

Hydrolytic polycondensation of trimethoxymethylsilane under ultrasonic irradiation

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^1H and ^{29}Si NMR, FTIR and GPC analysis data: ^1H NMR spectrum of blocked MTMS HPC product (CDCl_3 , δ): 0.03—0.21 (m, SiMe); 3.48—3.53 (m, OMe); 5.70—6.21 (m, SiVin).

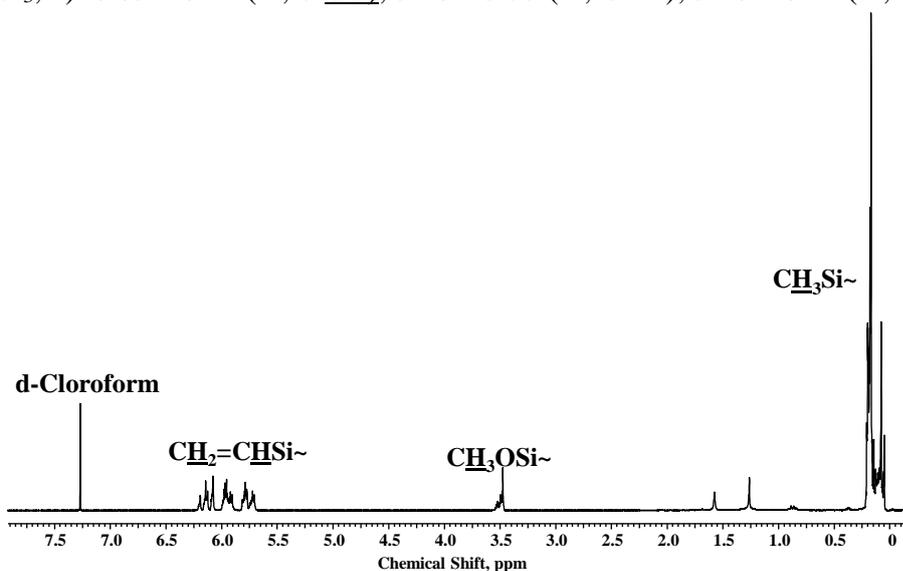


Figure S1 The ^1H NMR spectrum of the non-volatile TMMS hydrolysis product (see Table 1 of the main text, entry 3) after blocking with chlorovinyltrimethylsilane.

^{29}Si NMR spectrum of blocked MTMS HPC product (CDCl_3 , δ , ppm): -1.96, -2.85, -3.63, -3.77 ppm (m, $\text{O}_{0.5}\text{SiMe}_2\text{Vin}$); -56.17, -56.28, -57.00, 58.46 ppm (m, $\text{OSi}(\text{Me})\text{OMe}$); -63.98, -65.33, -65.63, -66.93, -67.26 ppm (m, $\text{O}_{1.5}\text{SiMe}$).

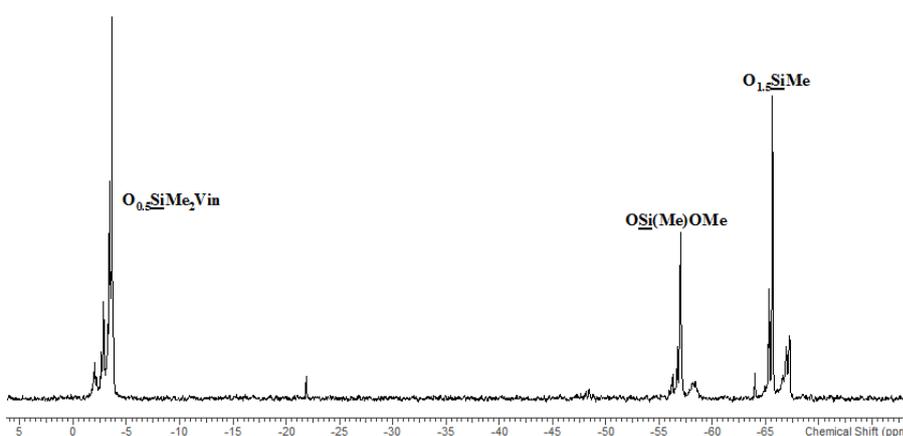


Figure S2 The ^{29}Si NMR spectrum of the non-volatile TMMS hydrolysis product (see Table 1 of the main text, entry 18) after blocking with chlorovinyltrimethylsilane.

IR spectrum of blocked MTMS HPC product (CCl_4), v/cm^{-1} : 3051 w, 2961 (C–H) w; 1595 (C=C) w; 1406 (C=C) w; 1267 w, 1252 (Si–C) w; 1029 s, 954 (Si–O) w; 835 m, 780 s, 704 w, 518 (Si–C) w.

