Supplementary Material (ESI) for Journal of Materials Chemistry

Preventing sintering of Au and Ag nanoparticles in silica-based hybrid gels using phenyl spacer groups

Nina M. Wichner[a], Jurriaan Beckers[b], Gadi Rothenberg[b,] * and Hubert Koller[a,] *

[a] *Institut für Physikalische Chemie, Westfälische Wilhelms-Universität,Corrensstrasse 28/30, 48149 Münster Germany.* E-mail hubert.koller@uni-muenster.de Fax. +49 251 8323409.

[b] *Van 't Hoff Institute for Molecular Sciences, University of Amsterdam, Nieuwe Achtergracht 166, 1018 WV Amsterdam, The Netherlands.* E-mail g.rothenberg@uva.nl Fax. +31 (0)20 525 5604.

SUPPORTING INFORMATION

Tab. 1, Supporting Information: Composition of metal nanoparticles stabilized by phenylethylthiol in hybrid silica gels, composed of TMOS and PhTS; the metal to Si ratio (M / Si) is the atomic ratio of the synthesis mixture

sample	metal source	M / Si =	pH	color after calcination
A	$Au_{x1}(SC_2H_4Ph)_{y1}$	0.012	7	violet
B	$Au_{x1}(SC_2H_4Ph)_{y1}$	0.014	6	red
C	$Au_{x1}(SC_2H_4Ph)_{y1}$	0.035	6	red
D	$Ag_{x2}(SC_2H_4Ph)_{y2}$	0.033	6	brown
E	$Ag_{x2}(SC_2H_4Ph)_{y2}$	0.009	6	beige
F	$Ag_{x2}(SC_2H_4Ph)_{y2}$	0.016	7	beige
G	---	0	6	colorless
H	$Ag_{x2}(SC_2H_4Ph)_{y2}$	0.014	5	brown
I*	$Ag_{x2}(SC_2H_4Ph)_{y2}$	0.016	7	grey-brown
J**	$Ag_{x2}(SC_2H_4Ph)_{y2}$	0.016	7	brown
K	$AgNO_3$, $NaBH_4$	0.016	7	grey-brown

* silver particles are adsorbed post-synthetically on the surface of the silica gel
** no PhTS

