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Preparation and Characterization of Magnetite – Silica Core – Shell Nanoparticles

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Abstract—In this study, two types of ligands were introduced onto the surface of magnetite nanoparticles by hydrolysis and condensation of organosilicon reagents: organosilane-tetraethoxysilane (TEOS) and aminoorganosilane - aminopropyltriethoxysilane (APTES). It is shown that coatings based on SiO_2 solve a double problem: first, they prevent the aggregation of nanoparticles and the oxidation of magnetite; secondly, they allow the surface to be modified with various specific ligands for biomedical applications due to terminal groups. It was shown, that after the modification of TEOS and APTES (in argon and in air), the Fe_3O_4 content decreases to 66, 42, and 36%, respectively. The formation of a silicon framework on the magnetite surface due to Fe-O-Si and Si-O-Si bonds was determined by IR spectroscopy. The identification of surface amino groups is complicated due to the superposition of absorption bands of NH_2- and OH-groups. This opens new prospective for creation of tailored nanocomposites containing magnetite nanoparticles. These materials can be further used as sorbents for various applications.

Keywords—Nanoparticles, core-shell, magnetite, silica.

I. Introduction

In order to solve possible problems with the introduction of magnetite nanoparticles into a living organism, such as instability under physiological conditions [1]-[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]-[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]-[35].

Magnetite nanoparticles Fe_3O_4 have a wide range of applications - from magnetic separation of universal technical media to the preparation of materials for biomedicine. The properties of magnetic nanoparticles are significantly affected by phase transformations as a result of their modification with various compounds, as a result of which the modified surface layer of magnetic nanoparticles can have completely different magnetic characteristics than the particle core [36]-[43]. In this regard, the methods of obtaining magnetic nanoparticles are combined with various methods of their stabilization, using protective shells of different nature [44]-[53]. Modification of the surface of nanoparticles allows not only to ensure their stability in biological media with high ionic strength, but also to control the nature of their interaction with objects, which determines the biocompatibility of nanoparticles [54]-[76]. In this case, already surface characteristics stand out as one of the most important, if not the main, determining factors of the bioactive properties of nanoparticles. For this purpose, magnetite nanoparticles of the "core-shell" type are widely used, which have an inner core of iron oxides (Fe_3O_4) with an outer protective shell made of silicon dioxide. The most

widely used are two types of silanol ligands: with terminal OH-groups and terminal NH_2-groups.

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]-[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]-[63].

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]-[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]-[57], which can fix a large number of biologically active substances [32]. The most common way to obtain LF 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].

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.

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).

II. Preparation and Study of the Properties of Magnetite Nanoparticles Coated with Silicon Dioxide

By studying the processes that occur during the interaction of ligands with nanoparticles, it is possible to understand the stabilization mechanism, and most importantly, the nature of the bond at the forming interface [32]-[38]. It is also necessary to take into account the fact that the processes of nanoparticle enlargement and the adsorption of macromolecules on the surface of both initial and formed particles proceed in parallel and, accordingly, influence each other. The size and polydispersity can be controlled within a fairly short nucleation period, because the end of the nucleation process means a finite number of particles. Nucleation, i.e. nucleation is the key to the crystallization process by controlling crystal shape and nanoparticle size distribution.

The first stage of modification of the surface of silica using APTES in an aqueous medium consists in the hydrolysis of alkoxysilyl groups with the formation of silanol groups Si-OH. Further, the silanol groups of the modifier react with OH-groups on the silica surface, releasing water, with the formation of an anchor bond Si-O-Si-C. The introduction of APTES on the surface of Fe3O4 nanoparticles in our case is justified by the presence of reactive amino groups, which can subsequently interact with any classes of compounds in order to obtain functional and hybrid materials. The disadvantages of the method for modifying the surface of silicas with organosilanes in an aqueous medium include the possibility of a side process of polymerization of functional organosilanes.

III. Results and Discussion

To study the crystal structure and lattice parameters of the synthesized powder, X-ray phase analysis (XRF) and analysis of the X-ray line profile were carried out on a DRON-UM-2 diffractometer in the Bragg-Brentano geometry using CrKα radiation. The values of the current and voltage across the X-ray tube were 20 mA and 40 kV, respectively. The set of spectra was carried out in the continuous scanning mode at a detector movement speed of 1 rpm. The analysis of the phase composition was carried out in a platinum cell, in two modes of temperature control and a set of X-ray spectra. Consider the diffraction patterns of the obtained samples.

