Study of Electrokinetic Properties of Magnetite – Silica Core – Shell Nanoparticles

YU. A. UTKIN[1], MINGGONG SHA[2],
[1]Moscow Aviation Institute (National Research University),
Moscow, Volokolamskoe shosse, 4, 125993,
RUSSIA
[2]School of Civil Aviation, Northwestern Polytechnical University (NPU),
Xi'an Shaanxi, 127 West Youyi Road, Beilin District, 710072,
CHINA

Abstract—In this work, the electrokinetic properties of Fe_3O_4 nanoparticles modified with various alkoxysilanes (tetraethoxysilane and 3-aminopropyltriethoxysilane) in various media were investigated. The determined values of the zeta potential of the Fe_3O_4/SiO_2 samples indicate the complete coverage of nanoparticles with a tetraethoxysilane shell, as well as in the case of the Fe_3O_4/aminopropyltriethoxysilane. The data obtained on the zeta-potentials of modified nanoparticles with various ligands make it possible to predict the efficiency of subsequent functionalization by target molecules.

A decisive role in the study of surface properties is played by cleaning from low molecular weight impurities that can screen the surface of nanoparticles or bind with an indifferent electrolyte. Thus, dispersion on a magnetic stirrer leads to an increase in the sorption capacity of the sample in comparison with ultrasonic dispersion, which causes irreversible destruction of the core- shell nanoparticle structure due to an increase in temperature and pressure in the cavities. This opens the prospective for practical application of modified nanoparticles for creation of tailored composite materials.

Keywords—Nanoparticles, composites, magnetite, silica.

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1. Introduction

In order to solve possible problems with the introduction of magnetite nanoparticles into a living organism, such as instability under physiological conditions [1],[2],[3],[4],[5],[6],[7],[8],[9],[10],[11],[12], the formation of free radicals dangerous for the body, as well as insufficient strong bond with ligands during targeted drug delivery, nanoparticles cover a protective a shell that should ensure their stability, reduce toxicity to a minimum, and have the ability to form strong bonds with various types of ligands that are used to functionalize the surface of nanoparticles [13],[14],[15],[16],[17],[18],[19],[20],[21],[22],[23],[24],[25].

Serious concern is also caused by the behavior of materials based on Fe_3O_4 nanoparticles, which are widely used as detoxicants in the restoration of the environment, in particular, for the removal of chlorine-containing compounds, organic dyes, heavy metals from technogenic and natural aqueous media. Fe_3O_4 nanoparticles are subject to oxidation in air and easily aggregate in aqueous systems. The necessary stabilization of iron oxide nanoparticles by surface modification should pursue a double goal: control of the size and polydispersity during synthesis and stabilization of nanoparticles against aggregation after synthesis [26],[27],[28],[29],[30],[31],[32],[33],[34],[35]. Modification of nanoparticles is carried out using various inorganic or organic compounds by their non-covalent or covalent immobilization on the surface of iron oxide nanoparticles, which leads to a change in their primary properties and allows expanding the areas of their potential application [36],[37],[38],[39],[40],[41],[42],[43],[44],[45]. There is a wide range of substances capable of forming a protective shell on the surface of magnetite nanoparticles; among them, alkoxysilanes are of interest as inert, biocompatible, and functional inorganic ligands.

Modern composites have not only a wide range of physical and mechanical properties, but are also capable of directionally changing them, for example, increasing fracture toughness, regulating rigidity, strength, and other properties. These possibilities are expanded when fibers of different nature and geometry are used in composites, i.e., when creating hybrid composites. In addition, these materials are characterized by the appearance of a synergistic effect (coordinated joint action of several factors in one direction) [45],[46],[47],[48],[49],[50],[51],[52],[53],[54],[55],[56],[57],[58],[59],[60],[61],[62],[63],[64],[65],[66],[67],[68],[69],[70],[71],[72],[73],[74],[75].

The properties of the interface or interfacial zone, first of all, the adhesive interaction between the fiber and the matrix, determine the level of properties of composites and their retention during operation. Local stresses in the composite reach their maximum values just near or directly at the interface, where material destruction usually begins. The interface must have certain properties to ensure efficient

transfer of the mechanical load from the matrix to the fiber. The adhesion bond at the interface should not be destroyed under the action of thermal and shrinkage stresses arising from the difference in the temperature coefficients of linear expansion of the matrix and fiber or as a result of chemical shrinkage of the binder during its curing.

