

Supplementary information

Impact on silica particle physical characteristics of co-condensed alkoxide precursors

*Francisco Bevilacqua,¹ Cynthia Cibaka-Ndaya,¹ Paula Sanz Camacho¹, Sabrina Lacomme²,
Etienne Durand,¹ Jean-Bernard Ledeuil³, Joachim Allouche³, Cédric Boissière,⁴ Clément
Sanchez,^{4,*} Glenna L. Drisko^{1,*}*

¹ Univ. Bordeaux, CNRS, Bordeaux INP, ICMCB, UMR 5026, F-33600 Pessac, France.

² Univ. Bordeaux, CNRS, INSERM, BIC, UAR 3420, F-33600 Pessac, France

³ Univ. Pau et des Pays de l'Adour, E2S UPPA, IPREM/CNRS, UMR 5254, 64000 Pau, France

⁴ Laboratoire Chimie de la Matière Condensée, UMR UPMC Collège de France-CNRS 7574,
Paris, France.

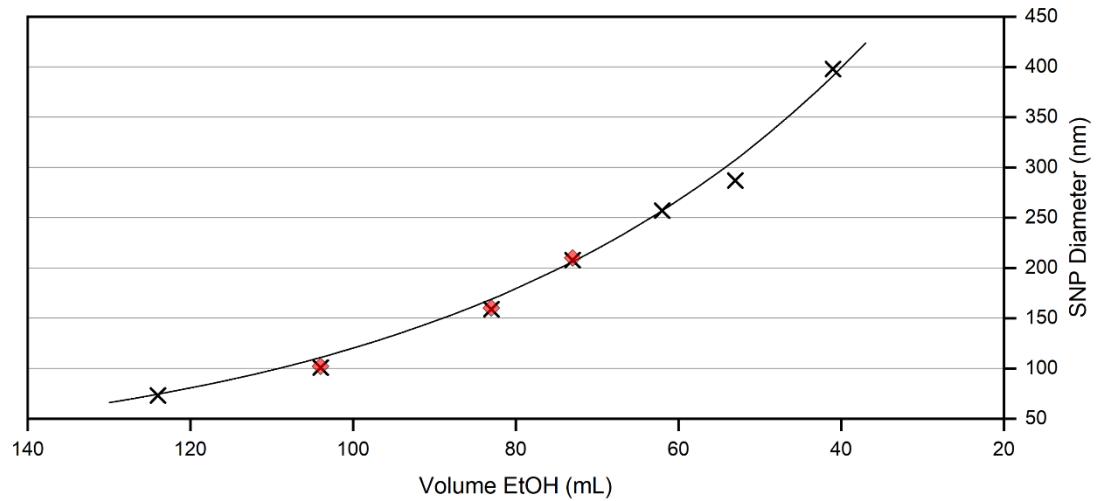


Figure S1. Evolution of the SiO_2 particle size as a function of the ethanol volume. In red is data that we produced by reproducing the methods reported by Gao, *et al*, figure adapted from W. Gao, M. Rigout, H. Owens, *J Nanopart Res.* 2016, **18**, 387.

