

## Supplementary Information

# Expanding the Range: AuCu Metal Aerogels from H<sub>2</sub>O and EtOH

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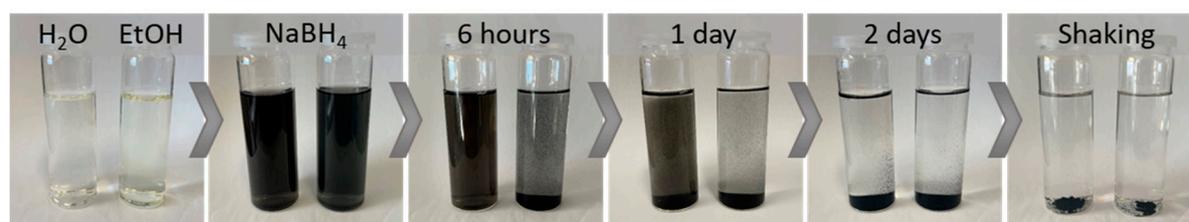
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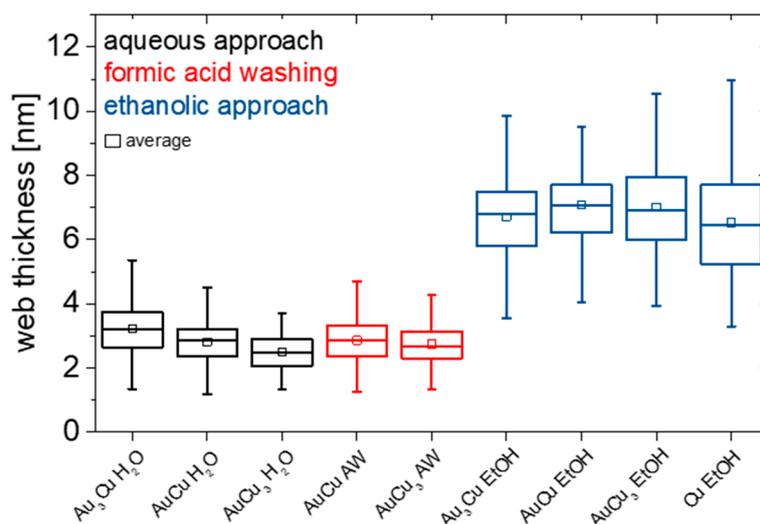
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**Table S1.** Properties of all Au<sub>x</sub>Cu<sub>y</sub> aerogels prepared via a synthesis in H<sub>2</sub>O and EtOH. Element compositions were determined via SEM-EDX, the average web thicknesses were determined from individual measurements of the TEM images and SSA/TpV via N<sub>2</sub> physisorption.

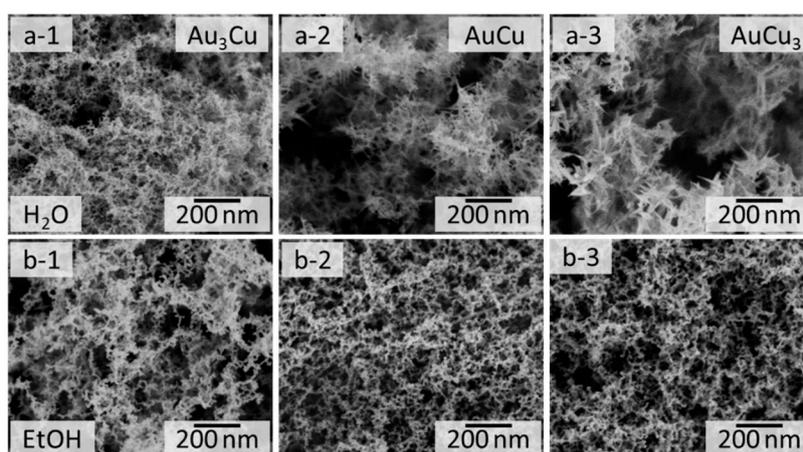
Solvent	Sample	Element composition [at%]			Web Thickness [nm]	SSA [m <sup>2</sup> g <sup>-1</sup> ]	TpV [cm <sup>3</sup> g <sup>-1</sup> ]
		Au	Cu	Standard Deviation			
H <sub>2</sub> O	Au <sub>3</sub> Cu	73.4	26.6	0.7	3.2 ± 0.8	56	0.121
	AuCu	49.9	50.1	1.9	2.8 ± 0.6	59	0.140
	AuCu <sub>3</sub>	24.9	75.1	1.6	2.5 ± 0.7	59	0.213
	AuCu AW	62.0	38.0	2.5	2.9 ± 0.7	71	0.186
	AuCu <sub>3</sub> AW	62.2	37.8	2.5	2.7 ± 0.7	69	0.226
EtOH	Au <sub>3</sub> Cu	76.0	24.0	1.0	6.7 ± 1.3	28	0.122
	AuCu	52.0	48.0	0.8	7.1 ± 1.5	40	0.294
	AuCu <sub>3</sub>	26.9	73.2	0.3	7.0 ± 1.5	64	0.490
	Cu	-	-	-	6.6 ± 1.6	43	0.101



