Electronic Supplementary Material (ESI) for RSC Advances.

Supporting info for

Novel Porous materials based on Oligospiroketals (OSK)

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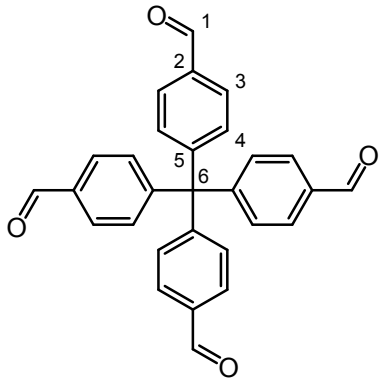
Synthesis of 4,4',4'',4'''-methanetetrayltetrabenzaldehyde (**1d**)



A solution of Tetrakis-(4-bromophenyl)methan (1.25 g, 1.97 mmol) in 75 mL dry THF was cooled to -78 °C and at this temperature was added slowly *n*-buthyllithium (1.6 M in hexane, 9.8 mL, 15.72 mmol) at -78 °C. After stirring the reaction mixture for 8 hour at this temperature dry DMF (5 mL, 64.86 mmol) was added and stirred over night while the temperature was allowed to rise to room temperature. The reaction mixture was concentrated under reduced pressure, 1M HCl was added and extracted with methylene chloride. The organic layer was dried over $MgSO_4$ and the solvent removed under reduced pressure. The raw product was purified by column chromatography with 28 % yield.

^{1}H-NMR (δ / ppm, $CDCl_3$, 300 MHz): 7.43 (d, ^{3}J = 8.4 Hz, 8 H, H-4), 7.84 (d, ^{3}J = 8.4 Hz, 8 H, H-3), 10.01 (s, 4 H, H-1)

^{13}C-NMR (δ / ppm, $CDCl_3$, 75 MHz): 66.3 (C-6), 129.6 (C-4), 131.3 (C-3), 135.0 (C-2), 151.0 (C-5), 191.2 (C-1)

MS (ESI): 432.1360 $[M]^{+}$

MALDI-TOF spectra were measured with the MALDI-TOF massspectrometer *Axima Assurance* from *Shimadzu Biotech* and were plotted with the software *Launchpad 2*. The Matrix was 2,5-dihydroxybenzoic acid (DHB) with addition of sodium iodide.

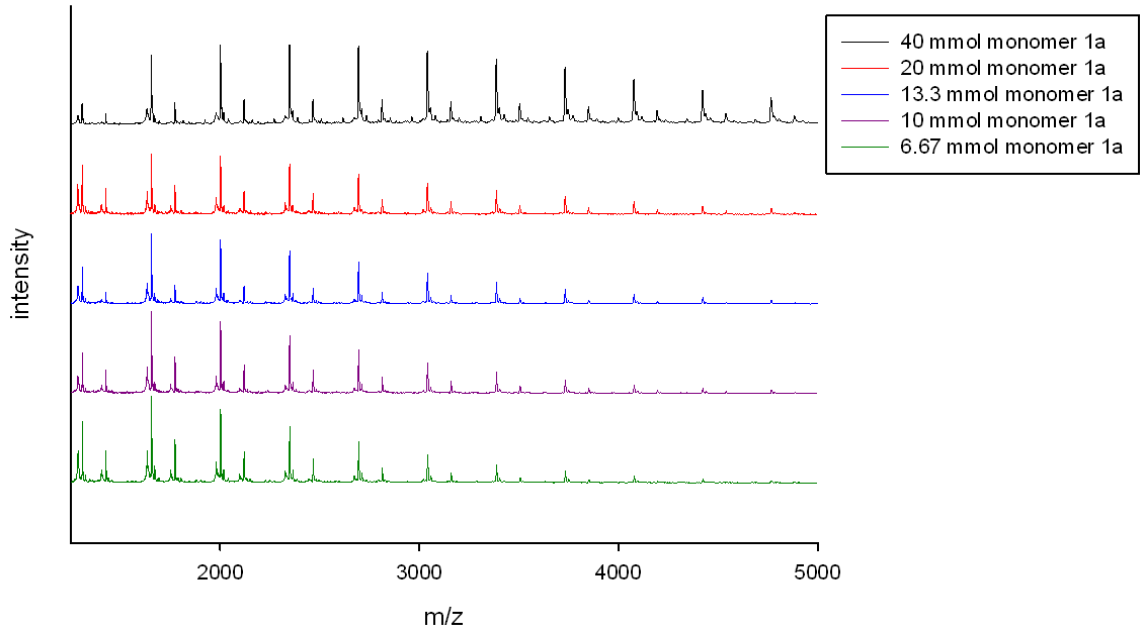


Fig. S1. MALDI-TOF MS spectra demonstrating the influence on the monomer concentration of polyacetal **TOSU-PA-a**.