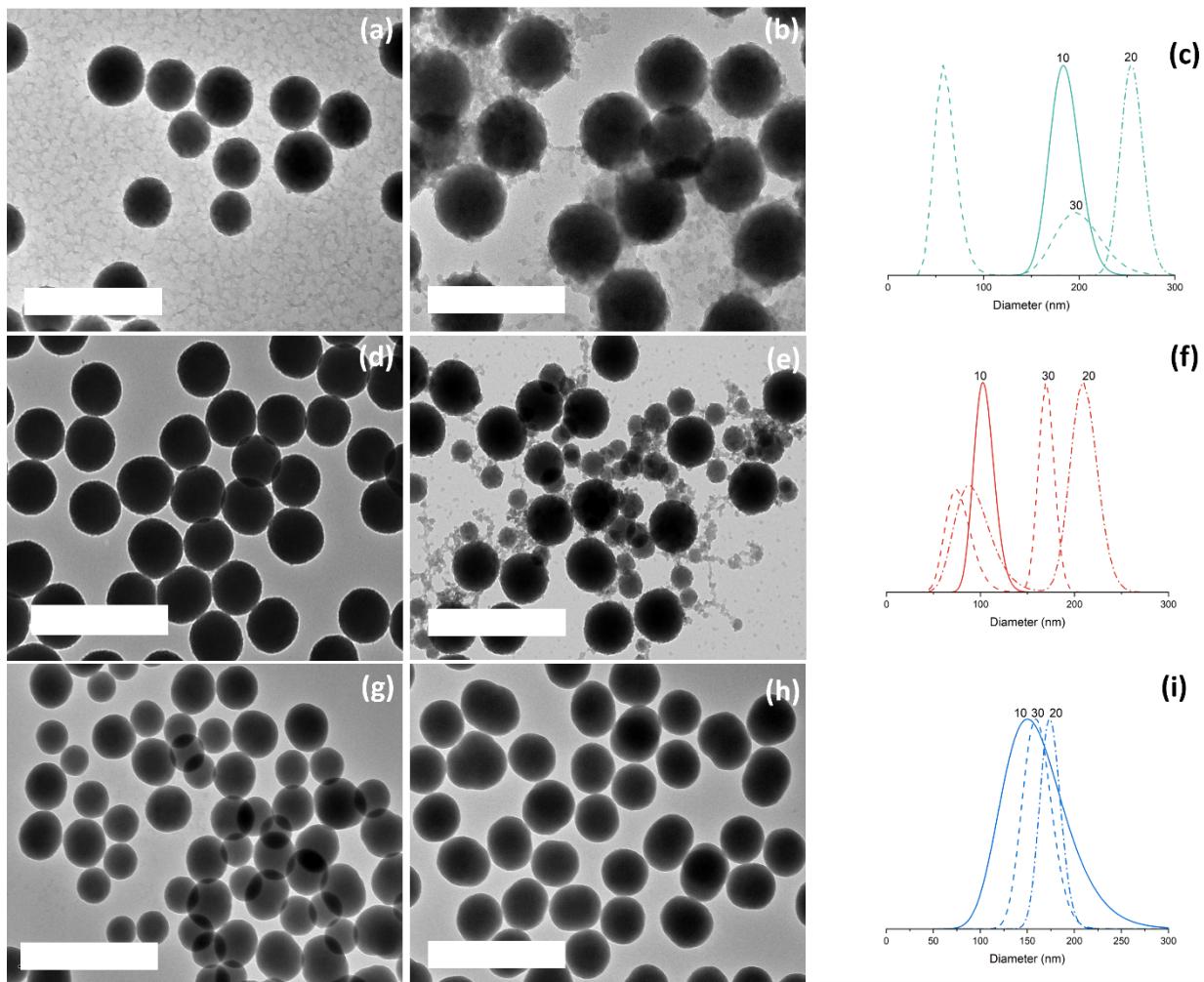


Figure S2. TEM micrographs of the particles produced by modifying the molar ratio of (a,d,g) 90:10 mol%, and (b,e,h) 80:20 mol% TEOS:organoalkoxide, for the different precursors: (a,b) CPTES, (d,e) MPTES, and (g,h) APTES. Scale bars represent 500 nm. Size distribution analysis from TEM images of the different hybrids prepared with (c) CPTES, (f) MPTES, and (i) APTES. The three prepared percentage are presented, in full line 10 mol%, with dash and dot line 20 mol%, and dashed line 30 mol%.

	10%	20%	30%
CPTES	176±16 nm	260±10nm	56±10nm
MPTES	105±8 nm	85±15nm	206±16nm
APTES	146±42nm	165±13nm	157±12nm

Table S1: Size of the particles and dispersion of the samples prepared with different percentage of the organo alkoxide.