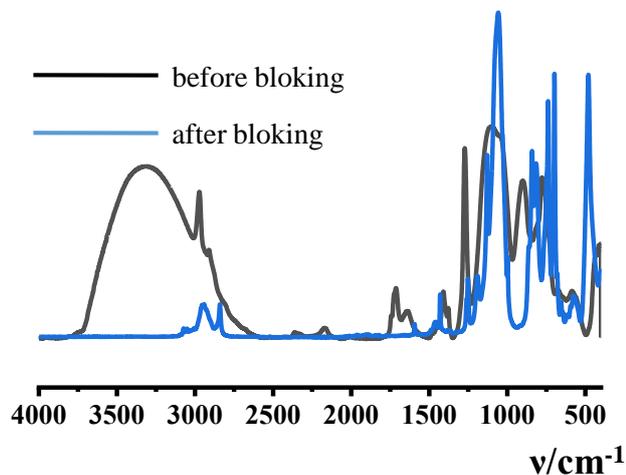


Figure S3 IR spectra of the TMMS hydrolysis product (see Table 1 of the main text, entry 1) before (black line) and after blocking (blue line) with chlorodimethylvinylsilane.

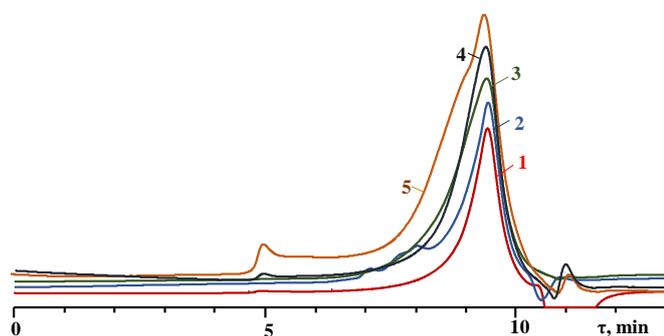


Figure S4 GPC curves of TMMS HPC products depending on the sonication temperature: (1) 15°C; (2) 25°C; (3) 35°C; (4) 45°C; (5) 55°C.

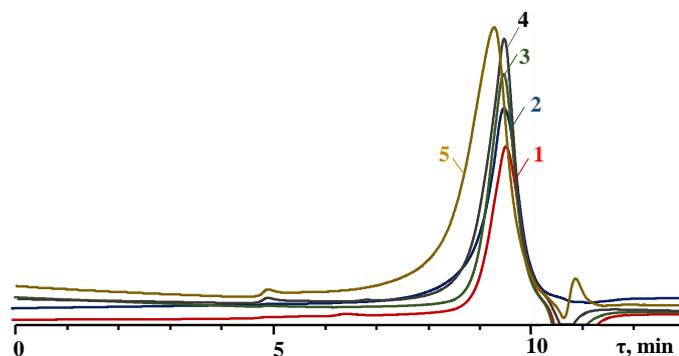


Figure S5 GPC curves of TMMS HPC products depending on the sonication power: (1) 150 W; (2) 300 W; (3) 600 W; (4) 900 W; (5) 1200 W.

Experimental

Methods for studying and monitoring the processes

GC analysis was performed on a Khromatek-Analitik-5000 chromatograph (Russia) with a catharometer detector. The column length was 2 m and the diameter was 3 mm. Helium was used as the carrier gas; the gas rate was 30 ml min⁻¹. SE-30 (5%) deposited on Chromaton-H-AW was used as the stationary phase.

GPC analysis was performed on a chromatographic system consisting of a STAIER series II high-pressure pump (Aquilon, Russia), a RIDK 102 refractometric detector (Czech Republic), and a JETSTREAM 2 PLUS column thermostat (KNAUER, Germany). The temperature was controlled at 40°C (+/- 0.1°C). THF was the eluent, the flow rate was 1.0 ml min⁻¹. A 300 x 7.8 mm column filled with Phenogel sorbent (Phenomenex, USA), particle size 5 µm, pore size 10³ Å was used (passport separation range - up to 75000 D). Recording and processing of data was carried out using UniChrom 4.7 software (Belarus).

¹H NMR spectra were recorded on a Bruker AC-250 spectrometer in CDCl₃.

IR spectra were recorded using a Nicolet iS50 IR spectrometer with ATR set-up (diamond) with resolution of 4 cm⁻¹ at 32 scans.

Equipment

PSB – Gals ultrasonic bath, PSB–4035–05m model, with an operating frequency of 35 kHz and a radiator power of 150 W. Inlab ultrasonic generator, model I10–2.0, with an operating frequency of 22 kHz and power adjustable from 0 to 2000 W, equipped with a magnetostrictive converter Inlab model PMS–2.0/22, with a Lagen flowing water cooling system for the converter, model LSO–156M, and an immersion waveguide probe.

Synthesis techniques. HPC using an ultrasonic generator (ultrasonic bath)

Weighed samples of TMMS and water corresponding to the ratios to be studied were placed in the reactor. The process was carried out under the specified conditions such as temperature, power, irradiation time, and ratio of the starting reagents (see Table 1 of the mail text). The temperature mode was implemented using an oil bath or flow-through water cooling of the reactor. Upon process completion, the mixture was blocked with chlorodimethylvinylsilane as described [A. A. Kalinina, D. N. Kholodkov, I. B. Meshkov M. A, Pigaleva, I. V. Elmanovich, Y. A. Molodtsova, M. O. Gallyamov and A. M. Muzafarov, *Russ. Chem. Bull.*, 2016, **65**, 1104]. The volatile fractions of the blocked product were analyzed by GC. The non-volatile fractions were analyzed by GPC, NMR and IR spectroscopy.