

SDA-free hydrothermal synthesis of high-silica ultra-nanosized zeolite Y

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Table S1. Products obtained from initial molar gel compositions 31 SiO₂ : 1 Al₂O₃ : 17 Na₂O : β H₂O (β varying between 360 and 615) with an aging time of 20 days at room temperature and a heating step at 60 °C during 16 hours.

Sample	β	Zeolite type	X-ray diffraction a ₀ ^a (Å)	Si/Al ^b	V _{micro} ^c (cm ³ /g)	V _{tot pore} ^d (cm ³ /g)	S _{BET} ^e (m ² /g)	Average particle size ^f (nm)
P	360	Zeolite Y	24.7252(15)	2.12	0.21	0.80	617	114±14
Q	485	Zeolite Y	24.7292(19)	2.10	0.31	1.16	859	171±25
R	550	Zeolite Y	24.688(3)	2.35	0.04	0.90	199	ND ^g
S	615	Zeolite Y	24.692(4)	2.33	0.04	0.95	182	ND

^a a_0 : Lattice parameter

^bSi/Al ratio obtained by XRD and using the Breck and Flanigen equation: $((192 \cdot 0.00868)/(a_0 - 24.191)) - 1$

^c V_{micro} : Micropore volume

^d $V_{\text{tot pore}}$: Total pore volume

^e S_{BET} : Specific surface area

^fAverage particle size determined from transmission electron microscopy images

^gND: Not determined

XRD patterns

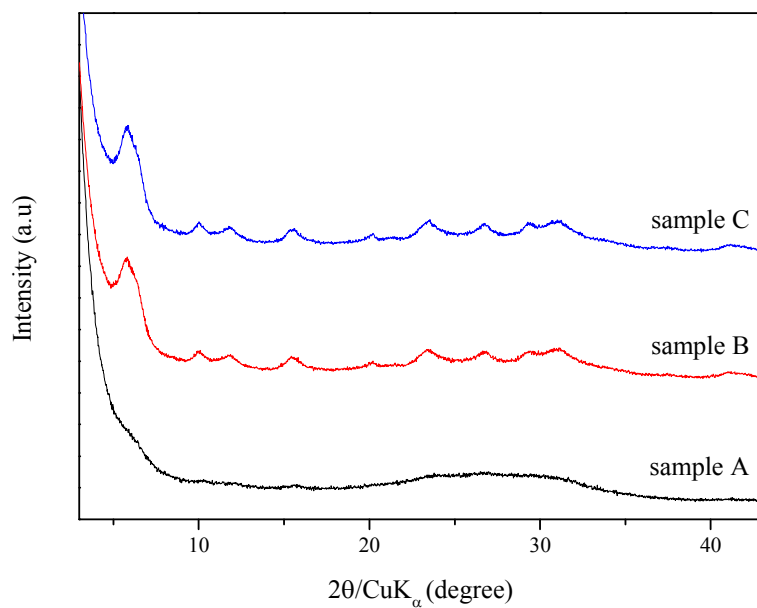


Figure S1. XRD patterns for samples obtained from the initial molar gel composition 15 SiO₂ : 1 Al₂O₃ : 17 Na₂O : 360 H₂O with an aging time of 7 (sample A), 10 (sample B) and 13 (sample C) days and with no heating step.

