Electronic Supplementary Information

POSS as building-blocks for the preparation of polysilsesquioxanes through an innovative synthetic approach

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Contents:

Experimental Section

1) Materials:

The partially condensed heptaisobutyl-POSS, purchased from the Hybrid Plastics Company, was used as a precursor for the preparation of PSQ-IsobutylPOSS samples.

2) PSQ-IsobutylPOSS synthesis:

1 g of heptaisobutyl-POSS was introduced in the alumina crucible and introduced inside a tube furnace and heated at 170 °C for different reaction times (1, 2, 4 and 6 hours) under nitrogen flow (rate of 100 $mL \cdot min^{-1}$). The thermal treatment was carried out following this program: ramp from 25 to 170 °C with rate of 5 °C min^{-1}, isotherm at 170°C for different hours and ramp from 170 to 25 °C with rate of 25 °C min^{-1}. Finally, the products obtained in form of transparent gel-like film were recovered.

3) Characterization:

FTIR analyses were carried out using a Thermo Electron Corporation FT Nicolet 5700 Spectrometer equipped with a pyroelectric detector (DTGS type) with a resolution of 4 cm^{-1}. Prior to the analysis, the samples were mixed with KBr matrix (5 wt%).

X-ray diffraction (XRD) patterns were obtained on a ARL XTRA48 diffractometer using Cu Kα radiation (λ = 1.54062 Å).

^{29}Si NMR spectra were acquired on a Bruker Avance III 500 spectrometer with a wide bore 11.7 Tesla magnet with operational frequencies of 99.35 MHz for ^{29}Si. The chemical shift values (δ [ppm]) in the NMR spectra are reported with respect to the internal/reference standard tetramethylsilane (TMS). The sample were dissolved in in deuterated chloroform ($CDCl_3$) at room temperature.

Thermogravimetric analyses were carried out with Setaram SETSYS Evolution thermobalance. The data were collected in the range 30-800 °C with scan rate of 10 °Cmin^{-1} under argon flow (20 mL/min).

MALDI-TOF analysis was performed by using the Mass Spectrometer Voyager DE Pro (AB-Sciex) equipped with a MALDI-TOF source and a quadrupole analyzer. For MALDI-TOF analysis, the sample were analyzed without the use of resins. After thorough drying in the dark, 100 shots per spectrum were acquired. The data were processed using the Data Explorer software from AB-Sciex.

Contact angle measurements were performed using an optical thensiometer Attension Theta with the sessile drop method in static conditions, using ultrapure water for HPLC as liquid for the analysis. The contact angle data were processed using the OneAttension software.

Figures

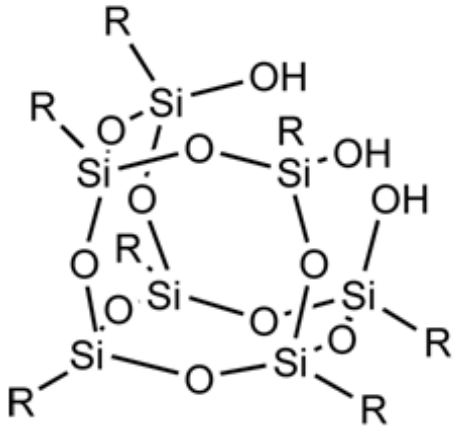

Figure S1: Schematic view of the partially condensed heptaisobutyl-POSS. R = isobutyl group.

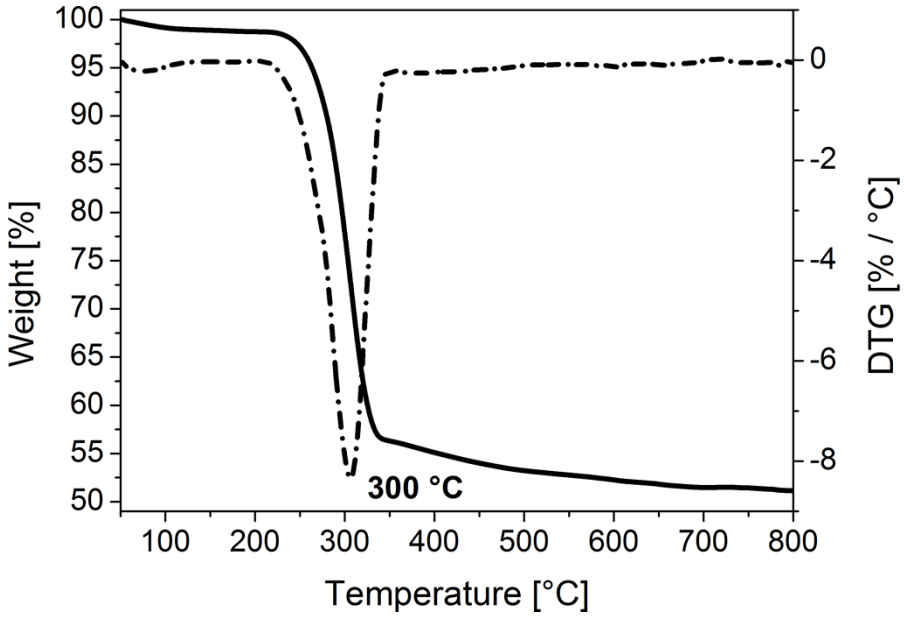

Figure S2: TG (solid line) and DTG (dash line) curves of PSQ-IsobutylPOSS-6h measured under argon flow.

