

Supplementary Information

Synthesis of inorganic aerogels via rapid gelation using chloride precursors

Helmut Schäfer*,† Barbara Milow and Lorenz Ratke*,‡

† University of Osnabrueck, Institute of Chemistry of New Materials, Physical Chemistry, Barbarastraße 7, 49069 Osnabrueck, Germany

‡ German Aerospace Center, Institute of Materials Physics in Space, Linder Höhe, 51147 Cologne, Germany

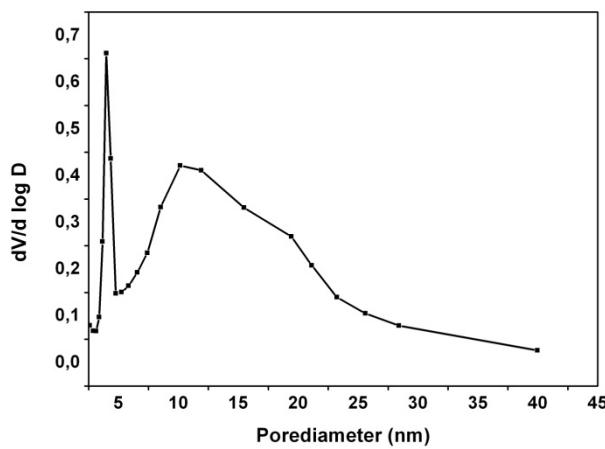


Fig. S1: BJH plot from silica aerogel sample HS_8

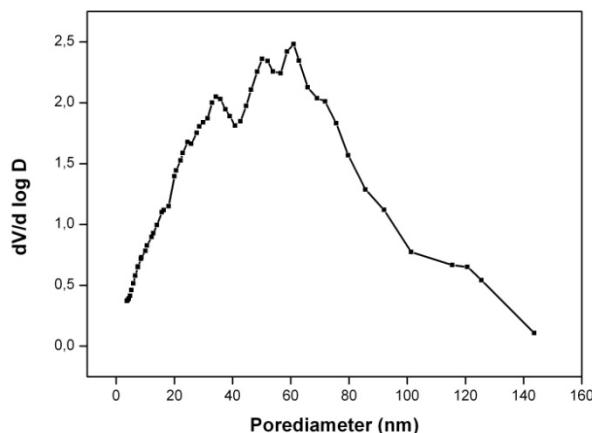


Fig. S2: BJH plot from silica aerogel sample HS_97

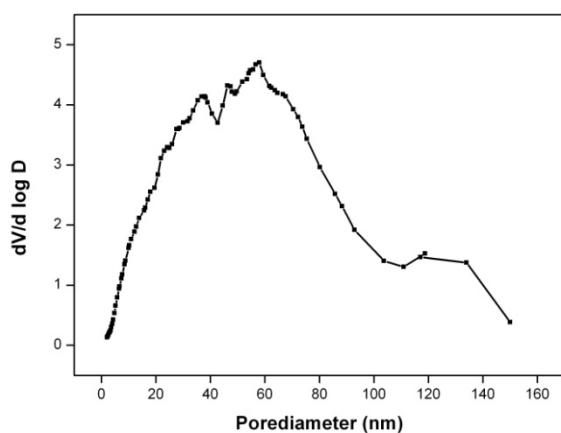


Fig. S3: BJH plot from silica aerogel sample HS_128

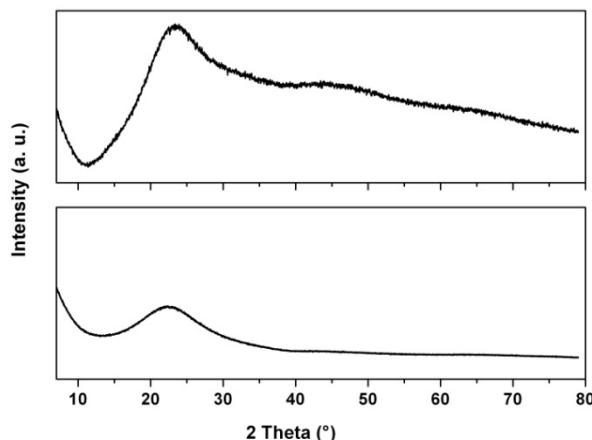


Fig. S4: Powder Diffraction pattern from silica aerogel HS_16.2 as-prepared (upper figure), respectively heat-treated (lower figure)

