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Morphology Control of Iron Oxide Nanoparticles

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Super paramagnetic Fe_3O_4 nanoparticles have been studied in several science fields, especially in biomedical applications. Coated magnetic nanoparticles are studied as non-conventional methods in cancer treatment. Magneto hyperthermia is considered as an alternative in cancer therapy [1]. This method claims to selectively destroy cancer cells and to concurrently use the coated magnetic nanoparticles as part of a drug delivery system. The present work aims at establishing the synthesis of the magnetic iron oxide nanoparticles with controlled size and shapes.

The nanoparticles were obtained by co-precipitation from iron salts - $FeSO_4$ and chlorines - $FeCl_3$ in the ratio Fe^{2+}:Fe^{3+} = 1:2, with the addition of a NH_4OH solution. There are several factors that control the final size of nanoparticles. In previous research [2], the size of the particles was reported to be pH controlled. The type of hydroxide used (NaOH, NH_4OH, etc) has an effect on the final size of the particles [3]. Solutions with different concentration of NH_4OH (0.15; 0.28; 0.50 and 1.M) were added into solutions containing a mixture of $FeCl_3$ (0.45 M) and FeSO4 (0.225 M). The magnetic nanoparticles were washed five times, alternately with water and ethanol in the order to remove residual chemicals reagents; then collected by centrifugation; and finally were magnetically separated. These nanoparticles were washed and dried at ~ 60 ºC for 12 h in a furnace. The structure of these nanoparticles was studied by transmission electron microscopy (TEM), scanning electron microscopy (SEM) and X-ray diffraction (XRD). The magnetic behavior was observed by Hall Effect magnetometer. Two different morphologies were observed: faceted nanoparticles and nanowires.

The produced Fe_3O_4 nanoparticles were observed by TEM. A Jeol 2010 instrument, operating at 200 kV under diffraction and phase contrast modes was used as main tool. FIG 1 shows typical bright field TEM images of the nanoparticles produced with (a) 0.15, (b) 0.28, (c) 0.50 and (d) 1.0 M of NH_4OH, dispersed on a thin carbon supporting film. The sample produced with 0.15 M of NH_4OH shows elongated shapes around 100 nm of length (FIG. 1.a), the sample produced with 0.28 M presents elongated shapes of length around 50 nm (FIG. 1.b). The samples produced with concentrations of 0.5 and 1.M (FIG.1.c and 1.d), did not present elongated shapes, the particles have an average size in the order of 10 nm and display a spheroidal morphology. Heterogeneous particle sizes were observed in the aggregate shown in FIG.1.a and FIG.1.b. The population of elongated shapes is seems to be reduced with the increase of NH_4OH concentrations, and eliminated when concentrations are above 0.5 M. In addition, the average size and aspect ratio of the particles is reduced with the increment of the NH_4OH concentration. Fig 2 shows a typical high resolution image of a 60 nm long nonowire obtained with an aberration corrected TEM, Zeis Libra at 80 kV. This nanowire produced with 0,28 M of NH_4OH exhibits atomic planes with about 0,37 nm. In summary, TEM has proved to be a fundamental tool in the controlled synthesis of Fe_3O_4 nanoparticles via co-precipitation.

References:

[1] R. Hergt, et al., J. Magn. Magn. Mater. 270 (2004) 345.
[2] J. P. Jolivet, et al., C. R. Geoscience 338 (2006) 488–497.

[3] G. Gnanaprakash, et al., Materials Chemistry and Physics 103 (2007) 168–175.
[4] This research was supported by the National Council for Scientific and Technological Development (CNPq)

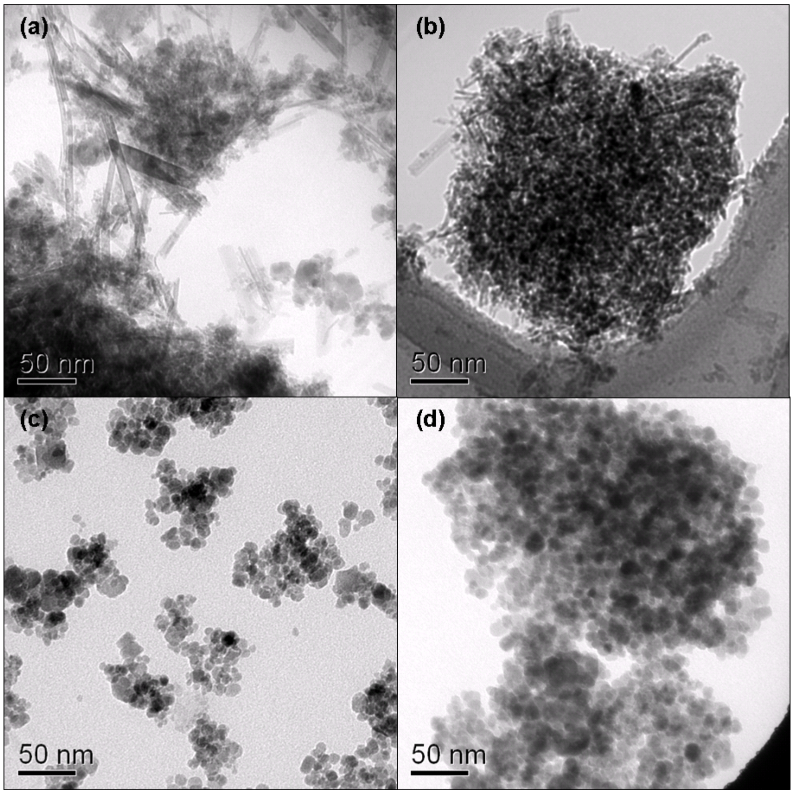

FIG. 1. Bright field micrographs of the nanoparticles produced with (a) 0,15, (b) 0,28, (c) 0,50 and (d) 1,0 M of NH_4OH, it shows variation of size and shape of the particles.

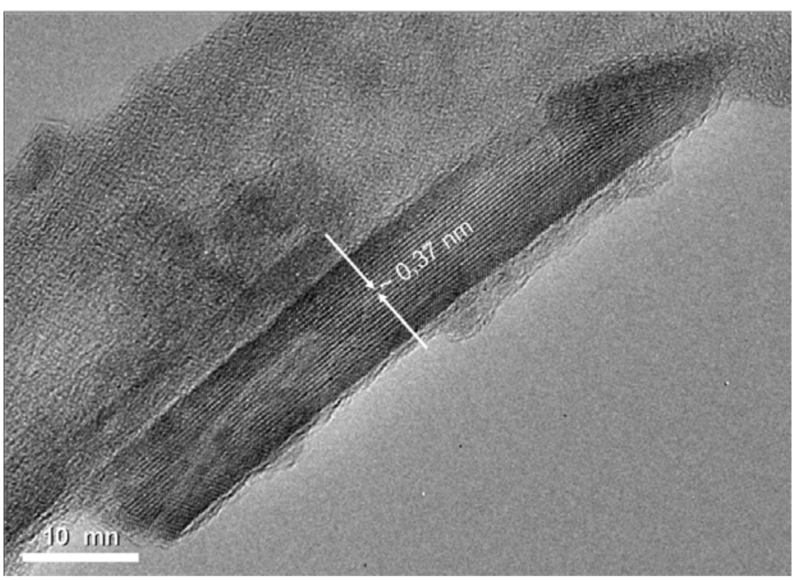

FIG. 2. HRTEM image of 60 nm nanowire in a sample produced with concentration of 0,28 M of NH_4OH. Atomic planes about 0,37 nm spacing can be observed.