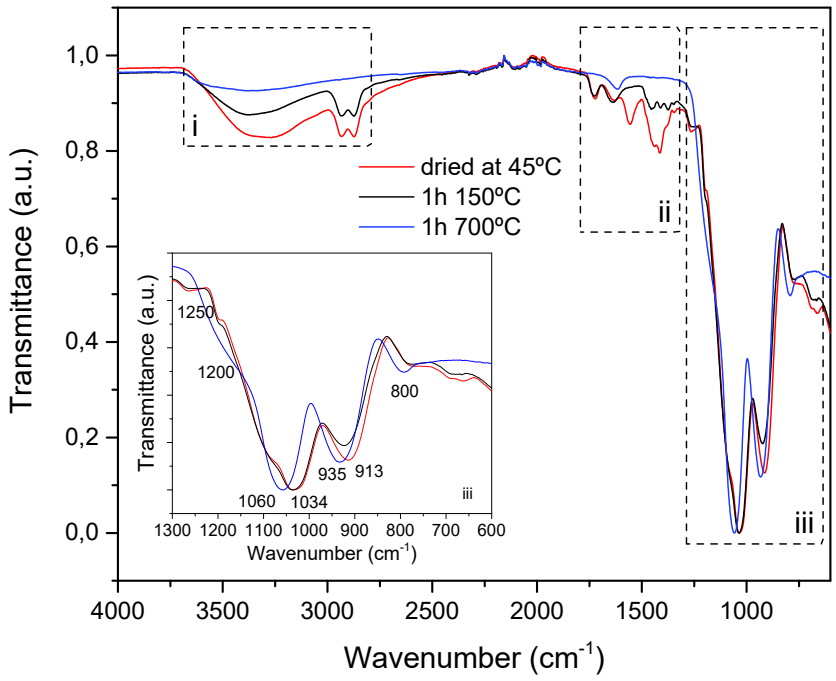
Electronic Supplementary Material (ESI) for RSC Advances.

Supplementary File

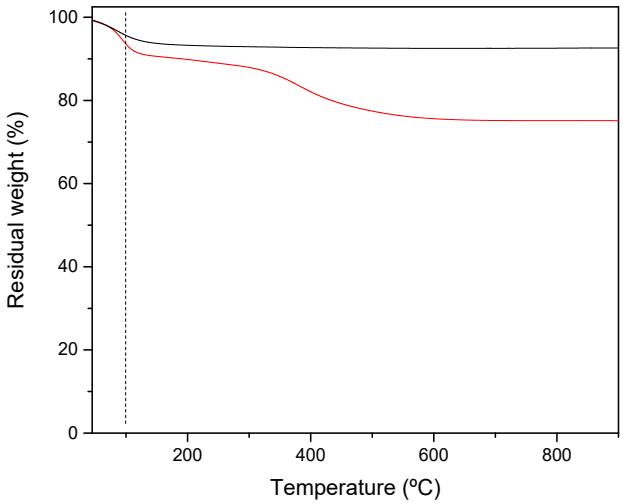
Multicomponent oxide microspheres with designed macroporosity (MICROSCAFS®): a customized platform for chemicals immobilization

Mário Vale[1], Sofia Orišková[1], António Mariquito[1], Luís Reis[2], Moisés Pinto[1], and Ana C. Marques[1,*]

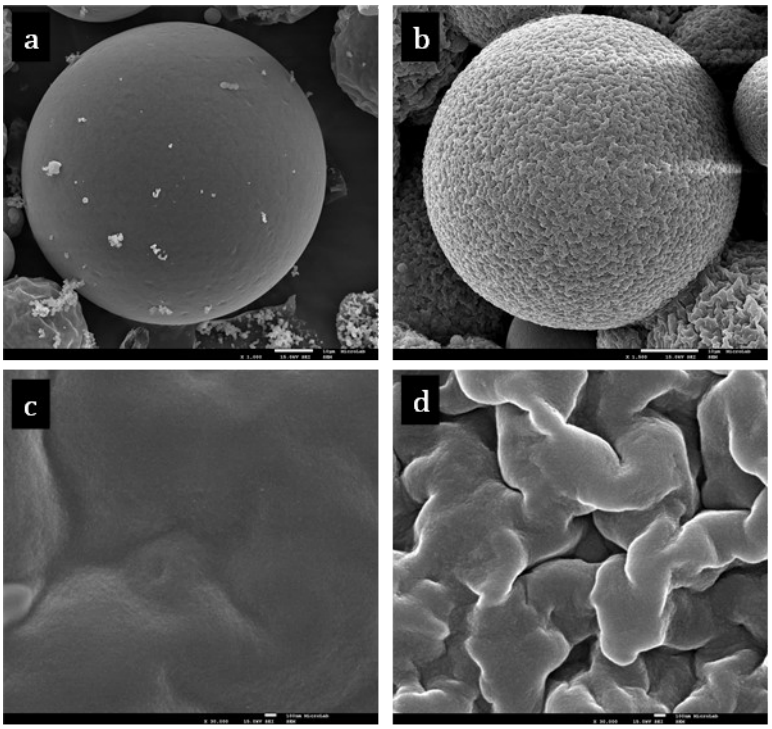
[1] CERENA, DEQ, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, 1049-001, Lisboa, Portugal
[2] IDMEC, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, 1049-001, Lisboa, Portugal
*ana.marques@tecnico.ulisboa.pt