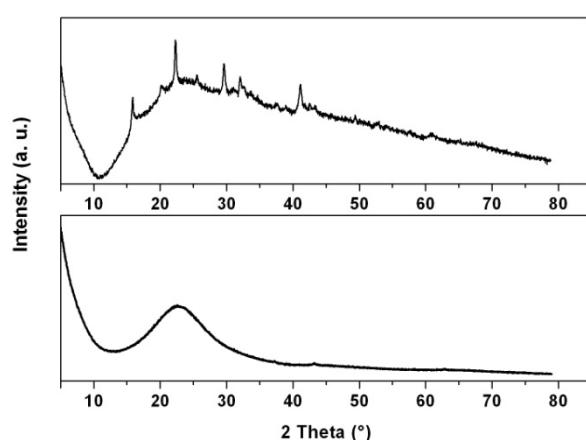


Fig. S5: Powder Diffraction pattern from silica aerogel HS_36 as-prepared (upper figure), respectively heat-treated (lower figure).

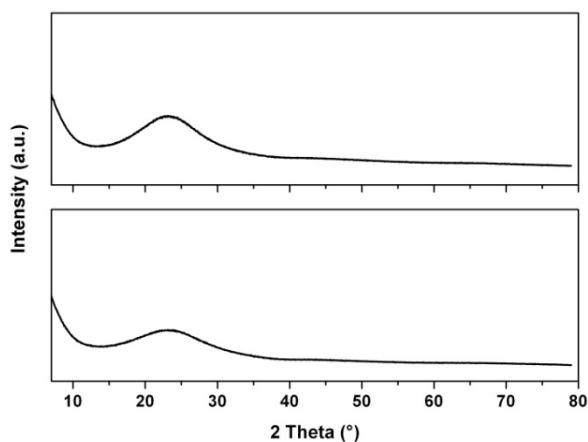


Fig. S6: Powder Diffraction pattern from silica aerogel HS_8 as-prepared (upper figure), respectively heat-treated (lower figure).

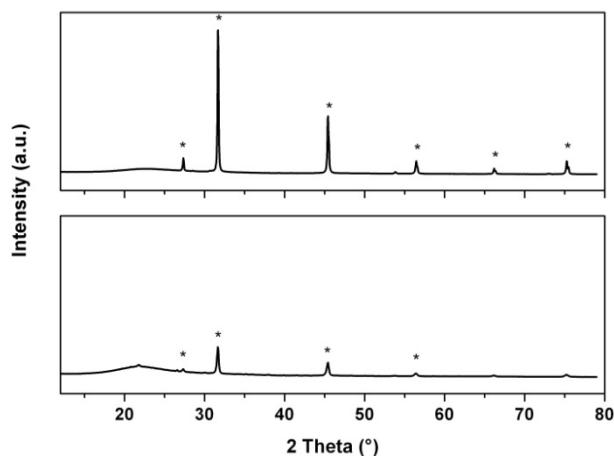


Fig. S7: Powder Diffraction pattern from silica aerogel HS_97 as-prepared (upper figure), respectively heat-treated (lower figure). Peaks corresponding to NaCl (pdf card 01-077-2064) are marked with an asterisk.

