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Maghmite nanoparticles stabilization by chlorine segregation at interfaces

Caliman, L.B.(1); Roulin, T.(2); Gouvêa, D.(1);

(1) USP; (2) EEIGM;

Maghmite, (gamma)-Fe₂O₃, is one of iron oxide polymorphs. Due to its ferromagnetic properties, its nanoparticles can be used in many applications such as biotechnology and biomedicine. At room temperature, (alpha)- Fe₂O₃ is the thermodynamically stable phase at this temperature but additive-induced surface energy changes can stabilize nanoparticles of the (gamma) phase, like interface segregation. When additives are used, generally segregation at the interfaces is observed (i.e. free surface or grain boundary) which modify the interface energy changing the thermodynamic stability of the nano phases. Surface modification is usually made by adding a different cation into the matrix of a material. In this study, stabilization of maghmite using an anion (Cl⁻) was investigated. Fe₂O₃ powders were prepared by the polymeric precursors method and doped with NH₄Cl at target molar concentrations of 0, 1, 3, 5 and 10 mol% and calcination temperature was 300 °C for 15 h. XRF showed that chlorine is difficult to be incorporated to the system and needed to be added in excess (about 3x times target concentrations). XRD and Rietveld refinement characterization showed that samples with retained chlorine showed a stabilization of maghmite (23 w%) mixed with hematite (77 w%). Maghmite has a final crystallite size of 24.1 nm and hematite of 39.5 nm (Rietveld refinement), supported by SEM images. The specific surface area increased for the samples with higher content of chlorine. Surface selective lixiviation was performed with water as a solvent. Interestingly, samples with the higher concentrations of chloride (shown by XRF) presented less chlorine segregated at the surfaces, which indicates that the chlorine might be trapped in the grain boundaries. Magnetic force was considerably increased in the samples with higher content of chlorine.