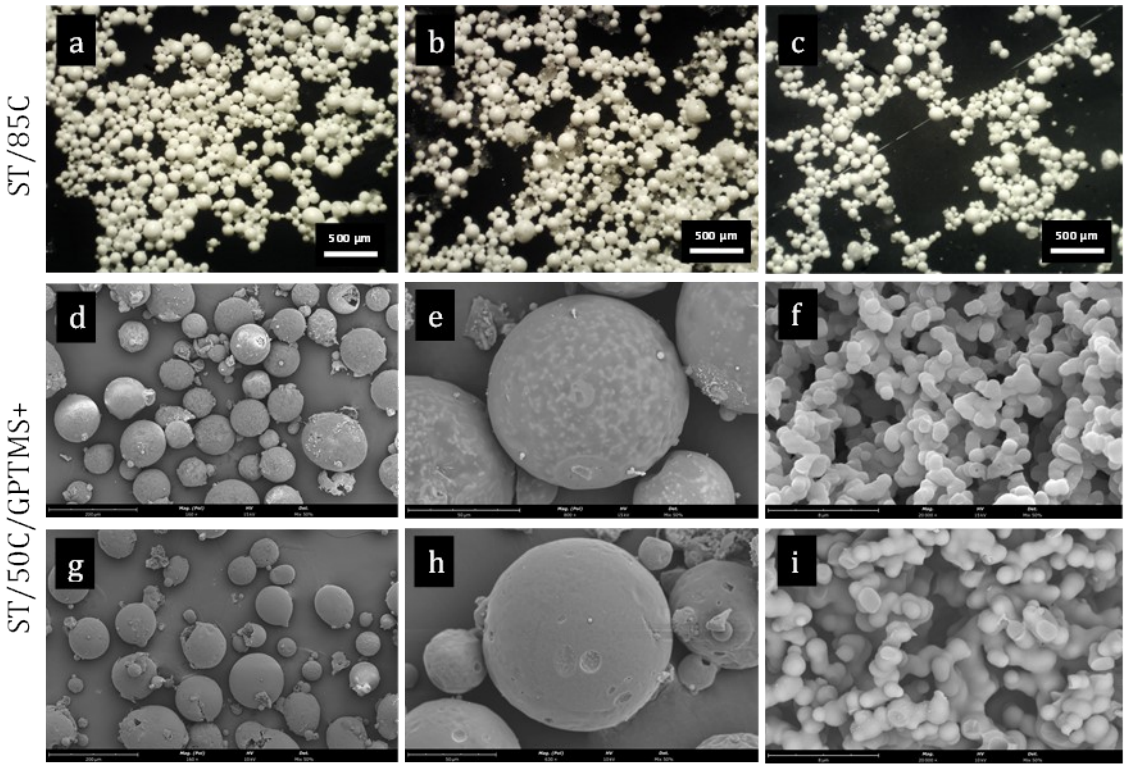
Supplementary Figure S1. ATR-FTIR spectra of MICROSCAFS® ST/50C, dried at 40°C, with heat treatment for 1h at 150°C and 700°C.



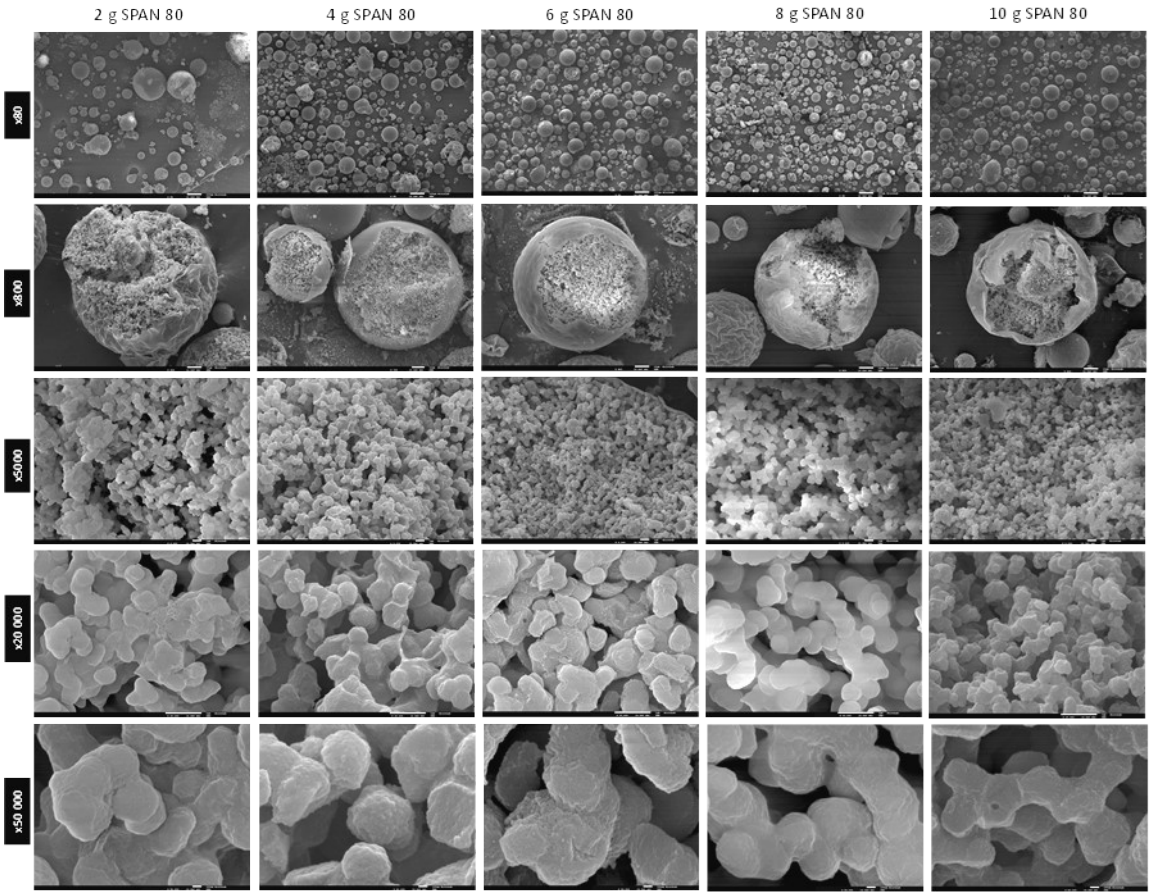
Supplementary Figure S2. Thermogram of MICROSCAFS® ST/50C dried at 45 ºC (red) and heat treated for 1 hour at 700 ºC (black).