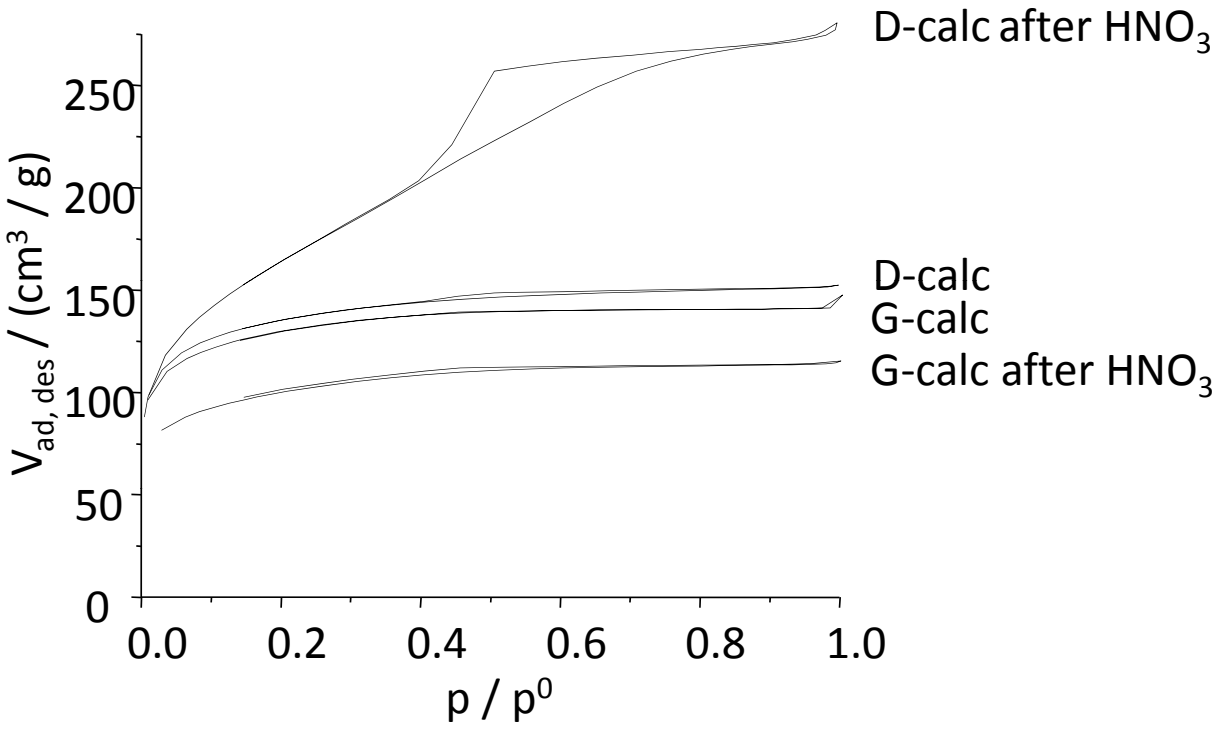

Fig. 1, Supporting Information: N_2 adsorption of calcined silica gel with and without silver particles before and after treatment with HNO_3 under reflux.