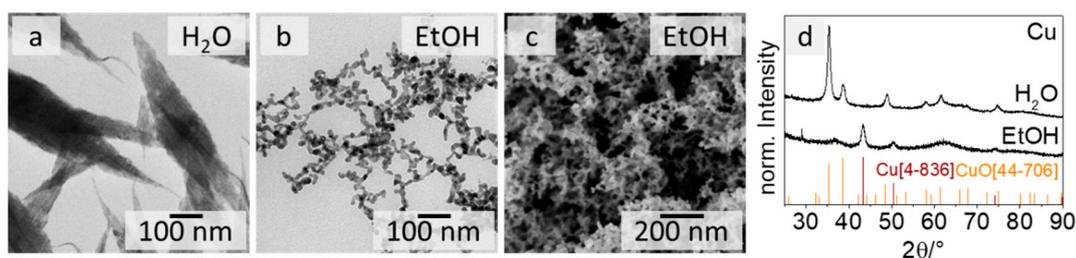
**Figure S1.** Time lapse photographs of the Au<sub>3</sub>Cu solvogel gelation process in water (left) and ethanol (right). The metal salt solutions turn dark upon NaBH<sub>4</sub> addition. The intermediately formed aggregates settle at different rates and form gels.



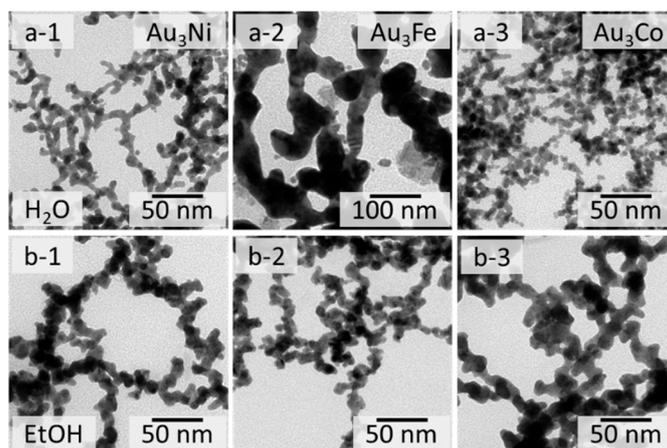
**Figure S2.** Overview of the web thicknesses and their size distribution of all prepared Au<sub>x</sub>Cu<sub>y</sub> aerogels, determined from bright-field TEM images and visualized in form of boxplots. The average web thickness of the gels prepared in aqueous solution is 3 nm, while that of the gels obtained in ethanolic solution is about 6 nm. In EtOH, the web thickness' size distribution becomes broader.