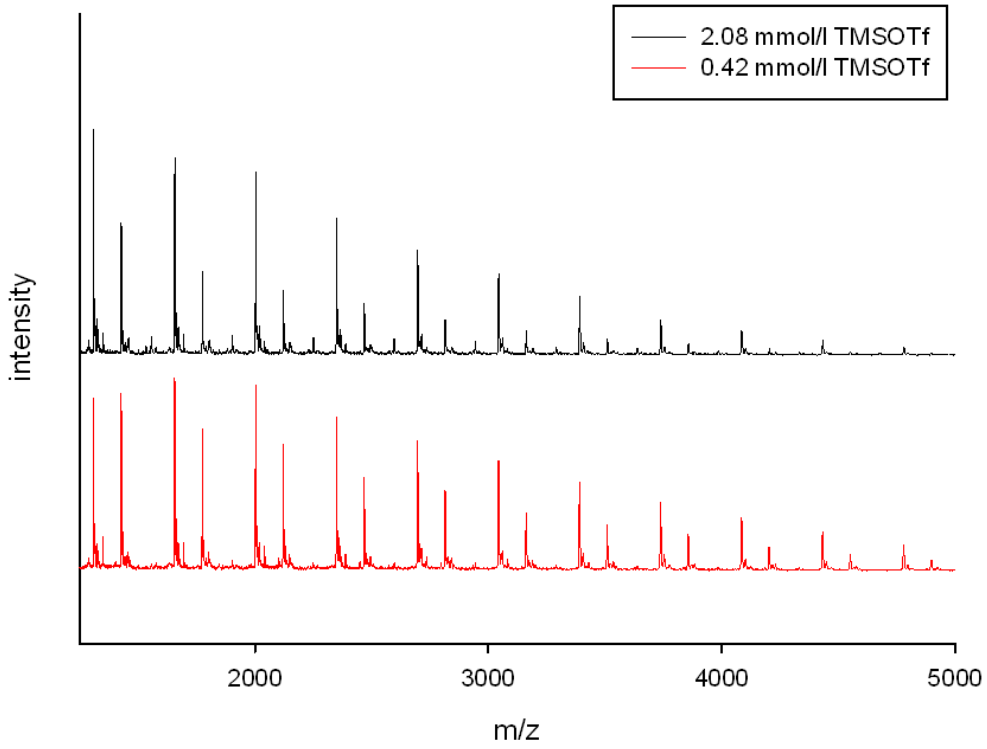


Fig. S2. MALDI-TOF MS spectra demonstrating the influence on the catalyst concentration of polyacetal **TOSU-PA-a**.

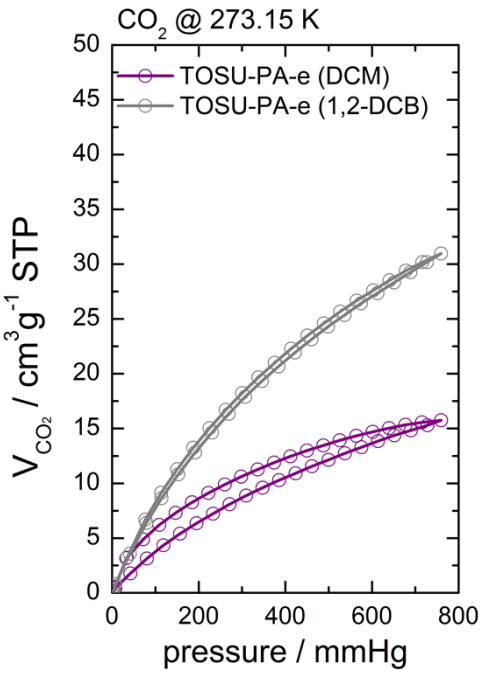


Fig. S3. CO_2 (273 K) adsorption/desorption isotherms (adsorption with filled spheres, desorption with hollow spheres) of TOSU-PA-e material synthesized under different conditions.

