



Figure S1. FT-IR spectra (range 4000–2750 cm⁻¹) of the reference material and the hybrid xerogels of: (a) MPhTEOS series and (b) Ph(TEOS)₂ series.

Table S1. Chemical shifts and integral areas of the Q signals in the ²⁹Si NMR spectra of both series of hybrid xerogels.

Precursor	Molar Percentage (%)	²⁹ Si NMR (ppm)				Band Area		
		Q ²	Q ³	Q ⁴	Q	Q ²	Q ³	Q ⁴
TEOS	0	-91.8	-100.8	-110.0	100.0	18.2	66.4	15.4
	1	-91.7	-100.8	-110.0	100.0	17.2	67.3	15.5
	3.5	-91.7	-100.7	-110.0	95.6	14.6	64.0	17.0
	7.5	-91.8	-100.8	-110.0	93.0	12.4	61.9	18.7
	10	-91.6	-100.5	-109.6	88.7	12.9	58.5	17.3
	12.5	-91.8	-100.7	-109.7	89.8	11.7	61.5	16.6
Ph(TEOS) ₂	1	-89.8	-98.7	-108.0	100.0	11.1	72.0	16.9
	3.5	-91.6	-101.1	-110.5	85.5	10.4	57.4	17.7
	7.5	-91.5	-101.1	-110.8	83.7	10.5	54.1	19.2
	10	-91.7	-100.8	-110.0	78.9	12.1	50.2	16.5
	12.5	-91.5	-100.6	-109.8	70.8	12.6	45.0	13.2

Table S2. Bragg angles (2θ) and bond distances (d₁ and d₂ (nm)) calculated from XRD maxima of both series of hybrid xerogels.

Precursor	Molar Percentage (%)	Peak 2 θ < 10°		Peak 10° > 2 θ < 30°	
		2θ ₁ (°)	d ₁ (nm)	2θ ₂ (°)	d ₂ (nm)
TEOS	0	a	a	23.48	0.38
	1	a	a	23.35	0.38
	3.5	a	a	23.82	0.37
	7.5	4.30	2.06	24.61	0.36
	10	4.43	1.99	23.78	0.37
	12.5	3.75	2.35	23.13	0.38
MPhTEOS	1	a	a	24.16	0.37
	3.5	a	a	23.23	0.38
	7.5	a	a	23.28	0.38
	10	a	a	24.14	0.37
	12.5	a	a	23.80	0.37

a, Non-detected.