When creating nanocomposites, the key tasks are the development of efficient, reliable, and affordable production technologies for mass production, which make it possible to obtain materials with stable characteristics. The hand lay technique, also called wet lay, is the simplest and most widely used process for producing flat reinforced composites. The process consists of laying layers of a polymer in successive layering using an epoxy matrix. Wet-laying is a molding process that combines layers of reinforced carbon fiber with epoxy to create a high-quality laminate. Before starting the installation process, you must prepare the appropriate form. This preparation consists of cleaning the table and applying a release agent to the surface. The manual laying process can be divided into four main steps: mold preparation, epoxy coating, laying and curing. Form preparation is one of the most important steps in the installation process. This process requires dry reinforcement layers and the application of a wet epoxy matrix. They are connected together - reinforcing material, impregnated with a matrix

Coatings on nano and micro-sized particles can serve for many purposes. First of all, modification of the surface with coatings makes it possible to make the particles compatible with various matrixes [14],[15],[16],[17],[18], [19],[20],[21],[22],[23],[24],[25],[26],[27],[28],[29],[30]. For medical purposes, the biocompatibility with the environments of a living organism is of crucial importance. It is equally important that coatings can significantly enhance or decrease the sorption properties of magnetically controlled sorbents. This provides prerequisites for the creation of magnetically controlled particles with specific sorption properties. It is also known that the coatings prevent the core from leaching out. The presence of a coating also often facilitates the stabilization of particles in an environment with an alkaline pH or significant salt concentration. For example, the isoelectric point of SiO_2 is reached at pH 2-3. Therefore, the particles coated with silica are negatively charged at the pH of the blood, which causes electrostatic repulsion, which avoids the formation of clumps.

The ability of the adsorbent to absorb the adsorbate is characterized by the amount of adsorption. The amount of adsorption is the excess mass of the adsorbate in the boundary layer over its mass in an equal volume of the environment, referred to the unit surface of the adsorbent.

Sometimes the adsorption value is expressed in moles of adsorbate per 1 m^2 (or 1 cm^2) of the adsorbent surface. Since quite often the surface of the adsorbent is unknown, the value of adsorption is expressed in moles of adsorbate per 1 g of adsorbent (mol/g). It is customary to evaluate the process of toxin sorption by the adsorbing surface using the curves of Langmuir sorption isotherms.

Silanol binding agents are applied directly to the surface of Fe_3O_4 nanoparticles by copolymerization of monomers or by direct silanization. The developed surface of nanoparticles leads to a high density of surface functional groups [48],[49], [50],[51],[52],[53],[54],[55],[56],[57], which can fix a large number of biologically active substances [32]. The most common way to obtain Fe_3O_4/SiO_2 with a core-shell structure is the sol-gel method (Stober method), which consists in hydrolysis and polycondensation under alkaline conditions in ethanol [33].

In this regard, in this work, we performed a comparative analysis of the microstructure of magnetite nanoparticles synthesized by various methods before and after their modification with 3-aminopropyltriethoxysilane under various reaction conditions (in argon and during oxidation).

Analyzing the works where the authors provide data on the electrokinetic properties of magnetite nanoparticles coated with silanes, it can be noted that under various conditions for the preparation of nanoparticles (different sample preparation, temperature and time of preparation, drying conditions), the authors obtained samples identical in structure and composition according to the IR data and the method of electrophoretic light scattering. However, the lack of uniformity in the characteristic absorption bands and the position of isoelectric points for the same samples does not make it possible to correctly evaluate the physicochemical data and the success of the preparation.

The aim of this work was to study the electrokinetic properties of Fe_3O_4 nanoparticles upon their modification with various alkoxysilanes (tetraethoxysilane and 3-aminopropyltriethoxysilane) in various media, and also to determine the zeta potential and hydrodynamic diameter of the obtained nanocomposites by the method of dynamic and electrokinetic light scattering.

2. Study of the electrokinetic properties of nanoparticles

An examination of the data obtained makes it possible to show that systematic changes are observed in the experimental spectra, which are the same at different temperatures. Moreover, when passing from a sample of native particles to a sample of coated particles, they are less than from a sample of coated particles to a sample of oxidized particles. Thus, the treatment of the initial sample of magnetite with aminopropyltriethoxysilane (APTES) leads to a narrowing of the external profile of the relaxation sextet, apparently due to the sharpening of the internal sextets. The processing of a sample of APTES coated particles with nitric acid caused the removal of components from the right side of the spectra, and the appearance of components in the left side of the spectrum, i.e. in fact, we are talking about a shift of some sextet to the region of low isomeric shifts, i.e. oxidation of iron.

