

Tuning Size And Morphology Of Nanostructures: The Fundamental Role of Transmission Electron Microscopy In Synthesis Control

Muñoz, Vanesa ^{1*}, González, Jimena S. ¹, and Hoppe, Cristina E. ¹

¹ Institute of Materials Science and Technology (INTEMA), University of Mar del Plata and National Research Council (CONICET), Colón 10890, 7600. Mar del Plata, Argentina.

* Muñoz, Vanesa.: vanem1973@gmail.com

In this work, the paramount importance of synthesis variables on shape, size and polydispersity of metal and oxide nanostructures is shown with an especial focus put on the role of electron microscopy as a powerful tool of characterization. Two types of systems, ferrogels and gold nanostructures were analyzed.

Ferrogels with well-dispersed magnetic nanoparticles (NPs) were obtained by the infusion of iron salts in cross-linked polyvinyl pyrrolidone (PVP) hydrogels followed by coprecipitation. Two different PVP concentrations (PVP 8% and PVP 16%) in the initial solution were used and the gelation of the polymer was carried out with ammonium persulfate.

On the other hand, gold nanostructures were obtained by mixing oleylamine, HAuCl₄ and tri(isopropylsilane) in tetradecane, the solution is left without agitation for 12hs at room temperature and 80 °C.

Magnetic gel microstructure was studied by scanning electron microscopy in a JEOL JSM-6460 at 15 kV (Figure 1A-D). In order to observe iron oxides nanoparticles inside, magnetic gel was ground, resuspended in water and sonicated. Dispersions were placed on 200 mesh carbon-stabilized Formvar-film copper and observed by transmission electron microscopy, in a JEOL JEM 2100 at 200 Kv (Figure 1B-C-E-F). Final size of NPs was controlled by concentration of the polymer in the initial aqueous formulation.

Both gold dispersions, room temperature (Figure 2A₁₋₂) and 80°C (Figure 2B₁₋₂) were placed on 200 mesh carbon-stabilized Formvar-film copper grids and were studied by transmission electron microscopy in a JEOL JEM 2100 at 200 kV. Completely different structures were obtained by changing reaction temperature between room temperature and 80°C. Difference in morphology was attributed to a change in the stability of a reaction intermediate complex.

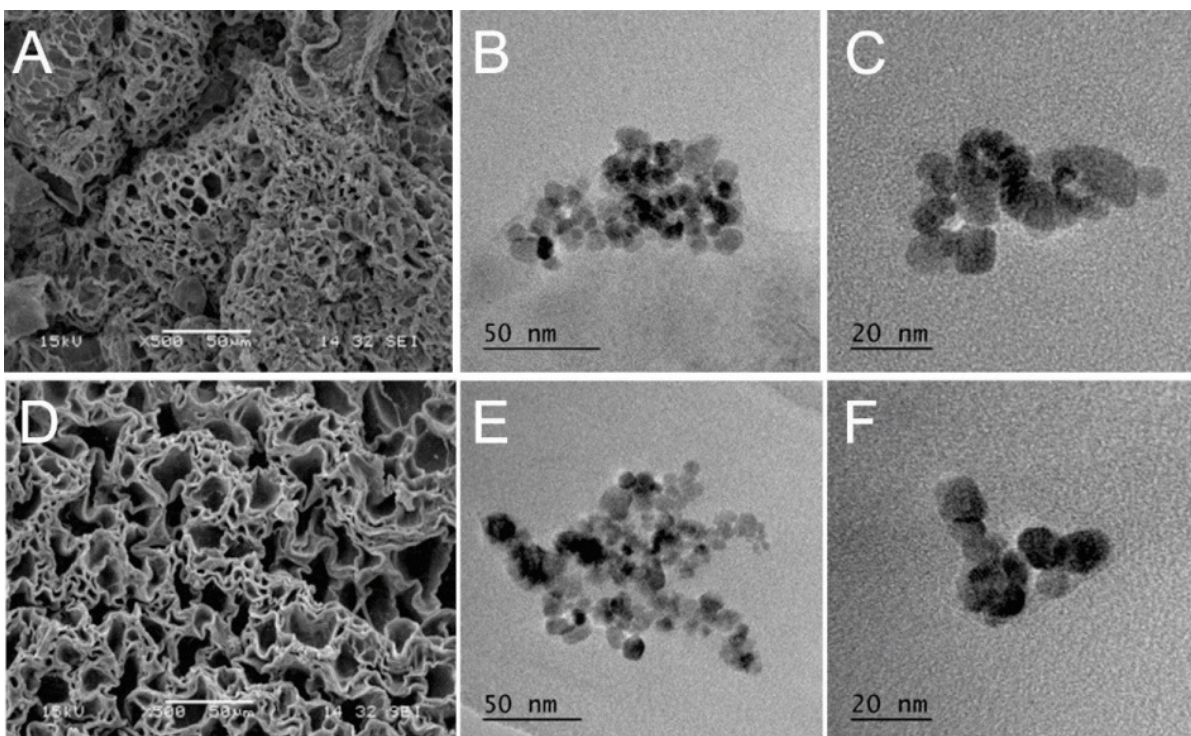


Figure 1. A) SEM image of 8% PVP magnetic gel, B) TEM image of NPs inside de 8% PVP ferrogel, C) Higher magnification of iron oxides nanoparticles inside de 8% PVP gel, D) SEM image of 16% PVP magnetic gel, E) TEM image of NPs inside de 16% PVP ferrogel F) Higher magnification of iron oxides nanoparticles inside de 16% PVP

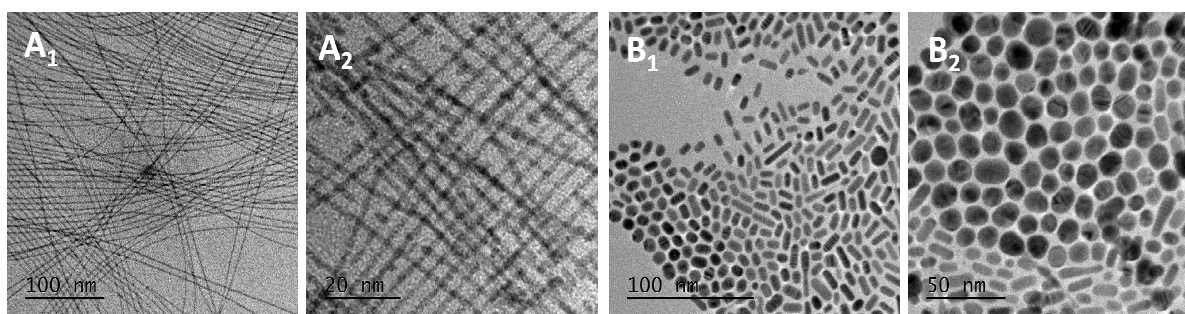


Figure 2. TEM image showing formation of A₁₋₂) Au ultrathin nanowires obtained by heating the reaction media at 25°C and B₁₋₂) Au nanoparticles obtained at 80°C.