Electronic Supplementary Material (ESI) for Nanoscale.

Supporting Information

Article:

" Nanometer precise adjustment of the silver shell thickness during the automated Au/Ag core-shell nanoparticle synthesis in micro fluid segment sequences"

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Experimental:

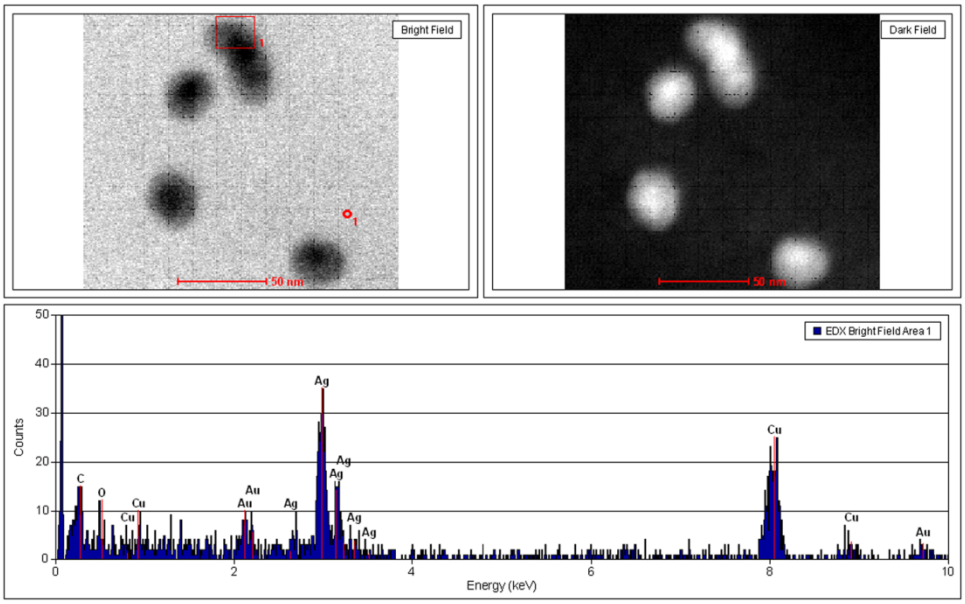
Characterization: The obtained core/shell particle samples were characterized by the usage of the methods and instruments listed below. The optical properties of both, Au core nanoparticles and Au/Ag core-shell nanoparticles, were characterized additionally to the in situ spectrophotometry by offline UV–vis spectrophotometry (Specord 200, Analytik Jena AG, Jena, Thuringia, Germany). The electron microscopic methods SEM (Hitachi S-4800 FE-SEM, Hitachi High Technologies America, Inc., Schaumburg, IL) and TEM (JEOL JEM-1400, Tokyo, Japan, Philips Tecnai S20, FEI, Hillsboro, USA/OR) were used to elucidate the shape and size of the obtained core-shell particles. Both techniques were furthermore utilized to verify the measured narrow size distributions. The particle size distribution spectra were measured by differential centrifugal sedimentation (DCS, DC 20000, CPS Instruments Inc., Newtown, PA). To give further evidence of closed silver shells around the Au nanoparticles, XPS measurements were performed in an UHV chamber (base pressure < 2×10^{-10} mbar) using monochromated AlKα radiation (hν = 1486.7 eV) from a PHI 10-610 X-ray source combined with an Omicron XM1000 monochromator. The spectra were recorded by a hemispherical electron energy analyser (Omicron, Type EA 125) equipped with 7 channeltrons. The escape depth of the emitted photoelectrons from the sample surface is only a few nm and the detection limit is about 0.1 at.%. XPS core level spectra were recorded under normal emission, with a pass energy of 15 eV resulting in a total energy resolution < 0.6 eV (FWHM of $Ag3d_{5/2}$ of a clean polycrystalline silver sample).
For the recording of the EDX (energy dispersive X-ray spectroscopy) data, the particle sample was dropped on a Cu-C-grid with a mesh size of 200 µm. The chosen particle population had an average shell thickness of 6 nm. The analyses were carried out using a Philips Tecnai S20 (FEI, Hillsboro, USA/OR) TEM.

