

# From Au(I) organometallic hydrogels to well-defined Au(0) nanoparticles

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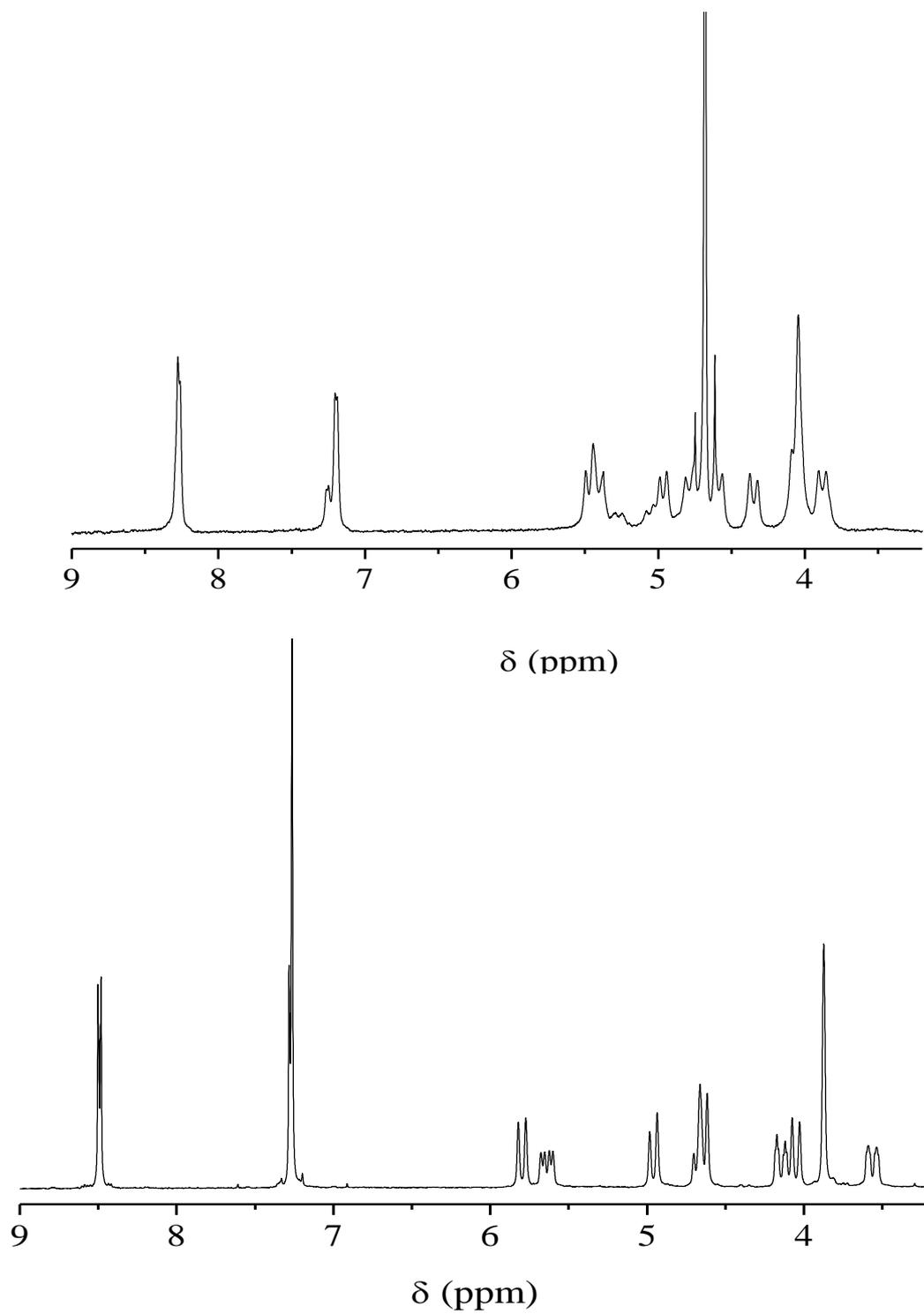
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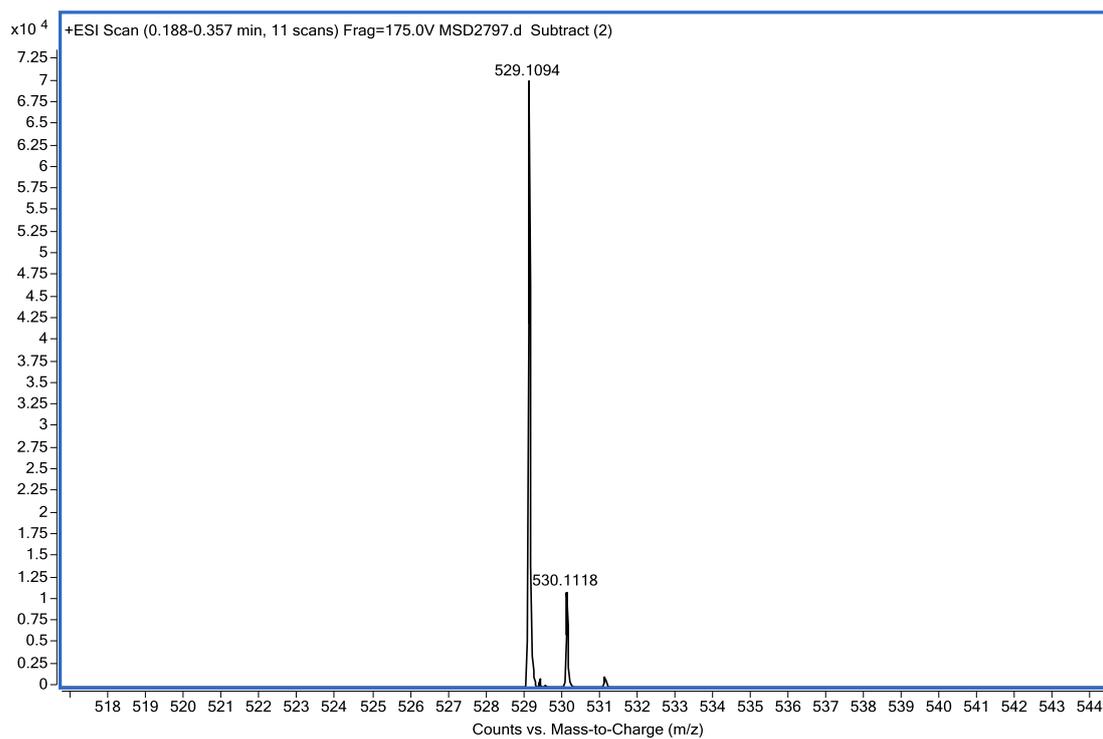
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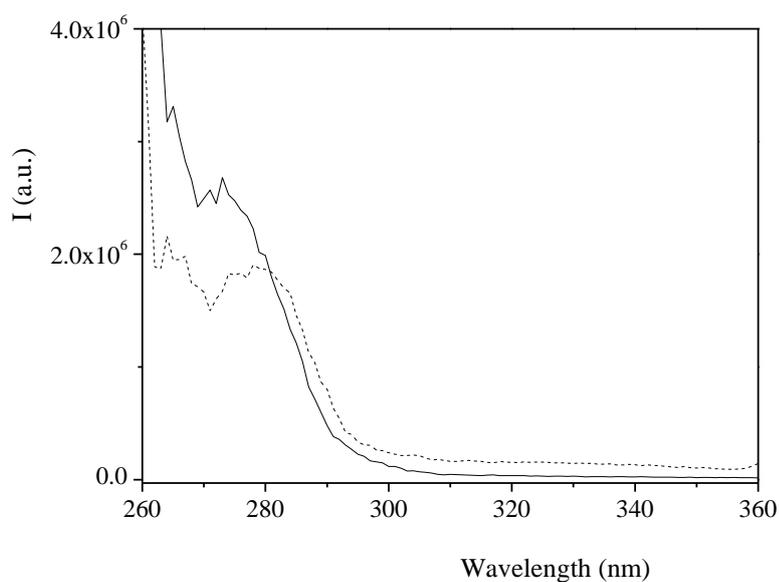
## Supporting Information