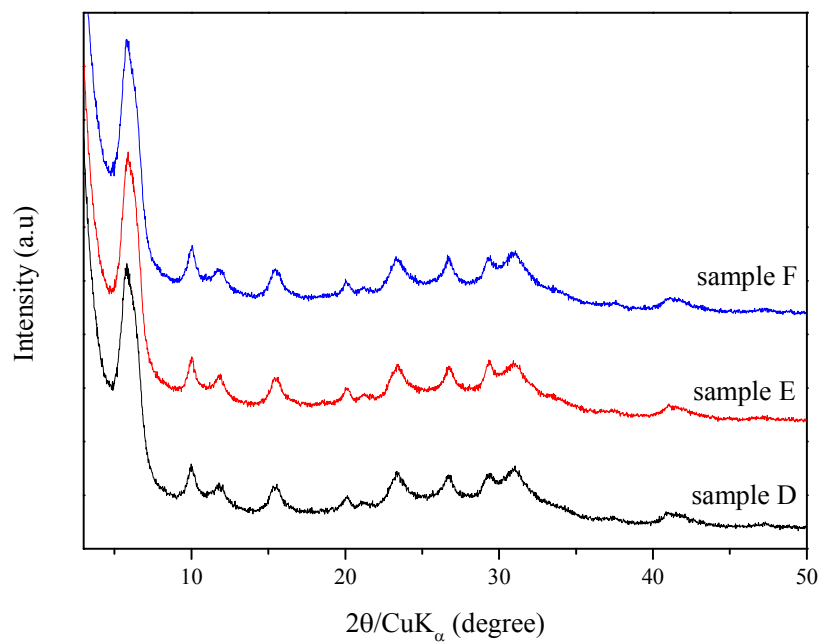


Figure S2. XRD patterns for samples obtained from the initial molar gel composition 15 SiO_2 : 1 Al_2O_3 : 17 Na_2O : 360 H_2O with an aging time of 7 (sample D), 10 (sample E) and 13 (sample F) days and with a heating step at 60 °C during 16 hours.

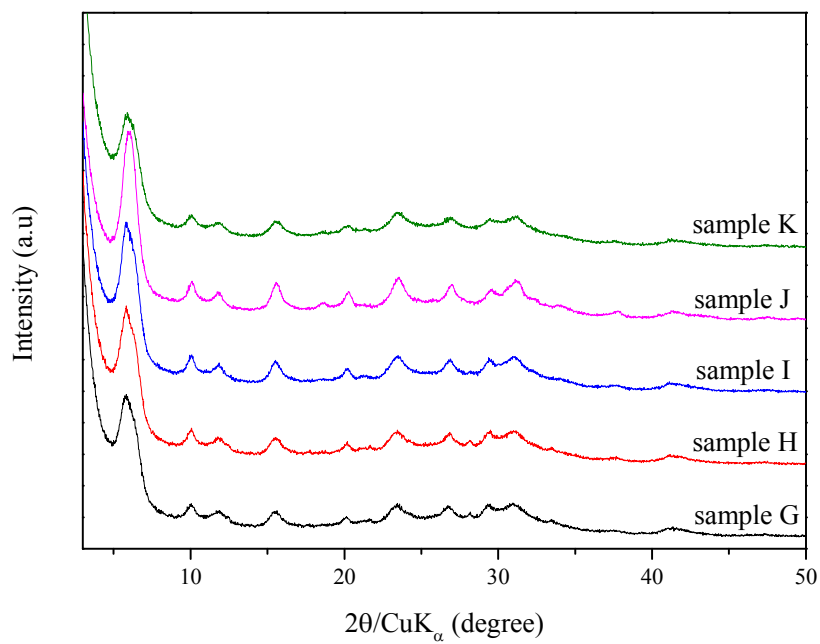


Figure S3. XRD patterns for samples obtained from final molar gel compositions α SiO_2 : 1 Al_2O_3 : 17 Na_2O : 360 H_2O (α = 21 (sample G), 26 (sample H), 31 (sample I), 36 (sample J), 41 (sample K)) with an aging time of 13 days and a heating step at 60 °C during 16 hours.

