

Supporting Information

Synthesis of Silver Nanocubes in a Hydrophobic Binary Organic Solvent

Sheng Peng, and Yugang Sun^{*}

ADDRESS: Center for Nanoscale Materials, Argonne National Laboratory, 9700 South Cass
Avenue, Argonne, Illinois 60439

CORRESPONDING AUTHOR'S EMAIL ADDRESS: ygsun@anl.gov

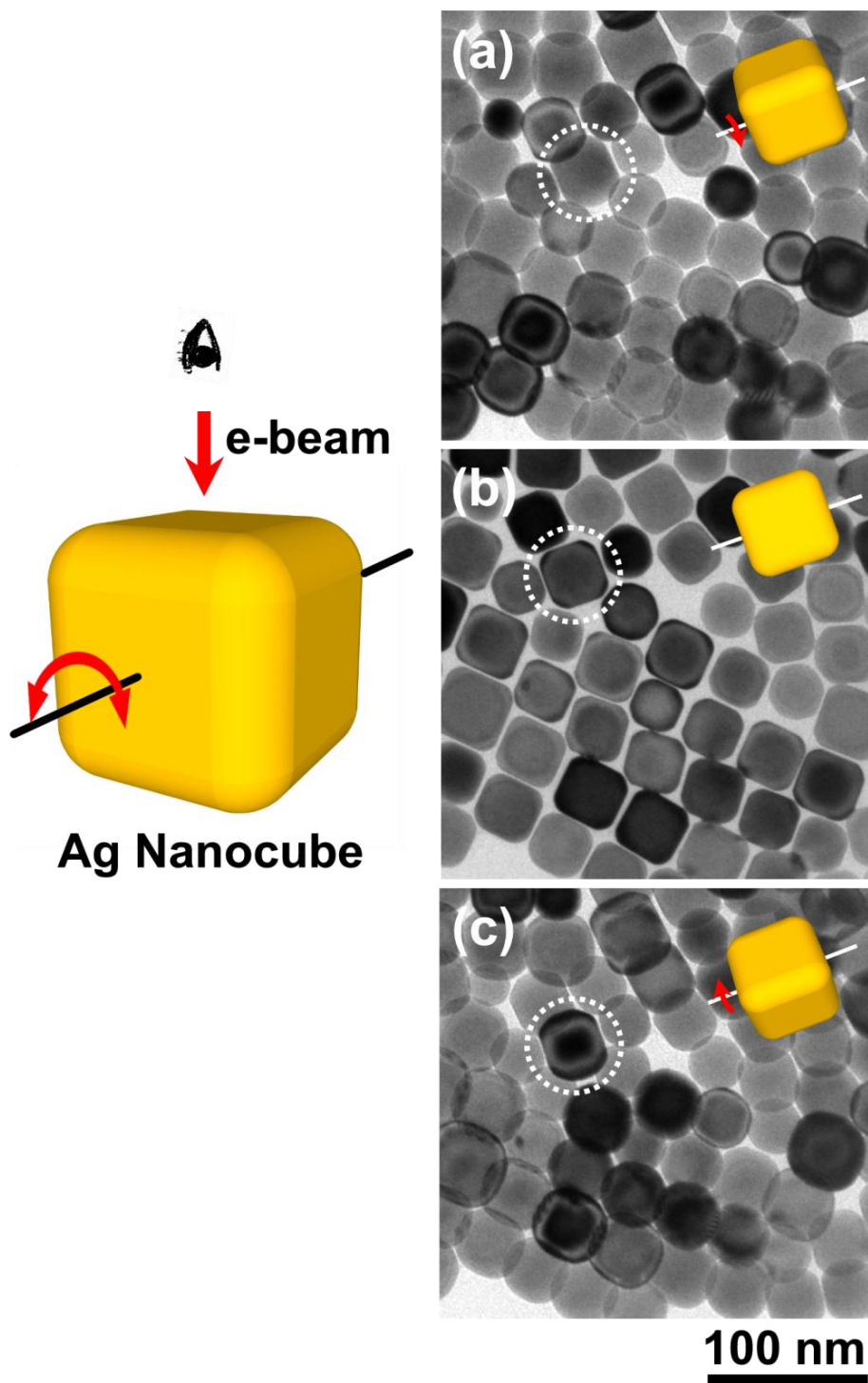


Figure S1. TEM images of the as-synthesized Ag nanocubes (same as those shown in Figure 1) at different tilting angles: (a) -35° , (b) 0° , and (c) 35° . The schematic illustrations in (a-c) correspond to the nanocube (highlighted by dotted circles) at different tilting states.