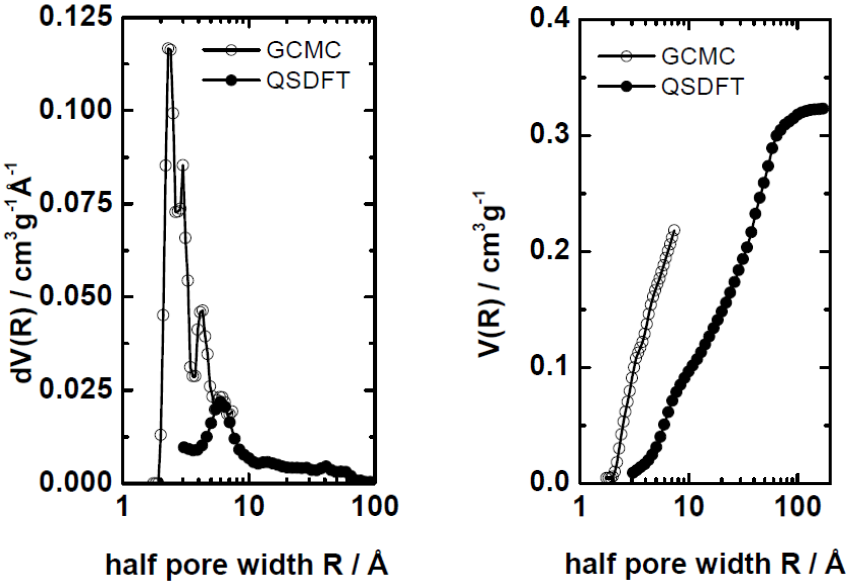


Fig. S4. Pore size distribution (PSD, left-hand side) and respective cumulative pore volume of TOSU-PA-c, determined by analysis of the N_2 adsorption branch using a quenched-solid density functional theory (QSDFT) kernel (assuming slit pores, carbon-N_2 interaction) together with the Grand-Canonical Monte-Carlo (GCMC) kernel based analysis of the CO_2 adsorption data. The CO_2 data can give a better description of the micropores (which are underestimated somewhat by N_2 analysis), while it cannot describe the mesoporosity of the material (better described by QSDFT analyis of N_2 data). The second inflection point of the cumulative pore volume calculated by QSDFT model can give information on the average mesopore size (this information is typically a bit blurred in the PSD plot if micropores are also present) and a mesopore size of ~ 8-10 nm can be determined.

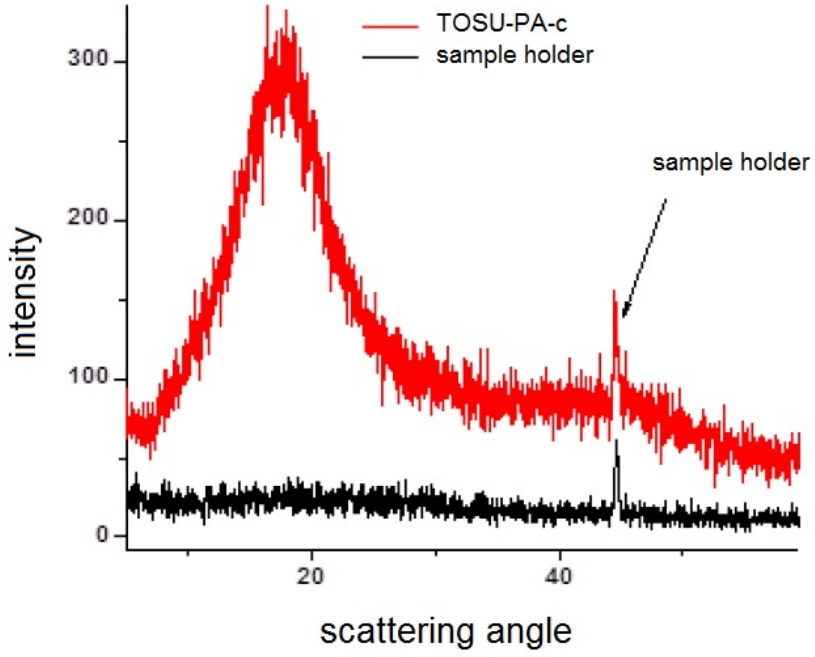


Fig. S5. representative wide-angle X-ray scattering (WAXS) pattern of TOSU-PA-c (red) and the sample holder (black), indicating that the materials are totally amorphous.