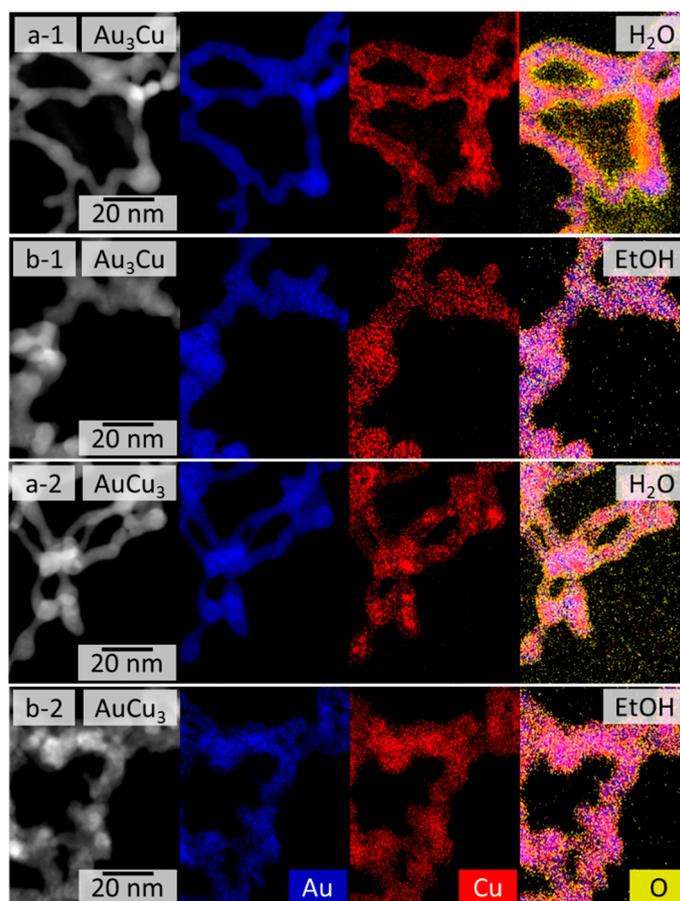
**Figure S3.** SEM images of Au<sub>3</sub>Cu (1), AuCu (2), and AuCu<sub>3</sub> (3) aerogels from an aqueous (a) and ethanolic (b) synthesis. The aerogels form three-dimensional, porous networks. In a-2 and a-3 the gels are covered with a needle-like substructure.



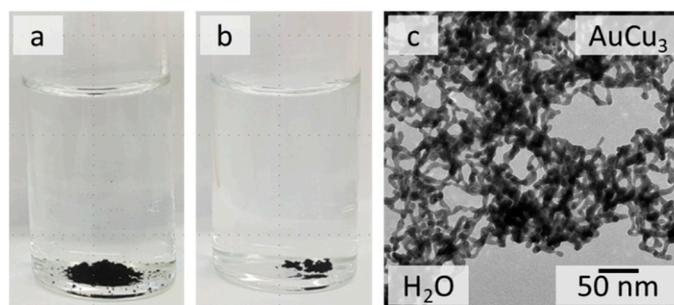
**Figure S4.** TEM (a + b) and SEM (c) images as well as XRD analysis (d) of reduced CuCl<sub>2</sub>, following the synthesis parameter of the Au-Cu gels. In water (a) the reaction leads to the formation of CuO-needles (d). In EtOH the formation of a three-dimensional Cu aerogel (b + c) can be observed. The gel keeps its metallic character and an additional CuO phase (d).