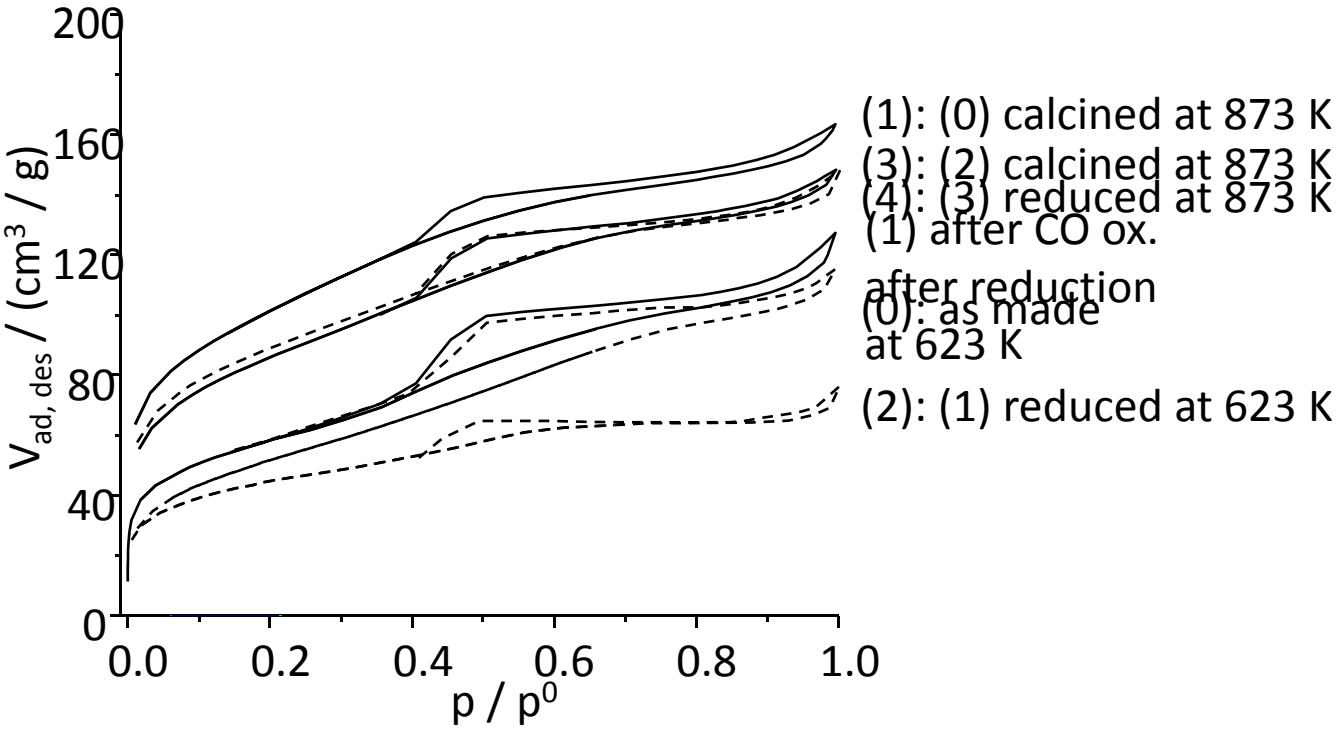

Fig. 2, Supporting Information: Nitrogen sorption isotherms of sample **F** (pH=7) after calcinations steps (solid lines), reduction steps (dashed lines) and after catalytic test and subsequent reduction (solid line). Trace (0) is the as-made sample, and trace (1) was already shown in Fig. 5 for the calcined sample, F-calc. Interestingly, the adsorption isotherm decreases after reduction of the silver particles (trace (2)). This effect can be attributed to a decrease in the mesopore volume, and a substantial decrease in the external surface area (Tab. 1). We suggest that the particles aggregate upon reduction, and their external surface is reduced, accordingly. Further analysis would be necessary to investigate this effect, but it is out of the scope of this work. Trace (3) shows the adsorption isotherm of the sample of trace (2) after another calcination step. A large recovery of pore volume can be identified after this treatment, and the isotherm then remains stable after another reduction step (trace (4)). The Figure also shows the adsorption isotherm of the sample of trace (1) after CO oxidation and reduction.

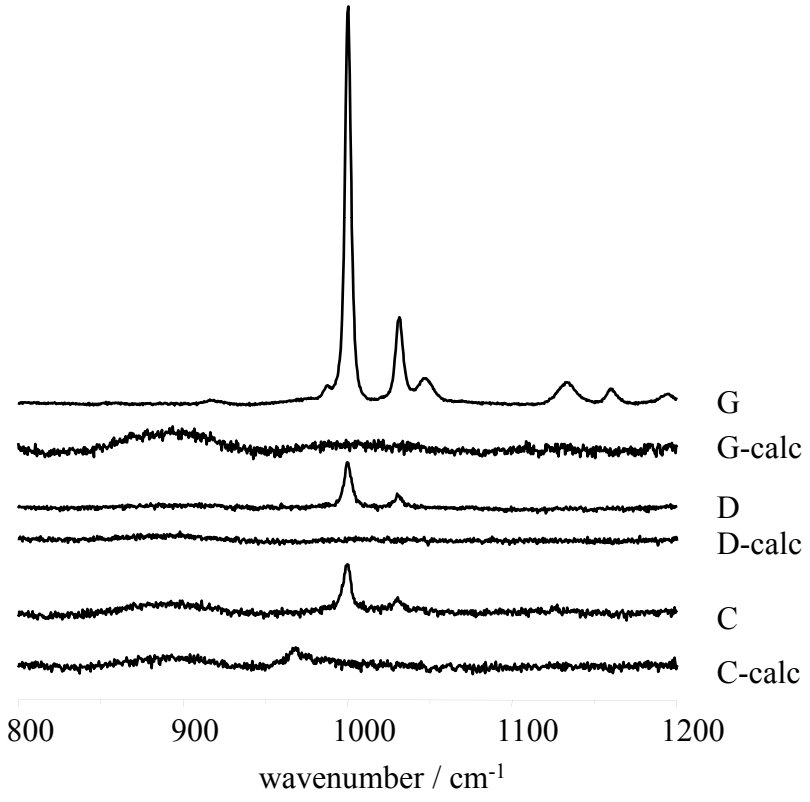

Fig. 3, Supporting Information: Raman spectra of a metal-free , a gold-containing and silver-containing hybrid gel before and after calcination at 923 K. The intense phenyl vibration at 1000 cm^{-1} can only be observed for the uncalcined samples.