LOW-TEMPERATURE, FREE- RADICAL PROMOTED COBALT OXIDE NANOPARTICLE SYNTHESIS

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Cobalt nanoparticles can be synthesized by thermal decomposition of octacarbonyl dicobalt in the presence of the right mixture of surfactants (usually long chain carboxylic acids combined with a long chain phosphine or phosphine oxide)[1,2]. This is a reproducible and clean method, but it requires high temperatures (around 180ºC) to ensure the total decomposition of the metal precursor. Other wet-chemistry methods, working in milder conditions use previously synthesized organometallic compounds[3] or reduction of cobalt salts[4], with the inherent reactant contamination.

Our research was aimed at developing a method that allows us to form the particles from the corresponding metal carbonyl (most of the metal carbonyls are commercially available) decreasing its decomposition temperature by the effect of a promoter.

Starting from the corresponding metal carbonyl, $Co_2(CO)_8$, cobalt oxide nanoparticles[5,6] were synthesised by the TEMPO free radical, which is also the responsible of the metal oxidation[7, 8, 9]. This mediated decarbonylation allowed the low-temperature (r.t. to 50ºC) decomposition of the organometallic precursor in the presence of oleic acid and trioctylphosphine oxide (TOPO) as surfactants.

The decarbonylation process at promoter controlled injection rate was monitored on line by ATR-FT IR with a silicon probe (Mettler Toledo React-IR 4000®). TEMPO compared with other oxygen donor promoters (N-oxides: NMO, PPNO and N,N-dimethyloctacecylamine N-oxide) caused an slower decarbonylation process (monitored absorption band: residual non-bridging carbonyl at 2022cm^{-1}). The samples obtained using TEMPO contained cobalt oxide particles with an average diameter size of 100nm.

IR experiments revealed that TOPO diminished the conversion of decarbonylation. The reaction seemed to be more efficient when the promoter was present in an equimolar ratio with the CO ligand and that an increase in the surfactant concentration, oleic acid, can also improve the decarbonylation conversion. Parameters as the injection rate, solvent employed and concentration of cobalt were also tested.

Figures:

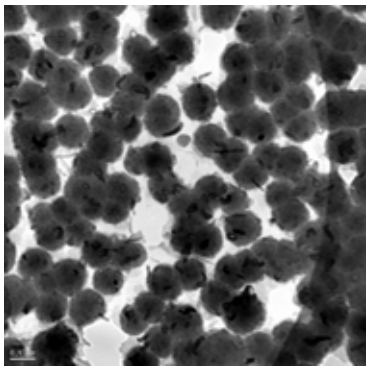

Figure1. TEM micrograph of cobalt oxide nanoparticles synthesized using TEMPO as a promoter.

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