

## Preparation and characterization of Ag, Pd and Pt nanoparticles for labeling of ultrathin sections in TEM microscopy

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Many methods of preparation and characterization of colloidal metals have been developed and a variety of metallic nanoparticles with different shapes and sizes were reported. For many applications size and shape of nanoparticles are important, but their size distribution is crucial. We plan to use colloidal metallic nanoparticles as immunospecific markers in TEM microscopy. The objective of our work was to prepare colloidal metallic nanoparticles with suitable size ranging from 5 to 12 nm.

Ag colloids were obtained by reduction of  $\text{AgNO}_3$  with  $\text{NaBH}_4$  [1]. Briefly, 3.5 mg of  $\text{NaBH}_4$  was dissolved in 75 ml of deionized water and cooled to 2 °C. After reaching this temperature, 9 ml of  $2.2 \times 10^{-3} \text{M}$  solution of  $\text{AgNO}_3$  was added dropwise during vigorous mixing. The resulting colloidal solutions were yellow.

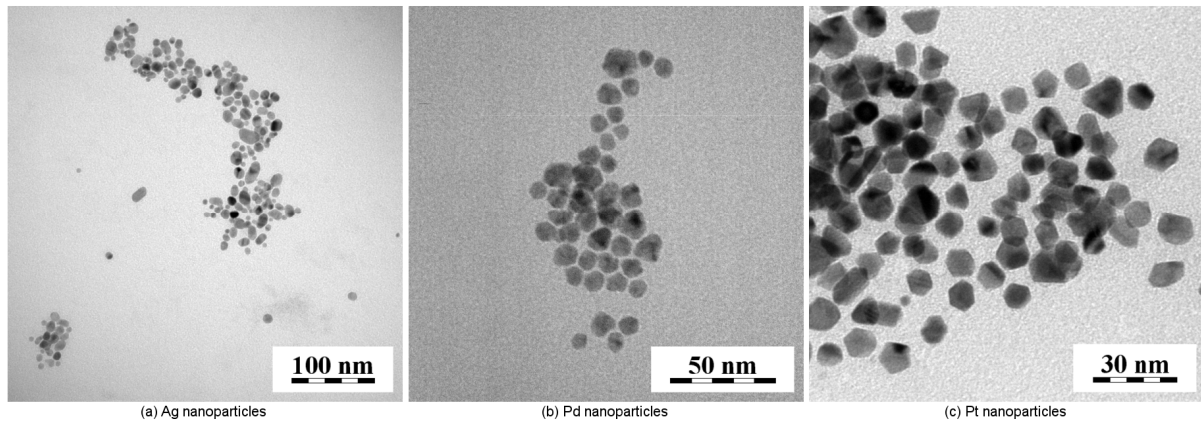
The syntheses of Pd colloids were based on a method consisting in reduction of  $\text{PdCl}_2$  with sodium citrate [2]. In the typical synthesis, 0.165 g of  $\text{PdCl}_2$  was dissolved in 20 ml of 1N HCl and 2 g of sodium citrate were dissolved in deionized water. Then 7.5 ml of prepared  $\text{PdCl}_2$  solution and 15 ml of sodium citrate solution were added to the 52.5 ml of deionized water, heated to the boiling point and kept under the reflux for 6 h. The resulting colloidal solutions were light yellow.

Pt colloids were synthesized as reported elsewhere [3]. Aqueous solution of  $\text{K}_2\text{PtCl}_4$  and  $\text{C}_{14}\text{TABr}$  were mixed in a 20ml vial and the mixture was heated at 50 °C until the solution became clear. Ice-cold  $\text{NaBH}_4$  was added and the vial was capped with a rubber septum. The  $\text{H}_2$  gas was released through a needle in the septum for 10 min. Then the needle was removed and the solution was kept at 50 °C for 6 hours. The resulting colloidal solutions were dark brown.

All prepared colloids were characterized by TEM microscopy (microscope Tecnai G2 Spirit Twin 12, FEI): 2  $\mu\text{l}$  of colloidal solution was dried on a microscopic grid covered with thin carbon film and observed in TEM. For all colloids, four representative micrographs with the same magnification were processed to obtain equivalent diameters for size distributions.

According to TEM micrographs we prepared colloidal silver nanoparticles with an average size of 8 nm, palladium nanoparticles with an average size of 10 nm and platinum nanoparticles with an average size of 12 nm. In all colloids the size distribution was quite sharp and colloidal solutions were stable for at least several weeks. The results indicated that all prepared colloidal nanoparticles are suitable for using as immunospecific labeling in TEM.

1. B. Vlkova et al., J. Phys. Chem. 97 (1993) p9719.
2. J. Turkevich et al., Science, 169 (1970), p873.
3. H. Lee et al., Angew. Chem. Int. Ed. 45 (2006) p7824
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**Figure 1.** Representative TEM micrographs of colloidal Ag (a), Pd (b) and Pt (c)