Results:

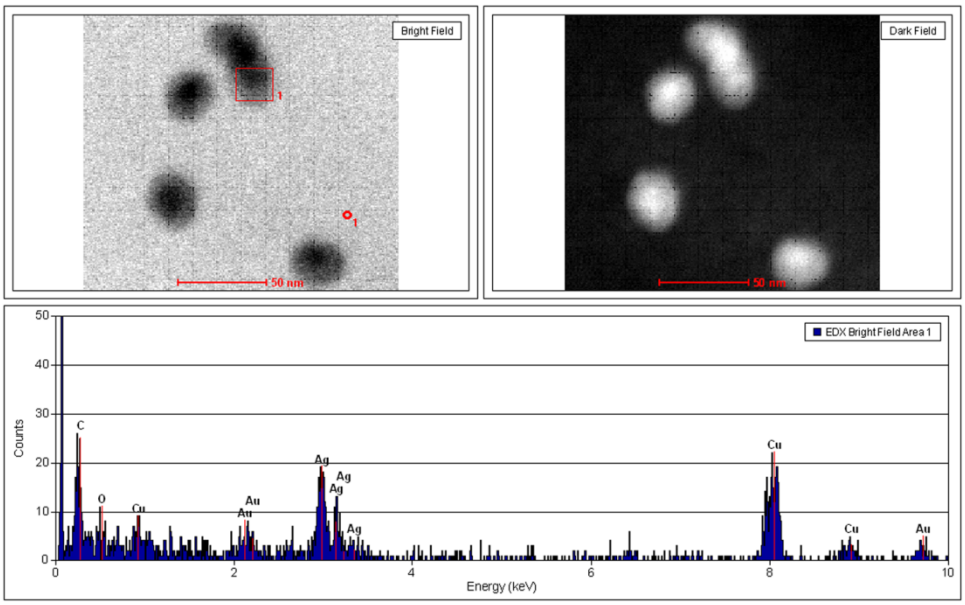
EDX: area scans on 5 different Au/Ag core-shell nanoparticles were carried out to confirm the presence of both metals within the core-shell nanoparticles. The elemental analysis shows distinct peaks of gold and silver. The integration was limited by a certain sample drift and was set to 60 seconds.

Fig. S1 a-e:

a


Bright Field
Dark Field
50 nm
EDX Bright Field Area 1
Counts
Energy (keV)
C
O
Cu
Au
Ag

b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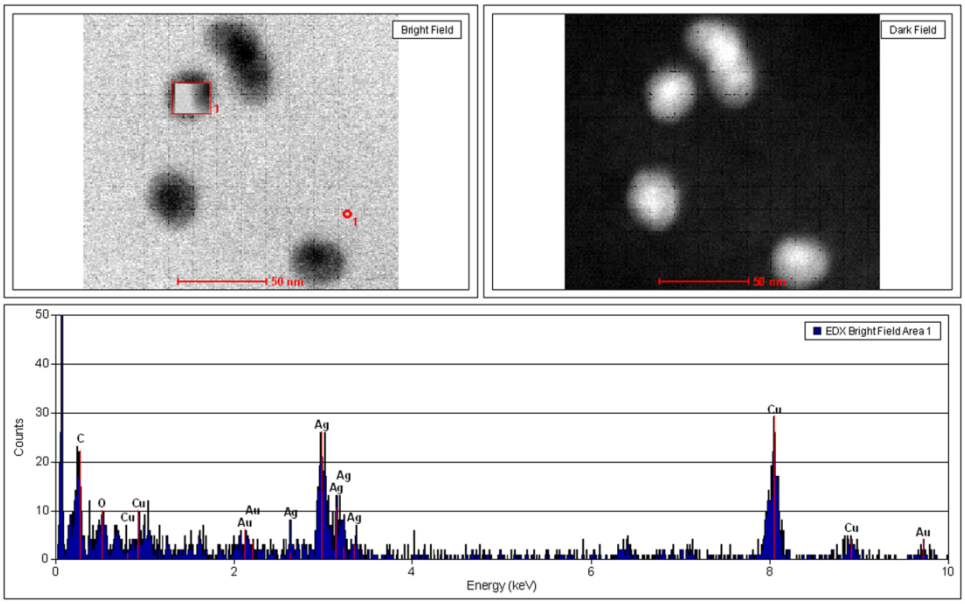
EDX Bright Field Area 1
Counts
Energy (keV)
C
O
Cu
Au
Ag