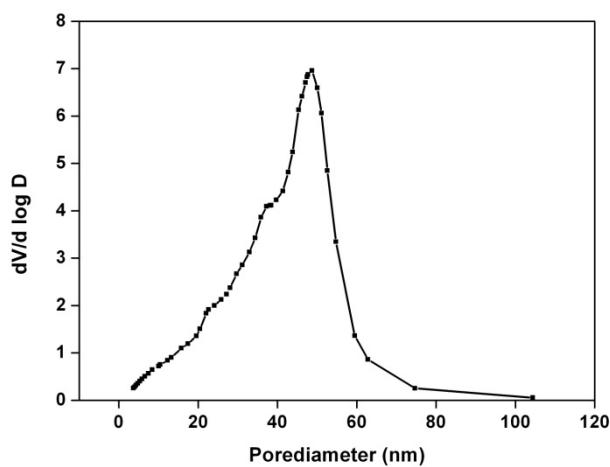


Fig. S8: BJH plot from silica-/titania aerogel sample HS_91

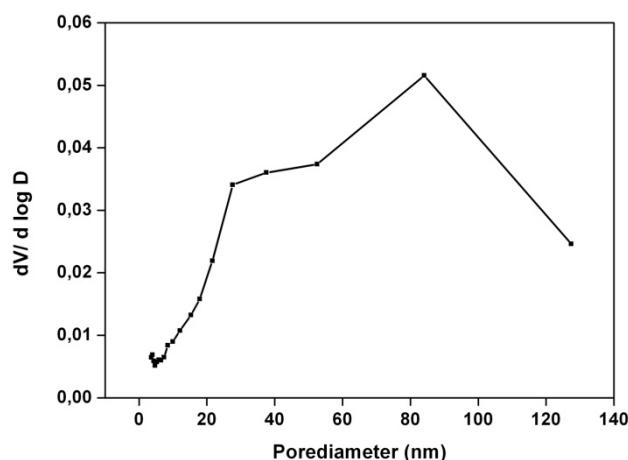


Fig. S9: BJH plot from silica-/titania aerogel sample HS_43.1

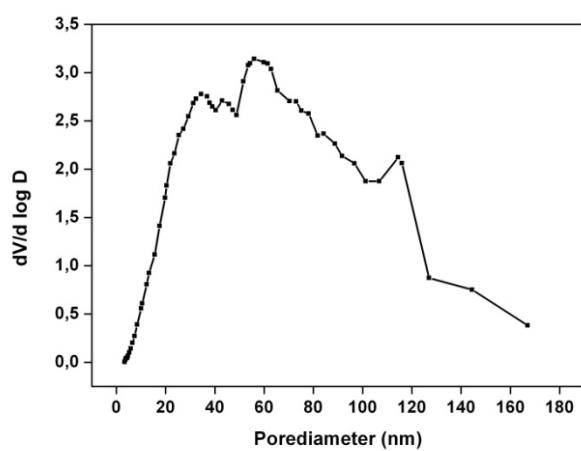


Fig. S10: BJH plot from silica-/titania aerogel sample HS_126

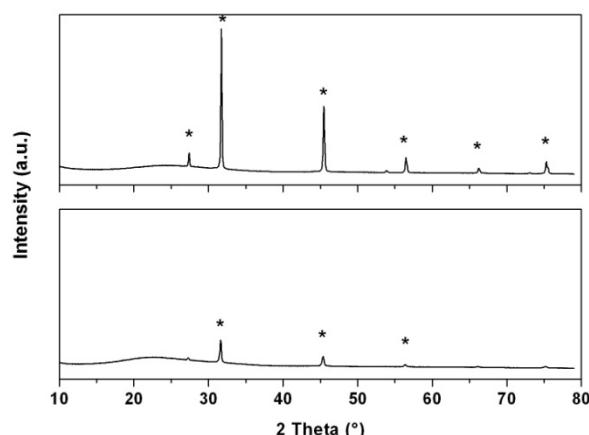


Fig. S11 Powder diffraction pattern from silica-titania aerogel HS_46 as-prepared (upper figure), respectively heat-treated (lower figure). Peaks corresponding to NaCl (pdf card 01-077-2064) are marked with an asterisk.

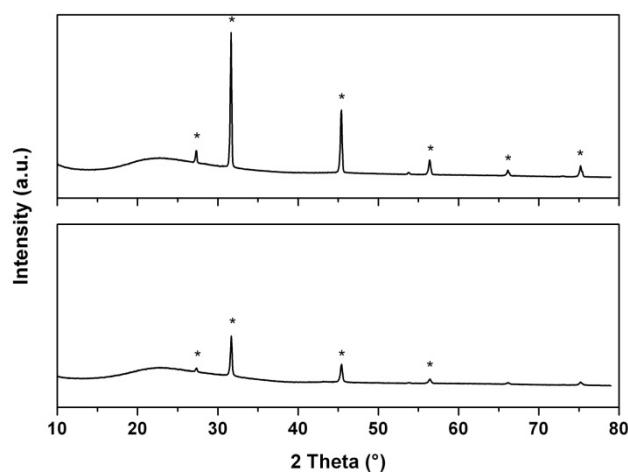


Fig S12: Powder diffraction pattern from silica-titania aerogel HS_91 as-prepared (upper figure), respectively heat-treated (lower figure). Peaks corresponding to NaCl (pdf card 01-077-2064) are marked with an asterisk.