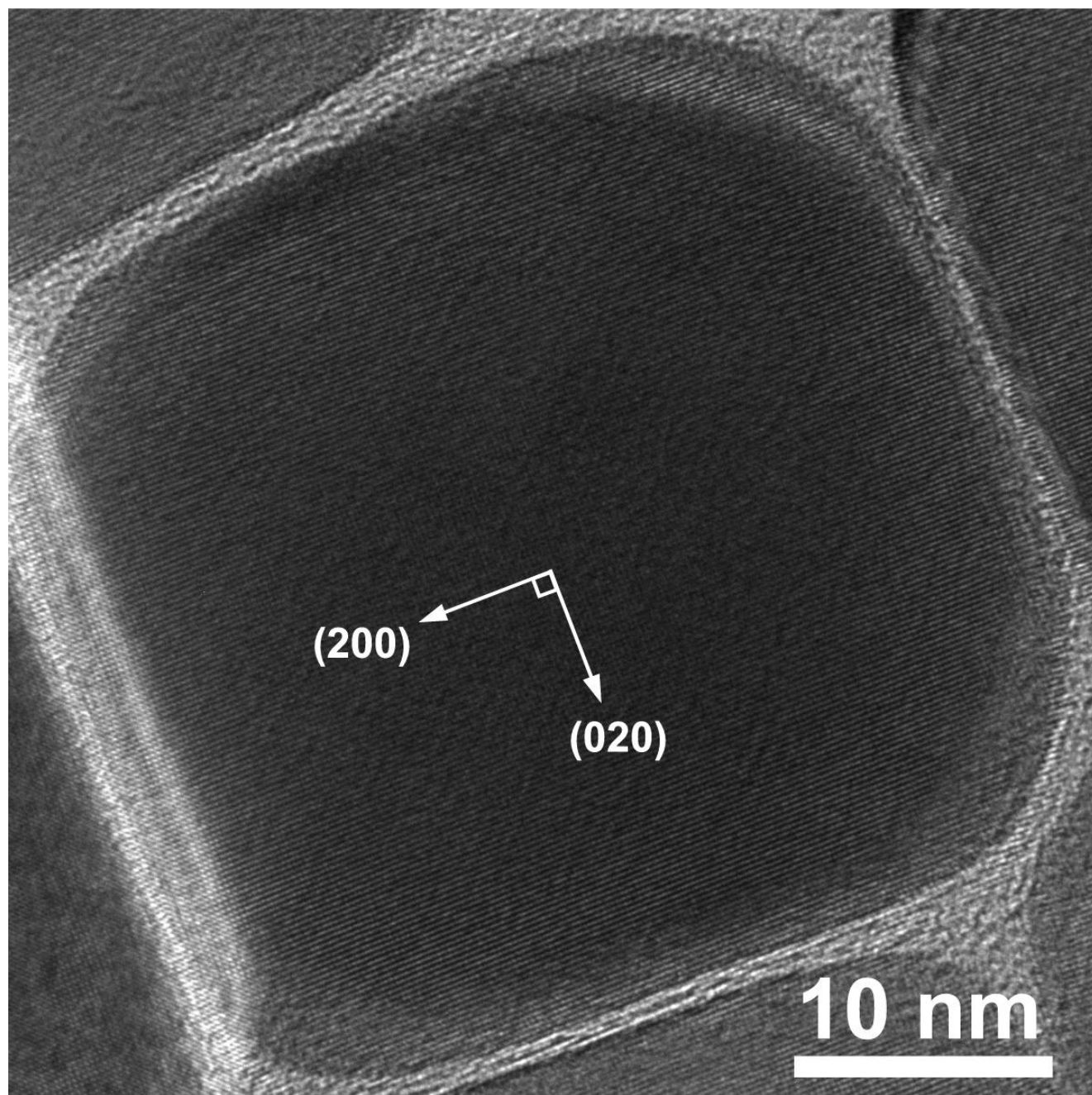


Figure S2. HRTEM image of an individual Ag nanocube.

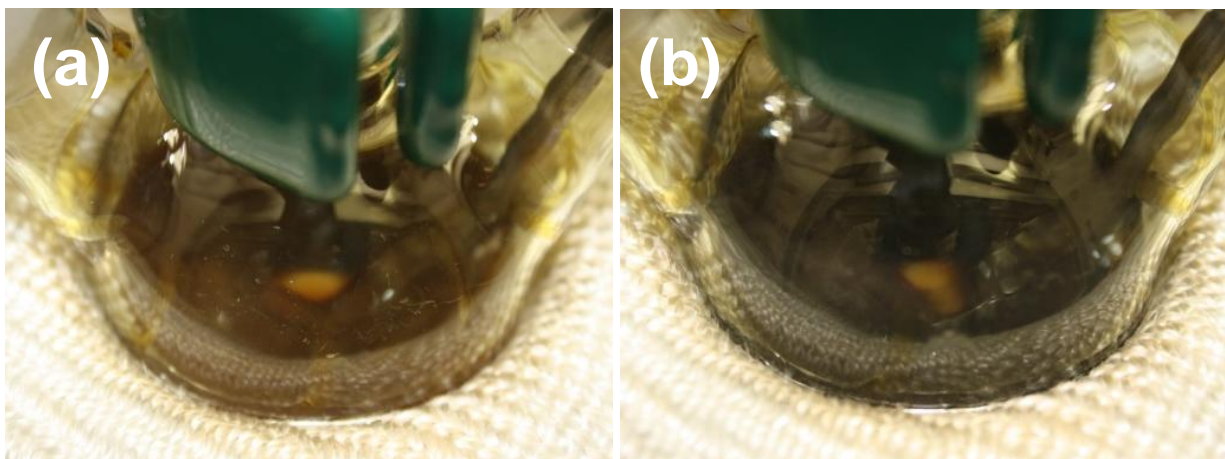


Figure S3. Photographs of the reaction solution at (a) 3 s