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Synthesis of spheres containing iron oxide superparamagnetic and aluminum oxide.

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Abstract – Spheres of superparamagnetic iron oxide nanoparticles and aluminum oxide were synthesized by a mixture of organic (chitosan) and inorganic (iron and aluminium hydroxide) materials. Scanning electron microscopy micrographs showed the presence of spheres with good regularity in the diameters .The presence of nanoparticles of iron oxide and aluminum oxide was observed by X-ray diffraction while the superparamagnetic effect was revealed by Mössbauer spectroscopy measurements.

The materials were synthesized by a method that consists of preparing a hybrid sphere, composed of metal ions (iron hydroxide and/or aluminum hydroxide) and organic polymer (chitosan), followed by a calcination process under a flow of air. Samples with different Al to Fe molar ratios were prepared and labeled AlFeX, where X denotes the Al:Fe molar ratio. The sample labeled Al contained only aluminum oxide.

The scanning electron microscopy (SEM) presented in Figure 1 (a, b), shows the presence of cracks in the sphere structure. Such morphology suggests a low mechanic resistance for these spheres. The average sphere diameter determined by SEM was 1.42 mm for the samples before the calcination process and 1.02 mm after the calcination process at 500ºC.

The calcinated samples were also analyzed with X-ray diffraction; results are presented in Figure 2-a. All patterns, except for sample AlFe0, present broad reflections, indicating that crystallites are rather small. Two crystalline phases of iron oxide were identified from the diffraction profile of sample AlFe0 (see Figure 2-a): α-Fe_2O_3 (hematite, JCPDS card # 87-1166) and γ-Fe_2O_3 (maghemite, JCPDS card # 25-1402). The diffraction patterns for samples AlFe15, AlFe6 and Al (Figure 2-a) show the occurrence of Al_2O_3 (JCPDS card # 10-0425); figures for sample AlFe6 and AlFe2 reveal also the co-existence of iron oxide phases, namely α-Fe_2O_3 (hematite, JCPDS card # 87-1166) and γ-Fe_2O_3 (maghemite, JCPDS card # 25-1402). The average crystallite diameter of the different phases as estimated with the Scherrer's formula [1], and the results showed the phases with a particle size between 2.0 and 9.0 nm.

The local environment of iron atoms in the iron-containing samples was investigated by Mössbauer spectroscopy. The room temperature spectra obtained for samples AlFe15, AlFe6 and AlFe2 (Figure 2-b) showed a similar feature, with a central doublet, suggesting that iron is in a superparamagnetic state. The Mössbauer signal for sample AlFe15 corroborates the assumption that Fe-based phases are not identifiable by XRD due to low Fe:Al ratio and to the extremely small particle sizes. Mössbauer spectra for sample AlFe2 taken at low temperatures confirm its superparamagnetic state.

Therefore, materials with useful characteristics were synthesized by a simple method. Additionally, a uniform spherical shapes and size was obtained.

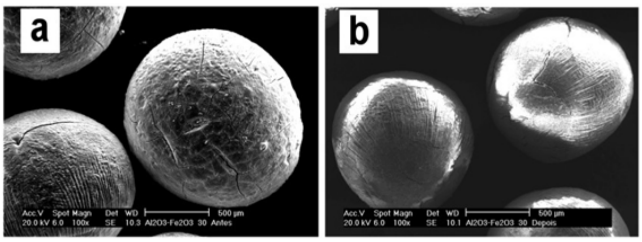

Figure 1: Scanning electron microscopy of the AlFe6 sample (a) before calcination, (b) after calcinations.

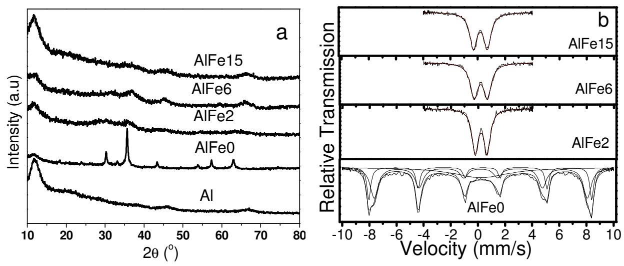

Figure 2: (a) X-ray power diffraction; (b) ^{57}Fe Mössbauer spectra of the spheres.

[1] L.V. Azaroff, Elements of X-ray Crystallography. McGraw-Hill Book Company: New York, 1968.