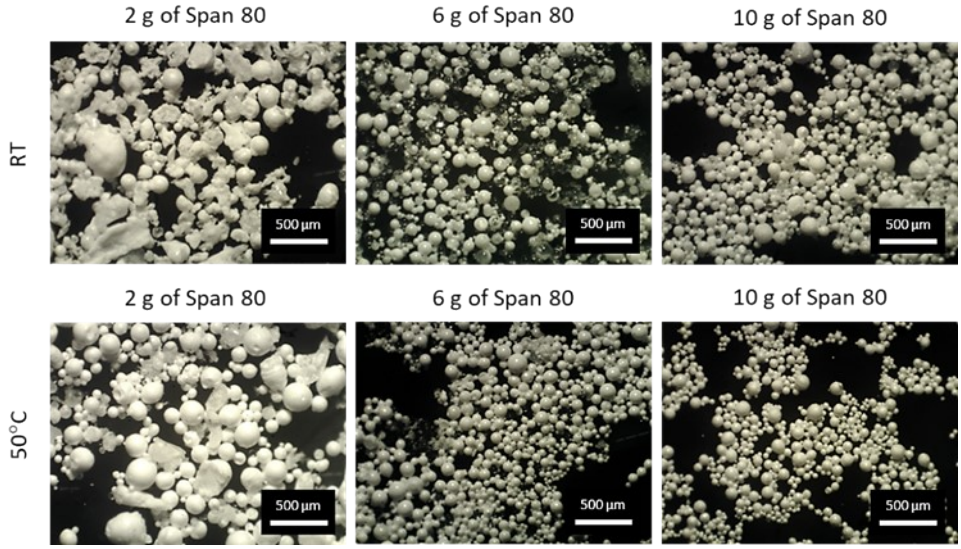
Supplementary Figure S3. SEM images of MICROSCAFS® ST/85C: without LiOH treatment (a) scale bar: 10 µm, (c) scale bar: 100 nm; with LiOH treatment (b) scale bar: 10 µm, (d) scale bar: 100 nm.



Supplementary Figure S4. Evidence for synthesis reproducibility: (a, b, c) Optical microscopy images of three different batches of the same synthesis of MICROSCAFS® ST/85C; (d, g) SEM images (scale bar: 200 µm) of two different batches of the synthesis of MICROSCAFS® ST/50C/GPTMS+; (e,h) same as (d, g) with scale bar = 50 µm; (f, i) same as (d, g) with scale bar = 5 µm. Note: d, e and f were acquired with a higher voltage (15 kV) than g, h and i (10 kV).



Supplementary Figure S5. SEM images of the MICROSCAFS® ST/85C, at different magnifications, obtained using different quantities of surfactant SPAN 80: 2, 4, 6, 8 and 10 g. Scale bar (from top to bottom): 100 µm, 10 µm, 1 µm, 1 µm, 100 nm.



Supplementary Figure S6. Optical microscopy images of the MICROSCAFS® ST/RT and ST/50C obtained using different quantities of surfactant Span® 80: 2, 6 and 10 g.

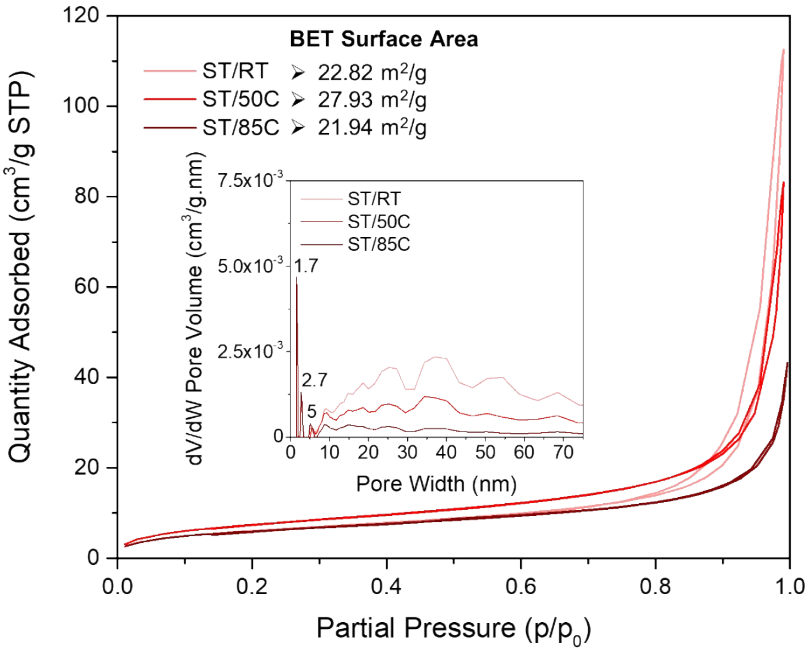
Supplementary Table S7. Particle size distribution data.

Sample acronym	D (0.1), µm	D (0.5), µm	D (0.9), µm	Span
ST/RT	40	89	169	1.45
ST/50C	44	102	220	1.72
ST/85C	47	96	172	1.31
ST/50C/GPTMS-	32	75	178	1.94
ST/50C/GPTMS+	66	102	190	1.11
ST/50C/AA+	64	134	242	1.33
ST/50C/W+	48	112	277	2.04
STH/50C/GPTMS+	44	103	253	2.03

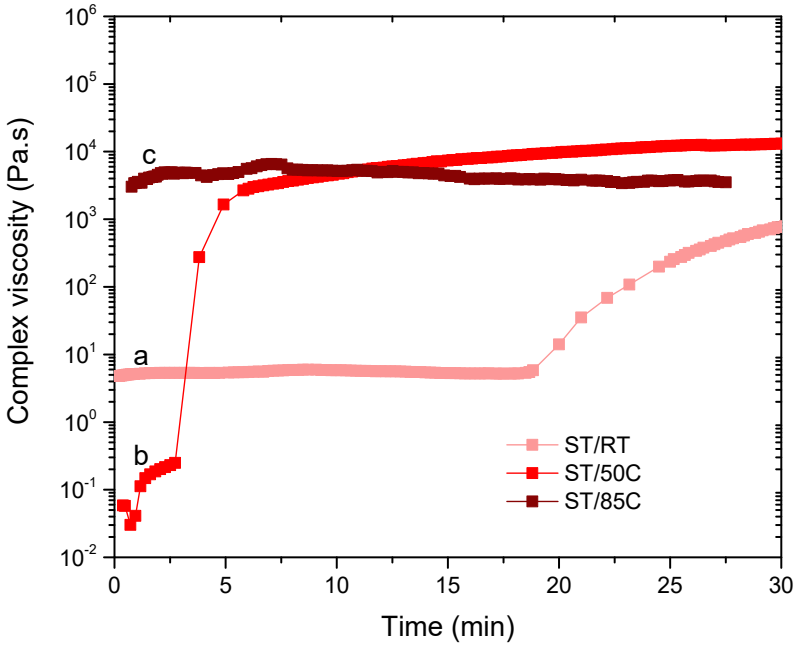
Supplementary Table S8. Average hydrolyzate viscosity and MICROSCAFS®' synthesis pH.

Sample acronym	Average hydrolyzate viscosity (cP)	Average synthesis pH
ST/RT	16.2 ± 0.8*	9.23 ± 0.02
ST/50C	16.2 ± 0.8	8.51 ± 0.04
ST/85C	16.2 ± 0.8*	6.67 ± 0.08
ST/50C/GPTMS-	25.0 ± 1.2	9.22 ± 0.01
ST/50C/GPTMS+	14.8 ± 0.4	8.97 ± 0.01
ST/50C/AA+	14.8 ± 0.6	7.61 ± 0.01
ST/50C/W+	16.2 ± 0.8*	8.62 ± 0.02
STH/50C/GPTMS+	45.3 ± 0.3	9.05 ± 0.01

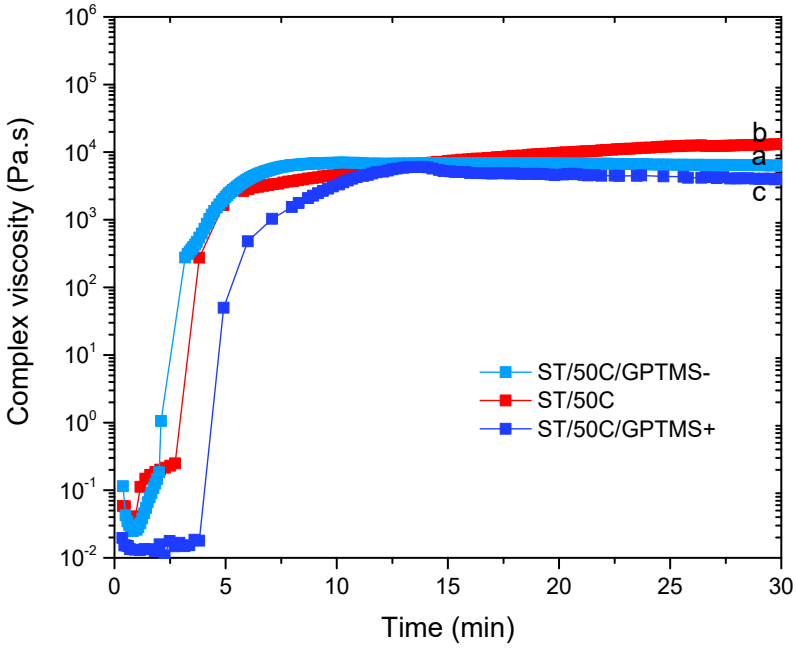
*same hydrolyzate as for ST/50C



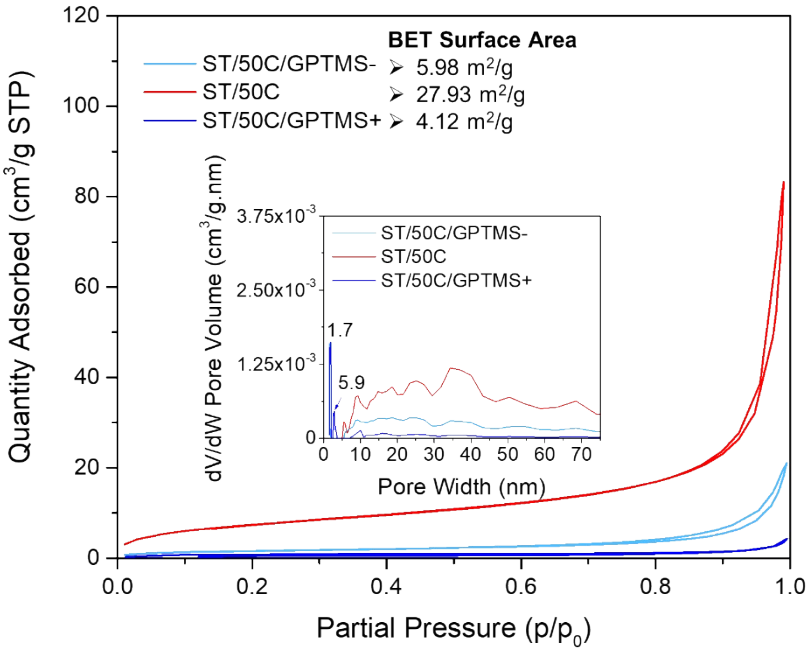
Supplementary Figure S9. BET adsorption isotherm and DFT pore size distribution (inset) of MICROSCAFS® synthesized at room temperature (ST/RT), 50 °C (ST/50C), and 85 °C (ST/85C).



Supplementary Figure S10. Complex viscosity evolution over time for the hydrolyzates at (a) room temperature, (b) 50 °C, and (c) 85 °C.



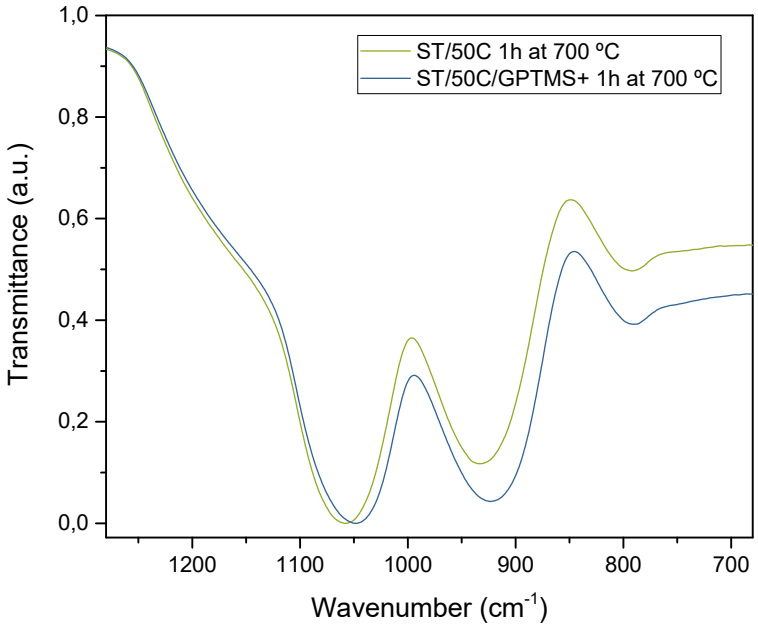
Supplementary Figure S11. Complex viscosity evolution over time of the hydrolyzates with (a) 0.65, (b) 0.86, and (c) 1.08 GPTMS/TEOS molar ratio.