and (b) 1 min after the injection of AgNO_3/OAm solution.

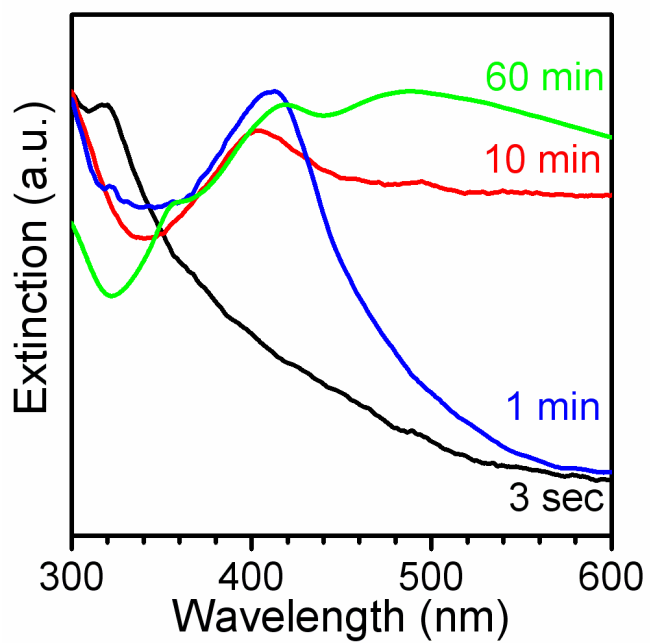


Figure S4. Extinction spectra of samples formed at various times: 3 s, 1 min, 10 min, and 60min.