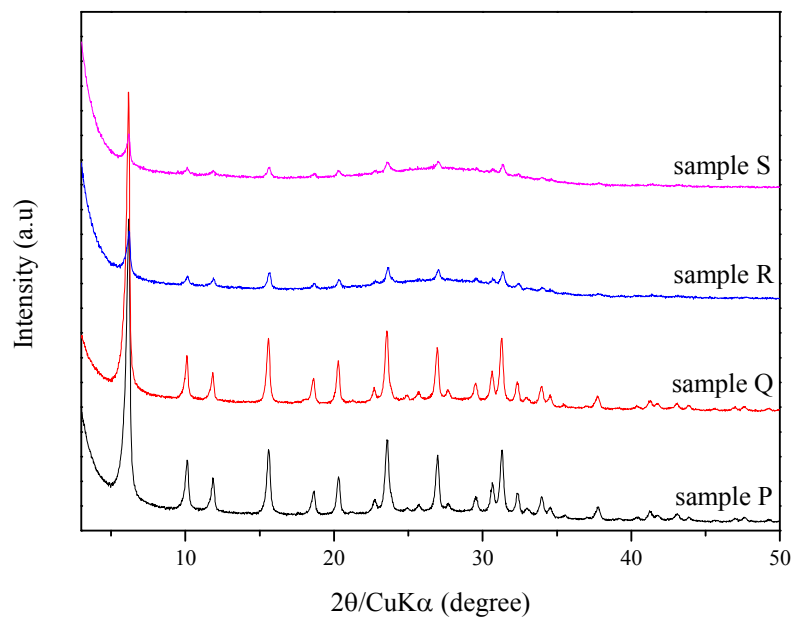


Figure S4. XRD patterns for samples obtained from initial molar gel compositions 31 SiO₂ : 1 Al₂O₃ : 17 Na₂O : β H₂O (β = 360 (sample P), 485 (sample Q), 550 (sample R), 615 (sample S)) with an aging time of 20 days and a heating step at 60 °C during 16 hours.

Nitrogen adsorption curves

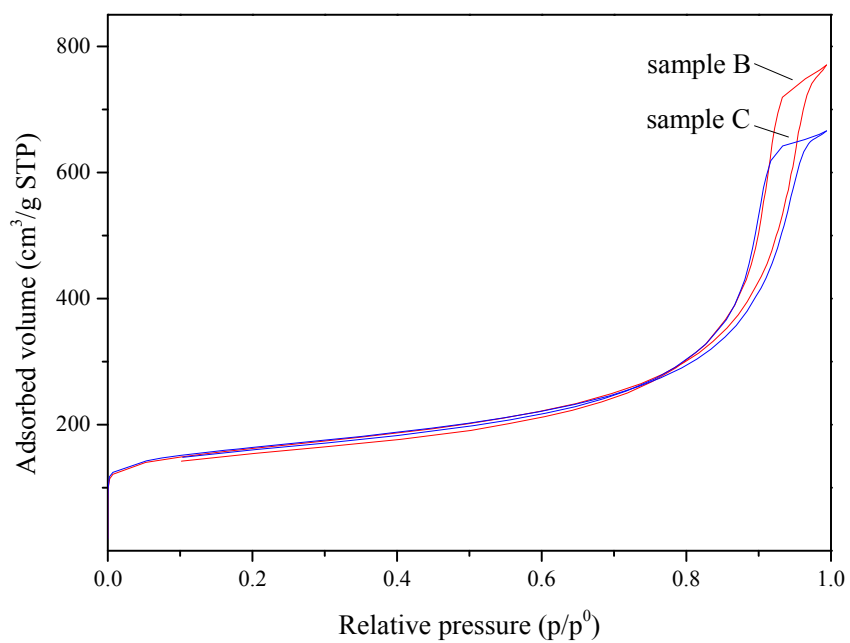


Figure S5. Nitrogen adsorption-desorption isotherms at -196 °C for samples obtained from the initial molar gel composition 15 SiO₂ : 1 Al₂O₃ : 17 Na₂O : 360 H₂O with an aging time of 10 (sample B) and 13 (sample C) days and with no heating step.

