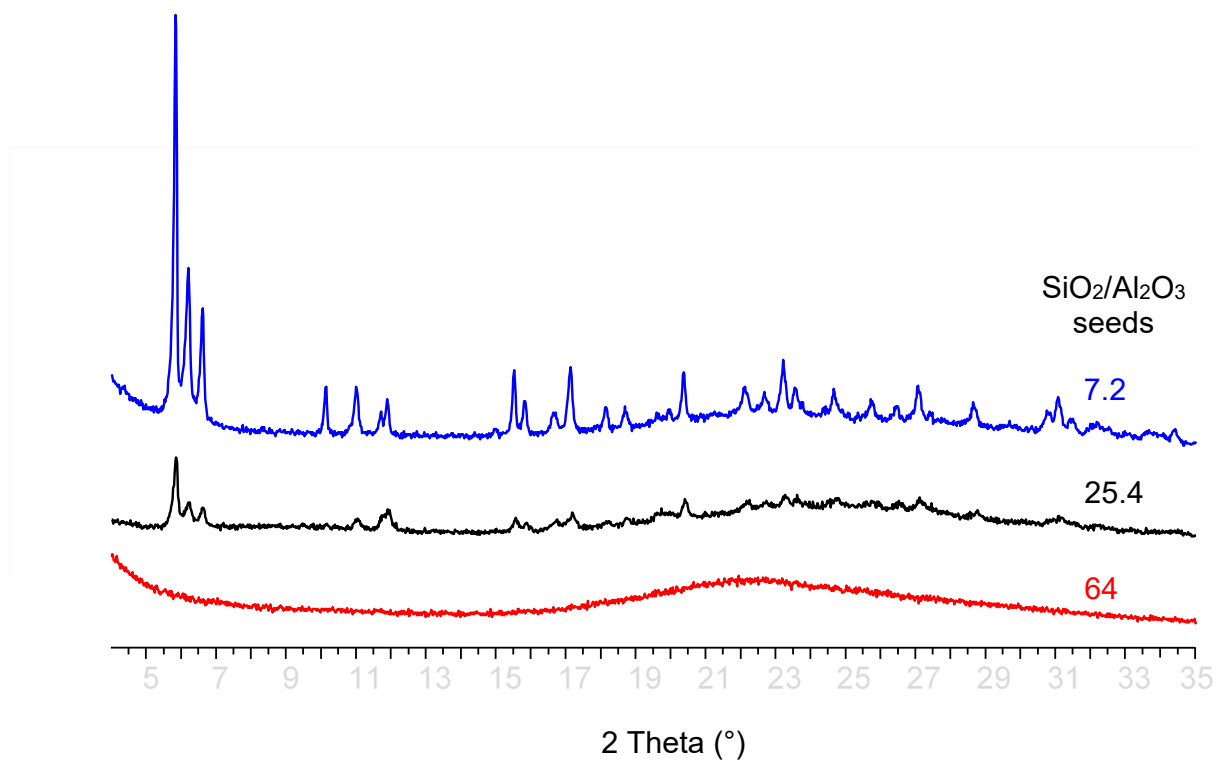


Figure S7 XRD patterns of as-made solids collected before the hydrothermal treatment ($t = 0$) during the crystallization of a gel with $\text{SiO}_2/\text{Al}_2\text{O}_3 = 36$ and using HEMT seeds with various $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratios.