Hydrophobic Organic Aerogels and Xerogels Based on a Phloroglucinol Ether

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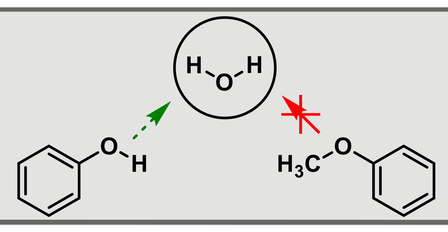
Motivation

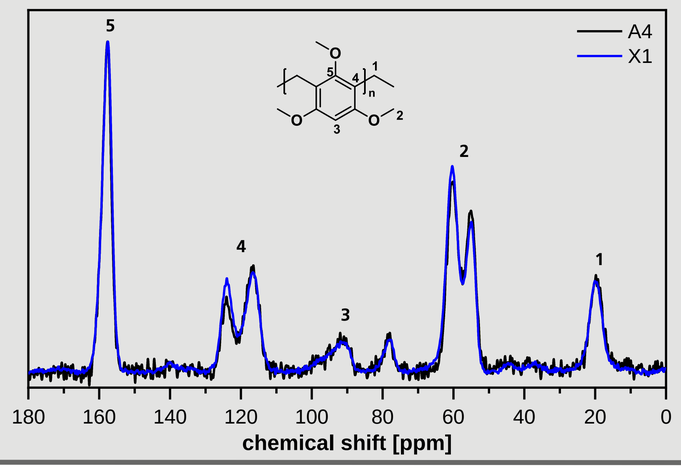
Aerogels are open-porous nanostructured solids obtained by a sol-gel process followed by drying of the wet gel. The most prominent example of an organic aerogel is the resorcinol-formaldehyde (RF) aerogel.[1] The phenolic nature of RF renders the material hydrophilic, a factor that partly limits its commercial use.[2] In order to address this issue, we report on the condensation of the phenolic ether 1,3,5-trimethoxybenzene (TMB) with formaldehyde (F) yielding inherently hydrophobic aerogels and xerogels.



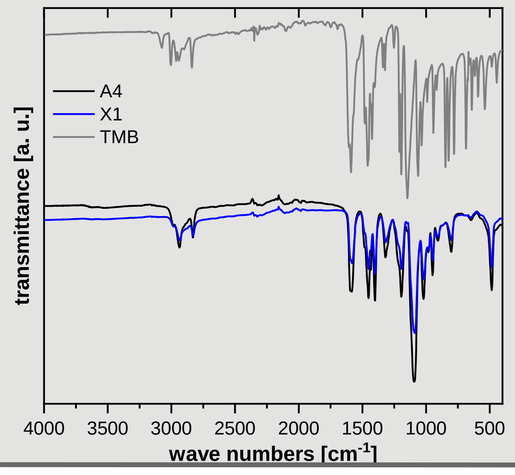
Experimental

Sample	Solvent	c(TMB) [mol/L]	HCl [mol-%]	Temp. [°C]	Time	Drying method
A1	Dioxane	0.60	12.5	60	7 d	sc-CO_2
A2	DMSO	0.60	12.5	60	7 d	sc-CO_2
A3	DMSO	0.60	12.5	80	7 d	sc-CO_2
A4	DMSO	0.60	1.5	80	7 d	sc-CO_2
A5	DMSO	0.30	12.5	80	7 d	sc-CO_2
X1	DMSO	0.60	50.0	100 + 80	1 h + 1 d	oven (60°C)
X2	DMSO	0.60	25.0	100 + 80	1 h + 1 d	oven (60°C)

Solid state ^{13}C-NMR Spectroscopy

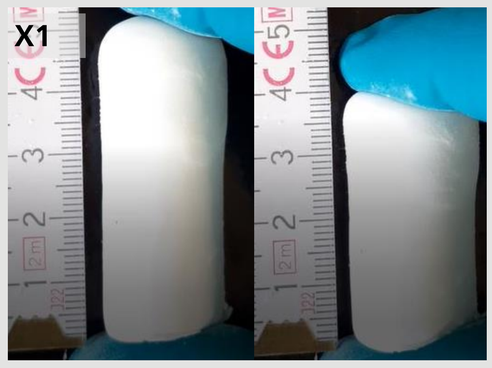


ATR-IR Spectroscopy



Elemental Analysis

Sample	C [%]
A1	65.91 ± 0.12
A2	67.00 ± 0.38
A3	66.59 ± 0.30
A4	67.52 ± 0.00
A5	66.32 ± 0.21
X1	67.51 ± 0.13
X2	68.54 ± 0.40