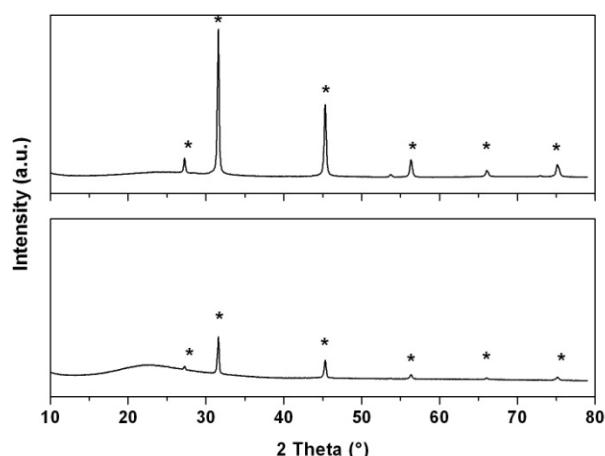


Fig. S13: Powder diffraction pattern from silica-titania aerogel HS_43.1 as prepared (upper figure), respectively heat-treated (lower figure). Peaks corresponding to NaCl (pdf card 01-077-2064) are marked with an asterisk.

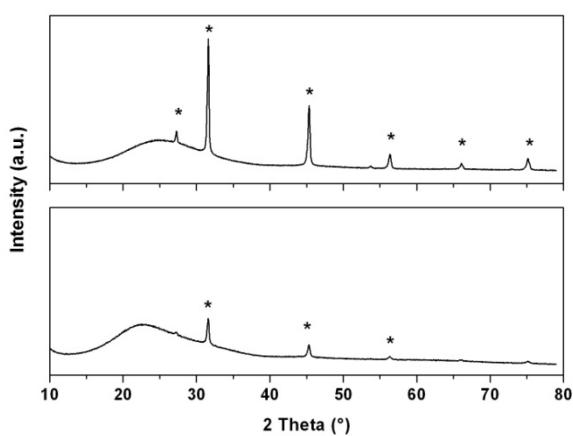


Fig. S14: Powder diffraction pattern from silica-titania aerogel HS_123 as prepared (upper figure), respectively heat-treated (lower figure). Peaks corresponding to NaCl (pdf card 01-077-2064) are marked with an asterisk.