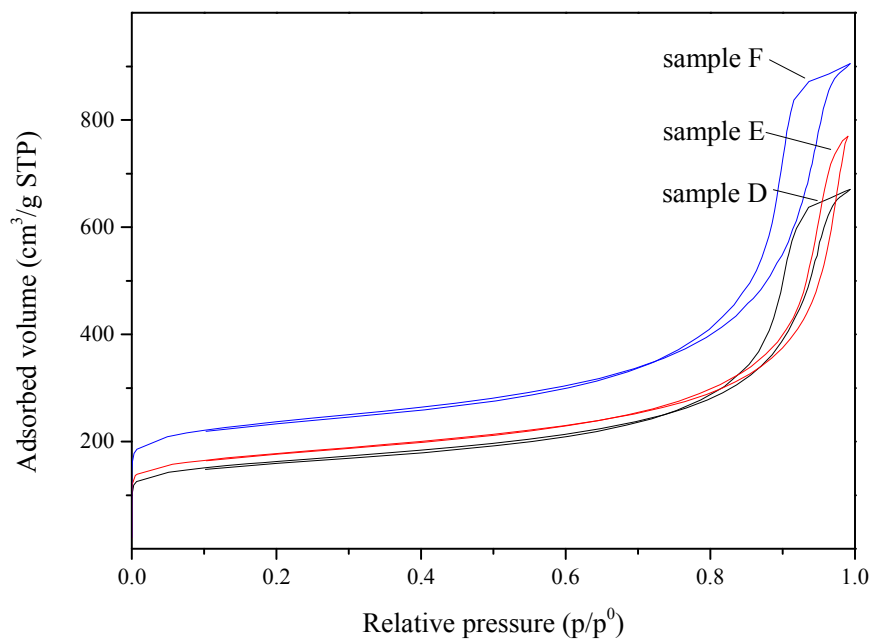


Figure S6. Nitrogen adsorption-desorption isotherms at -196 °C for samples obtained from the initial molar gel composition 15 SiO₂ : 1 Al₂O₃ : 17 Na₂O : 360 H₂O with an aging time of 7 (sample D), 10 (sample E) and 13 (sample F) days and with a heating step at 60 °C during 16 hours.

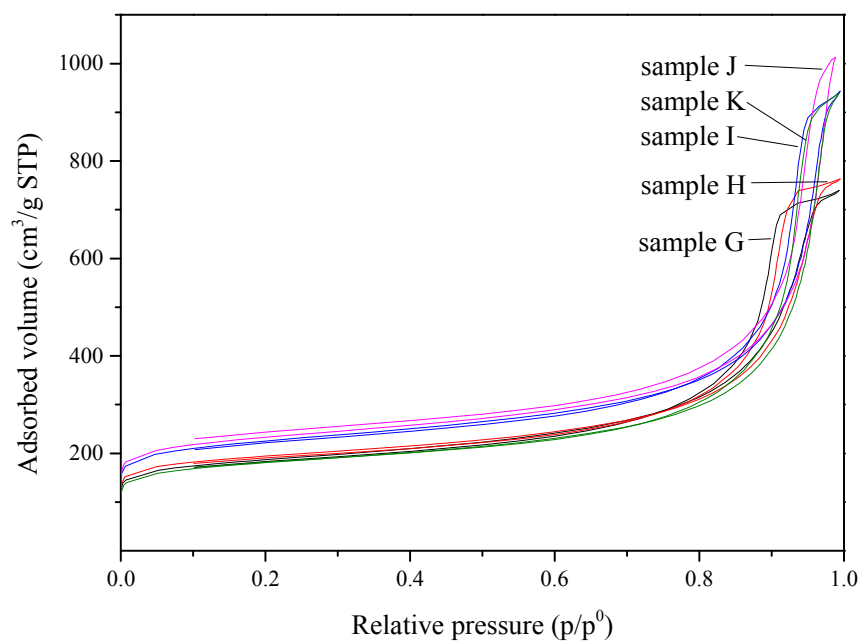


Figure S7. Nitrogen adsorption-desorption isotherms at -196 °C for samples obtained from final molar gel compositions α SiO₂ : 1 Al₂O₃ : 17 Na₂O : 360 H₂O (α = 21 (sample G), 26 (sample H), 31 (sample I), 36 (sample J), 41 (sample K)) with an aging time of 13 days and a heating step at 60 °C during 16 hours.