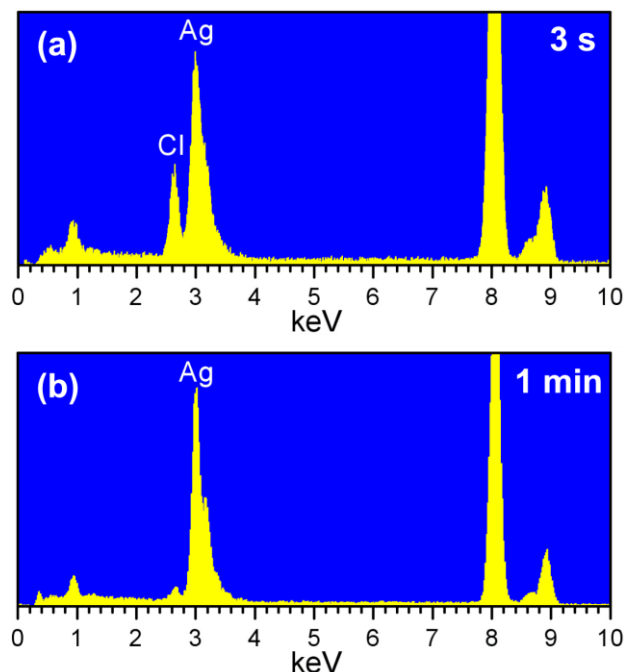


Figure S5. EDS analyses of an assembly of many nanoparticles over an area of $\sim 1 \mu\text{m}^2$ obtained at (a) 3 s and (b) 1 min after the injection. Quantitative analysis gives the atomic concentration of Cl $\sim 20\%$ for (a), which is smaller than the real Cl-content at this stage due to the quick reduction of AgCl under electron beam during the extended exposure (~ 60 s). In contrast, the atomic concentration of Cl is $\sim 1\%$ for (b), which is within the instrumental error range of the analysis, indicating that the AgCl nanoparticles have been completely reduced to Ag nanoparticles at 1 min. The Cu peaks at 0.93 keV ($L\alpha$), 8.04 keV ($K\alpha$) and 8.91 keV ($K\beta$) are from the TEM copper grids of which the nanocubes are deposited on. The small peak at ~ 2.6 - 2.7 keV in (b) is from the pronounced weak Ag L α line at 2.63 keV in the absence of Cl $K\alpha$ line at 2.62 keV.