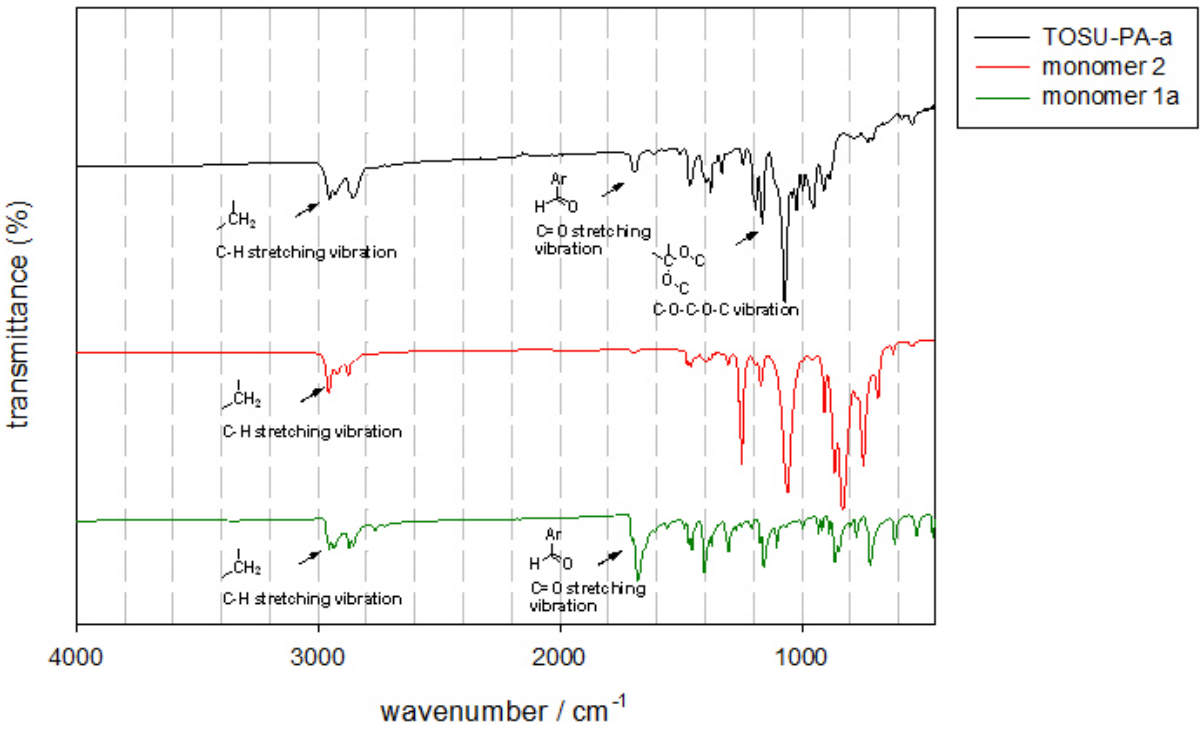


Fig. S6. FT-IR spectra of **TOSU-PA-a**, trimethylsilyl ether of pentaerythritol (**2**) and terephthalaldehydes **1a.**

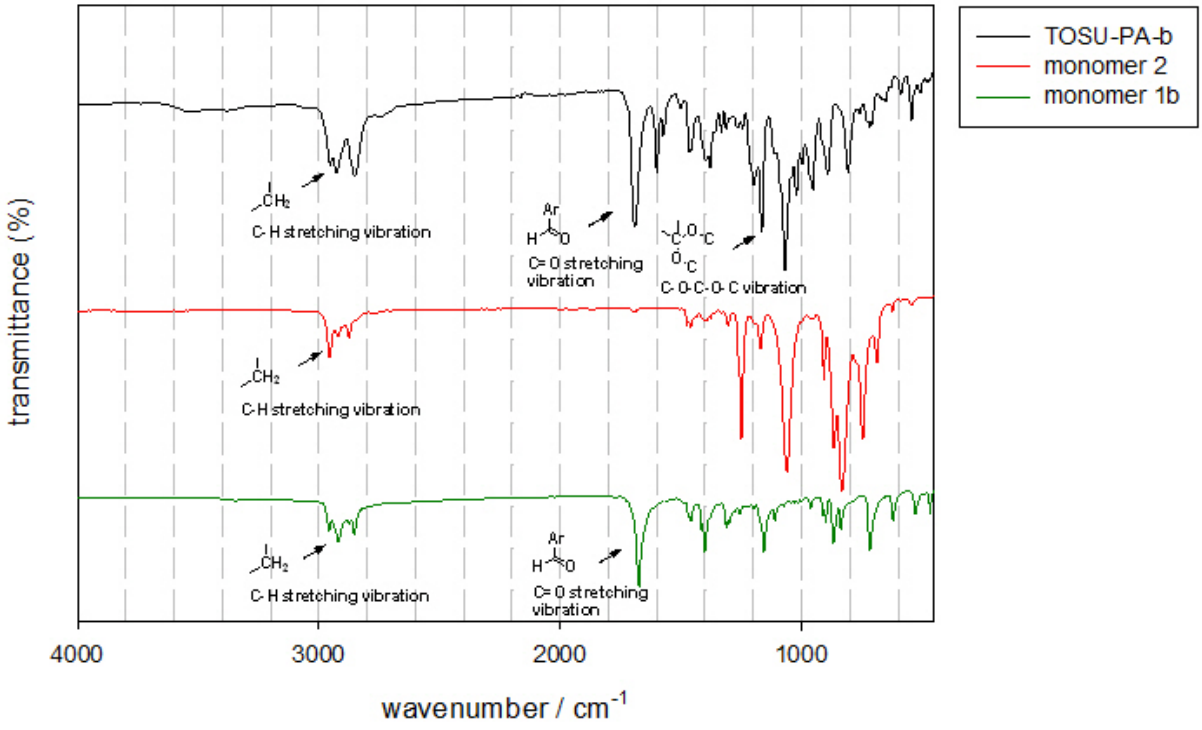


Fig. S7. FT-IR spectra of **TOSU-PA-b**, trimethylsilyl ether of pentaerythritol (**2**) and terephthalaldehydes **1b.**