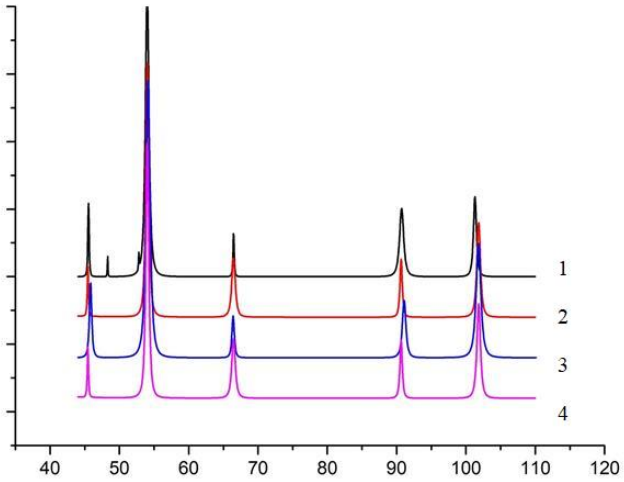

Fig. 1 Diffraction patterns of nanoparticles: 1) Fe_3O_4, 2) Fe_3O_4/TEOS, 3) Fe_3O_4/APTES (A) and 4) Fe_3O_4/APTES (B).

From the Fig. 1 it is seen that distinguished peaks of magnetite as well as silica can be observed, this proves the creation of nanocomposite material.

In accordance with the values of the Miller indices hkl and interplanar distances d obtained from the Match! Program, the crystal lattice parameters were calculated:

$$\text{a:}\frac{1}{d^2} = \frac{(h^2+k^2+l^2)}{a^2}$$

where a – unit cell parameter, angstrom, hkl – Miller indices, d – interplanar distance, angstroms. The interplanar spacing and Miller indices were obtained from the data of the Match! when processing spectra.

Magnetite can have a range of oxidation states depending on the amount of structural Fe^{2+}, which is the stoichiometry of magnetite (x = Fe^{2+} / Fe^{3+}). For magnetite with an ideal Fe^{2+} content (formula Fe_3O_4) stoichiometric parameter is x = 0,50. As magnetite oxidizes, the Fe^{2+} / Fe^{3+} ratio decreases (x <0,50), and this form is either nonstoichiometric or partially oxidized magnetite. Fully oxidized magnetite (x = 0) is maghemite (γ-Fe_2O_3). For nonstoichiometric magnetite, the structural formula is often written as $Fe_{3-\delta}O_4$, where δ can vary from zero (stoichiometric magnetite) to 1/3 (completely oxidized). Stoichiometric parameters can be calculated from the following relationship:

$$x = \frac{Fe^{2+}}{Fe^{3+}} = \frac{1-3\delta}{2+2\delta}$$

The x value is found from linear interpolation between two extreme points - magnetite and maghemite. For magnetite X=0.5, a=8.396–8.400 Å. For magnetite X=0 a=8.33–8.34 Å. The lattice parameters obtained in this work are less than those known for magnetite 8.396–8.400 Å.

The XRF data showed that the main phase formed during coprecipitation in the presence of TEOS is Fe_3O_4 (with a% content of 67%) in a γ-Fe_2O_3 shell with a% content from 33%). Upon functionalization of APTES%, the content of Fe_3O_4

decreases to 42% and 36%. The sizes of the regions of coherent scattering of X-rays corresponding to the sizes of nanoparticles were determined by the Debye-Scherrer formula:

$$D = \frac{k*\lambda}{\beta*\cos\theta},$$

where D - crystallite size, λ - x-ray wavelength, 2θ – diffraction angle, β– reflex width at half height after correction for instrumental broadening; k – Scherrer's constant (particle shape factor). We assume that the particles are spherical. For them k=0,94. The average sizes of nanoparticles calculated by the Scherrer equation are equal for magnetite 18 nm, Fe_3O_4/TEOS 12 nm, Fe_3O_4/APTES (A) 10 nm, Fe_3O_4/APTES (V) 9 nm. Thus, the Scherrer equation relates the crystallite size to the width of the diffraction peaks.

