

Electronic Supporting Information

Identification of the key steps in the self-assembly of homogeneous gold metal nanoparticles produced using inverse micelles

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1. UV-Vis Spectroscopy

UV-vis spectroscopy was employed to follow Au species evolution in solution during the reduction process.



Figure S1. Photograph of the UV-Vis cuvette with sample A-HCl showing precipitated Au nanoparticles formed after 24 hours of the reduction.

2. Small-angle X-ray Scattering (SAXS)

Figure S2-S3 show the 1D scattering profiles recorded at different synthesis times and corresponding fitted curves for samples A and A-HCl as well as the micelle core size and polydispersity extracted from the fit. 1D SAXS profiles from PS-*b*-P2VP micelles only and micelles loaded with the chlorauric acid are displayed in Figure S4.

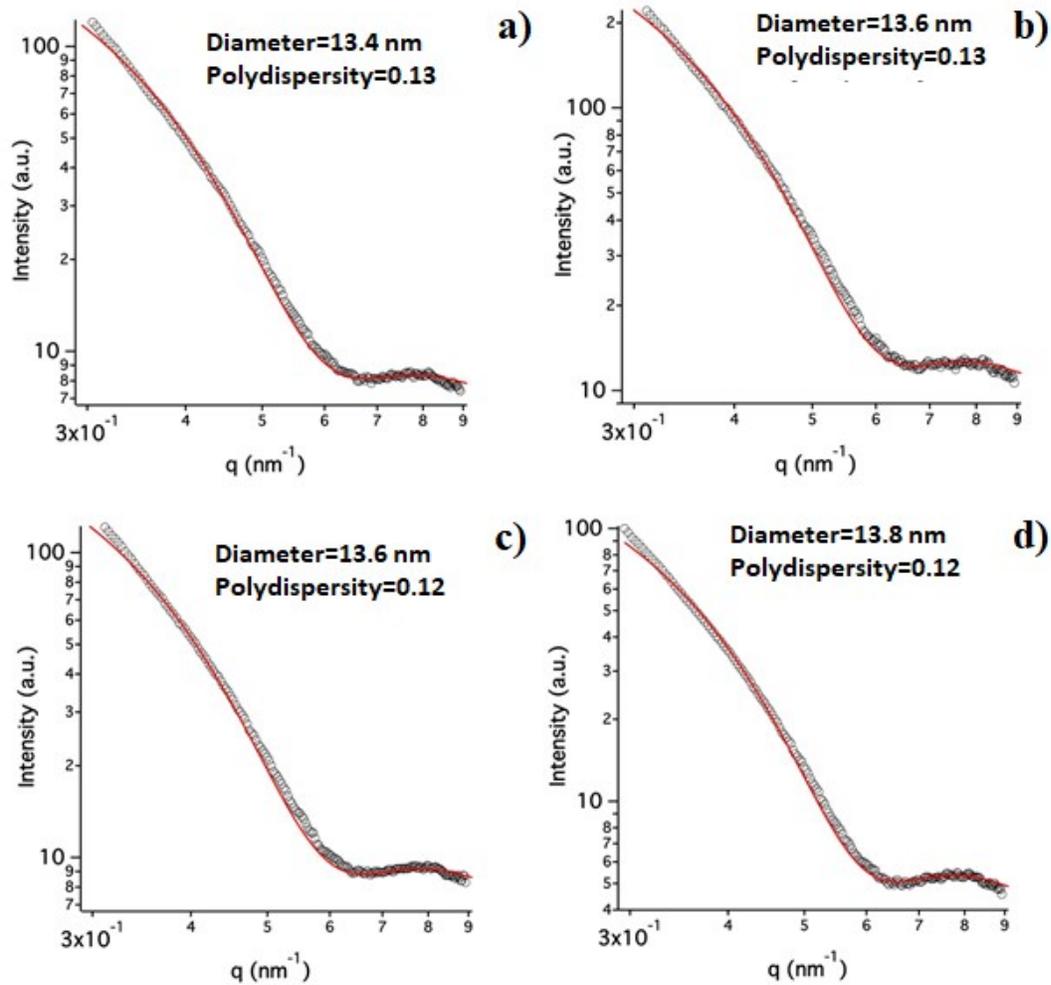


Figure S2. 1D SAXS curves (in green) and corresponding fitted curves (in blue) for sample A after a) 0h; b) 1.25 h; c) 2.25 h; d) 6.25 h.