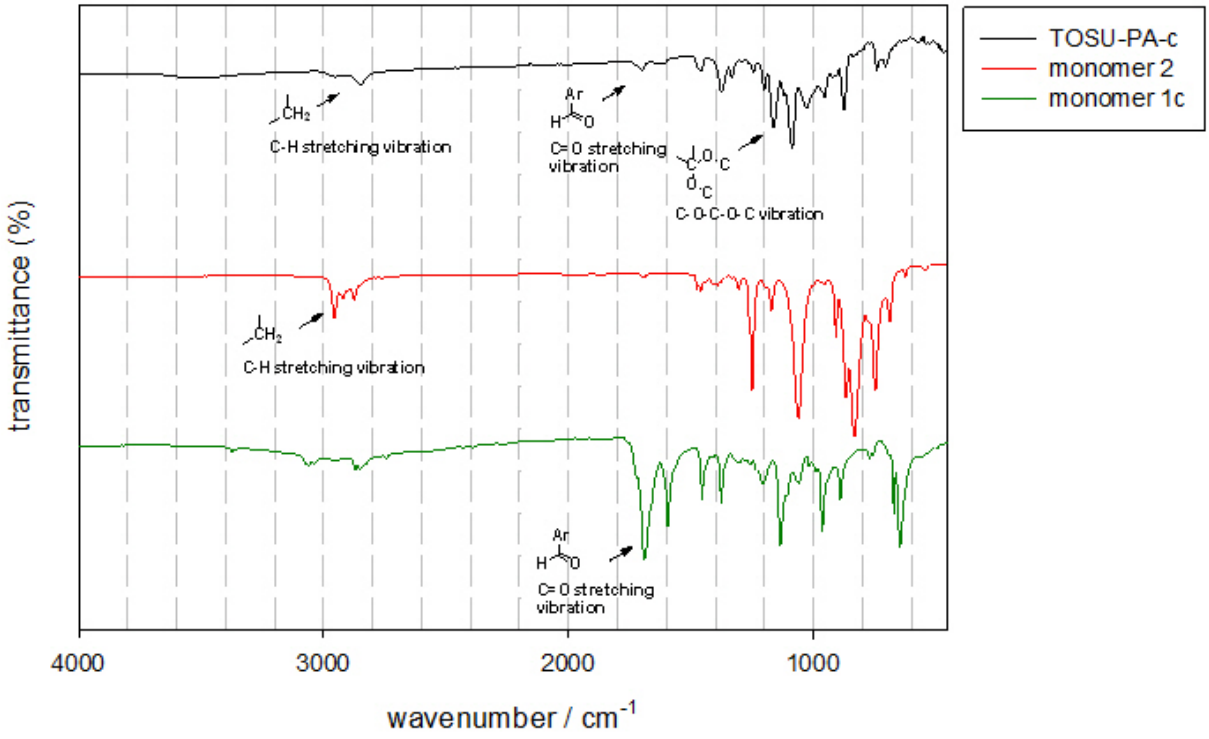


Fig. S8. FT-IR spectra of **TOSU-PA-c**, trimethylsilyl ether of pentaerythritol (**2**) and trialdehyde **1c.**

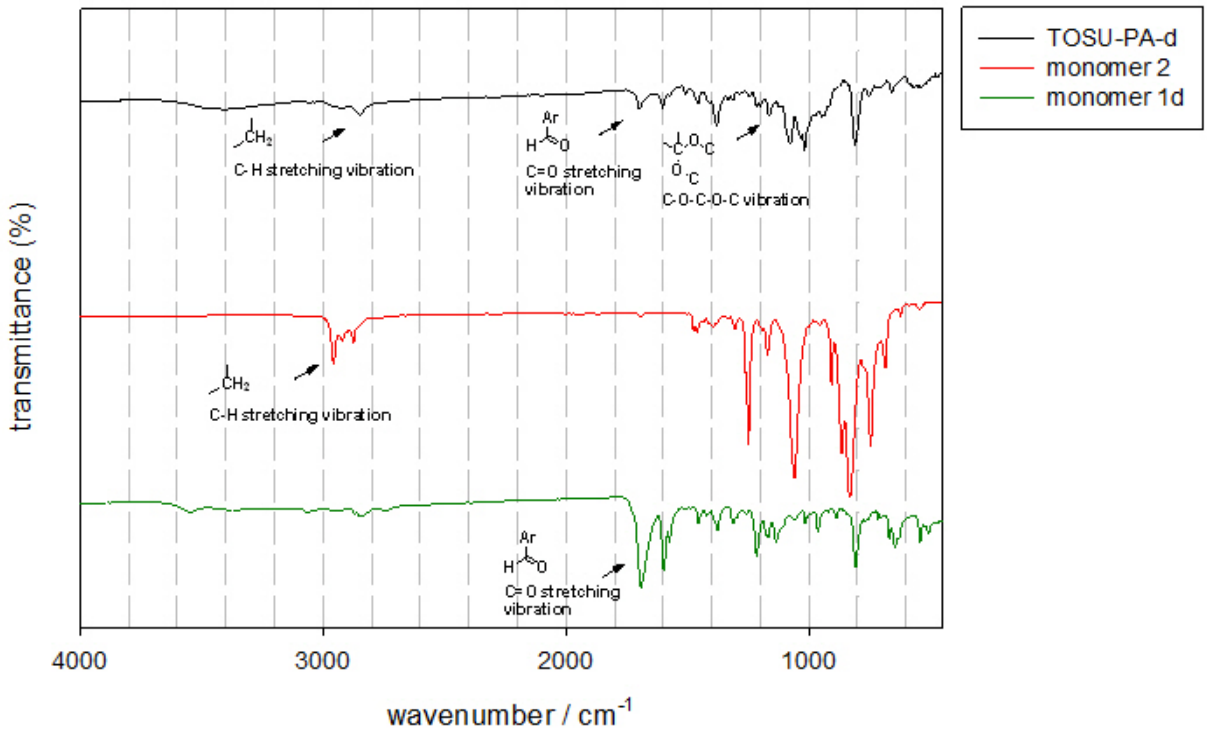


Fig. S9. FT-IR spectra of **TOSU-PA-d**, trimethylsilyl ether of pentaerythritol (**2**) and tetraaldehyde **1d.**

