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CHARACTERIZATION OF SILICA COATED SUPER PARAMAGNETIC IRONOXIDE NANOPARTICLES

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Abstract
Multifunctional superparamagnetic iron oxide nanoparticles (SPIONs) coated with silica are a promising research field for lots of biomedical applications. The scope of this work is a preparation of SPIONs and coating them with silica to form core- shell structured nanoparticles for nanomedicine applications. SPIONs were synthesized by two chemical methods - co-precipitation and thermal decomposition of organic iron precursor. Prepared nanoparticles were carefully characterized - average size, size distribution, morphology, crystallinity, colloidal stability and magnetic properties were studied. After comparing SPIONs synthetized by two routes the most suitable method for biomedical applicable nanoparticles preparation is determined. The nanomedicine requires nanoparticles of the highest quality.
KEYWORDS: [Nanoparticles, silica, paramagnetic substances, ironoxide.]

INTRODUCTION

Nanotechnologies are fast growing area of researches in all over the world. The reason of serious attention is a wide spectrum of possibilities to improve the quality of life. The key is new significant physical, chemical, biological properties of nanosize constructed materials - nanoparticles, nanotubes, nanowires, extremely thin films, nanocomposites etc. High surface to volume ratio allow to functionalize nanoparticles with different ligands, coatings and other useful tools for lots of biomedical applications. Nowadays medicine has great future opportunities based on use of engineered nanomaterials.

Magnetic nanoparticles

Magnetic properties of nanomaterials are powerful manipulation and detection tools which are studied for a long time. Since magnetic fields are not harmful to a human organism (but this question is still opened for high strength magnetic fields) magnetic nanoparticles can be used for biomedical in vivo and, of course, in vitro applications. By a material reaction to a magnetic field, all substances are categorised into several groups :

- diamagnetics,
- paramagnetics,
- ferromagnetics,

Iron oxides

Mostly used materials for the superparamagnetic core are iron oxides : magnetite Fe3O4 and maghemite ?-Fe2O3. Wustit FeO doesn`t show any significant magnetic properties. Both magnetite and maghemite have cubic spinel structure. Spins from these two sublattices are antiparallel, so magnetite net magnetization occurs due to Fe2+ ions from octahedral sublattice5. Magnetite is sensitive to oxidation - oxygen transforms it to maghemite by oxidizing of Fe2+ ions.

Thermal decomposition of iron organic precursor method

Highly monodispersed nanoparticles can be synthesised via thermal decomposition of organometallic precursors method. Some organic iron compounds (hydroxylamineferron [Fe(Cup)3], iron pentacarbonyl [Fe(CO)5], ferric acetylacetonate [Fe(acac)3], iron oleate [Fe(oleate)3]12 are decomposed at high temperature inside the non-polar boiling solvent with a presence of the capping agent (it is worth to say that most of these precursors are toxic and not friendly to environment). Narrow size distribution (?<10%), high crystallinity and shape control are the attributes of this route10.

Drug delivery systems

Engineering of high controllable drug nanocarriers able to transport and release very accurate amounts of therapeuticals is one of the most promising nanomedicine research fields. First mentioned in science fictions of early XX century, it is now developing with giant steps. An efficiency of drug delivery systems depends on 3 main parameters - neutrality to immune system, an ability to transport drug carriers directly into unhealthy tissue with a high precision, an ability to regulate drug release rate to provide an optimal concentration of medicaments - higher than minimal therapeutical level and lower than toxic dose.

Review of background literature:

This represents a review of the current developments in the area of synthesis and characterization of silica coated metal oxides particles prepared using sol gel technique and also preparation of coreshell nanocomposites. To give a background to discuss the reported studies on this topic, a brief description of the well-known sol gel systems is presented here. The reported studies on the preparation of coated-particles using different synthetic routes and formation of nanocomposites are presented here.

Preparation of ?-Fe2O3 Hydrosol: Linua Hua .et al have synthesized ?-Fe2O3 hydrosols with FeCl3.6H2O as the raw material and its thin films by dip coating technique .The results showed that uniform ?-Fe2O3 monolayer and multilayers could be obtained having pseudo-cubic shaped with mean particle size of nanocubic meterwith a certain dip-coating speed. Ying-Jie Zhu has reported on the microwave-hydrothermal ionic liquid method for the synthesis of a variety of iron oxide nanostructures such as a-FeOOH hollow spheres, b-FeOOH architectures and a-Fe2O3 nanoparticles.

Preparation of silica gel particles: Burtrand I. Lee .et al prepared silica gels by hydrolytic polycondensation of tetraethoxysilane (TEOS) with large amounts of nitric acid as the catalyst have been studied. The molar compositions were TEOS: H20: HNO 3 =1 : 10:x . In the presence of a large amount of nitric acid, the gels had higher specific surface areas and higher pore volumes as the drying temperature increased from 45°C to 60°C. Stober and fink.et al has synthesized silica nanoparticle by chemical methods from TEOS, ethanol and deionized water in presence of ammonia as catalyst at room temperature. Zeev Rosenzweig .et al developed a simple method to prepare bright and photo stable luminescent silica nanoparticle of different sizes and narrow size distribution in high yield. The method is based on the use of stober synthesis in presence of fluorophore to form bright silica nanoparticles.

