

On the Organic/Inorganic Interface between Mesoporous SBA-16 Silica and its Structural Directing Polymer: a Combined FT-IR and Solid State NMR study.

Fabio Carniato,^a Geo Paul,^a Chiara Bisio,^{a,b,*} Stefano Caldarelli,^c Leonardo Marchese^a

^aDipartimento di Scienze e Tecnologie Avanzate, Nano-SISTEMI Interdisciplinary Centre,
Università del Piemonte Orientale “A. Avogadro”, Viale Teresa Michel 11, 15121
Alessandria, Italy.

^bISTM-CRN Istituto di Scienze e Tecnologie Molecolari, via G. Venezian 21, Milano, Italy
corresponding author: chiara.bisio@mfn.unipmn.it

^cAix Marseille Université, ISm2 UMR 6263, Campus de Saint Jérôme, Service 511 F-13013
Marseille (France).

Supporting Information

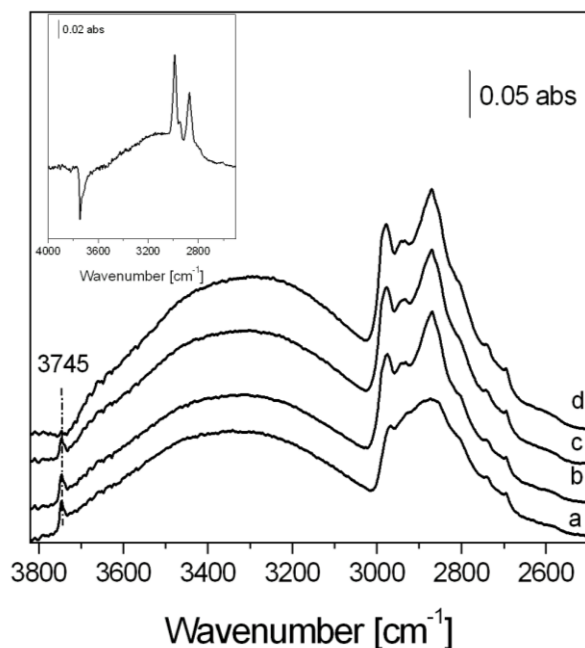


Fig. S1. FT-IR spectra of diethylether adsorbed at rt on the calcined SBA-16 sample mixed to Pluronic F127. Spectrum a) was collected in vacuum after treating the sample at 100°C for 2h, spectra b-d) were registered upon adsorption of 15 mbar of Et₂O and left to equilibrium for 1' (b), 10' (c) and 35 minutes (spectrum d). The inset shows the difference spectrum (d-a).

The ether adsorption of a sample prepared by mechanically mixing calcined SBA-16 and F127 co-polymer was also carried out and the obtained results are reported in Fig. S1. The sample treated at 100°C in vacuum for 2h present bands typical of F127 polymer. The bands in the 3000-2500 cm⁻¹ range appeared broader than those of as-synthesized sample, thus indicating that in the mechanically mixed sample the Pluronic is organised in a different fashion than in the as-SBA-16 and also probably less homogeneously distributed.

In addition, the spectrum of mechanically mixed sample showed a residual band at 3745 cm⁻¹ (not detected in the as-SBA-16 sample) due to SiOH species probably located in internal porosities of the sample and not accessible to the co-polymer added by mechanical grinding. These species are able to interact with diethylether (Fig. S1, curves b-d): the original band at 3745 cm⁻¹ is indeed converted in a new one at ca. 3200 cm⁻¹ upon exposing the sample to Et₂O for ca. 30 minutes. This effect is more evident in the inset of Fig. S1, where the spectrum of the sample before the adsorption has been subtracted from that of the sample exposed 35 min at 15 mbar of Et₂O vapour.

This experiment helped us to better understand the interactions of internal silanol groups with diethylether probe molecule.