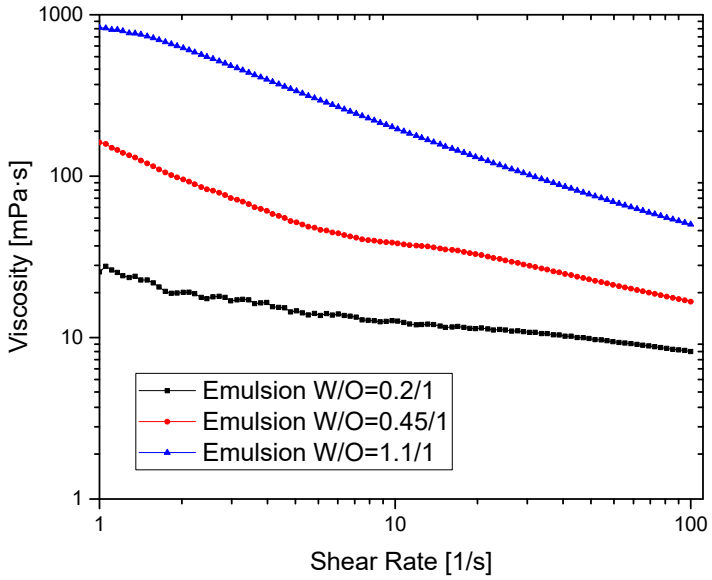
Supplementary Figure S12. BET adsorption isotherm and DFT pore size distribution (inset) of MICROSCAFS® synthesized using 0.65 (ST/50C/GPTMS-), 0.86 (ST/50C), and 1.08 (ST/50C/GPTMS+), GPTMS/TEOS molar ratio.

Supplementary Table S13. EDS relative atomic concentrations of the dried MICROSCAFS®.

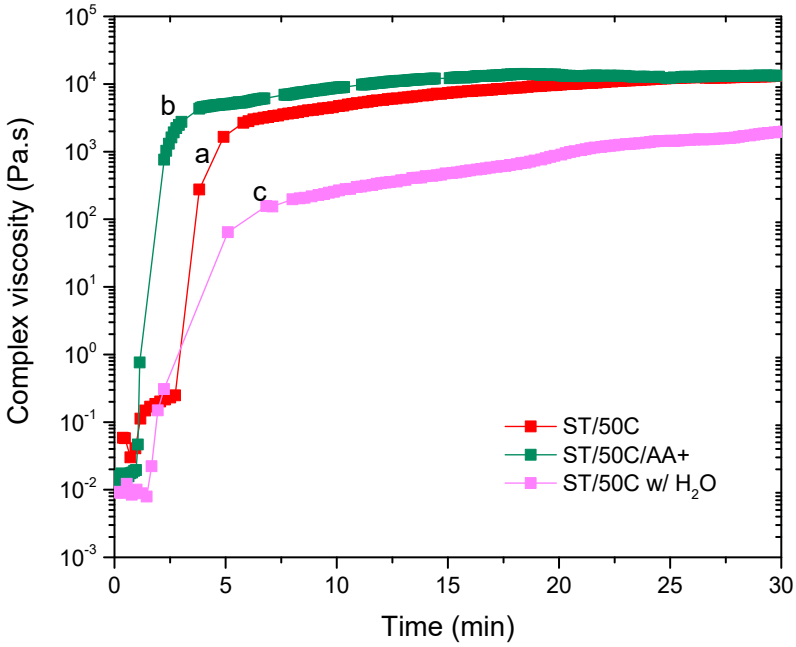
Sample acronym	Relative atomic concentration %		
	Si	Ti	Hf
ST/50C/GPTMS-	84.3	15.7	0
ST/50C	82.8	17.3	0
ST/50C/GPTMS+	79.7	20.3	0
STH/50C/GPTMS+	80.3	12.1	7.6



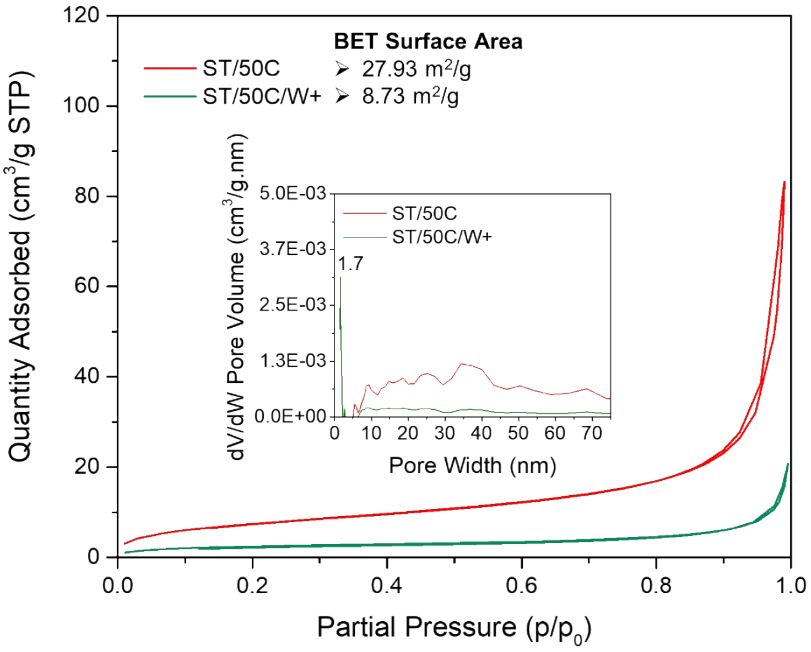
Supplementary Figure S14. ATR-FTIR spectra of MICROSCAFS® ST/50C and ST/50C/GPTMS+, with heat treatment for 1h at 700°C.



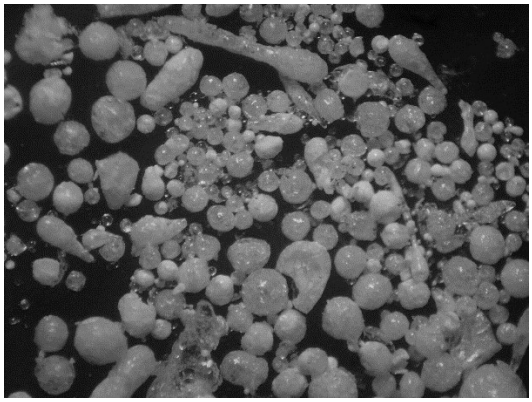
Supplementary Figure S15. Flow test curves of three groups of emulsion each belonging to different W/O ratios.