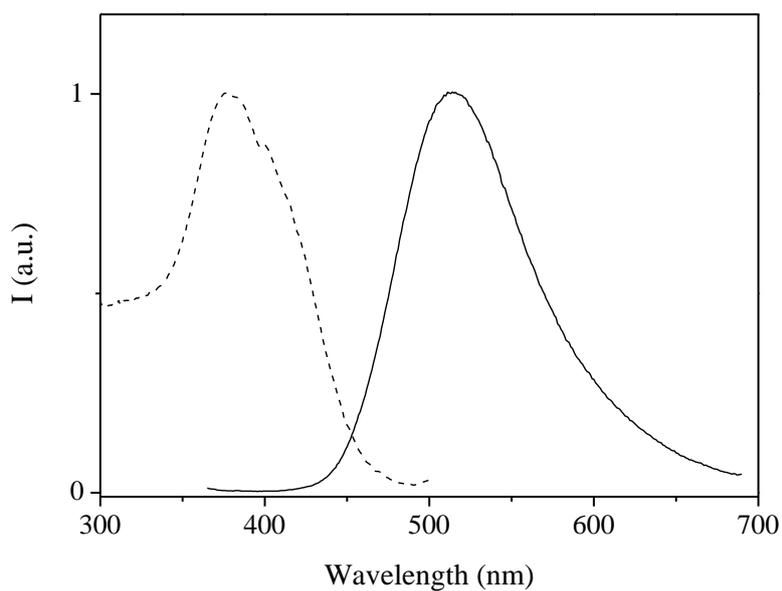
**Figure S1.** <sup>1</sup>H-NMR spectra of [Au(C≡C-C<sub>5</sub>H<sub>4</sub>N)(DAPTA)] in D<sub>2</sub>O (up) and CDCl<sub>3</sub> (bottom).