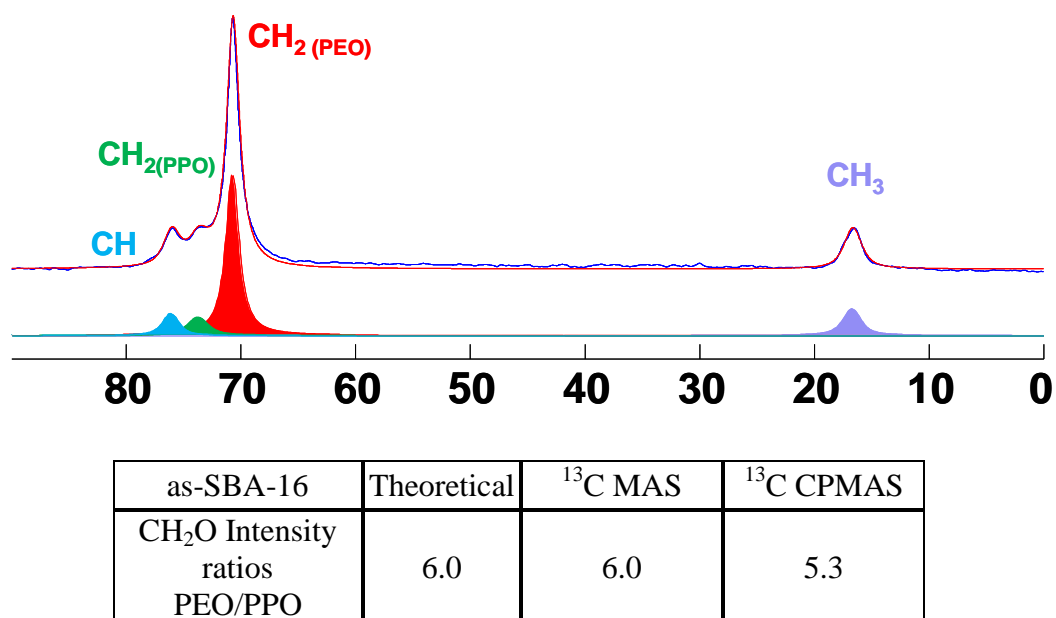


Fig. S2. ^{13}C NMR spectrum of as-SBA-16. The CH₂O Intensity ratio PEO/PPO estimated by ^{13}C MAS NMR and ^{13}C CP MAS NMR is reported in the Table.

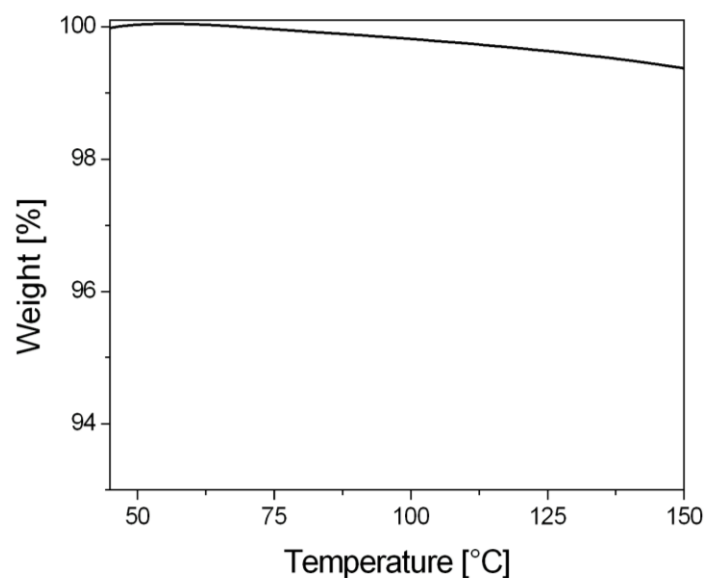


Fig. S3. TGA profile of as-synthesized SBA-16 collected under argon flow, after isothermal treatment at 80°C for 8 h in inert conditions.

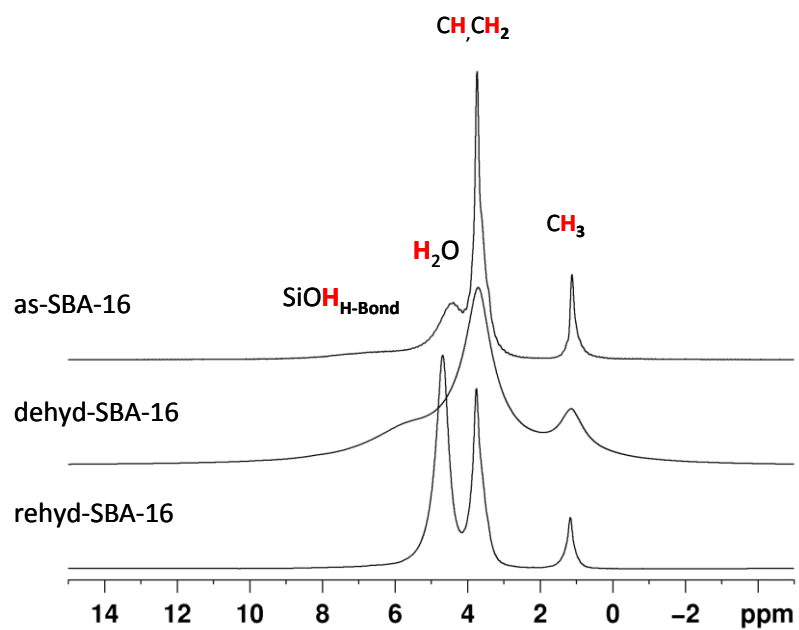


Fig. S4. ^1H MAS NMR spectra of as-SBA-16 sample and after dehydration (dehyd-SBA-16) and rehydration (rehyd-SBA-16) conditions.