Electronic Supplementary Material (ESI) for Materials Chemistry Frontiers.

Tailored structuring of functionalized silsesquioxanes in an one-step approach

Robert Winkler,*[a] Stéphane Pellet-Rostaing [a] and Guilhem Arrachart *[a]

ICSM, Univ Montpellier, CEA, CNRS, ENSCM, Marcoule, France.

Material synthesis

The silica hybrid material (SHM) synthesis was adapted from literature.[1]
In a typical synthesis, 1.5 mmoles of organosilane precursor PX were added to 50 mL of water and the pH was adjusted to basic (NH_4OH, pH 9) or acidic (HCl, pH 1.5) conditions. After stirring for two weeks in a sealed flask, the obtained precipitate was dried by freeze drying and washed with 60 mL (3x20 mL) EtOH/water (1/1) followed by 60 mL (3x20 mL) of EtOH and analysed. Afterwards, the materials were heated to 130 °C for 24 h to promote the condensation and washed in the same order to remove possible degradation products.

[1] Winkler, R.; Pellet-Rostaing, S.; Arrachart, G. Selective Extraction of REEs Thanks to One-Pot Silica Hybrid Materials. *Appl. Sci.* **2020**, *10* (21), 7558.

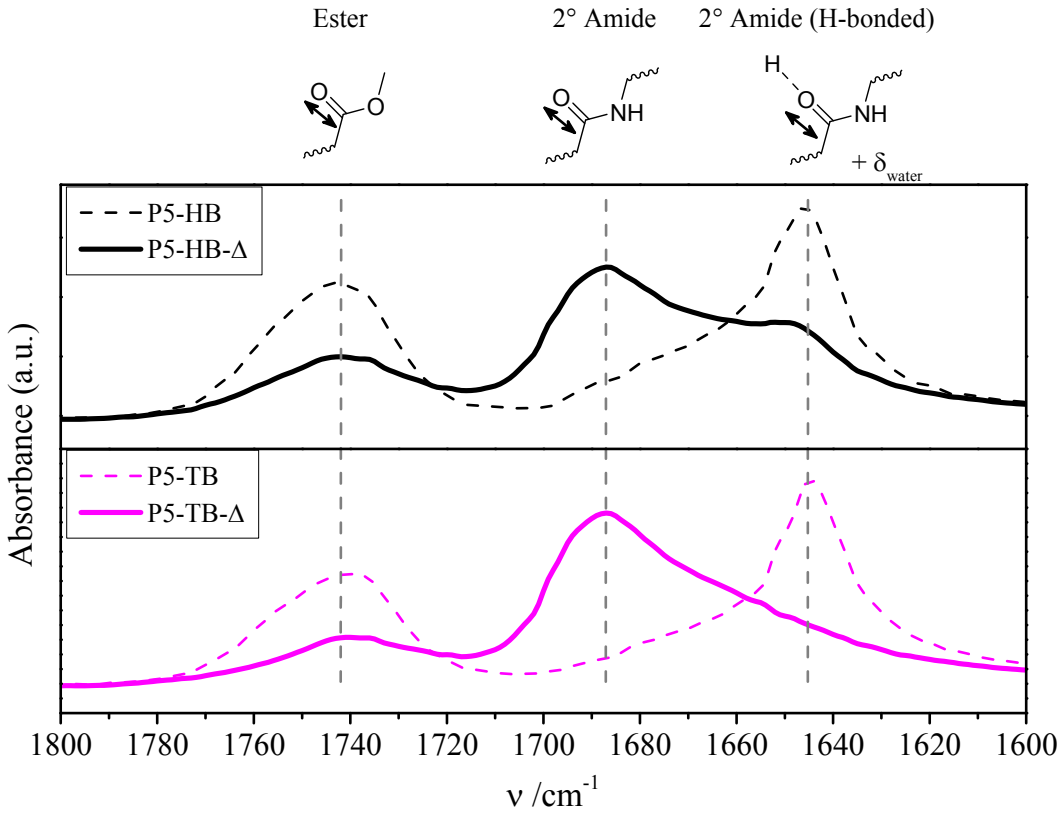

Figure S1. FT-IR spectra of the sample P5, ν=1800-1600 cm^{-1}.

