

Synthesis of Dense Silica Nanoparticles Containing Iron(III) Chloride

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Introduction

Nanotechnology is an area that has recently attracted considerable interest from researchers worldwide, because it allows scientists to work in the atomic scale. In this context, magnetic silica nanoparticles have received great attention: they are non-toxic and biocompatible [1]. Nevertheless, to synthesize silica nanoparticles, it is necessary to ensure a series of experimental conditions, which will affect features such as particle size, shape, and composition, among others, all of which are important in the medical field [2]. Applications of silica nanoparticles as drug delivery systems will depend on these features, since particle characteristics will dictate their diffusion in blood vessels and cells. In this context, the present work deals with the hydrolytic sol-gel synthesis and characterization of spherical silica nanoparticles in the presence or absence of magnetic materials obtained by the Stöber methodology, aiming at better application of these particles according to their size. Particle synthesis started by reacting the tetraethylorthosilicate (TEOS) alkoxide, ammonium hydroxide, distilled water, and isopropyl alcohol in the presence or absence of iron(III) chloride (magnetic compound). To this end, a digital ULTRA-TURRAX T-25 Disperser was used at 3400 rpm. The resulting particles were doped with europium(III) ions, which acted as structural probes. The final materials were dried and characterized by scanning and transition electron microscopy (SEM and TEM, respectively), infrared absorption spectroscopy (FTIR), photoluminescence (PL), X-ray diffraction (XRD), thermogravimetric analysis (TG), and electron paramagnetic resonance (EPR).

Results and Discussion

SEM and TEM showed that the spherical nanoparticles measured 250 nm, on average. Figure 1 illustrates the SEM micrographs of the two prepared samples.

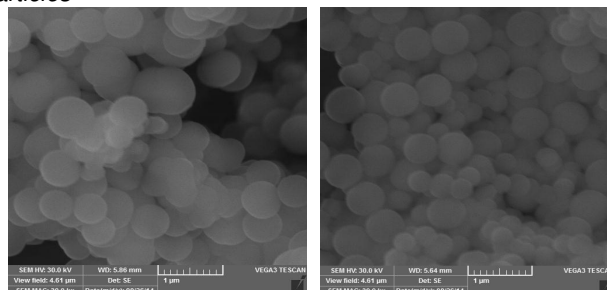


Figure 1. SEM recorded for the samples (a) SiO_2 , with $r = 250$ nm, and (b) $\text{SiO}_2 + \text{FeCl}_3$ with $r = 250$ nm.

Thermal analysis revealed total mass loss of 13 and 15% for samples SiO_2 and $\text{SiO}_2 + \text{FeCl}_3$ around 100 °C, respectively, due to loss of water molecules. The slightly larger mass loss observed for sample $\text{SiO}_2 + \text{FeCl}_3$ may be due to loss of chloride, which usually happens over 300 °C. EPR confirmed the presence of Fe^{3+} ions in the silica nanoparticles—sample $\text{SiO}_2 + \text{FeCl}_3$ displayed a signal in $g = 2.24$, which did not occur in the case of sample SiO_2 . Eu^{3+} PL also attested to the existence of Fe^{3+} ions in sample $\text{SiO}_2 + \text{FeCl}_3$, because the typical Eu^{3+} luminescence diminished in the presence of Fe^{3+} . Only the FTIR spectrum of sample $\text{SiO}_2 + \text{FeCl}_3$ exhibited a small shoulder in the band at 1630 cm^{-1} , attributed to water molecules, which indicated that Fe^{3+} interacted with water molecules in the silica.

Conclusion

Our results were satisfactory and may lead to the development of novel magnetic materials. Future tests using different dispersing rates, reaction times, temperatures, and other parameters shall afford particles for which it will be necessary to define applications.

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¹ Sanchez, C; Boissiere C, Cassignon S, Chaneac C, Durupthy O. Chem. Mater. (2013) 1-20.

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