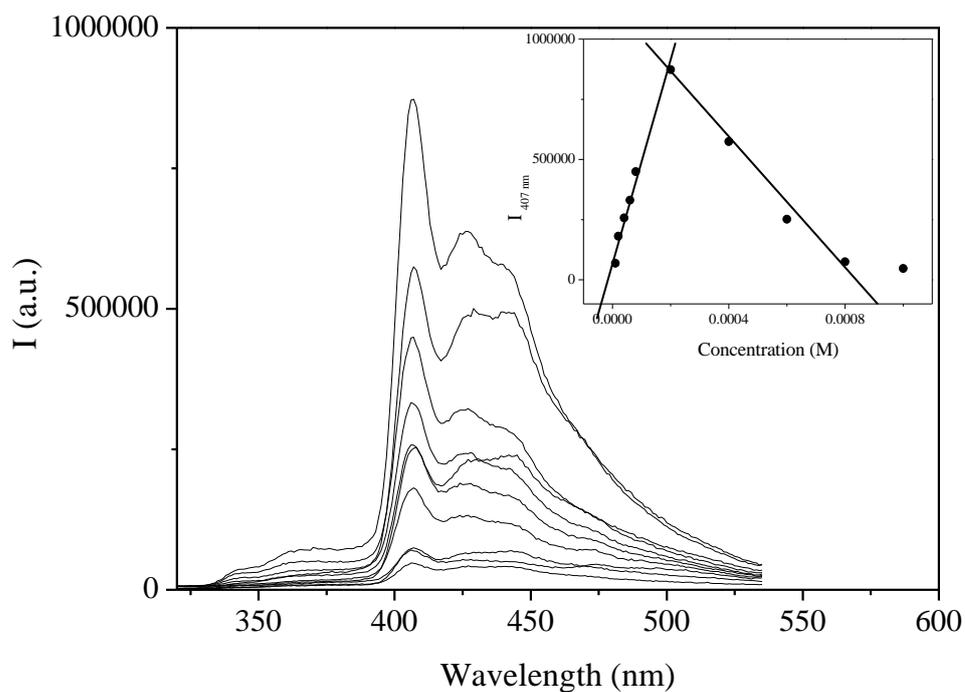
**Figure S2.** ESI-MS(+) spectrum of  $[\text{Au}(\text{C}\equiv\text{C}-\text{C}_5\text{H}_4\text{N})(\text{DAPTA})]$ .



