

Synthesis of maghemite nanoparticles by self-combustion: the atmosphere effect

Jean Pierre MIRANDA MURILLO - *University of Genova, Italy*

Generally, the sol-gel self-combustion method is a facile, inexpensive, and green synthetic approach, enabling to obtain the desired nanostructured oxide with a high degree of homogeneity and purity. In the literature, the synthesis of maghemite using a pre-heated oven as the heating source is reported. However, non-isothermal conditions (i.e., heating up the gel from room temperature instead of a pre-heated oven), has been reported to yield both maghemite ($\gamma\text{-Fe}_2\text{O}_3$) and hematite ($\alpha\text{-Fe}_2\text{O}_3$) phases. In this framework, this communication illustrates the effect of the reaction atmosphere on the formation of these magnetic phases. Here, a thick gel of Fe(III)-citrate(III) network, inside a three-necks balloon, is heated up by using a hot plate ($T. \text{ set} = 350^\circ\text{C}$) in air atmosphere (**A**) and in a closed line of Argon flow (**B**). The thermal treatment triggers a redox reaction that transforms the gel into a crystalline Fe_2O_3 powder. The X-Ray Diffraction (XRD) pattern of powder **A** was fitted with the MAUD program (<http://maud.radiographema.eu/>), estimating a 100 wt.% presence of $\alpha\text{-Fe}_2\text{O}_3$ phase. However, since the most intense peaks of the reference $\gamma\text{-Fe}_2\text{O}_3$ pattern overlap with those of $\alpha\text{-Fe}_2\text{O}_3$, the presence of $\gamma\text{-Fe}_2\text{O}_3$ cannot be excluded. Using the same fitting program, the fitting of pattern **B** reveals only the cubic reflexes of the tetragonal $\gamma\text{-Fe}_2\text{O}_3$ superlattice ($a, b_{\text{exp}} = 8.41 \text{ \AA}$ vs $a, b_{\text{th}} = 8.33 \text{ \AA}$). The pattern **B** shows a slight shift of the peaks towards lower scattering angle with respect to pattern **A**. This may be related to a tensed structure that alters the lattice parameter. The mean crystallites' size $\langle D \rangle$ of **B**, calculated through Scherrer equation, is 30 nm. To the best of our knowledge, self-combustion of Fe-citrate gel in Argon flow is presented here for the first time. Further analyses (e.g., magnetic characterization) are currently underway.