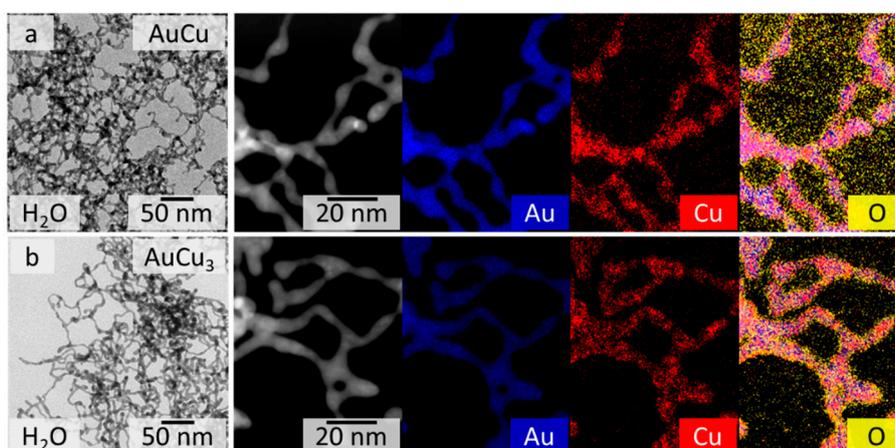
**Figure S5.** TEM images of  $\text{Au}_3\text{Ni}$  (1),  $\text{Au}_3\text{Fe}$  (2) and  $\text{Au}_3\text{Co}$  (3) gels, synthesized in  $\text{H}_2\text{O}$  (a) and  $\text{EtOH}$  (b). The synthesis results in irregular gel structures in water, while similar gel morphologies are obtained in ethanol.



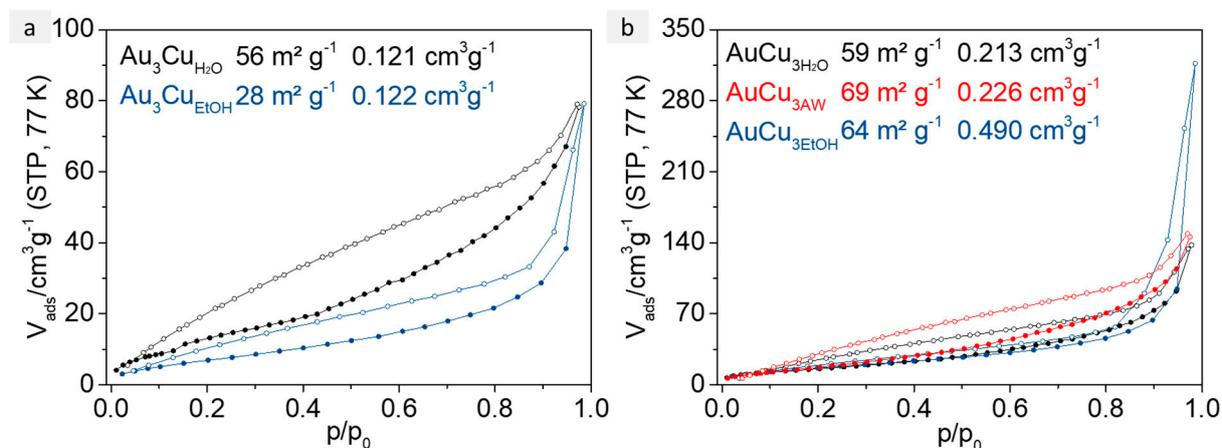
**Figure S6.** HAADF-STEM images (left) and EDX-based element distributions of the  $\text{Au}_3\text{Cu}$  (1) and  $\text{AuCu}_3$  (2) gels synthesized in water (a) and ethanol (b). The gels demonstrate a quite homogeneous distribution of Au and Cu over the whole gel networks.



**Figure S7.** AuCu<sub>3</sub> gel before (a) and after (b) the acid treatment in a 88.5 mM formic acid solution. TEM image (c) of the acid-treated gel. The treatment leads to a macroscopic shrinkage of the structure and an increase of the web thickness.



**Figure S8.** TEM micrographs as well as HAADF-STEM images and EDX-based element distributions of the AuCu (a) and AuCu<sub>3</sub> (b) gels from the aqueous approach after the acid treatment with HCOOH. CuO is not observable and the gel network remains untouched. Cu can still be found in the gel strands.



**Figure S9.** N<sub>2</sub> physisorption isotherms of Au<sub>3</sub>Cu (a) and AuCu<sub>3</sub> (b) aerogels synthesized in water (black), after acid treatment with HCOOH (red) and synthesized in EtOH (blue). All isotherms are combinations of type II and IV, indicating broad pore size distributions.