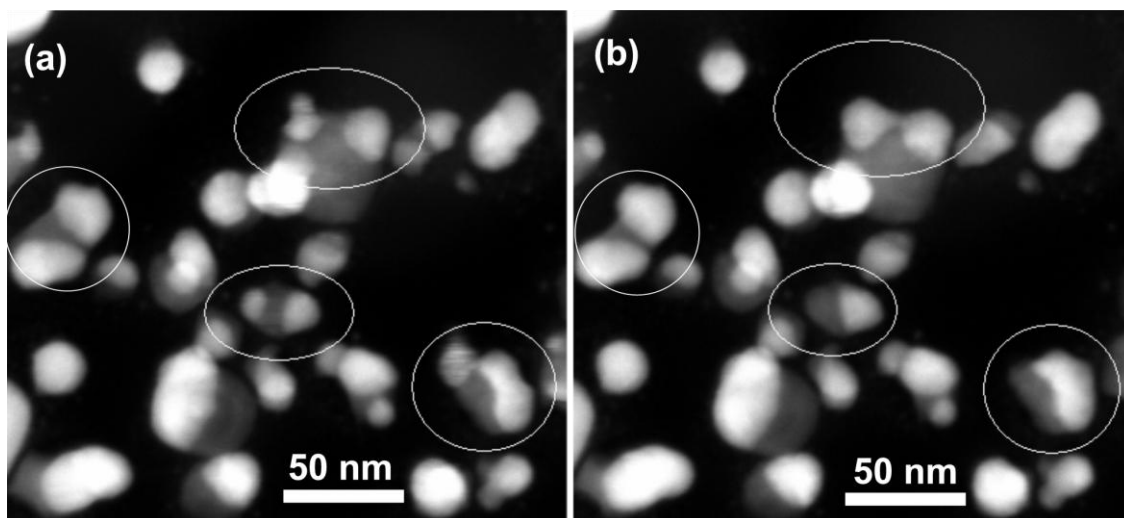


Figure S6. High-angle annular dark field scanning transmission electron microscopy (HAADF-STEM) images of the sample (same as that shown in Figure 2a) formed at 3 s after it was exposed to the electron beam for different times: (a) < 1 s and (b) 30 s. Ag components and AgCl components correspond to the spots with brighter and grayer contrasts, respectively. The white circles highlight several AgCl nanoparticles that were undergone quick morphological change under the electron beam. It is worth noting that the AgCl nanoparticles are very easily to be reduced with the electron beam. For example, the AgCl nanoparticles have already been partially reduced during the period (< 1 s) that the scanning TEM image was acquired (frame a).

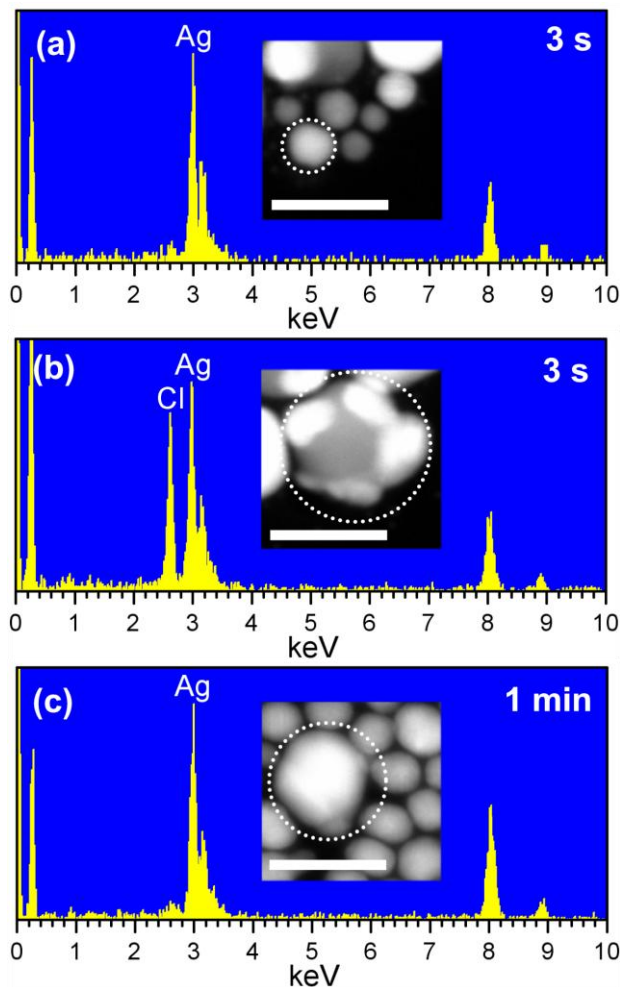


Figure S7. EDS spectra of the samples formed at different reaction times: (a, b) 3 s and (c) 1 min. Frames (a) and (b) show representative spectra of the smaller and the larger nanoparticles in the same sample, clearly showing that the smaller particles are composed of pure Ag and the larger ones are composed of both Ag and Cl. Frame (c) presents a representative spectrum of the sample formed at 1 min. Quantitative analysis gives the atomic concentration of Cl is $\sim 0.5\%$, which is within the $\pm 1\%$ error range of the analysis, indicating that the AgCl nanoparticles formed at 3 s are completely reduced to Ag nanoparticles at 1 min. Insets are the corresponding HAADF-STEM images and the dotted circles highlight the areas where the EDS spectra were taken. All the scale bars represent 50 nm. The Cu peaks at 8.04 keV and 8.91 keV are from the copper grids of the TEM samples. The small peak at ~ 2.6 keV in (a) & (c) is from the pronounced weak Ag L α line at 2.63 keV in the absence of Cl K α line at 2.62 keV.