EXPERIMENTAL METHODS

Synthesis of spions

Two SPION synthesis routes were performed - co-precipitation method and thermal decomposition method. As iron precursors for co- precipitation ferrous and ferric chlorides (both are hydrates) in a molar ratio 1:2 were chosen. Two solutions were prepared - ammonia hydroxide solution as a precursor of OH- ions and iron chlorides solution as an Fe2,3+ ionso precursor.

Thermal decomposition method

The thermal decomposition preparation of magnetite consisted of two steps : synthesis of iron oleate and high temperature decomposition of it. This route is relatively environment-friendly - sodium oleate and ferric chloride are the precursors for iron pleate complex.

Preparation of SiO2@Fe3O4

Water-in-oil microemulsion method was chosen due to a narrow size distribution of prepared nanocomposites, very low amount of fused together nanocomposites and an ability to coat hydrophobic nanoparticles via ligand exchange mechanism.

CHARACTERIZATION

The most important part of experiments is characterization - it gives an opportunity to analyze results of experiments and to choose the next step to achieve expected results. Different parameters need different characterization techniques, an efficiency of analysis strongly depends on how suitable was the chosen characterization technique for certain parameter.

Transmission Electron Microscopy

Transmission electron microscopy (TEM) is a powerful tool in exploring a shape, crystallinity, mean size and size distribution of pristine and silica coated SPIONs. Nanoparticles were investigated with a JEOL 2100 FEG-TEM using 200 kV electron acceleration voltage.

Dynamic Light Scattering

Dynamic light scattering (DLS) is the technique for hydrodynamic nanoparticle size explorations. When a nanoparticle is surrounded by media, it`s surface (with different ligands or without them) has a variety of interactions with solvent molecules and ions and this is strongly influencing a behavior of a nanoparticle.

RESULTS AND DISCUSSIONS

The main characterization technique for a morphology, structure and size of synthesized nanoparticles was TEM. On the pictures below SPIONs synthesized by co-precipitation and thermal decomposition methods are presented : SPIONs` size measurements were processed using ImageJ software. The average size for co-precipitation synthesized nanoparticles is 10.4 ± 3.6 nm (? = 34.6%). The average size for thermal decomposition prepared nanoparticles is 8.2 ± 0.8 nm (? = 9.7%). As it can be seen from the pictures, size and morphology are significantly different

- narrow size distribution and spherical shape for the thermal decomposition method, broad size distribution and random near- spherical shapes for the co-precipitation method. Despite co- precipitation synthesized SPIONs form aggregates on TEM images, they are stable and separated at certain pH range of a solvent. A sonication device can be used to restore nanoparticles from aggregated state. Core-shell nanoparticles with a diameter of 30.6 ± 4.9 nm (? = 16.0% were synthesized. Average silica shell thickness was calculated - 11.2 ± 1.7 nm (? = 15.1%). Prepared nanocomposites are quite homogeneous in size, separated and single-cored. However, some multi- core nanoparticles and fused together ones take place, but in a relatively small amount (less than 1%).

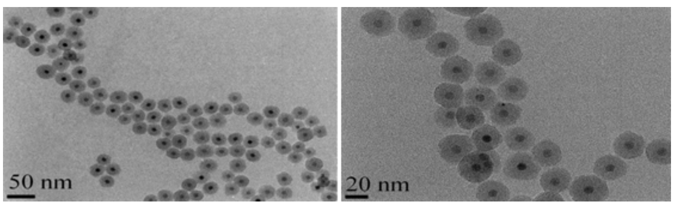

Figure 1 Core-shell structured nanoparticles

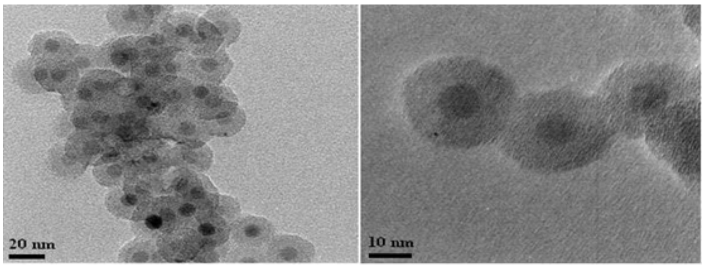

Figure 2 Necked nanoparticles

CONCLUSIONS

The aim of this work is to compare two synthesis methods of SPIONs, choose more appropriate one for preparing SPIONs applicable in biomedicine and to coat chosen SPIONs with silica layer. Every synthesis method was run many times in order to understand its main properties and features, aknowledge factors influencing experiments. Several characterization techniques were used to carefully analyze prepared nanoparticles. After this step I can conclude, that the thermal decomposition method synthesized SPIONs better fit to strict requirements of biomedical applications, than co-precipitation synthesized ones. They possess narrower size distribution, uniform spherical morphology, higher saturation magnetization and lower remanence magnetization and coercivity field - so they do not form significant aggregates.

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