Parametric study of iron and iron-oxide nanoparticle synthesis via Aerosol Spray Pyrolysis

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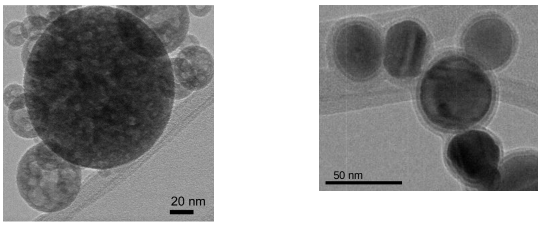
Emerging markets for energy (Karagiannakis et al, 2011), biomarker and magnetic data storage applications, drive the need for the production of iron and iron oxide based particles via continuous and scalable techniques. Aerosol synthesis routes have the advantage of being continuous, relatively simple and industrially-scalable techniques. More specifically, Aerosol Spray Pyrolysis (ASP) is a one-step, flexible and efficient method for the synthesis of tailor made nanoparticles (NP) (Karadimitra et al, 2001; Lorentzou et al, 2011).

In the current study, we examine the effect of different synthesis parameters such as the precursor material and its concentration, the solvent type, temperature, carrier gas, flow rate and exposure to different gases after synthesis on iron and iron oxide nanoparticle characteristics, such as morphology, crystallinity, mean particle size and composition. . The precursor materials used were commercial as well as appropriately purified precursors obtained from a low - cost mill-scale industrial by-product, in order to synthesize particles that could be used in a multitude of relevant applications.



A b

Figure 1:TEM image of (a) iron oxide particles from $Fe(NO_3)_3$ precursor (b) iron particles from industrial by-product derived precursor.

The synthesized particles have been post-characterized with Transmission Electron Microscopy (TEM), X-Ray Diffraction (XRD) and Energy Dispersive X-Ray Spectroscopy (EDS), in order to correlate synthesis parameters to resulting particle crystal structure and morphological characteristics. Particle crystallinity depends on the synthesis temperature, the residence time in the reactor and the temperature gradient applied after the aerosol spray generation. The different particle morphologies (such as dense, porous or core-shell) are determined largely by the composition of the precursor solution (type of iron salt, solvent, concentration) and carrier gas composition (O_2, N_2, H_2, etc). Knowledge obtained by the previous study was applied to synthesize iron nanoparticles from a low cost industrial by-product, normally treated as a waste.

The synthesized particles from different precursor concentrations (0.002-0.2M) were characterized on line with a Scanning Mobility Particle Sizer (SMPS) to study the effect of precursor concentration on particle size.

Particles made from a $Fe(NO_3)_3$ solution were studied under different carrier gases and were subjected to different reducing post treatments in order to obtain iron nanoparticle formation. Four different conditions with respect to the carrier gas were studied and were evaluated, with a representative result shown in Figure 2.

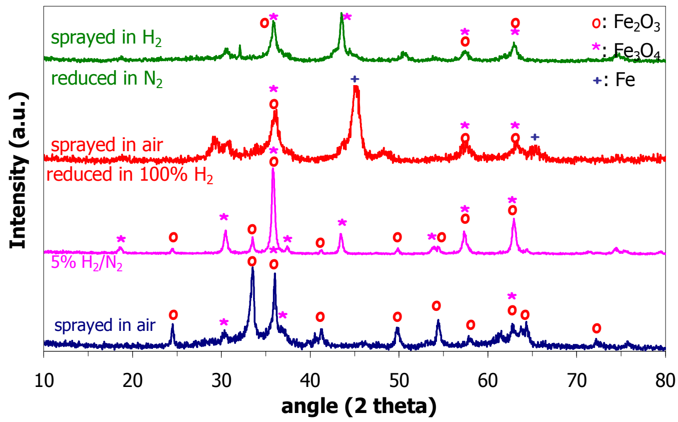


Figure 2. XRD patterns of ASP particles under different spraying and post treatment gas environments.

Aerosol Spray Pyrolysis is a flexible and promising technology for the production of various iron-based crystal structures and morphologies by controlling the parameters of the synthesis process.

Karadimitra K., Papaioannou E., Konstandopoulos A.G (2001), *J. Aerosol Sci*., **32**, Suppl. 1, S233-234.

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