Electrokinetic measurements provide independent information about the electric double layer of charged particles [62],[63],[64],[65],[66]. The measured electrophoretic mobility of particles can be converted to an electrokinetic (zeta) potential according to the equation:

$$\xi = \frac{\mu 3 \eta}{2\varepsilon}$$

where ξ – zeta potential, μ – electrophoretic mobility, η – medium viscosity, ε – the dielectric constant.

The value of electrophoretic mobility, measured in a dispersion of metal oxides, changes at a certain pH value (pH of the isoelectric point, IEP), at which amphoteric particles do not have an excess charge.

The zeta potential determined for the Fe_3O_4, Fe_3O_4/APTES (A), Fe_3O_4/APTES (B), and Fe_3O_4/TEOS samples is shown in Fig. 1 and found at pH = 2.8, pH = 7.1 and pH = 6.6, respectively. The Fe_3O_4/TEOS sample has a negative charge in the range of pH = 3-10. due to the presence on the surface of Si-OH groups characteristic of tetraethoxysilane (TEOS). The shift of the isoelectric point of magnetite from pH = 6.3 to pH = 2.8 indicates complete coverage of the magnetite surface with tetraethoxysilane. The presence of amino groups on the surface should shift the IEP towards higher pH values. The IEP value for Fe_3O_4/APTES (A) in the region of ~ pH 7 may be due to the presence of protonated amino groups on the SiO_2 surface or their small presence or absence. This is consistent with the position of the zeta potential curve, according to which in the alkaline region on the surface of Fe_3O_4/APTES (A) there are fewer negative charges than on the surface of Fe_3O_4 and Fe_3O_4/APTES (B). Reactions of surface centers Fe_3O_4-Si-O-NH_2 with H+ and OH ions lead to the formation of positive (Fe-Si-O-NH_3^+) and uncharged surfaces (Fe–Si-NH_2).

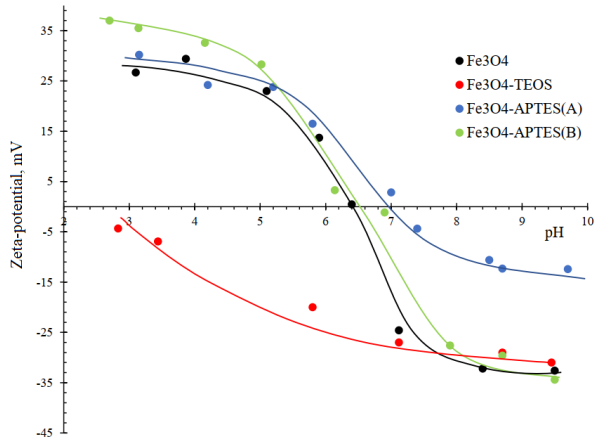

Fig. 1 dependence of the ζ-potential of Fe_3O_4, Fe_3O_4/TEOS, Fe_3O_4/APTES (A) and Fe_3O_4/APTES (B) nanoparticles on pH (0,0 KCl).

The value of IEP Fe_3O_4/APTES (B) near pH 6 is explained by the presence of protonated amino groups on the SiO_2 surface. However, in the alkaline region on the surface of Fe_3O_4/APTES (B) there are more negative charges than on the surface of Fe_3O_4/APTES (A), which may indicate a larger number of alcohol OH groups on the surface of nanoparticles. In the case of [65], the zeta potential is +30 mV over the entire pH range, which is an indicator of the stability of nanoparticles coated with APTES and may also indicate the presence of NH_3 + on the surface of nanoparticles. APTES has IEP at pH 10,05. The difference in the data for the zeta potential for Fe_3O_4/APTES samples in different works indicates the dependence of the zeta potential value on the method and conditions of synthesis, the ratio of components and purification of preparations, despite the same component composition of nanoparticles.

Samples Fe_3O_4 and Fe_3O_4/APTES (B) are unstable in a narrow range of pH = 5.6-7, however, in the range of pH 3–5.6 and above 7, the ξ-potential is ± 30 mV and the system remains stable. At the same time, the Fe_3O_4/APTES (A) sample obtained in an inert medium is stable only in a narrow pH range of 3-5 and then, with an increase in pH, the ξ-potential begins to decrease, not exceeding -10 mV, and the system loses stability. The Fe_3O_4/TEOS sample is stable in a wide range of pH = 6-10 and loses its stability in a strongly acidic medium in the region of the isoelectric point. When assessing the surface charge of the surface of modified magnetite samples, it was revealed that not only the synthesis conditions in various media (inert - in argon and not inert - with air access), but also the subsequent dispersion treatment of the samples to obtain suspended particles affect the value of the zeta potential.

