

## Synthesis and Characterization of Metallic Nanoparticles Stabilized with Polyvinylpyrrolidone

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The study of ultra-fine particles has been extensive in the last years for its unique properties and potential technological applications in different areas[1-3]. Magnetic ultra-fine metallic particles have been broadly investigated by its great coercive force and residual magnetization [2]. Many methods have been developed for the synthesis of nanoparticles, however there are several important problems to be solved: to prepare nanoparticles with a uniform size, to control the chemical and structural nature of these and to keep the particles well dispersed. In the objective of the present work is the synthesis and characterization of metallic nanoparticles of Fe, Co and Ni and its bimetallic compounds (FeCo, CoNi and FeNi) by the method of salt reduction stabilized with polyvinylpyrrolidone (PVP) and to study its magnetic properties by EPR.

The nanoparticles of Fe, Co and Ni and the FeCo were obtained by the reduction of the metal acetylacetonate: Ni(acac)<sub>2</sub>, Co(acac)<sub>3</sub> and Fe(acac)<sub>3</sub> with sodium boron hydride (NaBH<sub>4</sub>) in ethanol and they were stabilized with polyvinylpyrrolidone (PVP = 40.000) to 25 °C. The synthesis was carried out in inert atmosphere placing in a ball the precursor, the stabilizer, the reducing agent and the solvent under continuous agitation for 24 hours until the solution turns brown or black. The characterization of the obtained systems was carried out by: Transmission Electron Microscopy (TEM), Electron Paramagnetic Resonance (EPR) and Fourier Transform Infrared Spectroscopic (FT-IR). The samples for transmission electron microscopy were prepared by placing a drop of the solution on a holey carbon grid and immediately before observation in order to avoid possible oxidation effects. However, samples observed a period after prepared did not show any changes.

The IR spectra shows the disappearance of the bands (C = O and C = C) present in the precursors, this implies that in the reactions it is possible to displace the acac totally - to stabilize the particles in the polymeric matrix. The figure 1 show the dark field images for each metallic system. The system Fe/PVP presented a wide distribution of size particle in the range of 4-12 nm. In the Ni/PVP material the size and distribution was in the range of 7-15 nm. In the case of Co/PVP a narrow distribution (2-6 nm) was observed. For all bimetallic systems the distribution was narrow with particle size in the range of 2-5nm. Electron diffraction patterns of these samples identified the formation of the metallic compounds and the bimetallic compounds respectively. The formation of oxide was detected for the Fe nanoparticles, but not for the case of Ni, Co or the intermetallic compounds. For these the formation of the intermetallic compound was identified by electron diffraction. Figure 2 shows the unnormalized EPR spectra for Fe, Co and FeCo supported onto a PVP matrix. The spectrum for Fe shows clearly two Lorentzian signals, a high field signal very intense around  $g = 2.017$  and a  $\Delta H_{pp} = 18.6$  mT and a less intense one at  $g = 3.857$  with  $\Delta H_{pp} = 16.5$  mT. The supported Co on the PVP matrix shows a very wide Lorentzian EPR signal with  $\Delta H_{pp} = 156.8$  mT centered around  $g = 2.182$  this is characteristic of a strong dipolar interaction among the Co ions due to a high coordination to the PVP matrix. The bimetallic FeCo shows an unique Lorentzian EPR signal centered around  $g = 2.014$  and with a linewidth  $\Delta H_{pp} = 50.0$  mT whose value is between those obtained for the separated metals, which is indicative that the Fe has

diluted the strong dipolar interaction present for Co ions. Another interesting aspect standing out is that the intensity of the signal decreases considerably for the FeCo bimetallic compound. This could be related to an antiferromagnetic ordering of the iron and the cobalt that could finally be responsible for a ferrimagnetic order in the system.