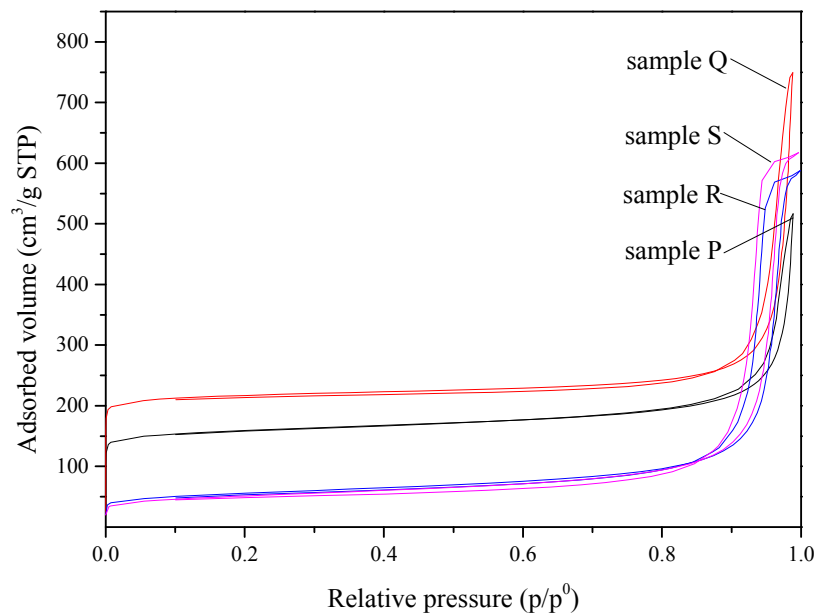


Figure S8. Nitrogen adsorption-desorption isotherms at -196 °C for samples obtained from initial molar gel compositions 31 SiO₂ : 1 Al₂O₃ : 17 Na₂O : β H₂O (β = 360 (sample P), 485 (sample Q), 550 (sample R), 615 (sample S)) with an aging time of 20 days and a heating step at 60 °C during 16 hours.

Si/Al framework ratio and silica content in the gel

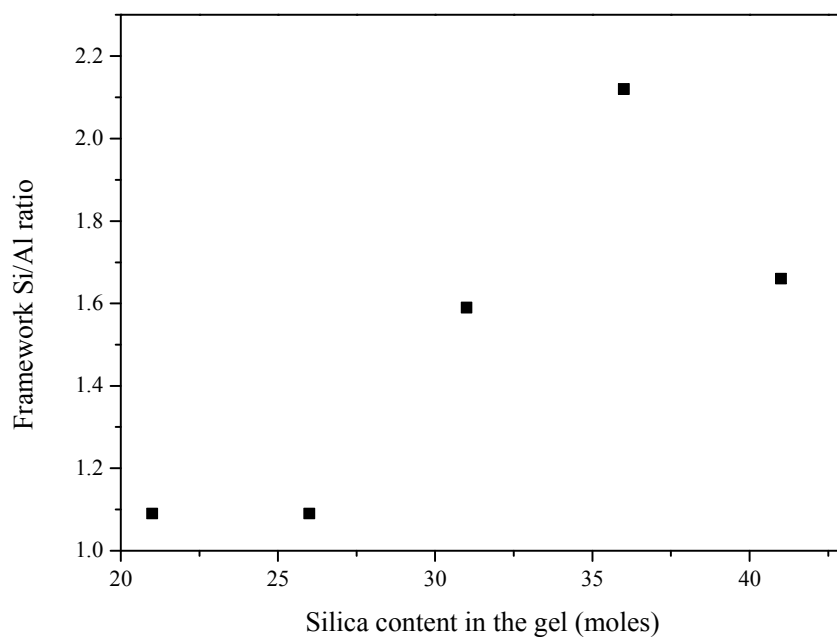


Figure S9. Evolution of the Si/Al framework ratio for samples obtained from final molar gel compositions α SiO_2 : 1 Al_2O_3 : 17 Na_2O : 360 H_2O ($\alpha = 21$ (sample G), 26 (sample H), 31 (sample I), 36 (sample J), 41 (sample K)) with an aging time of 13 days and a heating step at 60 °C during 16 hours.