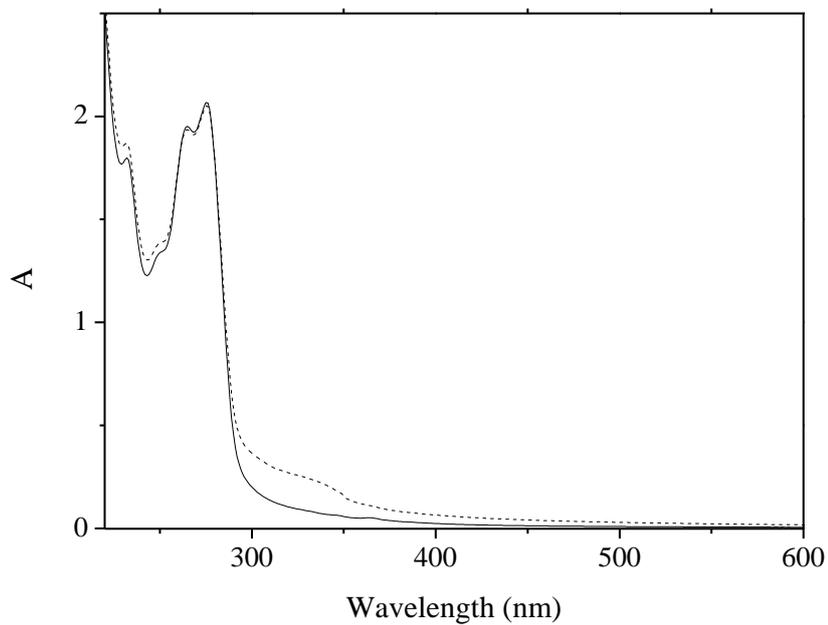
**Figure S3.** Excitation spectrum of a  $6 \times 10^{-4} \text{M}$   $[\text{Au}(\text{C}\equiv\text{C}-\text{C}_5\text{H}_4\text{N})(\text{DAPTA})]$  dichloromethane sample (dashed line) and aqueous sample (solid line).  $\lambda_{\text{em}} = 390 \text{ nm}$ .



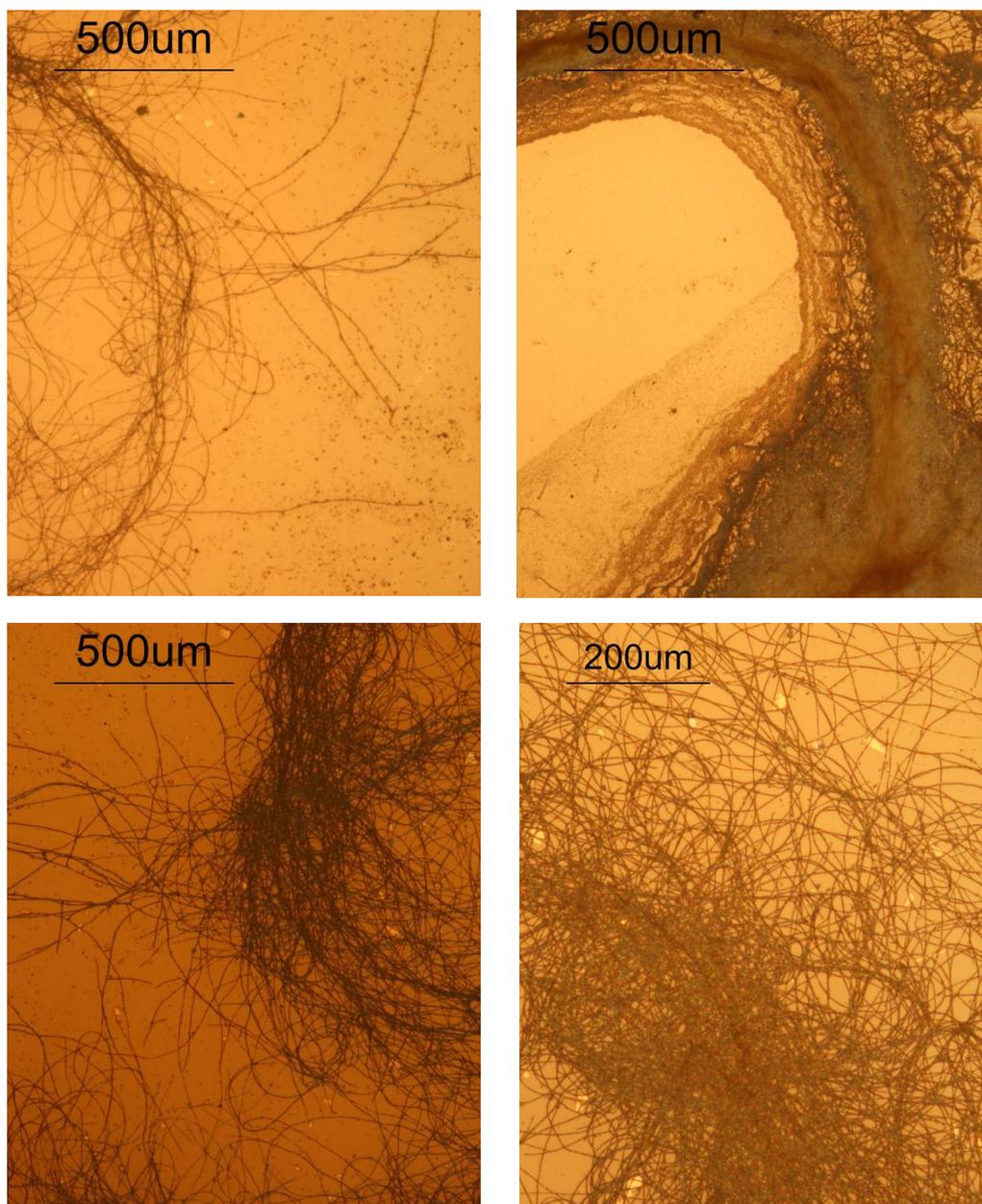
**Figure S4.** Normalized solid emission of  $[\text{Au}(\text{C}\equiv\text{C}-\text{C}_5\text{H}_4\text{N})(\text{DAPTA})]$  (solid line,  $\lambda_{\text{exc}} = 390 \text{ nm}$ ) and excitation spectra (dashed line,  $\lambda_{\text{em}} = 515 \text{ nm}$ ).