#### References

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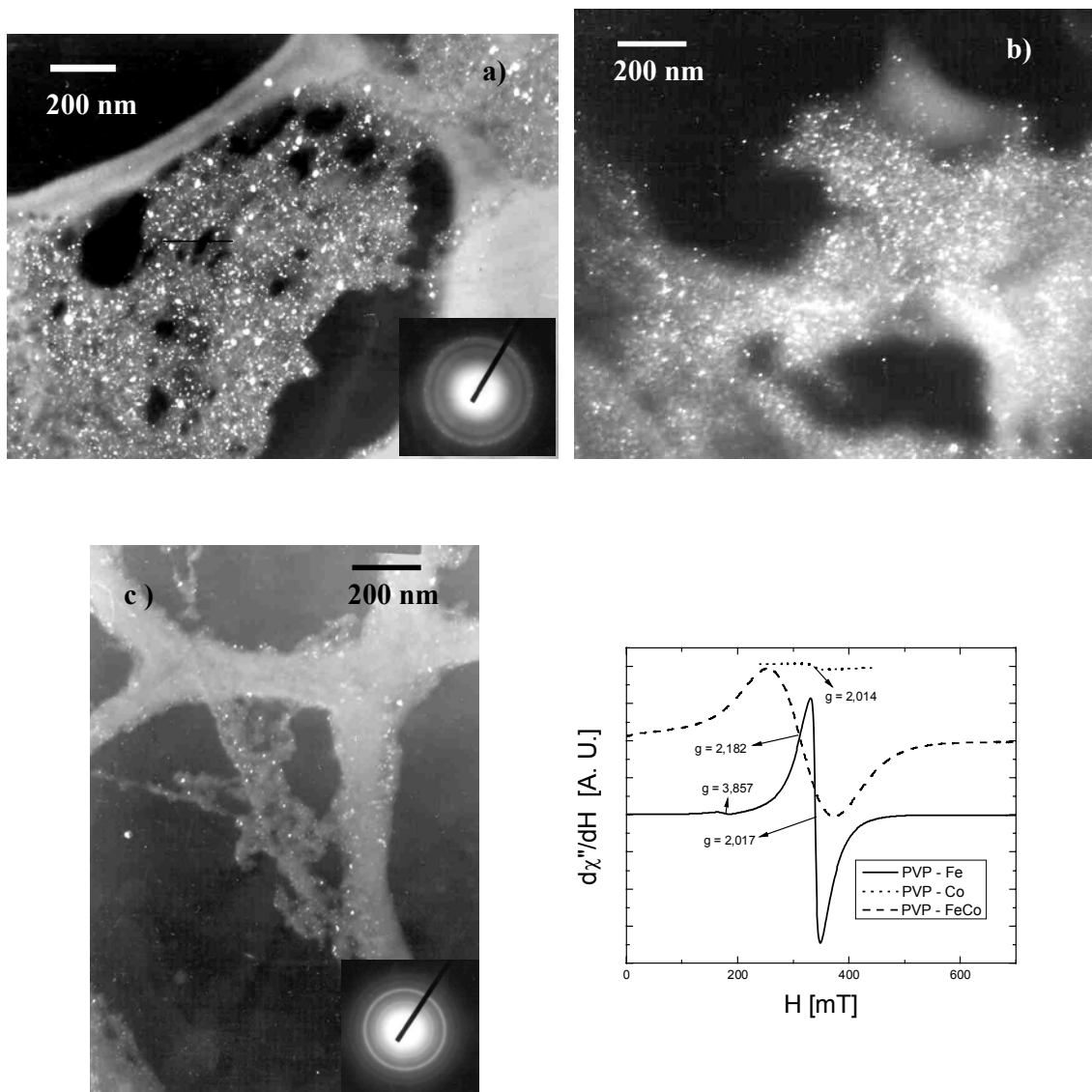


Fig. 1. Nanoparticles of  
 a) Fe b) Ni c) Co stabilized  
 with PVP

Fig. 2. EPR of Nanoparticles de  
 Fe, Co and FeCo stabilized with  
 PVP