Micropore volume and silica content in the gel

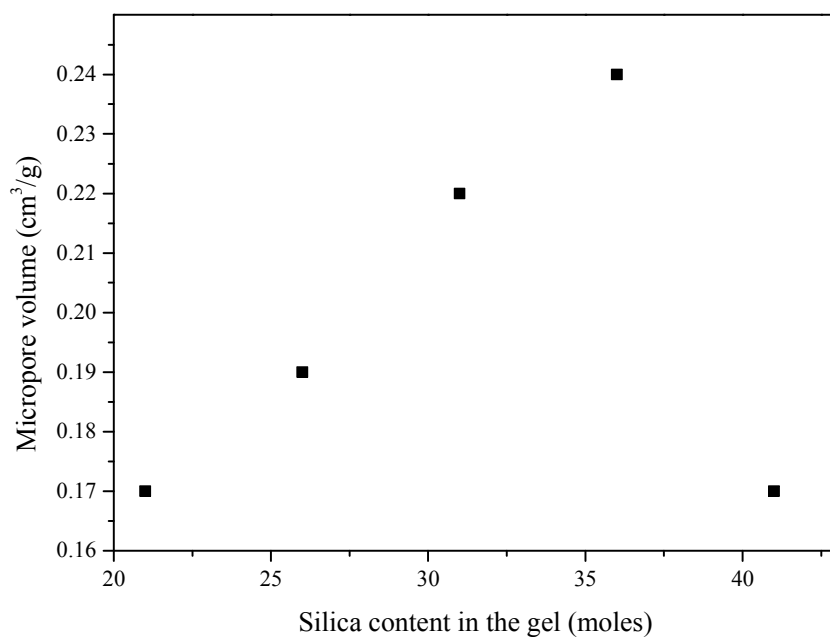


Figure S10. Evolution of the micropore volume for samples obtained from final molar gel compositions α SiO_2 : 1 Al_2O_3 : 17 Na_2O : 360 H_2O ($\alpha = 21$ (sample G), 26 (sample H), 31 (sample I), 36 (sample J), 41 (sample K)) with an aging time of 13 days and a heating step at 60 °C during 16 hours.