Fig. S15. Photo of silica aerogel HS_16.2 after supercritical drying



Fig. S16. Photo of silica aerogel HS_8 after supercritical drying



Fig. S17. Photo of silica aerogel HS_97 after supercritical drying



Fig. S18. Photo of silica aerogel HS_128 after supercritical drying



Fig. S19. Photo of silica aerogel HS_138 after supercritical drying

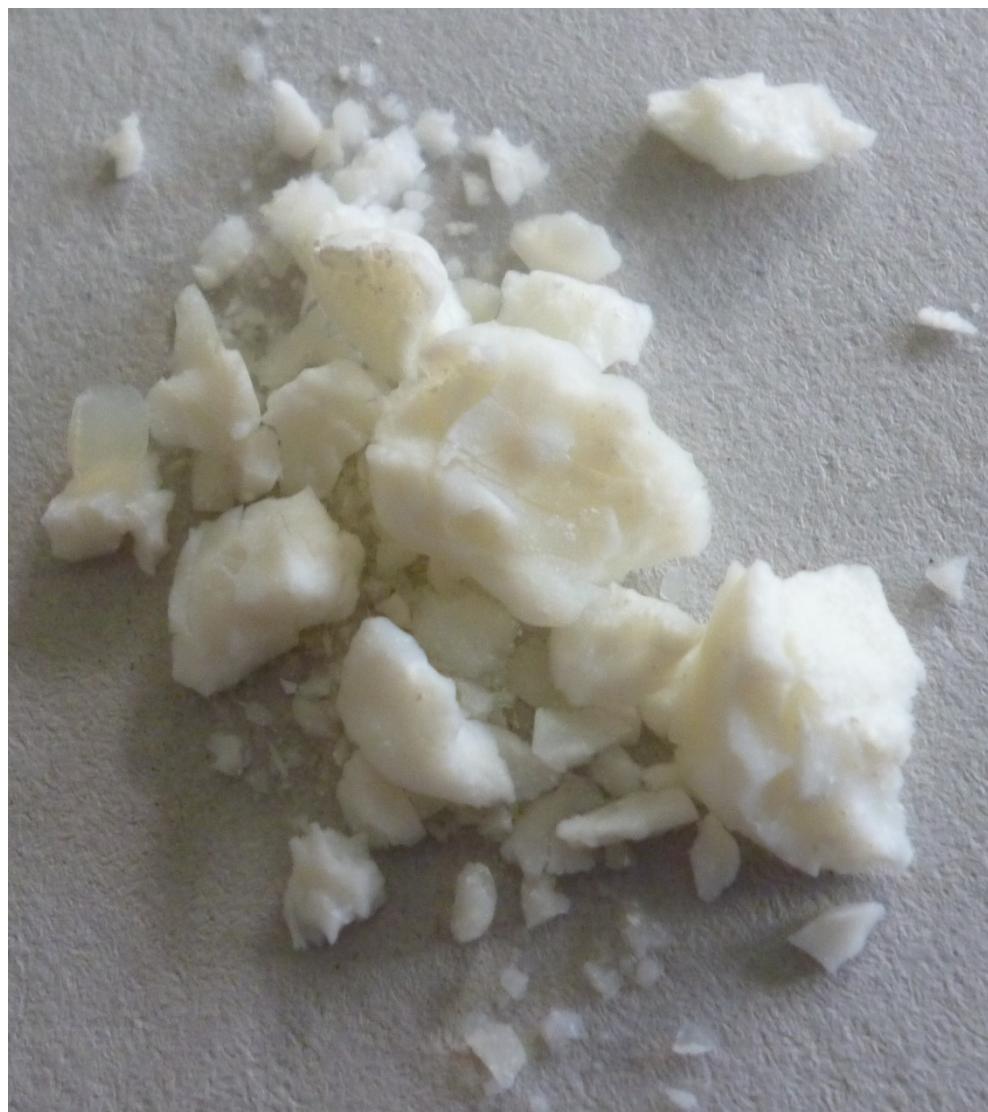


Fig. S20. Photo of titania aerogel HS_141 after supercritical drying

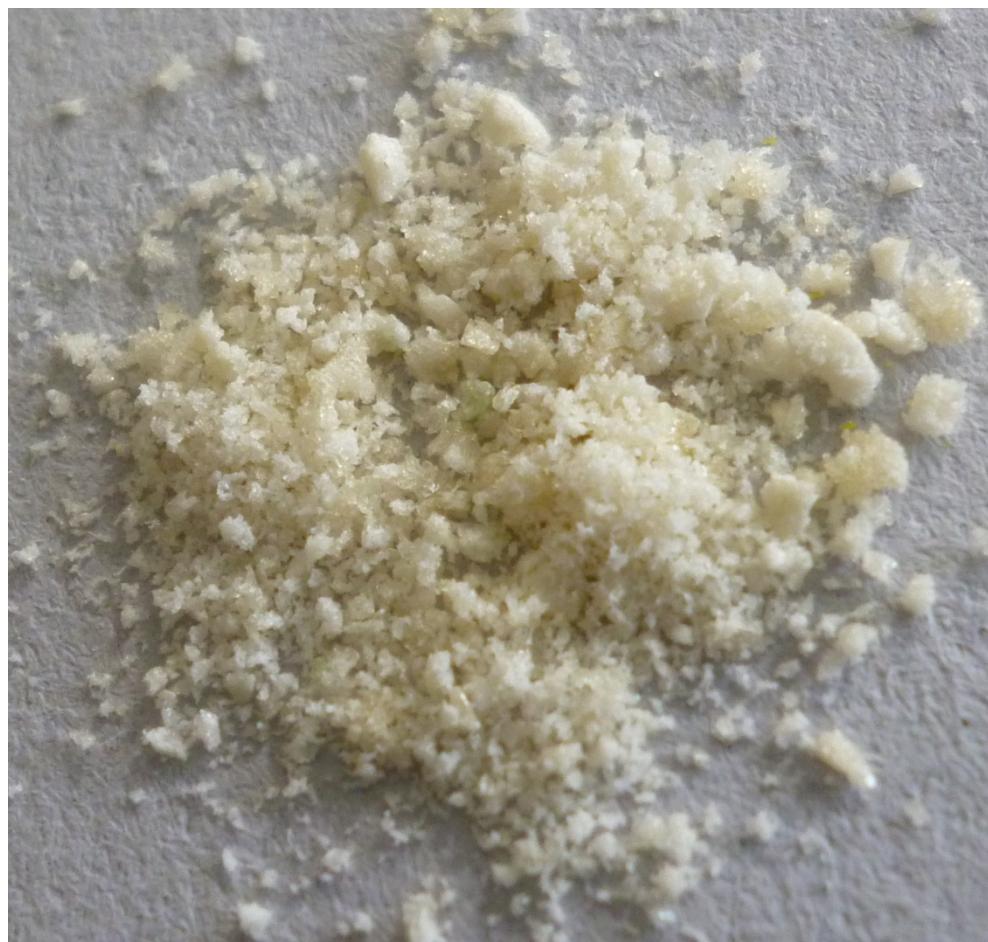


Fig. S21. Photo of titania aerogel HS_145 after supercritical drying



Fig. S22. Photo of zirconia aerogel HS_3.1 after supercritical drying

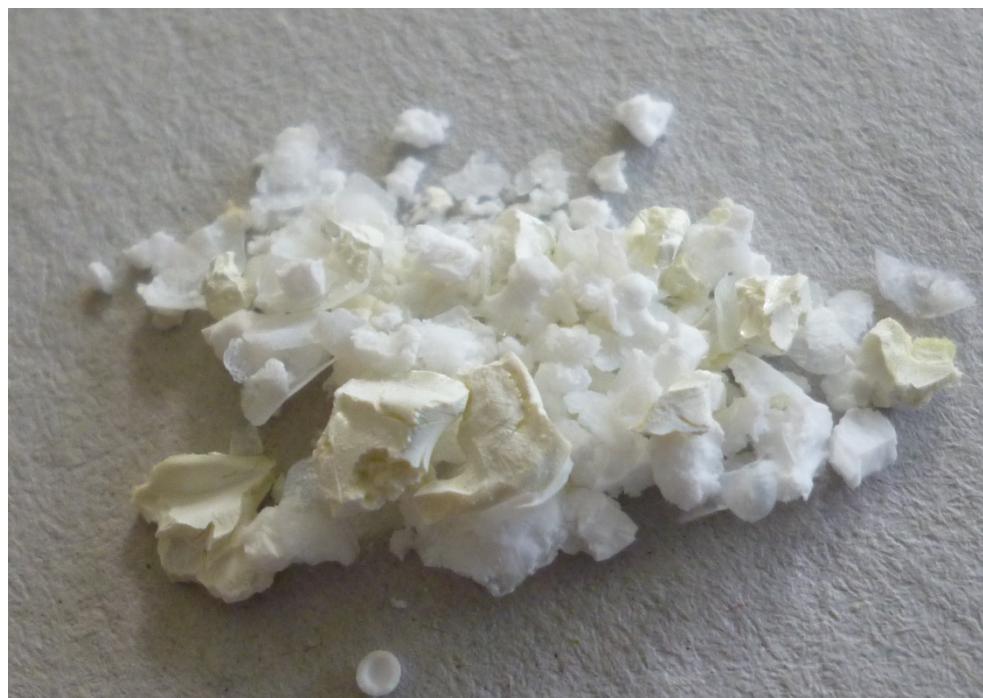


Fig. S23. Photo of zirconia aerogel HS_3.2 after supercritical drying



Fig. S24. Photo of zirconia aerogel HS_33 after supercritical drying

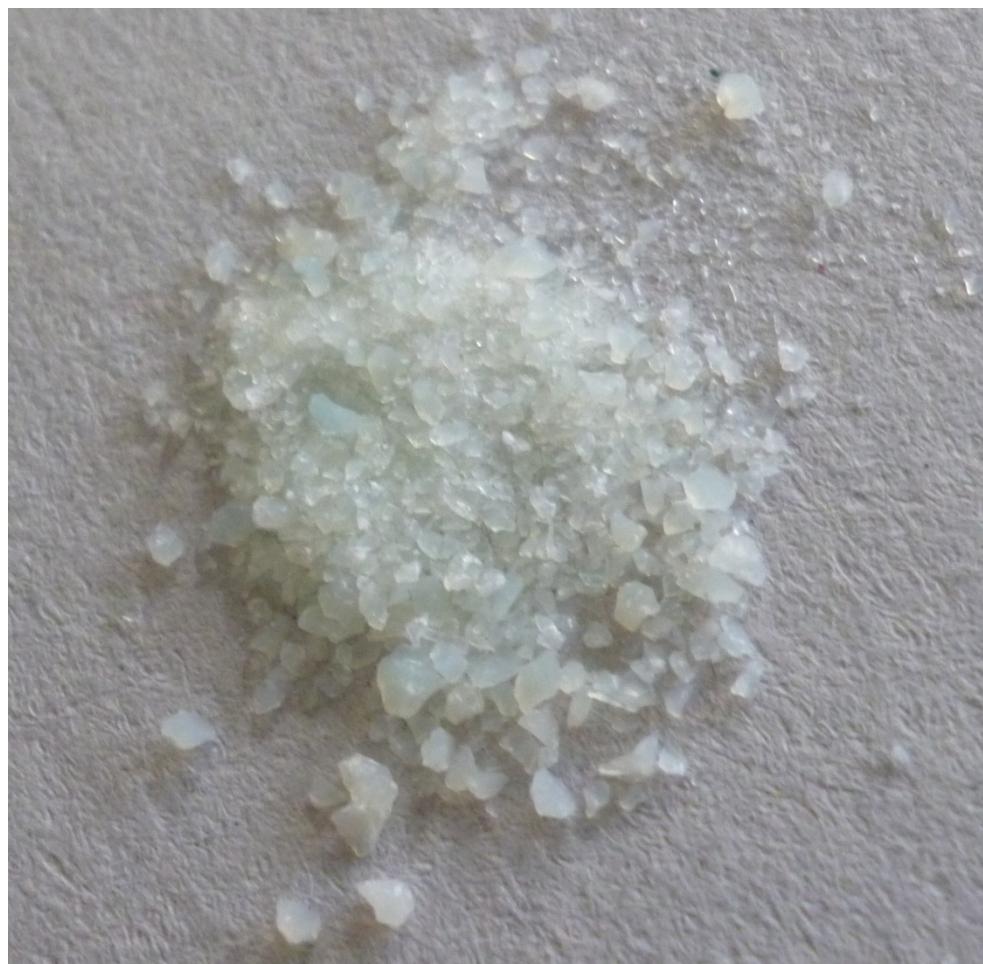