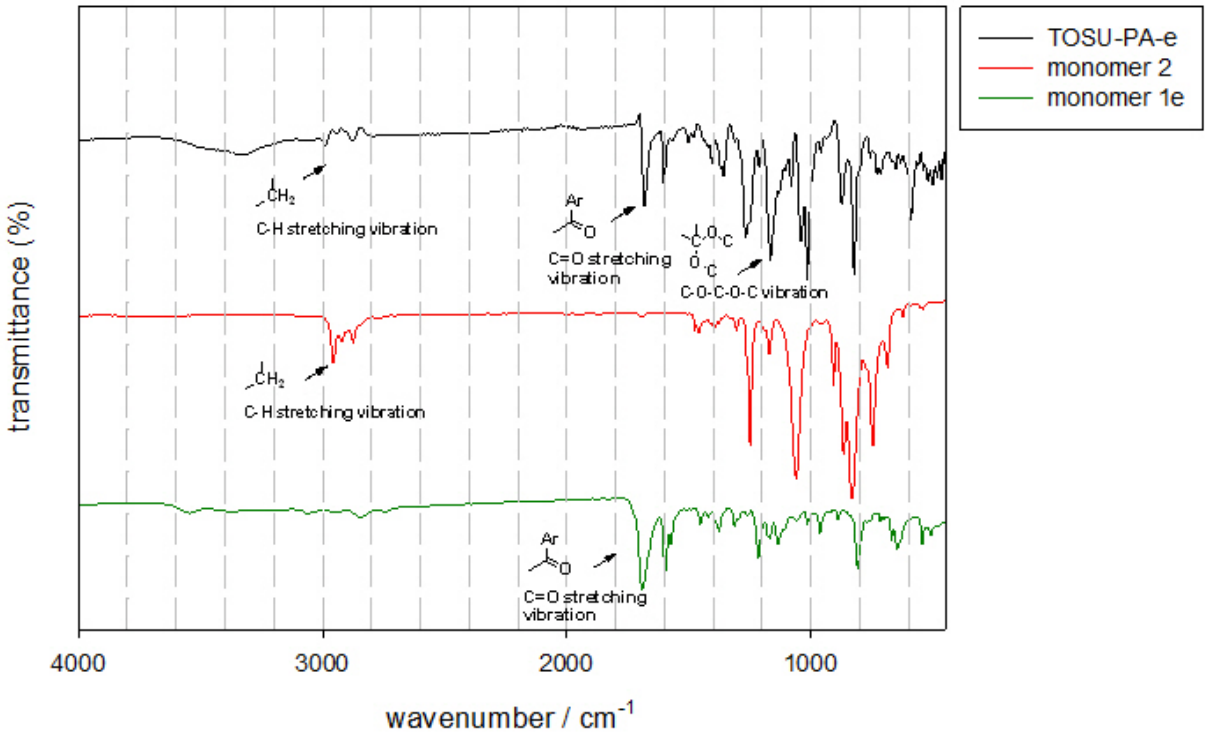
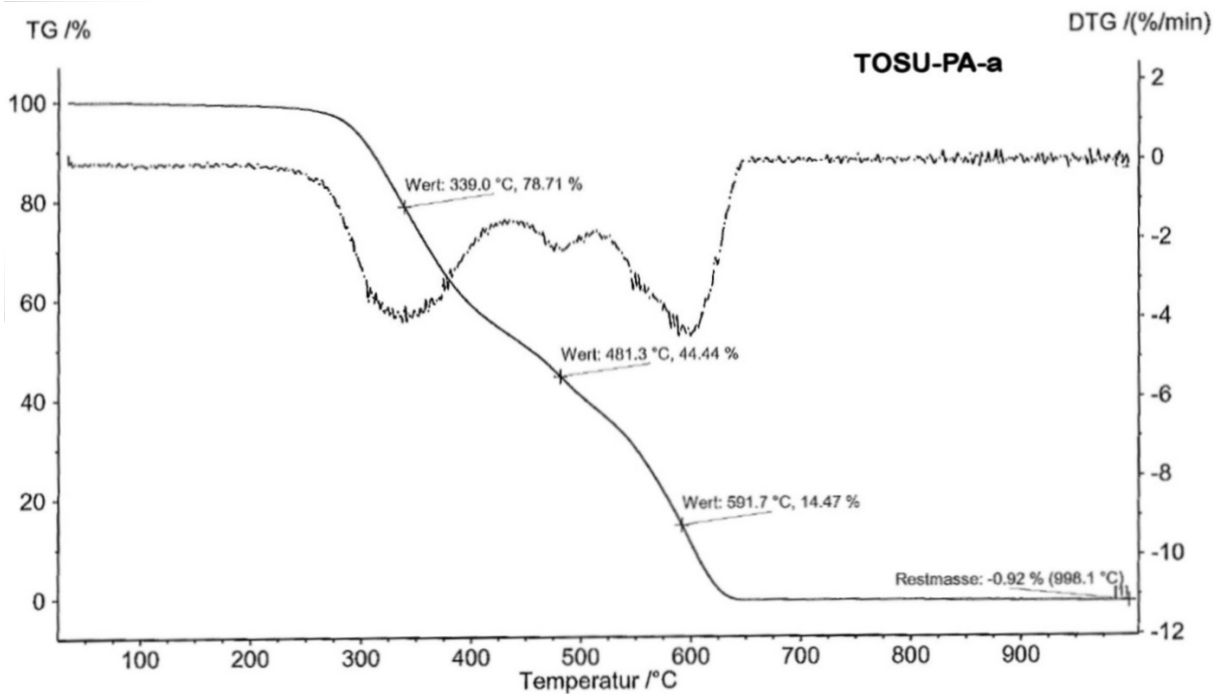
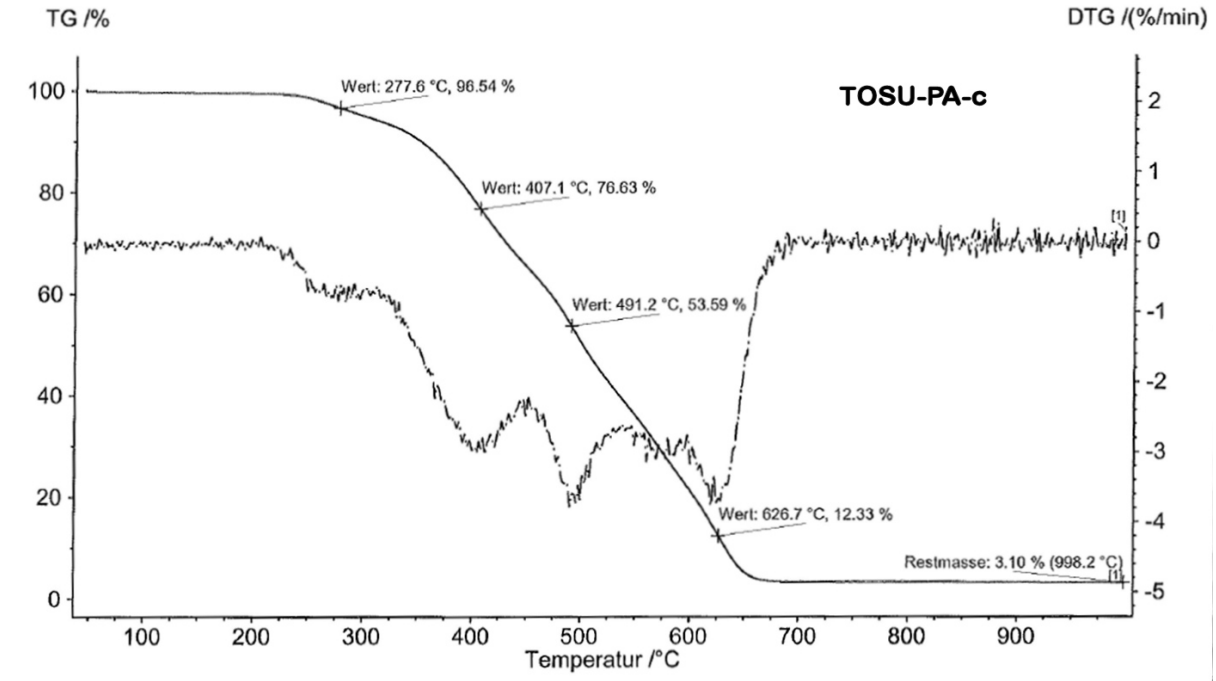