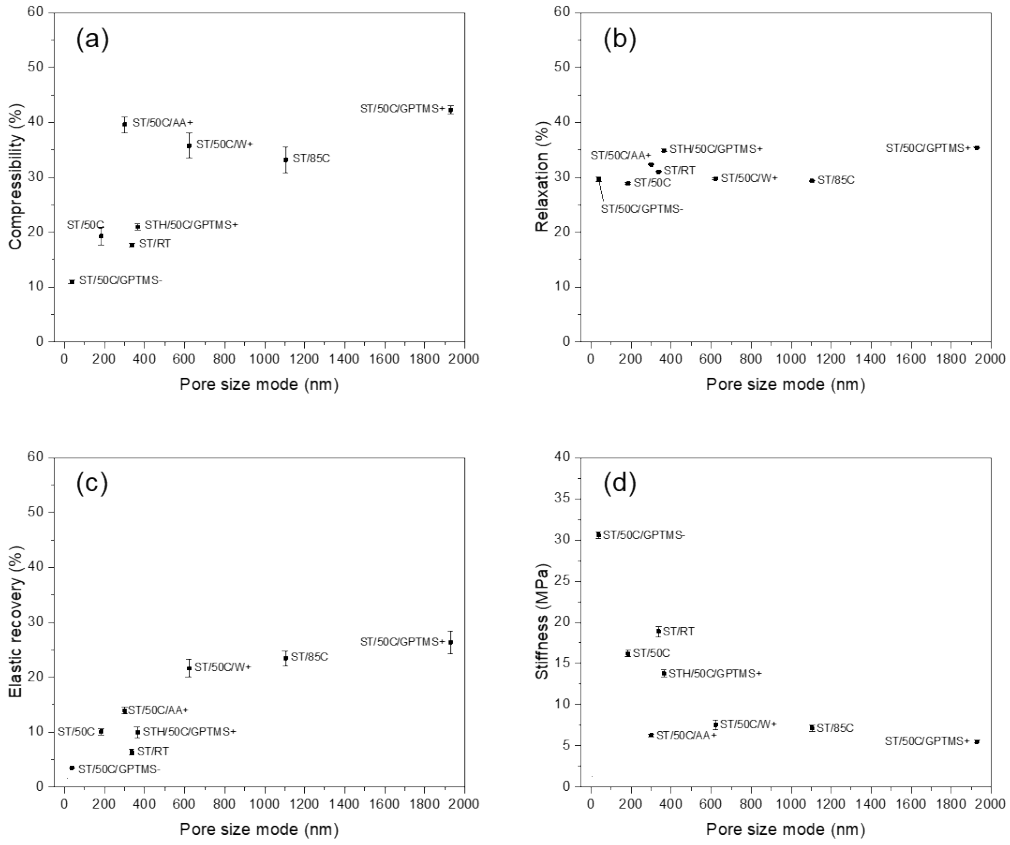
Supplementary Figure S16. Complex viscosity evolution over time of the hydrolyzates with (a) 1:4 TiPOT:CH3COOH molar ratio (sample ST/50C), (b) 1:6 TiPOT:CH3COOH molar ratio (sample ST/50C/AA+), and (c) 1:4 TiPOT:CH3COOH molar ratio, diluted with an equivalent amount of the excess of emulsion water.



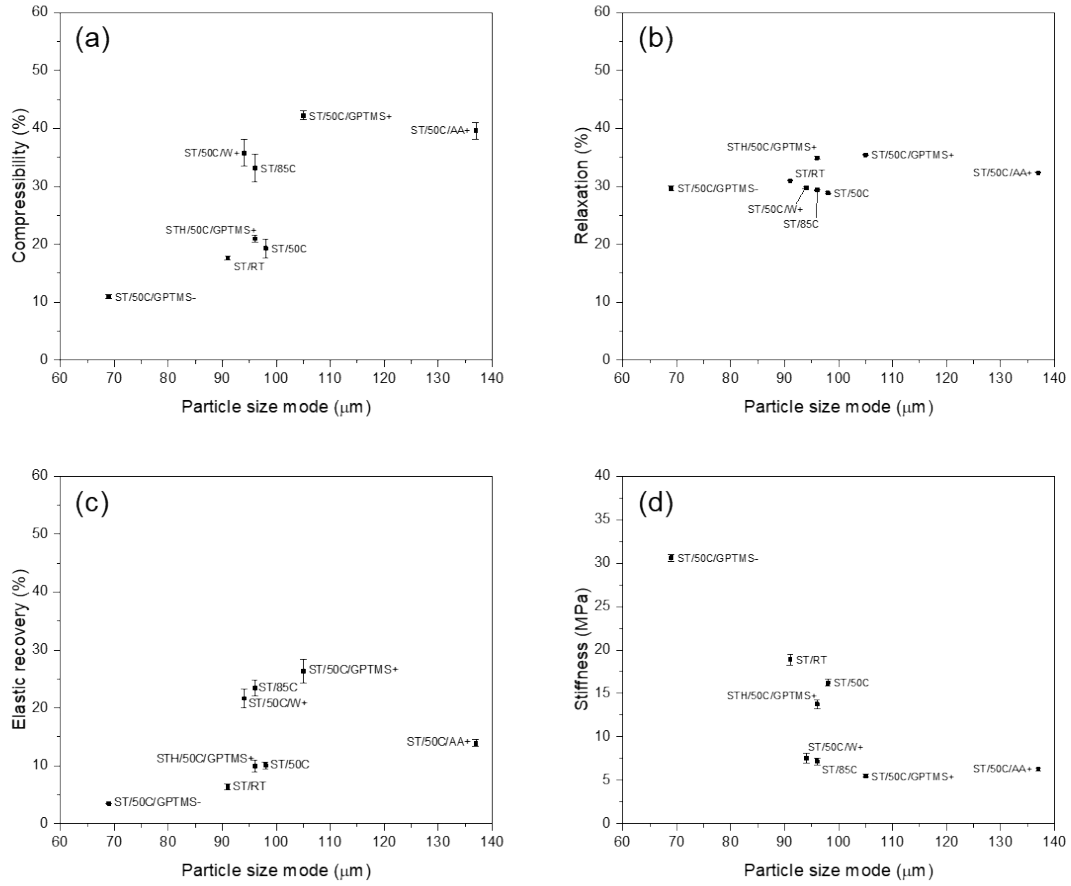
Supplementary Figure S17. BET adsorption isotherm and DFT pore size distribution (inset) of MICROSCAFS® synthesized at 0.45:1 (ST/50C) and 0.68:1 (ST/50C/W+) W/O mass ratios.



