Synthesis of Au-Ag alloyed nanoparticles

Adapted from J. Chem. Educ. 97 (2020), 3778 - 3783

Au, Ag, and alloyed particles were prepared by the Turkevich method [Frens, G. Controlled Nucleation for the Regulation of the Particle Size in Monodisperse Gold Suspensions. Nature, Phys. Sci. 1973, 241, 20−22].

- Place 25 mL of water into a 100 mL round-bottom flask and boil using a heating plate with dry-sin under air. Quickly add the solutions of $NaAuCl_4$ and $AgNO_3$ (totalling 0.25 mM) to the round-bottom flask and allow 2 min to equilibrate. Pure particles are synthesized by the addition of $NaAuCl_4$ or $AgNO_3$ only, while alloys are prepared by the addition of the two metal solutions in the appropriate proportion.
- Once boiling again, add a solution of sodium citrate (1.0 mL, 142.5 mM) quickly to the flask with stirring.
- The reaction is allowed to proceed for another 20 min, while students take notes over the color changes during the synthesis. Remove the reaction flask from heat and allow to cool to room temperature.

Different Au-Ag compositions must be prepared accordingly to the following Table.

Student group	Gold Molar Fraction (GMF)	Volume of 20 mM $NaAuCl_4$ (μL)	Volume of 20 mM $AgNO_3$ (μL)	Volume of 142.5 mM Sodium citrate (mL)
1	1.00	312.5	0.0	1.0
2	0.75	234.4	78.1	1.0
3	0.50	156.3	156.3	1.0
4	0.25	78.1	234.4	1.0
5	0.00	0.0	312.5	1.0

- *Acquisition of UV-Vis Spectra*. The blank and nanoparticle spectra were acquired by pipetting 1.5 mL of water (as a blank) or nanoparticle solution into a semi-micro cuvette and spectra were collected from 350 to 750 nm.
- *Analysis of the data*. Accordingly to J. Chem. Educ. 97 (2020), 3778 – 3783, the UV-vis spectra must be normalized with respect to the maximum of the localized surface plasmon resonance (LSPR) to observe the shift of the band with the gold molar fraction (GMF). The dependence of the LSPR peak wavelength on alloy composition is studied plotting the position of the maximum of the LSPR versus GMF. Find the best way to fit these data.

Optional:

If you want to study in deep the LSPR phenomenon, the paper J. Chem. Educ. 97 (2020), 3778 – 3783 (and its Supplementary Informations) presents the way to simulate the LSPR of Au-Ag nanoparticles using the software MiePlot. Check the agreement between our experimental data and those calculated using the software MiePlot.

Synthesis of SiO_2 by sol-gel

Adapted from J. Chem. Educ. 71 (1994), 599 – 602

- In a 10 mL vial, add 2.0 mL of TetraEthOxySilane (TEOS) and 2.0 mL of EtOH 99.8%. Stir vigorously for 1 minute.
- In an external becker, prepare the solution of the catalyst: put 10 mL of distilled water into the becker and add 3 dops of concentrated HCl (gropus 1 – 3) or concentrated NH_4OH (groups 4 – 5). Mix carefully the solution.
- Add 2.0 mL of the aqueous solution with the catalyst into the vial with TEOS and EtOH.
- Under vigorous stirring, put the open vial on a hot plate at 80 °C for at least 1 hour, until the solution becomes clear.
- To form the alcogel, the open vial must be left on the heating plate for at least 4 additional hours (operation performed by the teacher).
- The alcogel will be converted into xerogel by calcination at 500 °C for 5 h (this step will be performed during the following week).