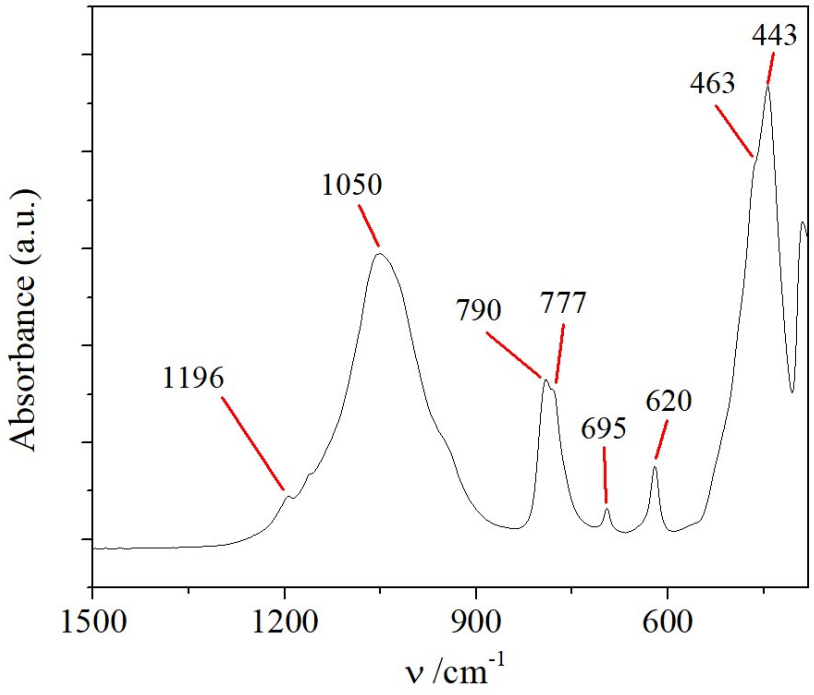

Figure S2. Typical FT-IR spectrum of the residual material after the TGA analysis.

The peaks indicate crystalline silica structures like *e.g.* β-cristobalite.