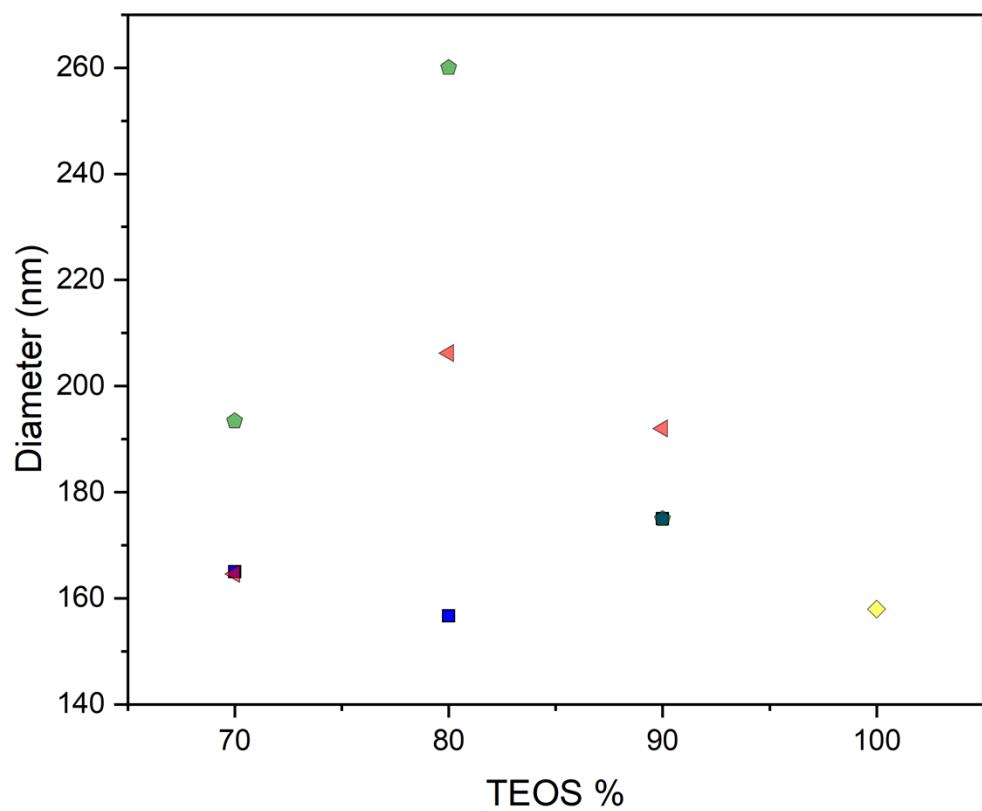


Figure S3: Evolution of the particle size with variation of precursor composition: CPTES (green hexagons), MPTES (red triangles), APTES (blue squares), and pure TEOS (yellow diamond).

Table S1. Relative percentage of the Tⁿ signals and the calculated degree of condensation. Relative percentage of the Q^m signals and the calculated degree of condensation for the hybrid system at 70:30 mol% TEOS:organoalkoxide and pure TEOS. The value is the isomeric displacement (ppm), with percentages in parentheses, δ(%).

Samples	T3	T2	T1	c(T)	Q4	Q3	Q2	Q1	c(Q)
TEOS - δ(%)	--	--	--	--	-110.42(56.97)	-101.6(41.92)	-91.63(1.11)	--	88.965
TEOS-APTES - δ(%)	-66.63(64.57)	-60.96(35.43)	--	88.19	-109.65(54.83)	-100.93(45.17)	--	--	88.7075
TEOS-MPTES - δ(%)	-66.05(54.96)	-59.87(45.04)	--	84.98	-110.39(54.5)	-104.7(42.69)	-91.97(2.81)	--	87.9225
TEOS-CPTES - δ(%)	-66.82(65.5)	-59.91(34.5)	--	88.5	-110.29(63.24)	-101.17(34.97)	-92.18(1.79)	--	90.3625