Impact has been identified:

- Dialysis. After dialysis, the IEP of the Fe_3O_4/APTES (A-DUs) sample shifts from pH = 7.2 for Fe_3O_4/APTES (A) to pH = 6.2

- Influence of the method of dispersion. For the Fe_3O_4/APTES (A-DM) sample, after stirring on a magnetic stirrer, the IEP value shifts to the acidic region to pH 6.6, and after ultrasound - to pH 6.2 (Fe_3O_4/APTES (A-D-Us) sample (Fig. 2).

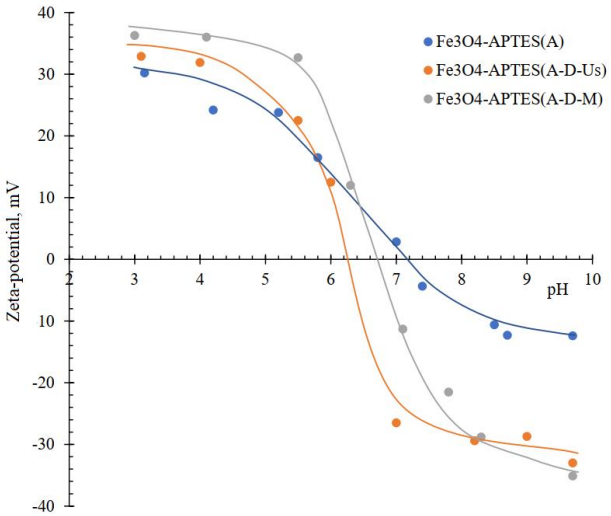

Fig. 2 dependence of the ζ-potential of Fe_3O_4/APTES (A), Fe_3O_4/APTES (A-D-Us) and Fe_3O_4/APTES (A-D-M) nanoparticles on pH (0,01 M KCl) after dialysis.

The average hydrodynamic diameter (Dh) of nanoparticles depends on the pH value. The pH at which NPs have the maximum size fully correlates with the IEP pH.

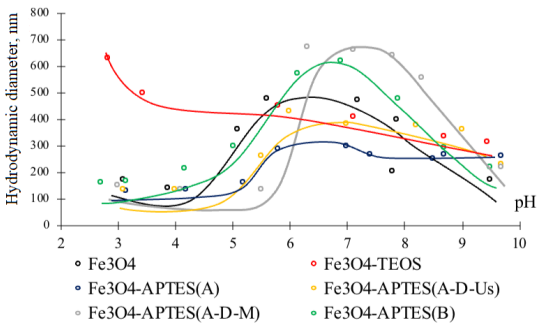

Fig. 3 change in the average hydrodynamic diameter of nanoparticles at different pH and modification agent.

In the range of pH >9, the average hydrodynamic size is 100–400 nm for all samples; at pH <5, the values remain the same except for TEOS, for which the size is 450–650 nm. An increase in the hydrodynamic diameter of Fe_3O_4/TEOS particles in the range of pH = 3 is due to the proximity to the isoelectric point and aggregation of nanoparticles. In addition, the difference in values between the Fe_3O_4/TEOS sample and the rest of the samples outside the isoelectric point (about 450 nm for Fe_3O_4/TEOS and about 200 nm for the rest of the samples) is probably due to the difference in the time of sample preparation and measurement of the hydrodynamic diameter. This means that the effective hydrodynamic diameter of the modified nanoparticles is strongly dependent on the modification agent and the way of synthesis. Lastly, the change in the average hydrodynamic diameter of nanoparticles at different pH and modification agent is presented in Fig. 3.

3. Conclusion

A decisive role in the study of surface properties is played by cleaning from low molecular weight impurities that can screen the surface of nanoparticles or bind with an indifferent electrolyte. The stage of dispersive post-preparation of samples is also important for the correct determination of the sorption capacity and hydrodynamic diameter of particles as it was clearly seen from the electrokinetic measurements. Thus, dispersion on a magnetic stirrer leads to an increase in the sorption capacity of the sample in comparison with ultrasonic dispersion, which causes irreversible destruction of the core-shell nanoparticle structure due to an increase in temperature and pressure in the cavities.

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