Fig. S25. Photo of zirconia/titania aerogel HS_32 after supercritical drying

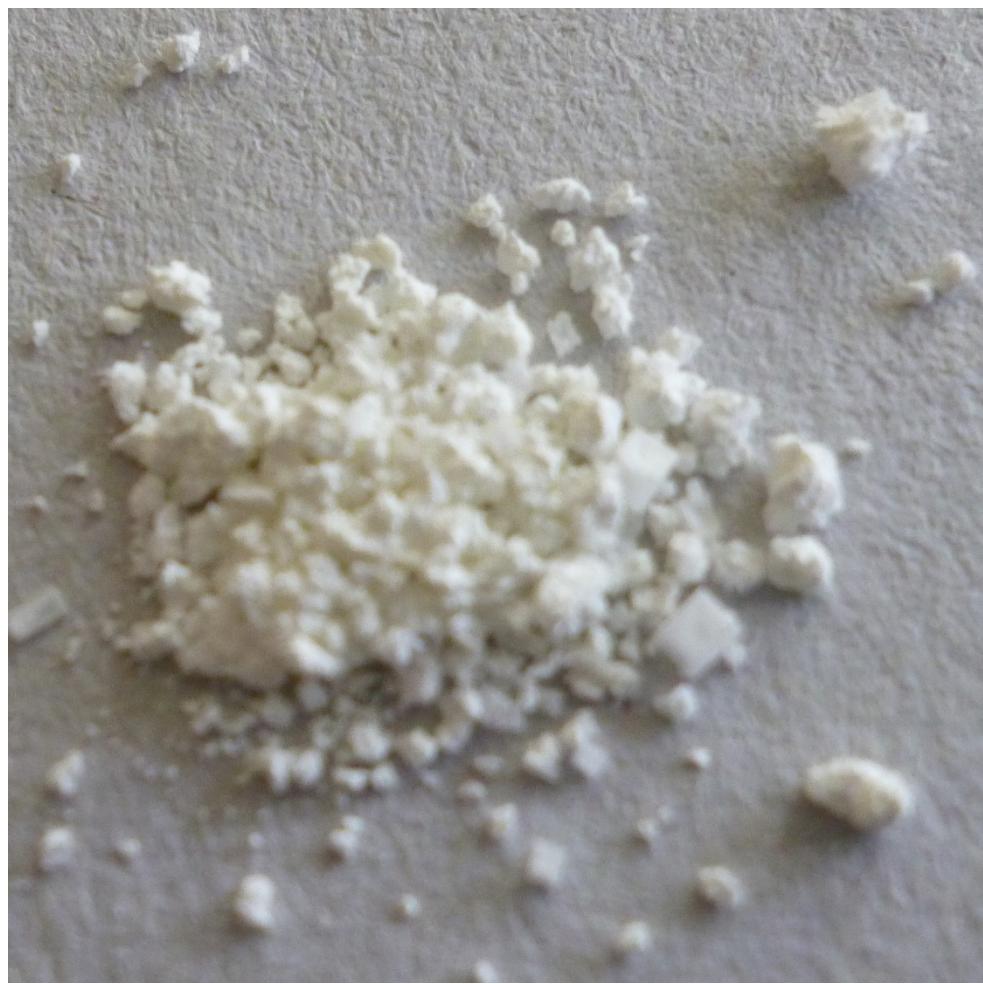


Fig. S26. Photo of silica/titania aerogel HS_46 after supercritical drying



Fig. S27. Photo of silica/titania aerogel HS_115 after supercritical drying



Fig. S28. Photo of silica/titania aerogel HS_126 after supercritical drying

sample number	silicon(IV) chloride	auxiliary agent	solvent	Gelation time
(HS_16.2)	0.9 ml (7.8 mmol)	-	water, 10 ml	< 30 s
(HS_36)	1.0 ml (8.73 mmol)	-	water, 10 ml	< 30 s
(HS_8)	5 ml (43.6 mmol)	-	58 ml THF + 2 ml water	2 days
HS_97	3 ml Sod.Sil. (18.4 mmol) + 0.3 ml SiCl ₄ (2.7 mmol)	-	water, 13 ml	30 s
HS_128	4 ml Sod.Sil. (24.6 mmol) + 0.4 ml SiCl ₄ (3.5 mmol)	-	water, 17 ml	10 s
HS_129	2.5 ml Sod.Sil. (15.3 mmol) + 0.2 ml SiCl ₄ (1.7 mmol)	-	water, 13 ml	30 s

Table TS1. Preparation conditions of wet silica- gels based on silicon (IV) chloride.