NMR spectra

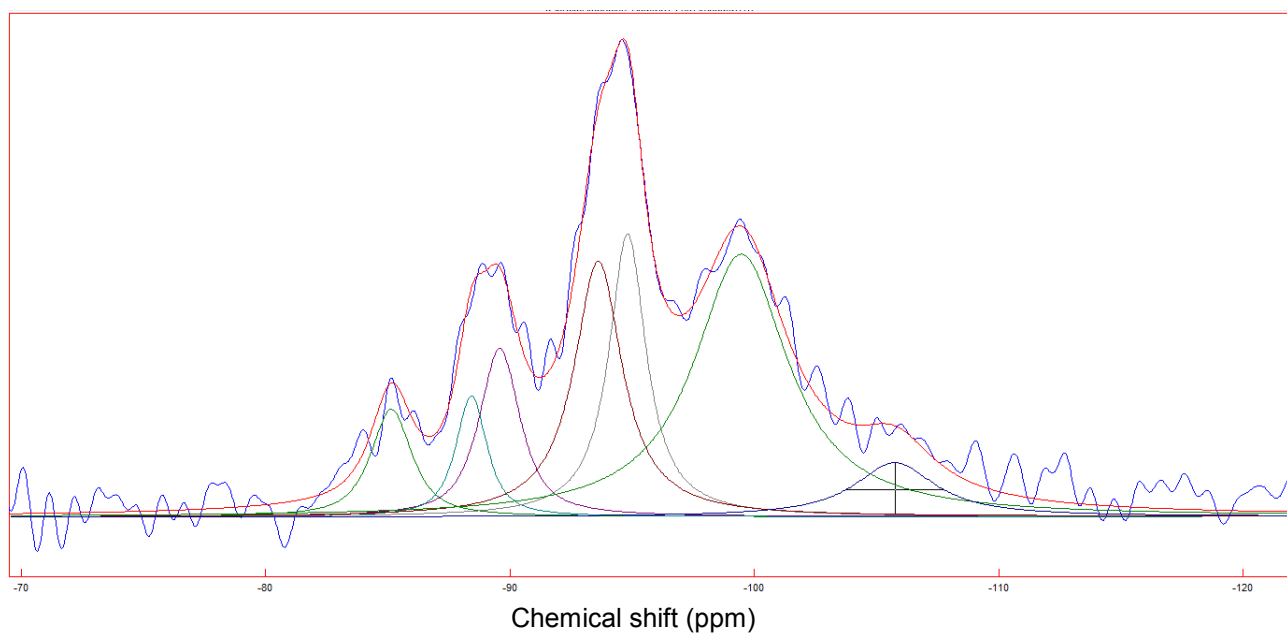


Figure S11. ^{29}Si solid-state MAS NMR spectrum for the sample obtained from the final molar gel composition 36 SiO_2 : 1 Al_2O_3 : 17 Na_2O : 360 H_2O with an aging time of 13 days and a heating step at 70 °C (sample N) during 16 hours.

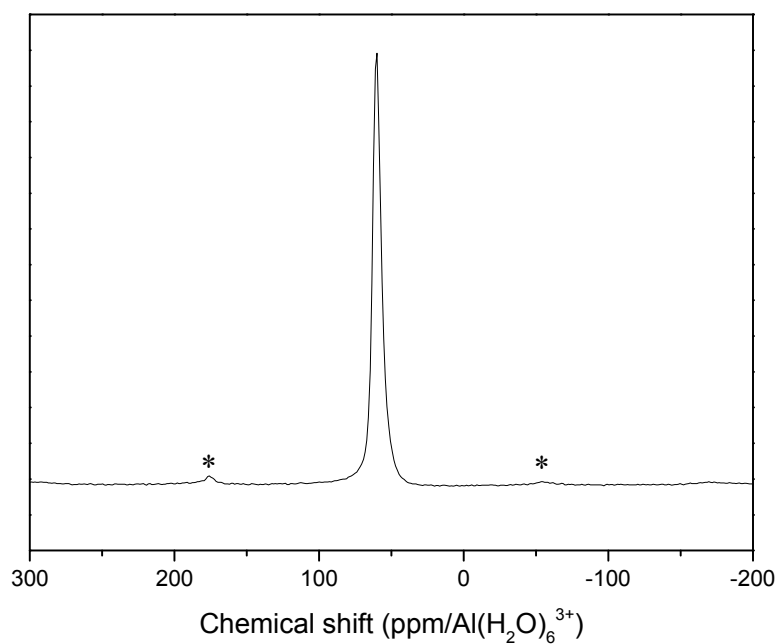


Figure S12. ^{27}Al solid-state MAS NMR spectrum for the sample obtained from the final molar gel composition $36 \text{ SiO}_2 : 1 \text{ Al}_2\text{O}_3 : 17 \text{ Na}_2\text{O} : 360 \text{ H}_2\text{O}$ with an aging time of 13 days and a heating step at 70°C (sample N) during 16 hours. The weak peaks indicated by an asterisk are spinning side bands.

