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Platinum nanoparticles obtained by two-phase method: synthesis, characterization and electro-catalytic properties.

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Abstract –. The two-phase method has been widely used to achieve well dispersed ligand-protected Au and Ag nanoparticles, but has never been described to synthesize Pt nanoparticles. In this work we present a procedure to synthesize dedocanothiol-capped platinum nanoparticles through the two-phase method (toluene-water). The obtained nanoparticles were characterized by XRD, FTIR, TEM, DLS, XPS, XANES and EXAFS. The use of these Pt-nanoparticles as catalyst to the electro-oxidation of methanol was evaluated by cyclic voltametry.

Nanosized particles of noble metals have attracted much interest in recent years due to their unique physical and chemical properties derived from small particle sizes and uniform size distribution. One of the most common and powerful method to synthesize metal colloids in organic media is reduction of transition metal salt in the presence of stabilizing agent[1,2]. This method was described for the synthesis of Au nanoparticles (NPs)[1] and further adapted to Ag-NPs[2], but there are no reports regarding their use to prepare Pt-NPs. In this work we report by the development of an alternative route to synthesize dodecanethiol-stabilized Pt NPs, with narrow and controllable size distribution, by a two-phase route. The synthesis was based on a phase-transfer (water to toluene) of $[PtCl_6]^{2-}$, followed by reduction and surface protection with dodecanethiol (DT). Controlling the time of reduction of metal precursor possibilities a control in the medium diameter of obtained samples. Three different samples have been obtained containing medium diameter of 1.8, 2.1 and 2.3 nm (medium size obtained by both TEM and DLS analysis).

Samples have been characterized by several techniques. The XRD patterns of these samples show that metallic Pt was obtained with a fcc structure. The high-crystallinity of the samples was confirmed also by HRTEM (Figure 1). FTIR analysis show the main bands of dodecanethiol, indicating that these particles are capped by the DT molecules; TEM images and dynamic light scattering analysis of the samples indicate the occurrence of spherical nanoparticles with narrow size distribution in all samples. The XPS spectra show the characteristic doublet of the Pt 4f energy binding, confirming the formation of metal platinum in these samples, as well as the typical S signal characteristic of thiols. Important information has been obtained by X-ray absorption measurements. The XANES spectra are characteristics of Pt^0 in all samples. The EXAFS spectra (Figure 2 FT-treated EXAFS spectra) indicate a reduction in the coordination number of the first shell Pt-Pt in the nanoparticles (in comparison with the Pt-foil), attributed to the high surface to volume ratio observed in small particles. The cyclic voltammograms obtained using this Pt nanoparticle samples as working electrode show that all samples present a good eletrocatalitic behavior regarding the electro-oxidation of methanol (Figure 3), which is very important in the technology of fuel cells.

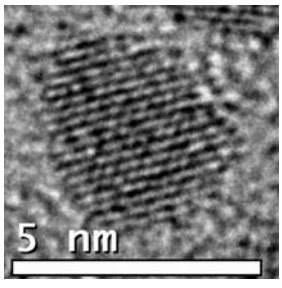

Figure 1: HRTEM images of one Pt nanoparticle.

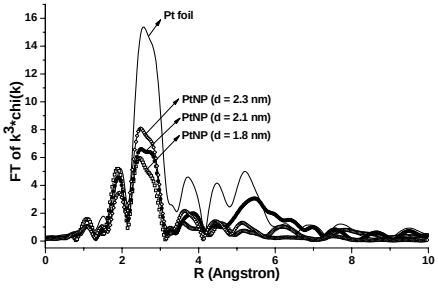

Figure 2: EXAFS spectra of all nanoparticles samples.

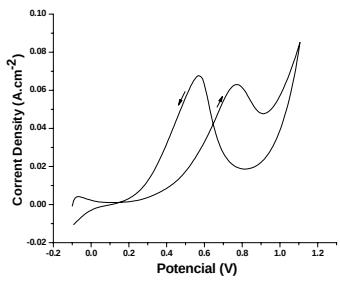

Figure 3: Cyclic voltametry in methanol-containing media.

References

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