Using the method of IR spectroscopy, the mechanism of stabilization and functionalization of Fe_3O_4 NPs by the organosilicon reagent APTES and TEOS was studied. It is believed that the surface silanol groups Si-OH located on the SiO_2 surface can easily react with various binding agents for covalent attachment of ligands to the surface of Fe_3O_4 nanoparticles. In order to form a coating based on APTES, a well-known technique was used, according to which a significant amount of the deposited substance is concentrated on the NP surface as a result of in situ hydrolysis and condensation of the sol-gel precursor.

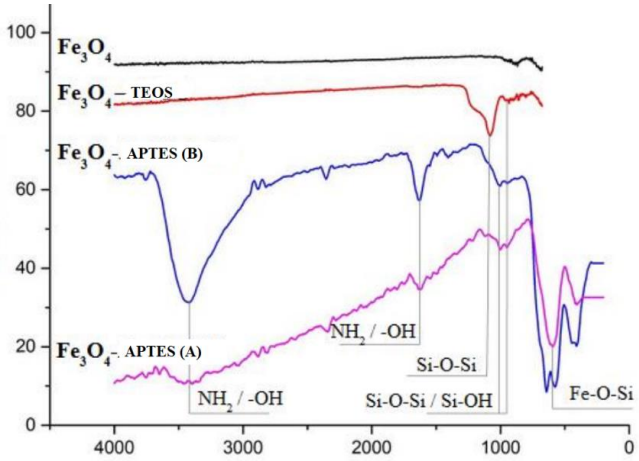

Fig. 2 ir spectra of samples: Fe_3O_4, Fe_3O_4/TEOS, Fe_3O_4/APTES (A), Fe_3O_4/APTES (B).

From the Fig. 2 it is seen that the ability of Fe_3O_4 to adsorb SiO_2 is due to the fixation of OH groups on the magnetite on the surface of Fe_3O_4, which is confirmed by the appearance in the spectrum for Fe_3O_4/APTES (A) and Fe_3O_4/APTES (B) of transmission bands at wavelengths of 886 and 944 cm^{-1}, characteristic of the Fe bond -OH (Fig. 2). The appearance of a band at 1080 cm^{-1}, characteristic of the Si-O-Si bond, confirms the formation of a silicon framework, which causes stabilization; however, different band depths indicate a different degree of polymerization of the samples. The low intensity of the band for the Si-O-Si siloxane bond in the 1130 cm^{-1} region and the absorption band characteristic of the Si-OH silanol group at 992 cm^{-1} indicates a low degree of polymerization of the Fe_3O_4/APTES sample (A). When TEOS is added, absorption bands appear at 869 and 960 cm^{-1}, which are characteristic of the Si – O group. The appearance of a new band of framework vibrations and Si – O – Si bonds in TEOS in the region of 1080 cm^{-1} is observed. Bands ~ 3500 cm^{-1} and ~ 1650 cm^{-1} may indicate the presence of OH groups, as well as free and bound amino groups.

The results obtained illustrate the formation of core-shell structures by the example of a high-tech method of sol-gel synthesis, in which silicon oxides in the process of polycondensation cause self-organization of the forming metal-polymer structure, including regulation of the size of polymer fragments at the level of iron oxide nanoparticles.

IV. Conclusion

Fe_3O_4 nanoparticles functionalized with TEOS under conditions of acid and alkaline catalysis and APTES in different atmospheres were obtained. For the first time using the XRF method using the OriginPro and Match software! the effect of the ligand on the content of stoichiometric Fe_3O_4 was determined. Thus, after the modification of TEOS and APTES (in argon and in air), the Fe_3O_4 content decreases to 66, 42, and 36%, respectively. The formation of a silicon framework on the magnetite surface due to Fe-O-Si (760 cm^{-1}) and Si-O-Si (1130 cm^{-1}) bonds was determined by IR spectroscopy. The identification of surface amino groups is complicated due to the superposition of absorption bands of NH_2- and OH-groups (~ 3500 cm^{-1} and ~ 1650 cm^{-1}). This opens new prospective for creation of tailored nanocomposites containing magnetite nanoparticles. These materials can be further used as sorbents for various applications.

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