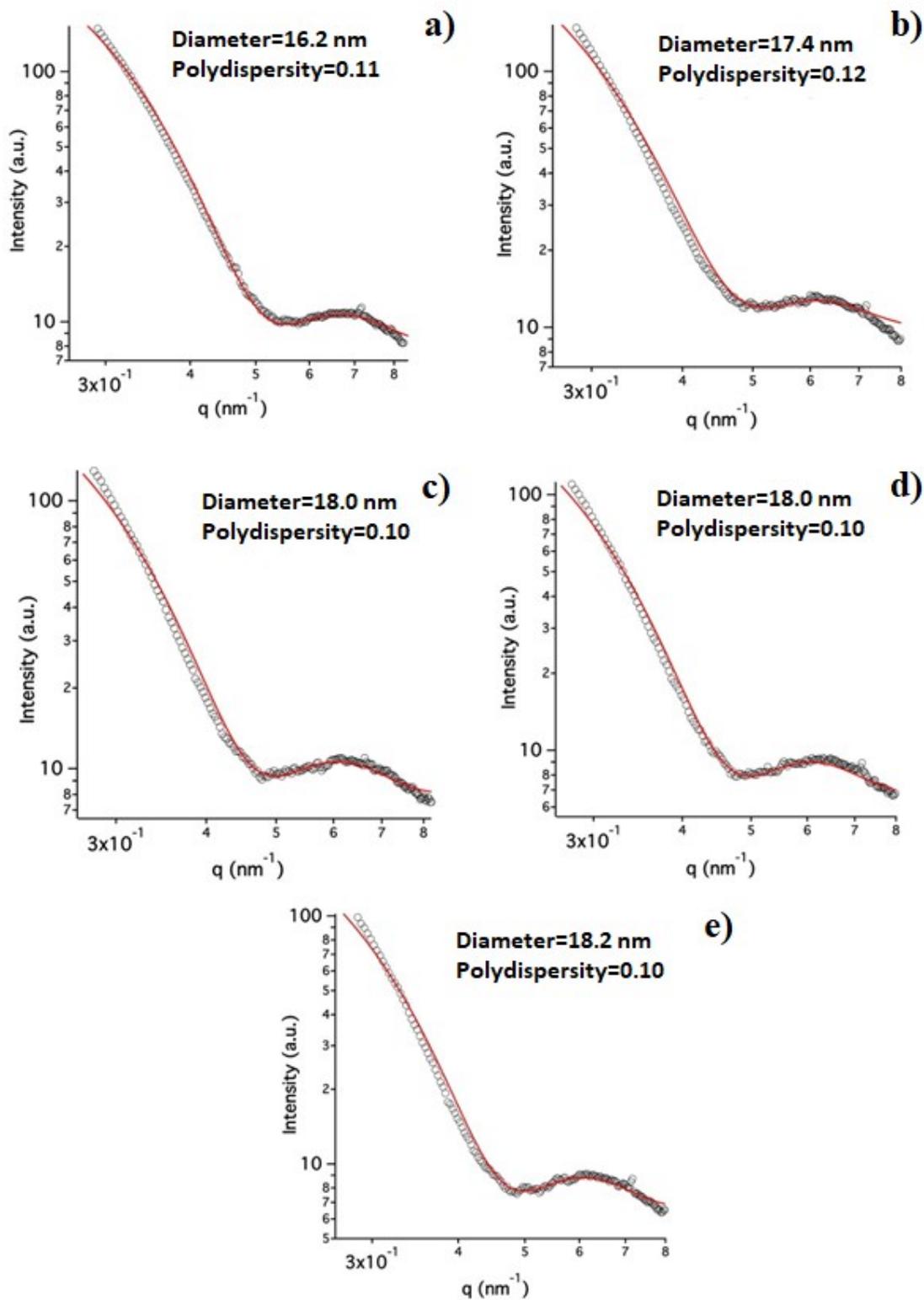


Figure S3. 1D SAXS curves (in green) and corresponding fitted curves (in blue) for sample A-HCl after a) 0h; b) 0.33 h; c) 1.25 h; d) 2.25 h; e) 6.25 h.

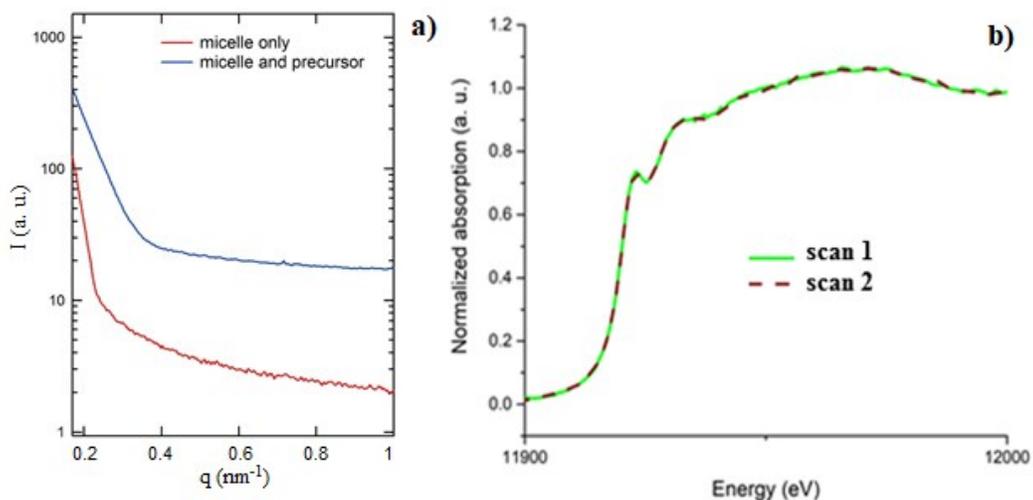


Figure S4. a) 1D SAXS profiles for polymer P18226-S2VP in toluene and micelles loaded with gold precursor. b) Two XANES spectra from sample A recorded one after the other (10 min. acquisition time). The data suggest no obvious influence of the X-ray beam (e.g. reduction of the Au precursor) within the acquisition time.

3. Transmission Electron Microscopy (TEM)

Figure S5 shows the micelles formed by dissolving the PS-*b*-P2VP polymer in toluene above the critical micelle concentration (CMC)¹. Spherical polymer micelles form a close-packed hexagonal array where P2VP cores are embedded in the PS matrix. As a result of the low Z-contrast it was not possible to obtain values of the micelles' core for sample A.

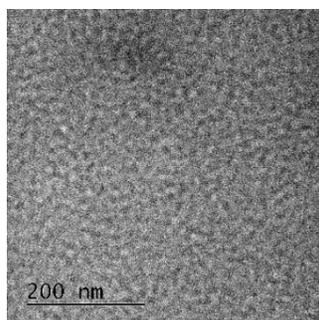


Figure S5. TEM micrograph of sample A without HAuCl_4 precursor. White P2VP cores are embedded in the PS matrix (dark).

4. References

- (1) Spatz, J. P.; Sergei, S.; Martin, M. L. Ion-Stabilized Block Copolymer Micelles: Film Formation and Intermicellar Interaction. *Macromolecules* 1996, 29, 3220–3226.

5. Raw data

Raw spectral data can be found at the following address: <http://tiny.cc/CP-ART-06-2019-003473>