Supplementary Figure S18. Optical microscopy photograph of STH MICROSCAFS® made using 13.7 g of hafnium dichloride oxide octahydrate salt.



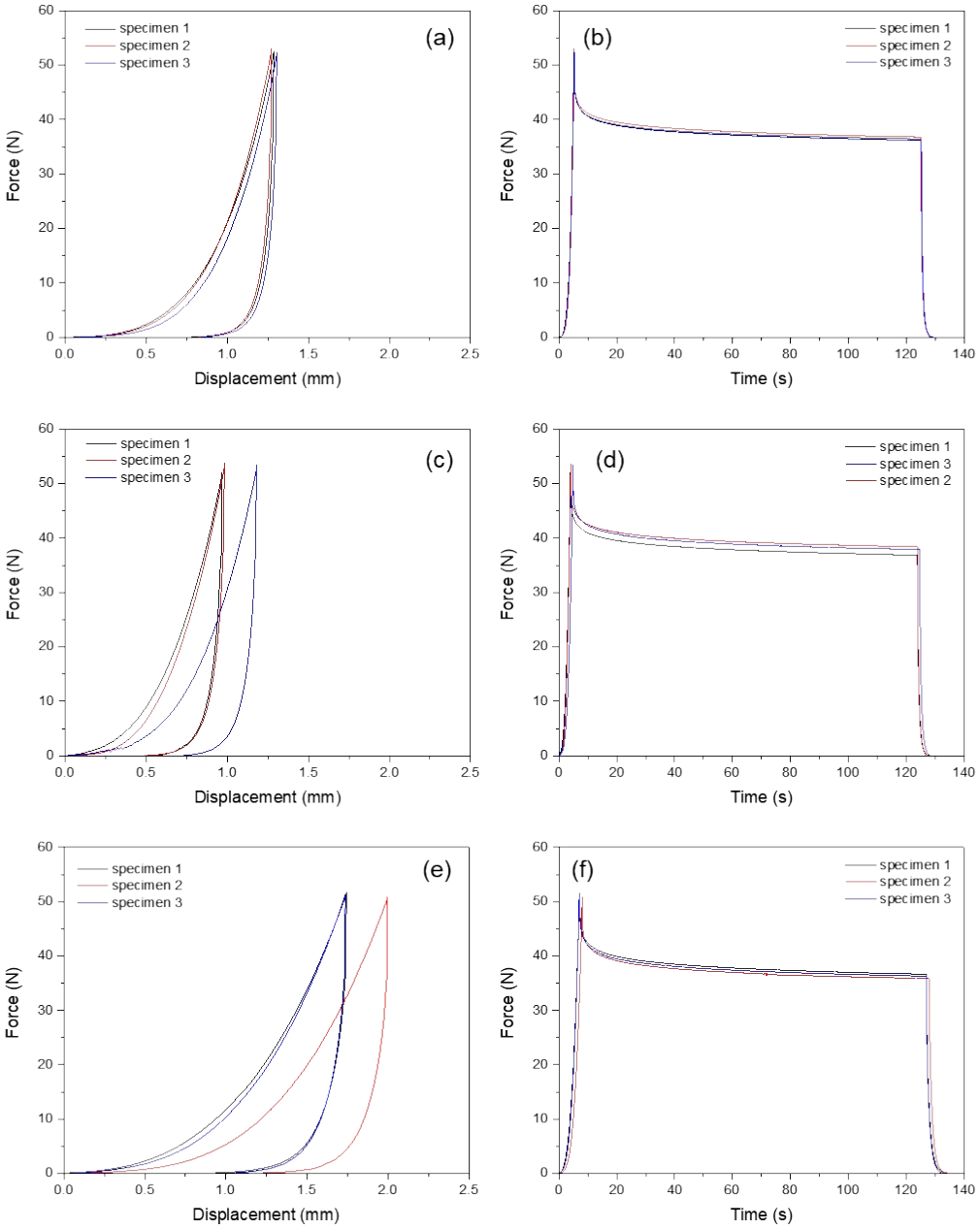
Supplementary Figure S19. Effect of MICROSCAFS®' pore size mode in (a) compressibility, (b) relaxation, (c) elastic recovery, and (d) stiffness.



Supplementary Figure S20. Effect of MICROSCAFS®' particle size mode in (a) compressibility, (b) relaxation, (c) elastic recovery, and (d) stiffness.

Supplementary Table S21. Mechanical parameteres of the dried MICROSCAFS®.

Sample acronym	Compressibility (%)	Relaxation (%)	Stiffness (MPa)	Elastic recovery (%)
ST/RT	17.6 ± 0.4	30.9 ± 0.1	18.9 ± 0.6	6.3 ± 0.5
ST/50C	19.3 ± 1.6	28.9 ± 0.2	16.2 ± 0.4	10.1 ± 0.6
ST/85C	33.2 ± 2.4	29.4 ± 0.1	7.2 ± 0.4	23.5 ± 1.3
ST/50C/GPTMS-	11.0 ± 0.4	29.6 ± 0.2	30.6 ± 0.4	3.5 ± 0.1
ST/50C/GPTMS+	42.2 ± 0.8	35.4 ± 0.1	5.5 ± 0.2	26.4 ± 2.0
ST/50C/AA+	39.6 ± 1.4	32.4 ± 0.1	6.3 ± 0.2	13.9 ± 0.6
ST/50C/W+	35.7 ± 2.3	29.8 ± 0.2	7.6 ± 0.5	21.6 ± 1.6
STH/50C/GPTMS+	20.9 ± 0.6	34.9 ± 0.3	13.8 ± 0.5	10.0 ± 1.0