Size distributions and transmission electron microscopy images

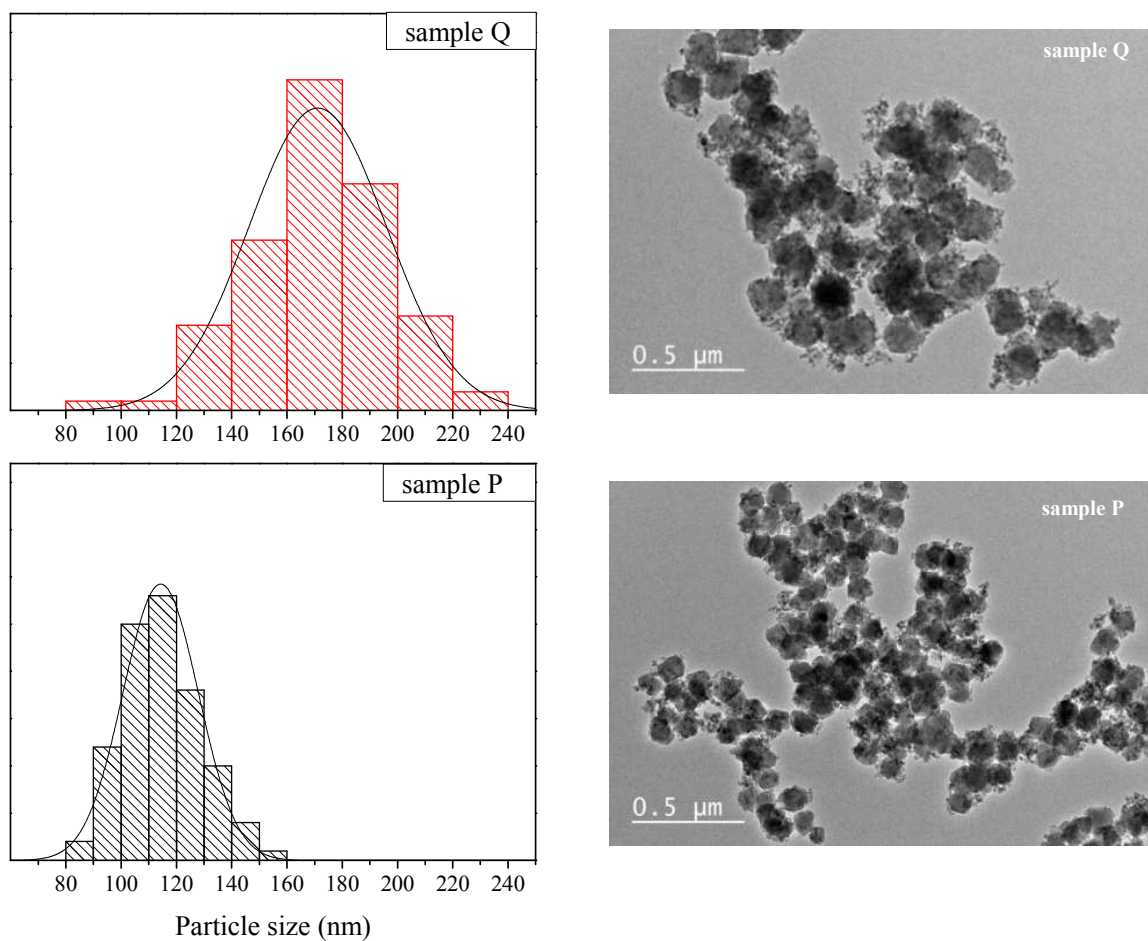


Figure S13. Size distributions (left) and transmission electron microscopy images (right) for samples obtained from initial molar gel compositions $31 \text{ SiO}_2 : 1 \text{ Al}_2\text{O}_3 : 17 \text{ Na}_2\text{O} : \beta \text{ H}_2\text{O}$ ($\beta = 360$ (sample P), 485 (sample Q)) with an aging time of 20 days and a heating step at 60°C during 16 hours.