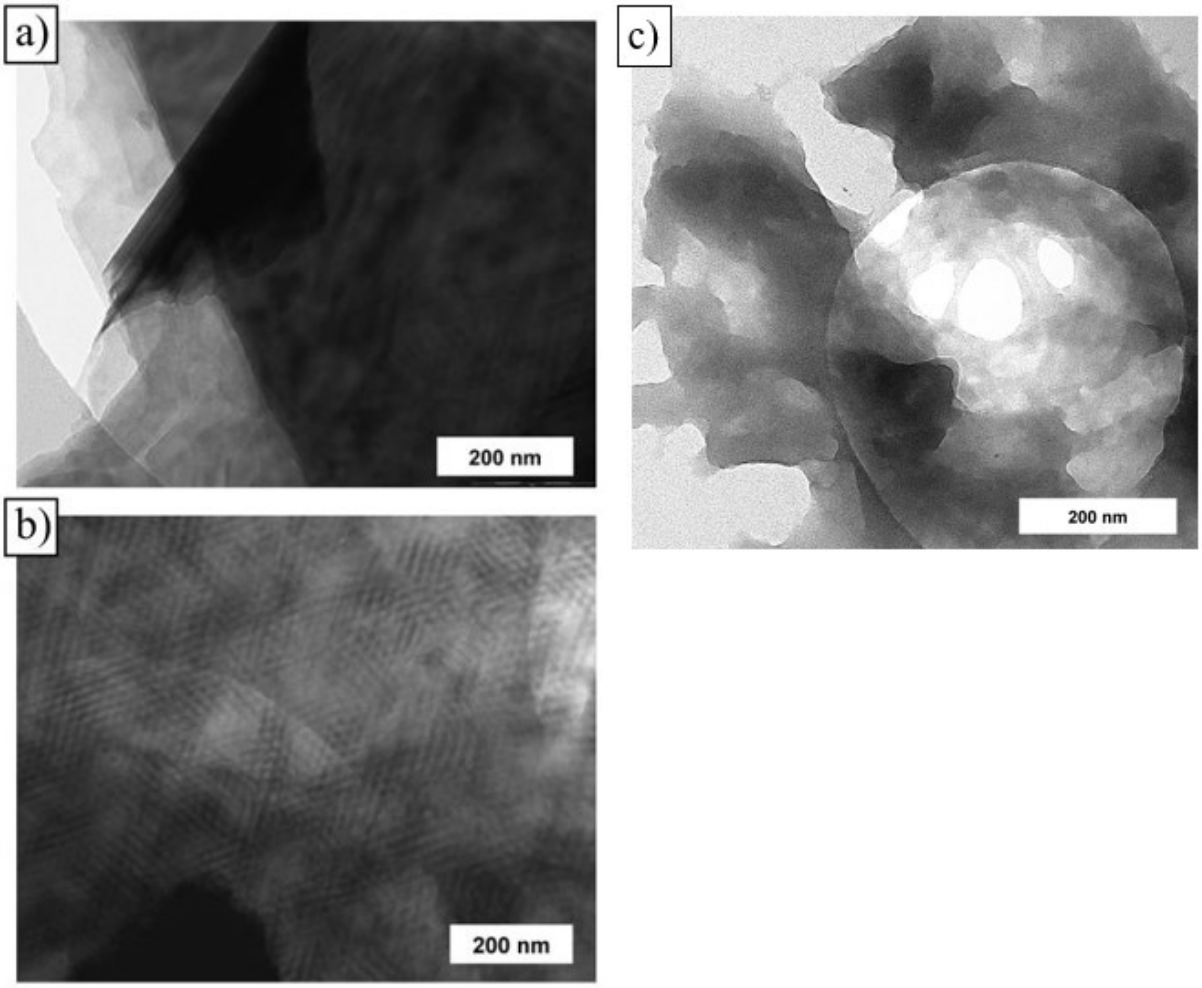

Figure S3. TEM images of samples: a) and b) P1-TA-Δ and c) P1-TB-Δ.

The scale is represented on the lower right.

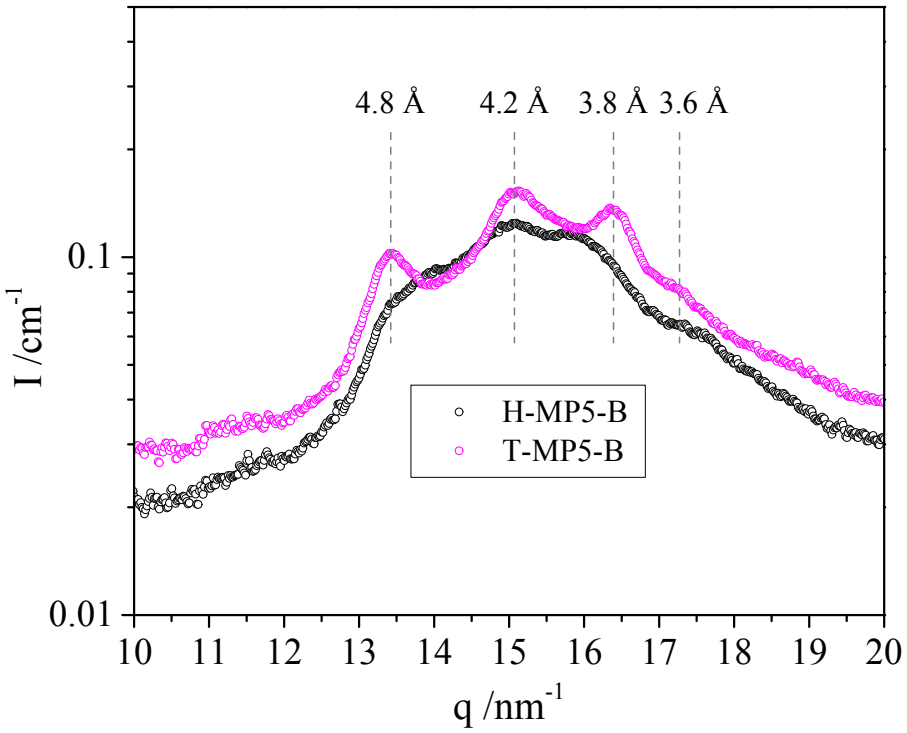

Figure S4. WAXS patterns of materials P5-HB and P5-TB.

Table S1. Fit parameters for the qualitative fit P5-TB/-Δ.

Parameter	Fresh material	After thermal treatment	Description
ρ_{CH2}	0.73E-10 cm^{-2}	0.73E-10 cm^{-2}	SLD of the alkyl chains
ρ_{SiOx}	1.23E-10 cm^{-2}	1.6E-10 cm^{-2}	SLD of the Si-O-Si network
ρ_{HG}	1.04E-10 cm^{-2}	0.85E-10 cm^{-2}	SLD of the headgroup
σ_{SiOx}	0.06 nm	0.06 nm	FWHM of the Si-O-Si network
σ_{HG}	1.3 nm	1.44 nm	FWHM of the headgroup
d	5.95 nm	6.7 nm	Separation distance