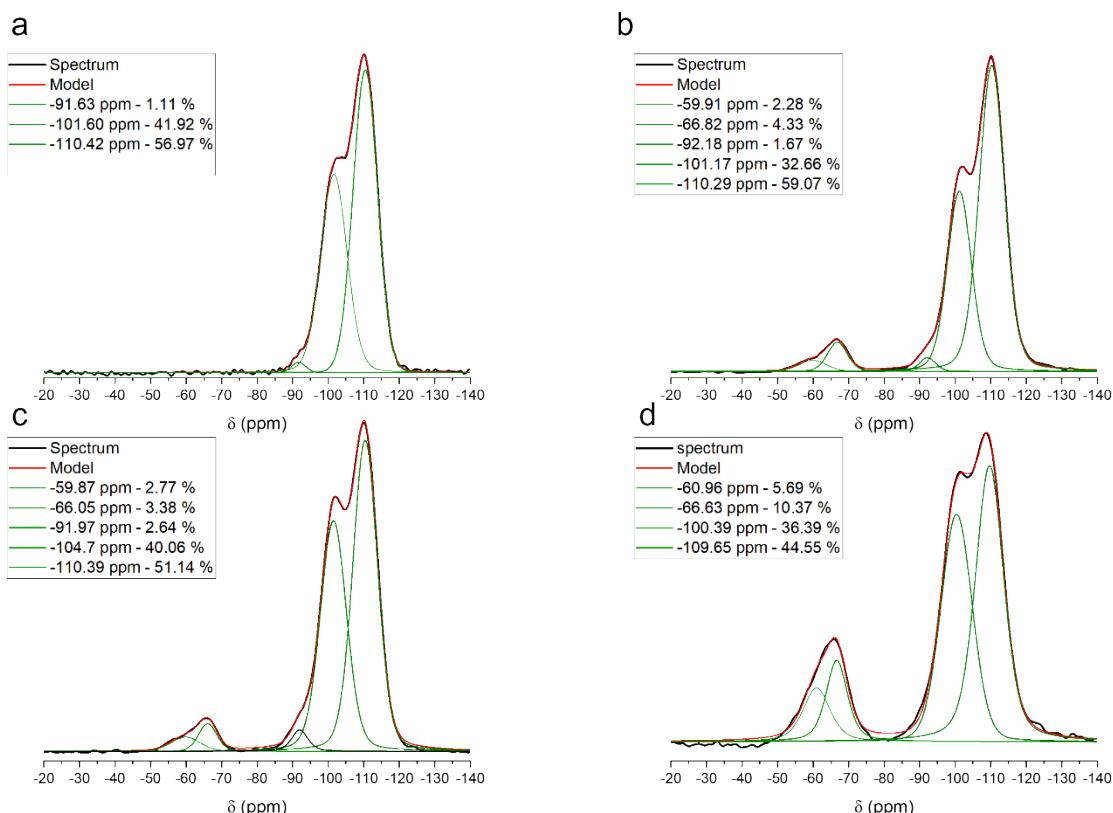


Figure S4: (a) Deconvolution fitting of the ²⁹Si MAS NMR spectra of pure TEOS, and the hybrids with 30 mol% of (b) CPTES, (c) MPTES, and (d) APTES.

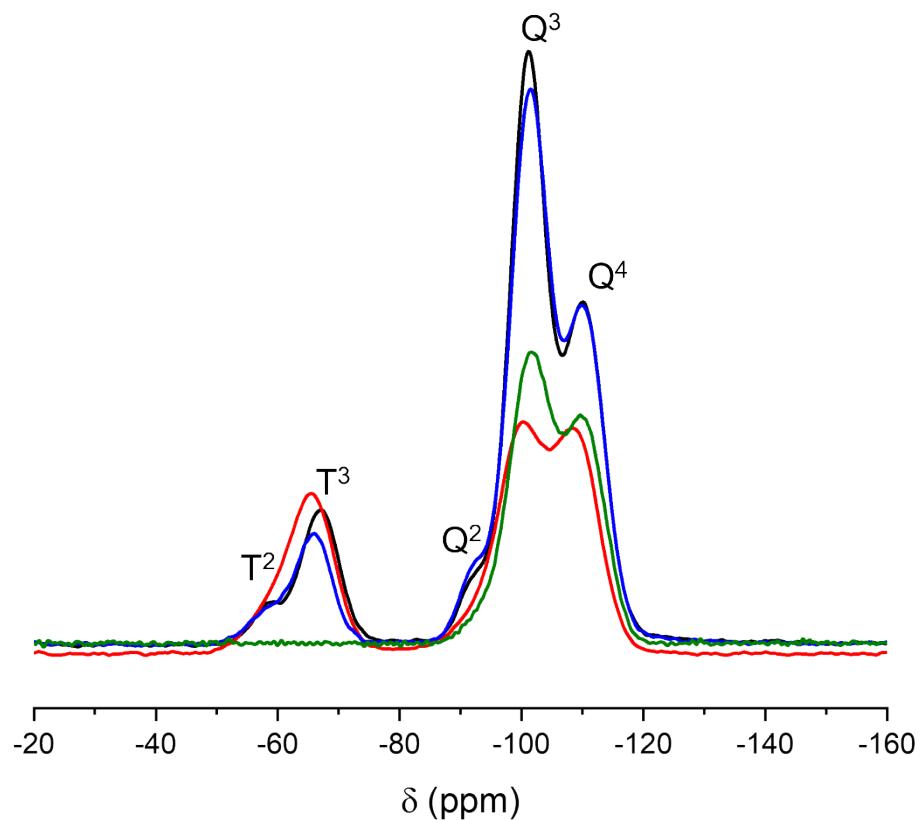


Figure S5. ^{29}Si CP MAS NMR spectra of the pure TEOS particles (black), and the particles produced with 30 mol% of 4-CPTES (green), 3-MPTES (red), and 3-APTES (blue).

