Silica Coating of hydrophilic SPIO nanoparticles via microemulsion

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Abstract

Silica coating is a well-known method for surface modification of various nano-sized materials in order to improve their colloidal and chemical stability. Silica coating can also be employed as an alternative strategy for preparing suspended particles surface for further surface modification.

In this research, SPIO nanoparticles were coating by silica condensed from TEOS using microemulsion based on cyclohexane/Igepal CO-520/ammonia system. The synthesized silica coated-SPIO nanoparticles were characterized using different techniques such as transmission electron microscope (TEM), dynamic light scattering (DLS) and zeta potential measurements. This process resulted in the complete coverage of the surface of SPIO nanoparticles with an ultra-thin silica layer, and 3 to 4 times aggregation.

Introduction

Superparamagnetic iron oxide (SPIO) nanoparticles are believed to offer superior properties for use in a broad range of applications in biology; the superior magnetic properties, colloidal and chemical stability in physiological environments has made SPIO as a potential candidate material to be used in the different fields of medical sciences.

The SPIO nanoparticles are generally used as a coated material with various surface chemistries; it is actually essential either for improving colloidal and/or chemical stability or to make a basis for further surface modification such as conjugating biological active entities. Base-catalyzed condensation of TEOS in micellar system has long been found to be a promising method for synthesis of monodisperse silica nanoparticles. The silica coating process of SPIO are also governed by the same principles except in the nucleation mechanism; unlike the formation of the silica nanoparticles, TEOS is condensed on SPIO nanoparticles surface via heterogeneous nucleation.

Experimental

Hydrophilic SPIO nanoparticles were transferred into an organic phase (cyclohexane) after surface modification by oleate ligands; Fig. 1 depicts the reactor.

In a typical experiment for silica coating, 1 g Igepal CO-520 was solubilized in 10 ml cyclohexane after 10 min sonication, followed by addition of 1 ml oleate-capped SPIO nanoparticles suspended in cyclohexane, and another 5 min sonication. Next, 180 µl ammonia solution (25 wt. %) was added into the reactor. Finally, silica coating reaction initiates after addition of 50 µl TEOS; Fig. 2 schematically describes different steps of this process.

Results & Discussion

The surface chemistry of SPIO nanoparticles was studied using thermogravimetric analysis (TGA); it was estimated to contain approximately 8 oleate ligands per nm².

The SPIO nanoparticles coated after different reaction times were characterized by TEM; see Fig. 3. It seems that after 6 hrs, a complete coverage of silica was achieved. The zeta potential was then measured in order to confirm this observation; see Fig. 4. The plot corresponding to SPIO nanoparticle coated for 3 hrs shows positive deviation that is contributed to presence of uncoated SPIO nanoparticles.

The SPIO nanoparticles shows positive zeta potential in all range of acidic pHs. The latter interpretation was evaluated by zeta potential measurements carried out on a mixture of silica NPs with small quantity of SPIO nanoparticles; See Fig. 5.

The hydrodynamic diameter of the silica-coated SPIO nanoparticles was also measured using DLS techniques. The obtained results is in agreement with the TEM observation; the number-average diameter of the nanoparticles is about 66 nm implying the occurrence of about 4 times aggregation. The volume-average size distribution reveals the formation of small quantity of very large particles after 23 hrs reaction implying the limited stability of the microemulsion; see Fig. 6.