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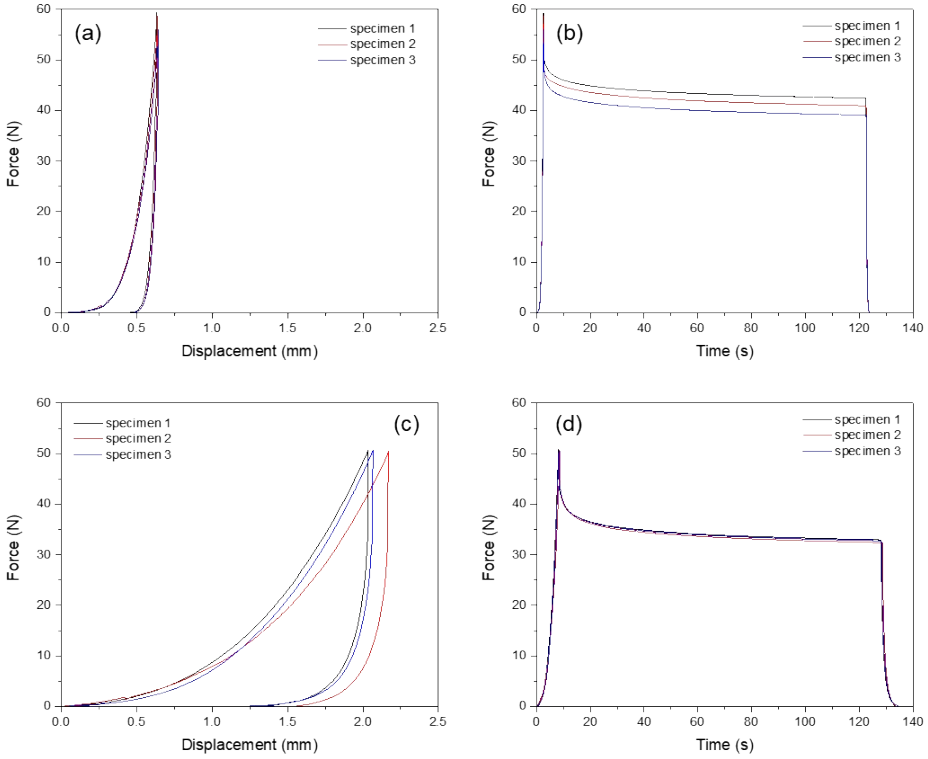
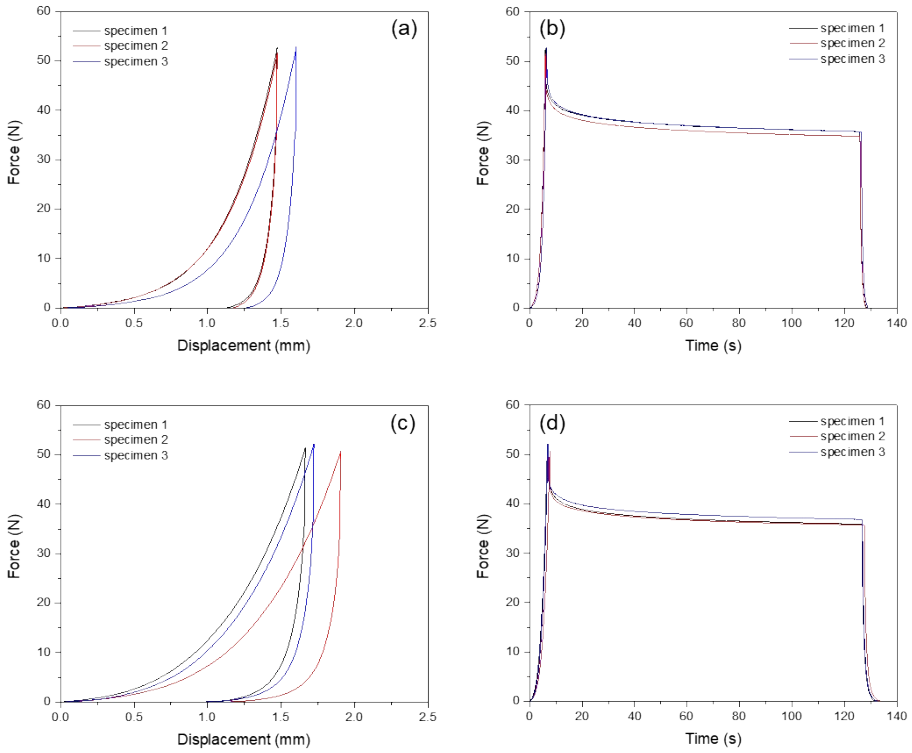
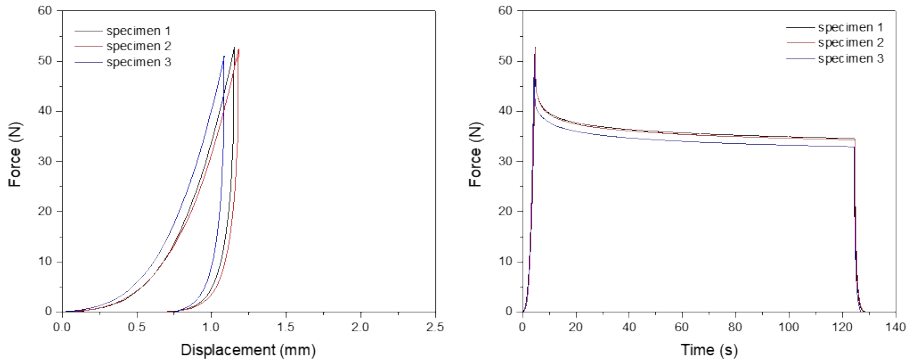
Supplementary Figure S22. Raw data from compression and relaxation tests of (a,b) ST/RT, (c, d) ST/50C, and (e, f) ST/85C dried MICROSCAFS®.



Supplementary Figure S23. Raw data from compression and relaxation tests of (a, b) ST/50C/GPTMS-, (c, d) ST/50C/GPTMS+ dried MICROSCAFS®.



Supplementary Figure S24. Raw data from compression and relaxation tests of (a,b) ST/50C/AA+, (c, d) ST/50C/W+ dried MICROSCAFS®.



Supplementary Figure S25. Raw data from compression and relaxation tests of STH/50C/GPTMS+ dried MICROSCAFS®.