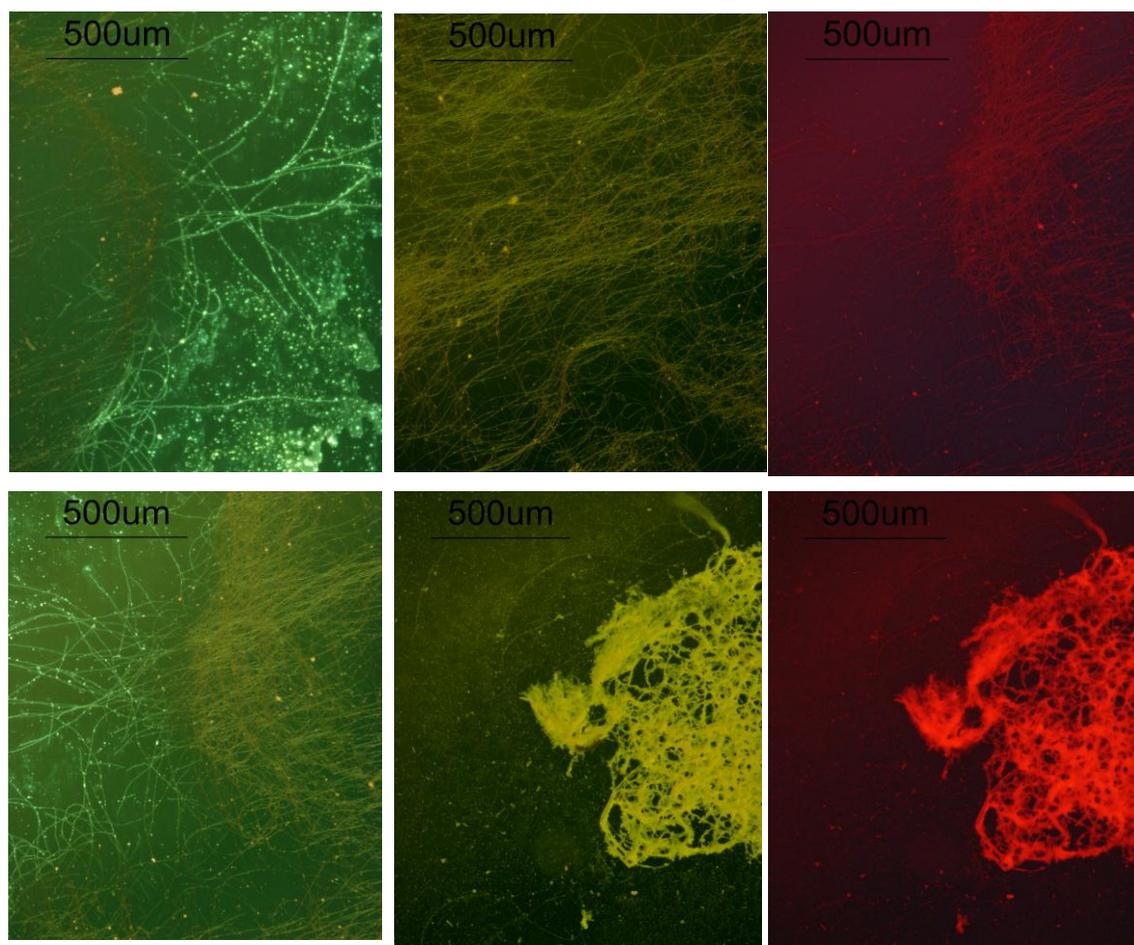
**Figure S5.** Emission spectra of  $[\text{Au}(\text{C}\equiv\text{C}-\text{C}_5\text{H}_4\text{N})(\text{DAPTA})]$  in water between  $1 \times 10^{-5} \text{ M}$  and  $1 \times 10^{-3} \text{ M}$  in water ( $\lambda_{\text{exc}} = 275 \text{ nm}$ ). Inset: Plot of  $I_{407\text{nm}}$  against concentration.



