

## ELECTROCHEMICAL SYNTHESIS AND STABILIZATION OF COBALT NANOPARTICLES

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Cobalt nanoparticles (NPs) were synthesized via electrochemical methods [1] in the presence of tetraalkylammonium salts. We describe in this work the preparation, growth, and stabilization of cobalt nanoparticles with a surfactant solution.

Size-selective synthesis of nanostructured transition metal cluster was developed by Reetz and Helbig [2] in the 90's using a simple electrochemical process. It is based on the dissolution of a metallic cobalt anode in an aprotic solvent in the presence of stabilizers like tetraalkylammonium salts, which also serve as supporting electrolyte. The nanometer dimensions of the cobalt colloids can be controlled in a simple manner by adjustment of the current density. The variation of the current density allows the production of metallic particles in the range (2-5) nm. The process is carried out in an inert atmosphere and vigorously agitated with a magnetic stirrer. It consists of the bulk metal to be transformed into metal clusters. The metal cations migrate to the cathode where reduction takes place with formation of metal particles (Fig 1).

The average radius of the CoNPs grows inversely with the current density. To get nanoparticles having a mean size of 2 nm and 5 nm we used current densities of 5.0 and 1.5 mA/cm<sup>2</sup>, respectively. The resulting colloidal cobalt dispersions are black and were prevented to oxidation keeping the particles under argon atmosphere.

The stabilization of the synthesized CoNPs was achieved by coating the particles with a long-chain capping molecule: oleic acid [3]. This kind of molecule produces enough entropic repulsion to overcome the strong short range van der Waals attraction that otherwise results in irreversible particle aggregation and the consequent colloidal instability of the NP dispersions. It was observed that combination of TPP and oleic acid binds tightly to the particle surface and separates each particle from its neighbour by the organic chain and makes easy the transfer of the metal nanoparticles from the acetonitrile solution to an organic system. In this way stable colloidal suspensions of Co colloids even against oxidation in heptane (C<sub>7</sub>H<sub>16</sub>) were prepared.

Transmission electron microscopy (TEM) was employed to determine the core size and the shape of metal nanoparticles. Narrow size distributions were obtained.

The chemical interaction of the surfactant with the Co nanoparticles was studied by using Fourier transform infrared spectroscopy (FTIR) and nuclear magnetic resonance (RMN).

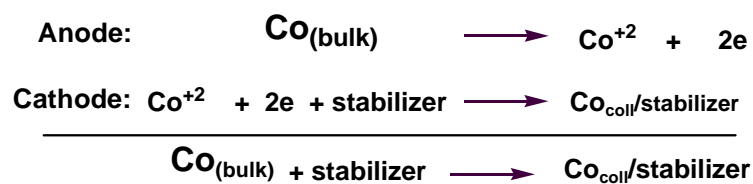
By thermogravimetric analysis (TGA) the elemental composition of the stabilized cobalt nanoparticles (2 nm) was determined to be CoTPP<sub>27</sub>OA<sub>34</sub>.

**References:**

- [1] Rodríguez-Sánchez, L.; López-Quintela, M. A.; Blanco, M. C. *J. Phys. Chem. B* **2000**, *104*, 9683.
- [2] Reetz Manfred T.; Helbig Wolfgang, *J. Am. Chem. Soc.*, *116*, 7401-7402, **1994**.
- [3] Nianqiang Wu; Lei Fu; Ming Su; Mohammed Aslam; Ka Chun Wong and Vinayak P. Dravid, *Nanoletters*, 2004, Vol 4, No 2, 383-386.

**Figures:**

(Fig 1) Electrochemical generation of cobalt colloids



(Fig 2) Variation of mean particle size with current density