Sample number	titanium(IV) chloride	Auxiliary agent	solvent	gelation time
HS_138	0.55 ml (5 mmol)	PPO, 6 ml (85.7 mmol)	water, 10 ml	< 30 s
HS_10.2	0.55 ml (5 mmol)	PPO, 4.5 ml (64.3 mmol)	water, 10 ml	< 30 s
HS_141	0.55 ml (5 mmol)	PPO, 3 ml (42.8 mmol)	water, 7 ml + ethanol, 3 mL	< 30 s
HS_145	0.65 ml (5.9 mmol)	PPO, 3 ml (42.8 mmol)	water, 8 ml + ethanol, 2.5 ml	20 min

Table TS2. Preparation of wet titanium gels based on titanium(IV) chloride.

Sample number	Zirconium-IV-chloride	Auxiliary agent	solvent	gelation time
(HS_3.1)	1.16 g = 5 mmol	PPO, 8 mL (114.3 mmol)	Water, 10 ml	30 s
(HS_3.2)	1.16 g = 5 mmol	PPO, 5 mL (71.4 mmol)	Water, 10 ml	30 s
(HS_33)	1.16 g = 5 mmol	PPO, 5 mL (71.4 mmol)	Water, 12 ml	30 s

Table TS3. Preparation conditions of wet zirconia gels based on zirconium(IV) chloride

Sample number	precursor	Auxiliary agent	solvent	Gelation time