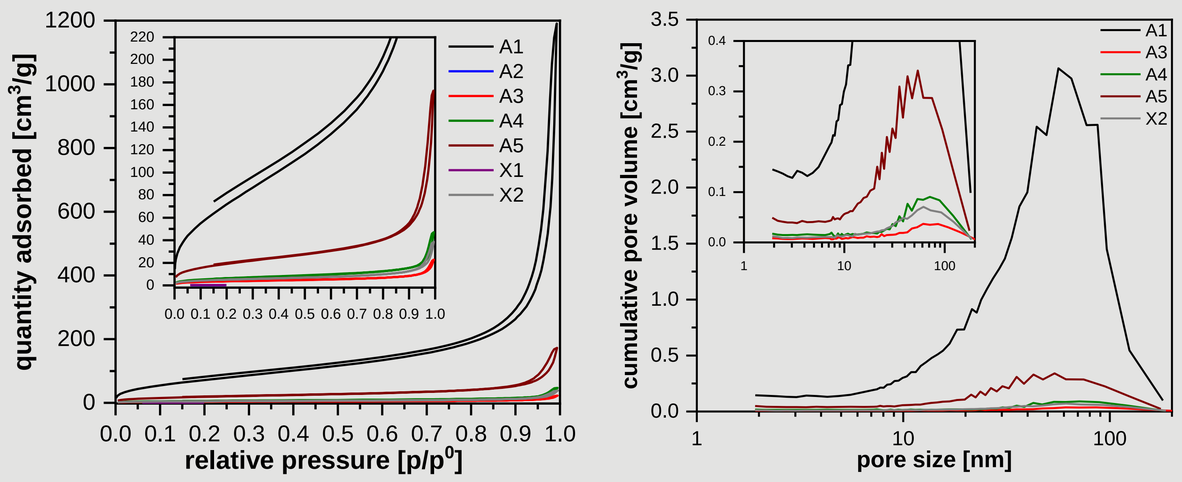


- ***Solvent influences cross-linking***
- ***Flexibility origins from microstructure***

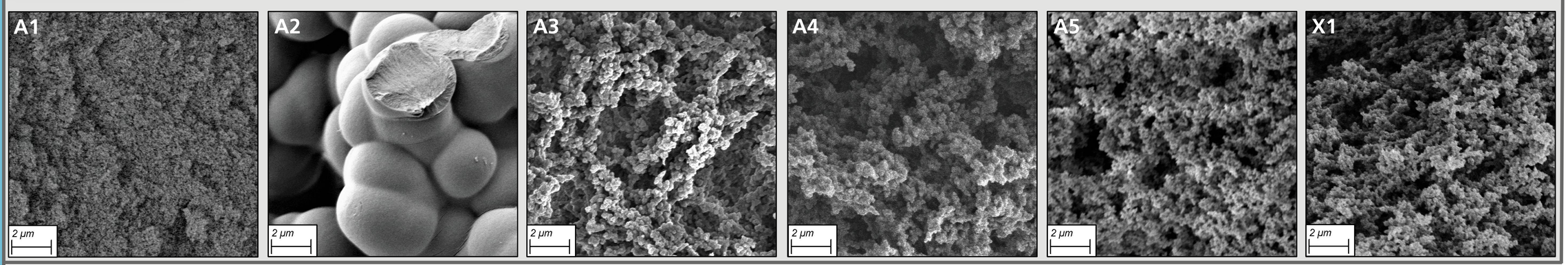
Results

Density-, Surface- and Pore Characteristics

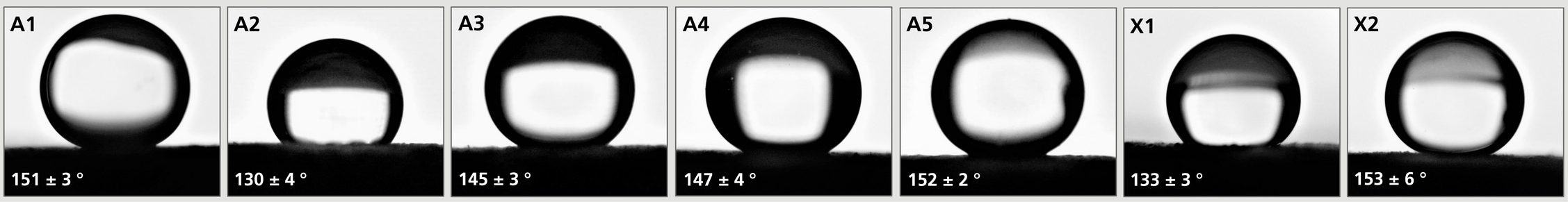
Sample	Radial Shrinkage [%]	Envelope Density [g/cm³]	Skeletal Density [g/cm³]	Porosity [%]	BET Surface Area [m²/g]
A1	29	0.30 ± 0.00	1.28 ± 0.01	77	260.0
A2	14	0.14 ± 0.00	1.32 ± 0.01	89	0.8
A3	9	0.14 ± 0.00	1.30 ± 0.02	89	13.1
A4	15	0.16 ± 0.00	1.29 ± 0.01	88	23.4
A5	18	0.08 ± 0.00	1.30 ± 0.02	94	70.5
X1	13	0.14 ± 0.00	1.33 ± 0.01	89	1.7
X2	20	0.16 ± 0.00	1.32 ± 0.01	88	19.8



Microstructure



Contact Angles



Conclusion

In summary, we have established an acid-catalyzed synthesis of 1,3,5-trimethoxybenzene formaldehyde (TMBF) aerogels and xerogels in organic solvents. The morphologies, densities, porosities, surface characteristics and contact angles can be tuned by variation of the synthesis temperature, solid- and catalyst concentration and the solvent. Furthermore, the chemical structure of the aerogels was investigated. The resulting materials can be considered as the (super-)hydrophobic analog to the well-studied resorcinol-formaldehyde (RF) aerogels. TMBF aerogels and xerogels are therefore a promising candidate to address the disadvantages caused by the intrinsic hydrophilicity of RF aerogels.

[1]: a) R. W. Pekala, J. Mater. Sci. **1989**, *24*, 3221-3227. b) S. Mulik, C. Sotiriou-Leventis, N. Leventis, Chem. Mater. **2007**, *19*, 6138-6144. c) A. M. ElKhatat, S. A. Al-Muhtaseb, Adv. Mater. **2011**, *23*, 2887-2903.
[2]: A. V. Rao, G. M. Pajonk, D. Y. Nadargi, M. M. Koebel, in Aerogels Handbook (Eds.: M. A. Aegerter, N. Leventis, M. M. Koebel), Springer New York, New York, NY, **2011**, pp. 79-101.

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