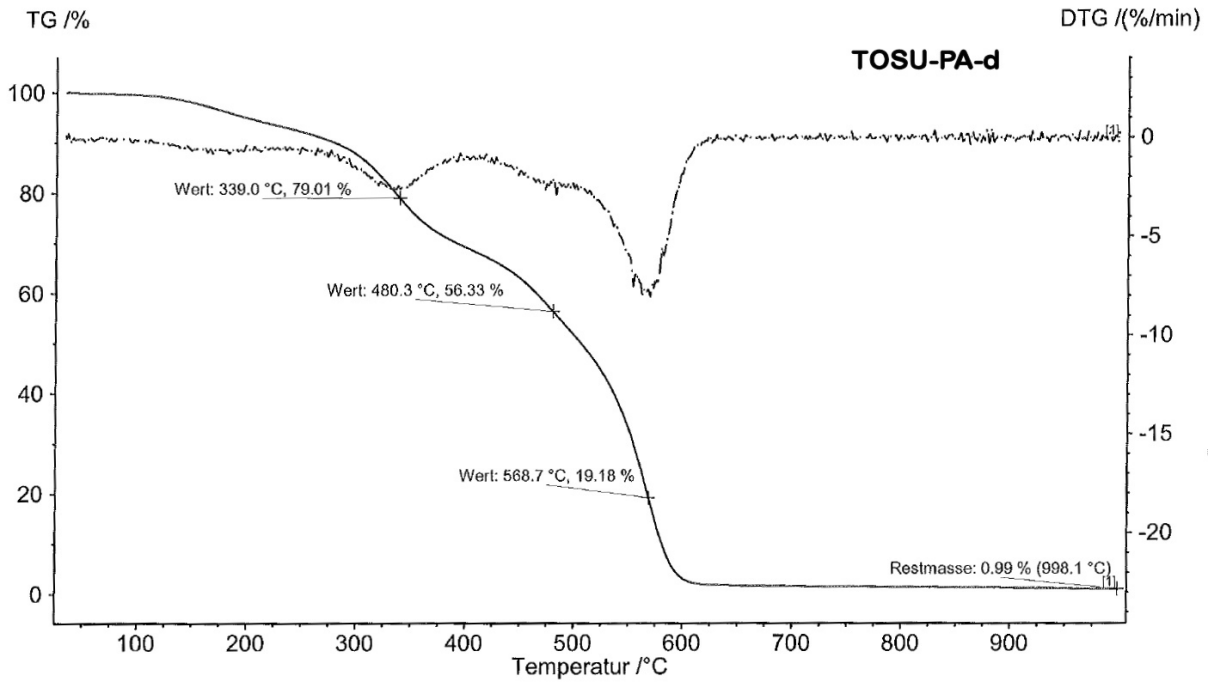
Fig. S10. FT-IR spectra of **TOSU-PA-e**, trimethylsilyl ether of pentaerythritol (**2**) and tetra(4-acetylphenyl)methane (**1e).**