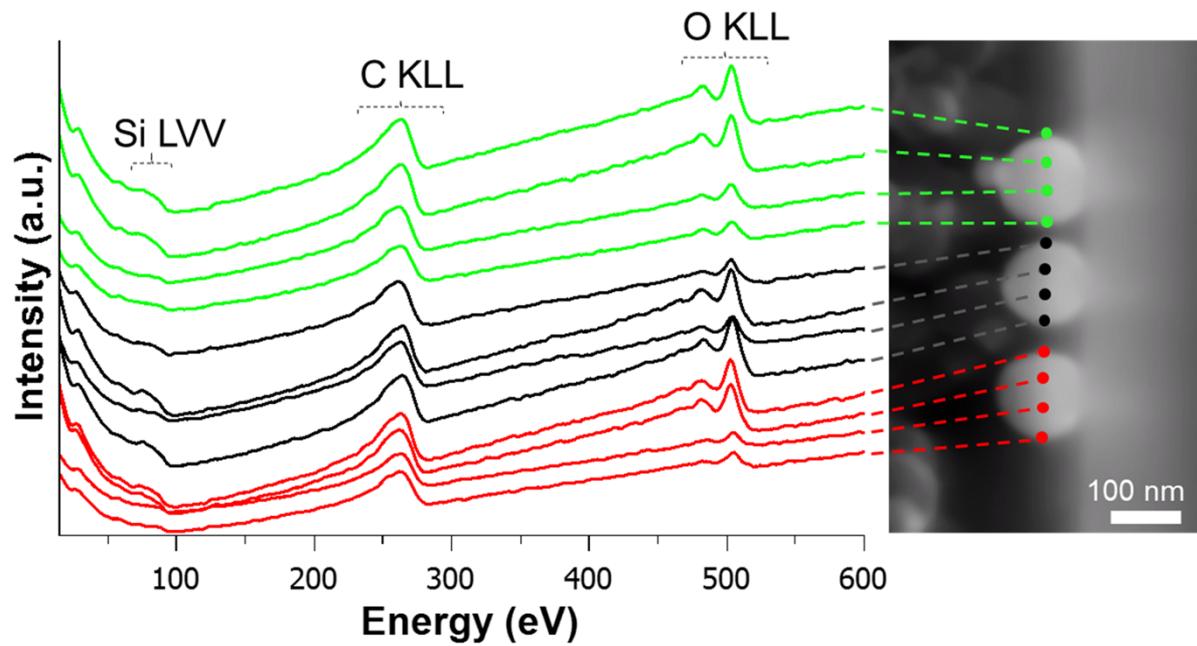


Figure S6. Auger electron spectra of three cross-cut particles with the corresponding target dots along the particle sections of a sample prepared with 30 mol% CPTES.

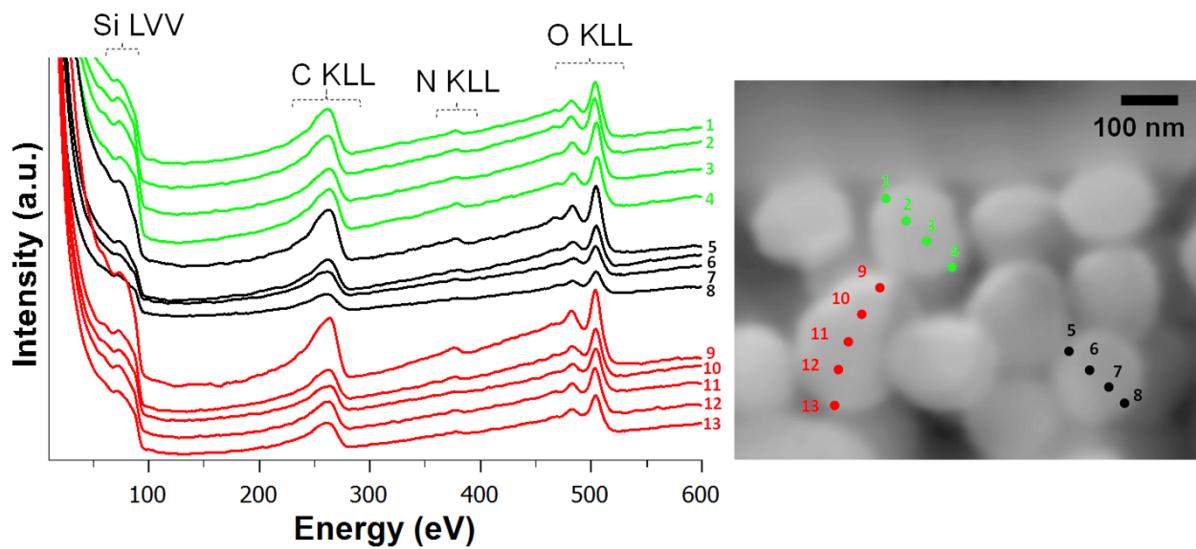


Figure S7. Auger electron spectra of three cross-cut particles with the corresponding target dots along the particle sections of a sample prepared with 30 mol% APTES.