c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50 nm
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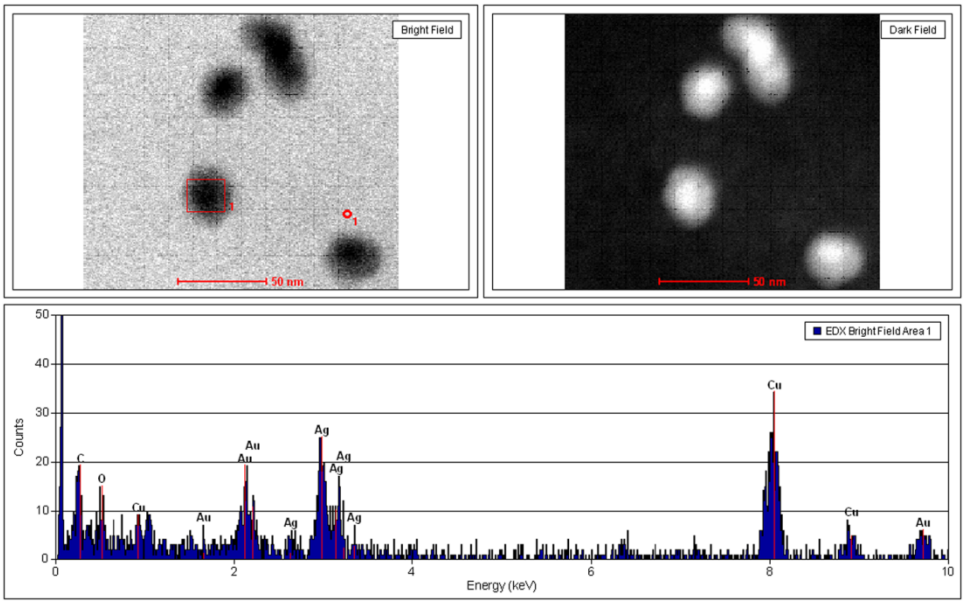
Counts
Energy (keV)
C
O
Cu
Au
Ag

d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Dark Field
50 nm
50 nm
EDX Bright Field Area 1
Counts
Energy (keV)
C
O
Cu
Au
Ag

e

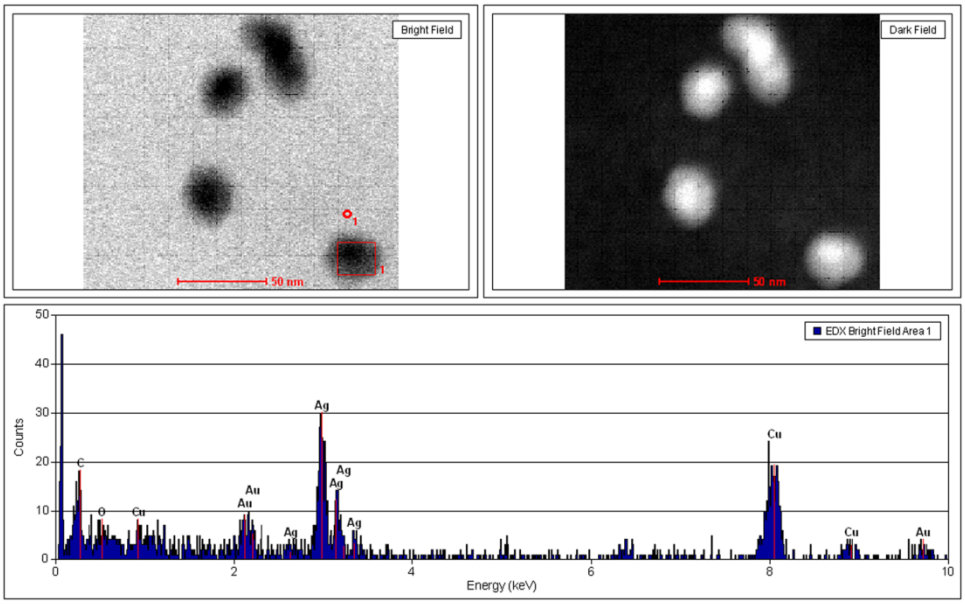

Fig. S1 a-e: EDX area scans on five different Au/Ag core-shell nanoparticles.