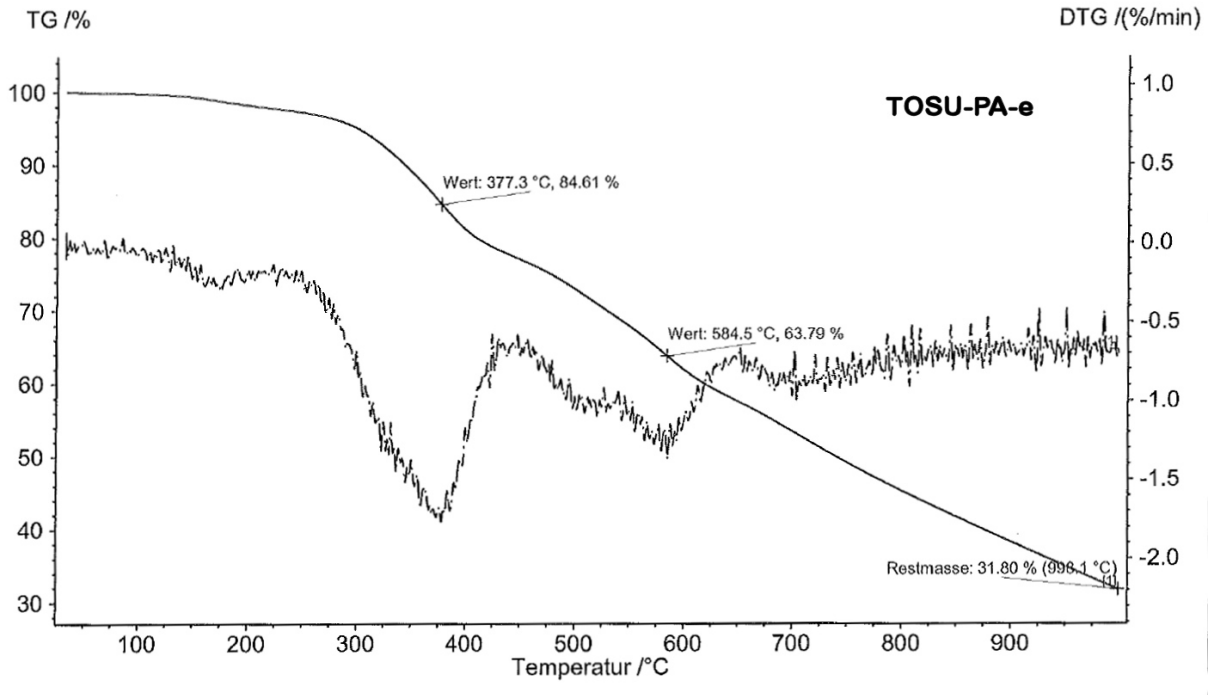
TGAs of TOSU-PA-a-e

Note: TOSU-PA-a-d were measured under air , TOSU-PA-e was measured under N_2






TG /%
DTG /(%/min)
TOSU-PA-d
Wert: 339.0 °C, 79.01 %
Wert: 480.3 °C, 56.33 %
Wert: 568.7 °C, 19.18 %
Restmasse: 0.99 % (998.1 °C)
Temperatur /°C


TG /%
DTG /(%/min)
TOSU-PA-e
Wert: 377.3 °C, 84.61 %
Wert: 584.5 °C, 63.79 %
Restmasse: 31.80 % (998.1 °C)
Temperatur /°C