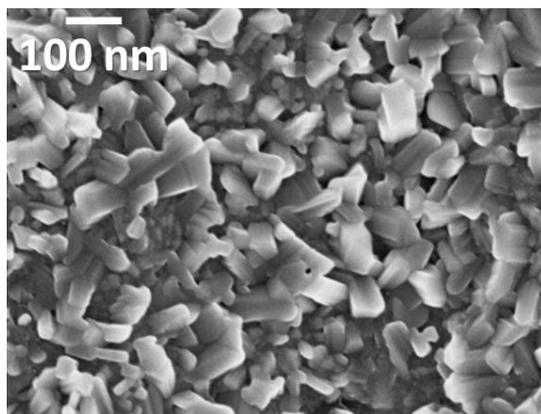
**Figure S6.** Absorption spectra of a freshly prepared  $6 \times 10^{-4} \text{ M}$   $[\text{Au}(\text{C}\equiv\text{C}-\text{C}_5\text{H}_4\text{N})(\text{DAPTA})]$  water sample (solid line) and after two days (dashed line).



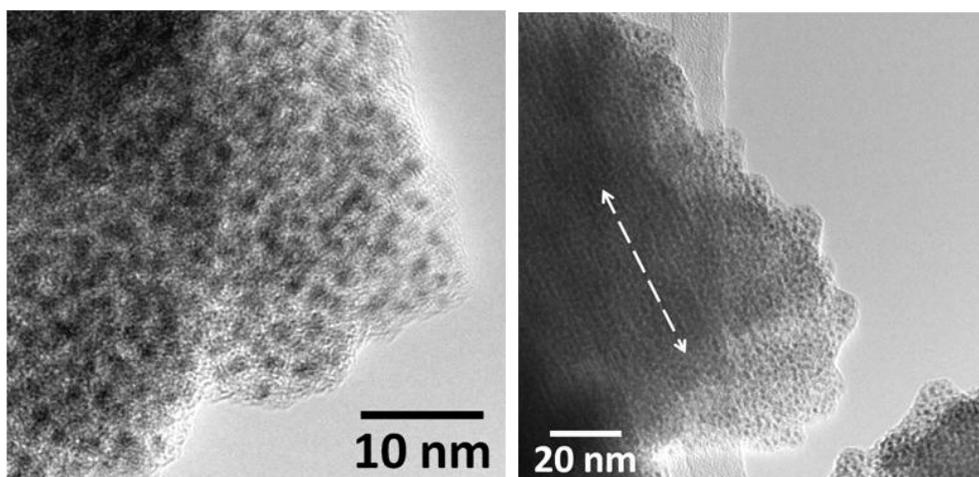
**Figure S7.** Optical microscopy images of the [Au(C≡C-C<sub>5</sub>H<sub>4</sub>N)(DAPTA)] fibers.



**Figure S8.** Images of [Au(C≡C-C<sub>5</sub>H<sub>4</sub>N)(DAPTA)] fibers and cross-linked aggregates under fluorescence optical microscopy with a 395-440 nm filter (left and middle) and 510-560 nm filter (right).



**Figure S9.** SEM images of the [Au(C≡C-C<sub>5</sub>H<sub>4</sub>N)(PTA)] xerogel on a rough (molybdenum) surface.



**Figure S10.** HRTEM images of xerogel upon electron beam irradiation.