HS_32	1.16 g ZrCl ₄ (5 mmol) + 0.55 ml TiCl ₄ (5 mmol)	PPO, 6 mL (85.7 mmol)	water, 10 ml	30 s
HS_46	2 ml Sod.Sil. (12.3 mmol) + 0.5 ml TiCl ₄ (4.56 mmol)	-	water, 17 ml	30 s
HS_91	3 ml Sod.Sil. (18.4 mmol) + 0.4 ml TiCl ₄ (3.65 mmol)	PPO, 3 ml (42.8 mmol)	water, 18 ml	30 s
HS_115	3 ml Sod. Sil. (18.4 mmol) + solution of 0.5 ml TiCl ₄ (4.56 mmol) in 10 ml water	-	water, 15 ml	30 s
HS_121	3 ml Sod. Sil. (18.4 mmol) + 0.7 ml TiCl ₄ (6.38 mmol)	3 ml 30w% NaOH (30 mmol), pH = 2	water, 18 ml	30 s
HS_43.1	3 ml Sod. Sil. (18.4 mmol) + 0.2 ml TiCl ₄ (1.82 mmol)	0.14 ml 37% HCl (1,7 mmol) pH~3	water, 13 ml	30 s
HS_123	4.5 ml Sod. Sil. (27.6 mmol) + 0.2 ml TiCl ₄ (1.82 mmol)	0.22 ml 37% HCl (2.65 mmol), pH ~4	water, 18 ml	30 s
HS_126	3 ml Sod. Sil. (18.4 mmol) + 0.4 ml TiCl ₄ (3.65 mmol)	0.14 ml 30 w% NaOH (1.4 mmol)	water, 13 ml	30 s

Table TS4. Preparation of wet mixed metal oxide gels