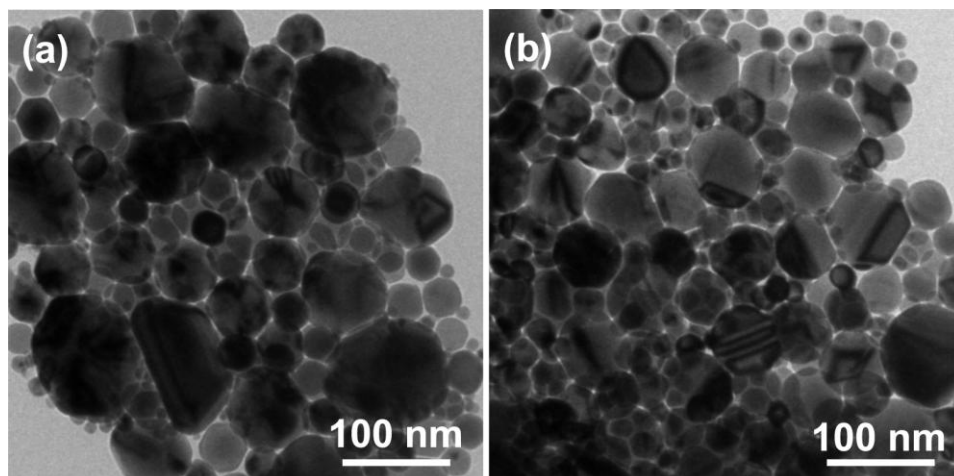


Figure S8. TEM images of the products synthesized through: (a) a synthesis by injecting 1 mL OAm solution of AgNO_3 (0.2 M) into a hot solution of 9.0 mL of OAm containing 0.3 mmol of DDAC and (b) a synthesis by injecting 0.4 mL OAm solution of AgNO_3 (0.5 M) into a hot solution of 9.6 mL of OE containing 0.3 mmol of DDAC. Other reaction conditions are the same as those of Figure 1.

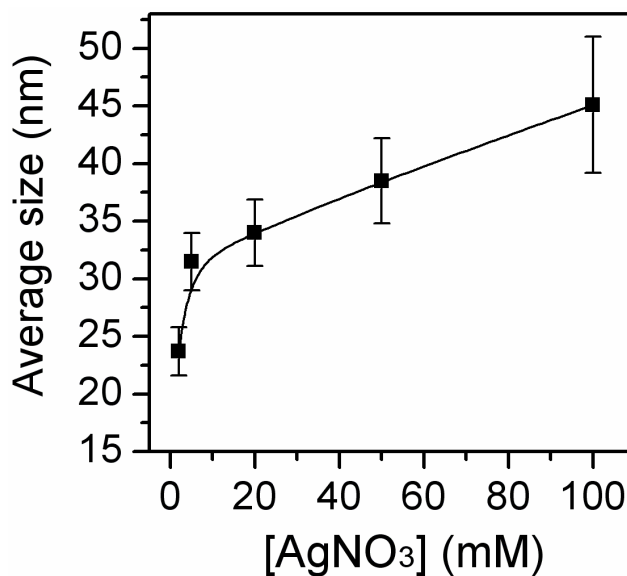


Figure S9. Dependence of average size and size distribution of the resulted Ag nanocubes (nm) on the AgNO_3 concentration ($[\text{AgNO}_3]$, mM) during the synthesis. The error margin represents standard size deviation of the synthesized nanocubes.