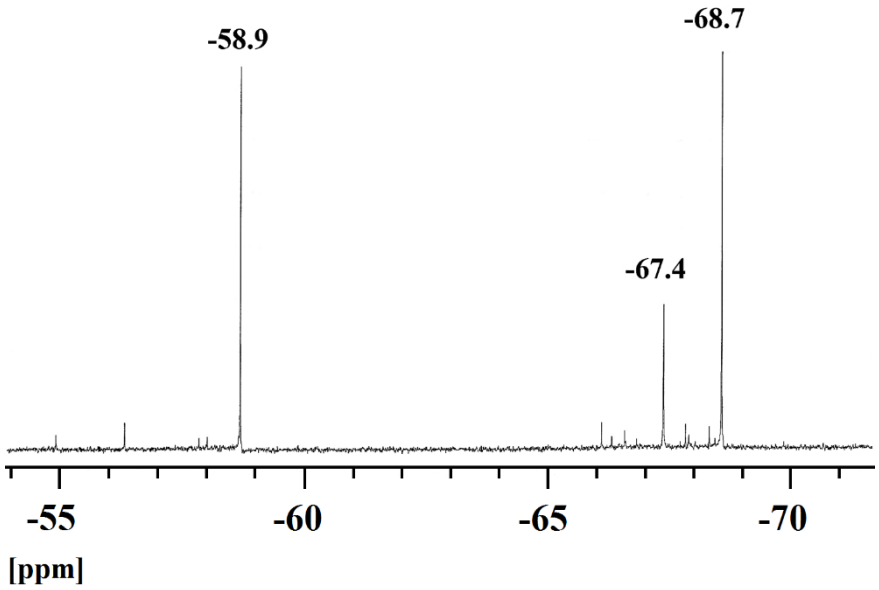

Figure S3. ^{29}Si-NMR spectrum of PSQ-IsobutylPOSS-1h sample.

Figure S4. Image of the PSQ-IsobutylPOSS-6h.

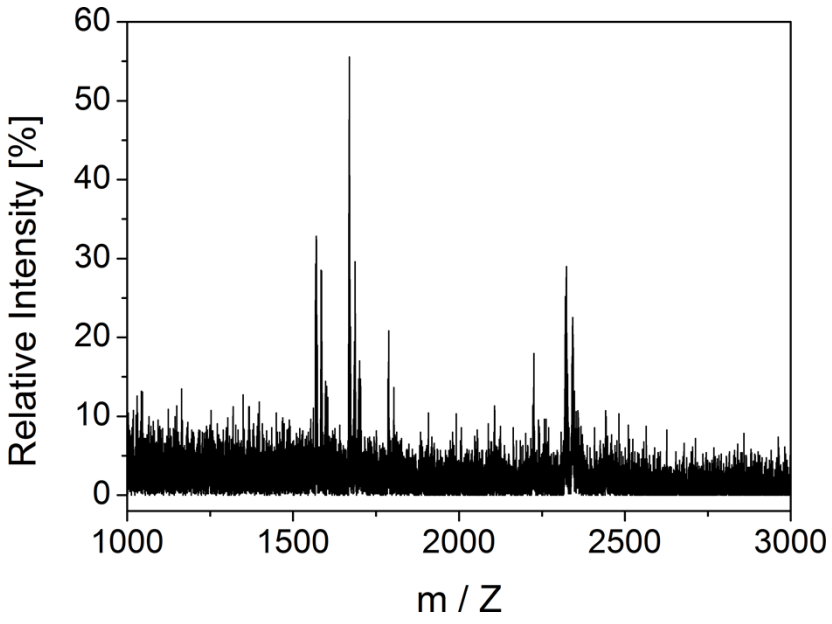

Figure S5. MALDI-TOF analysis of PSQ-IsobutylPOSS-6h.

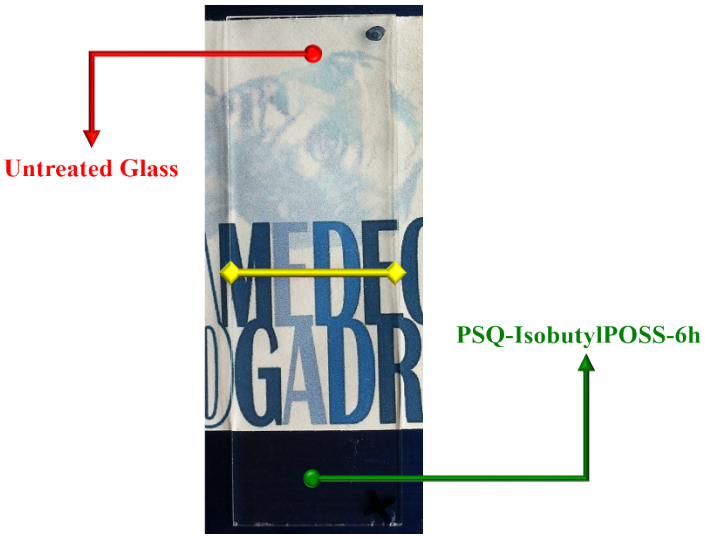

Figure S6. Photograph of PSQ-IsobutylPOSS-6h film coated on a glass substrate. The upper section of the slide is untreated.