Investigation of Adsorption Capacity of Magnetite Nanoparticles

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Abstract—In this work, the preparation of Fe_3O_4 nanoparticles modified with 3-aminopropyltriethoxysilane (APTES) was carried out under various synthesis modes (in air or in argon). The zeta potential and hydrodynamic diameter of Fe_3O_4-APTES nanoparticles were determined by the method of dynamic and electrokinetic light scattering. The effect of humic acids on the zeta potential, hydrodynamic diameter and colloidal stability of Fe_3O_4-APTES at different pH values was established by the method of dynamic and electrophoretic light scattering. It has been shown that changes in the conditions of the synthesis of nanoparticles of one component composition Fe_3O_4-APTES (argon) and Fe_3O_4-APTES (air) (in an inert medium and in an air atmosphere, respectively) lead to a change in the charge of the particle surface and a subsequent change in the sorption properties with respect to HA.

It was demonstrated that the decisive role in the study of surface properties is played by the purification 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.

Keywords—Nanoparticles, adsorption, magnetite, particle size.

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1. Introduction

AMONG the wide range of investigated nanoscale materials for various environmental and biomedical applications, magnetic nanoparticles have received considerable attention due to their intrinsic magnetic properties, making them successful as magnetically reduced catalysts, drug delivery agents, anti-cancer materials, magnetic resonance imaging, etc. This class of nanomaterials includes metallic, bimetallic nanoparticles, metal oxides, ferrites, and superparamagnetic iron oxide nanoparticles. Magnetic nanoparticles and nanocomposites have generated significant scientific and technological interest due to their potential applications in biomedicine, information technology, magnetic resonance imaging, catalysis, telecommunications, and environmental restoration [1],[2],[3],[4],[5],[6],[7],[8],[9],[10], [11],[12],[13],[14]. Magnetic nanocomposites usually contain magnetic nanoparticles embedded in a non-magnetic or magnetic matrix. However, magnetic nanoparticles dispersed in composites usually have a strong tendency to form agglomerates to reduce the energy associated with the high surface area to volume ratio of nanosized particles [14],[15]. To avoid aggregation of magnetic nanoparticles, protection strategies have been developed to chemically stabilize unprotected magnetic nanoparticles by grafting or coating with organic species, including surfactants or polymers, or coating with an inorganic layer such as silicon dioxide or carbon. Combining these functionalized magnetic nanoparticles in a polymer or other matrices to develop magnetic nanocomposite materials has proven to be more efficient [25],[26],[27],[28],[29],[30],[31],[32]. There are mainly four types of magnetic nanocomposites, i.e. inorganic core nanocomposites, self-assembled nanocomposites, silica-based magnetic nanocomposites, and organic-inorganic nanocomposites [33],[34],[35],[36], [37],[38],[39],[40],[41],[42],[43],[44],[45],[46],[47],[48],[49], [50]. Among them, organic inorganic magnetic nanocomposites have become more interesting due to the combination of the unique properties of the organic and inorganic components in one material. Hybrid organic inorganic magnetic nanocomposite materials can be obtained in situ, ex situ, microwave exposure, coprecipitation, melt mixing, ceramic glass treatment, and plasma polymerization methods.

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).

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.

Magnetic nanoparticles can act as a new class of non-toxic and effective flame retardants. Fe_3O_4 enhanced both thermal stability and flame retardant properties of polyvinyl alcohol. Nanoparticles were synthesized by a simple precipitation reaction without using an inert atmosphere at room temperature. The nanoparticles exhibited ferromagnetic behavior at room temperature. To obtain a magnetic nanocomposite, Fe_3O_4 nanoparticles were added to polyvinyl alcohol. Dispersed nanoparticles play the role of a magnetic barrier layer, which slows down the volatilization of the product and prevents the penetration of oxygen into the sample during polymer decomposition.

The effectiveness of magnetic nanoparticles for specific biomedical applications depends on the magnetic properties of the particles, their size, and most importantly, the surface chemistry [1],[2],[3],[4],[5],[6],[7],[8],[9]. Of particular importance in the biomedical application of colloidal magnetic systems is their stability, which depends on the effective stabilization of nanoparticles, which would ensure the presence of individual nanoparticles rather than agglomerates in solutions, while not significantly affecting the magnetic properties of the material and preserving the particle surface suitable for further functionalization [10],[11],[12],[13],[14],[15],[16], [17],[18].

For this purpose, magnetite nanoparticles of the "core-shell" type are widely used, having an inner core of iron oxides (Fe_3O_4) with an outer protective shell of silicon dioxide (SiO_2). SiO_2-based coatings solve a double problem: first, they prevent the aggregation of nanoparticles and the oxidation of magnetite (both problems are fundamental in the transport of nanomaterials through the bloodstream); second, they allow the surface to be modified with various specific ligands for biomedical applications [19],[20],[21], [22],[23],[24],[25]. In the present study, an aminoorganosilane - aminopropyltriethoxysilane (APTES) was introduced onto the surface of magnetite nanoparticles by hydrolysis and condensation of organosilicon reagents. The use of humic acids (HA) for the further functionalization of nanoparticles meets the requirements of "green" chemistry due to their unique properties, such as non-toxicity, biocompatibility, stability and polyfunctionality, which allows the binding of pharmaceuticals through the formation of hydrophobic bonds or metal ions by the mechanisms of physical sorption and ion exchange.

However, despite the unprecedented progress in the field of obtaining new data on nanoparticles based on HA and APTES, due to the specific ecological interests of the authors of the work, only an analysis of the sorption properties of the obtained materials is provided without considering and taking into account the properties in real aqueous systems where these drugs are used, are not studied processes transformation of the starting material under changing environmental conditions, ultimately causing a change in the properties of the nanoparticles used [56],[57],[58],[59],[60],[61],[62], [63],[64],[65],[66],[67],[68],[69],[70],[71],[72],[73]. These circumstances predetermined the goal and objectives of this study.

The aim of this work was to obtain and study the electrokinetic and sorption properties of Fe_3O_4/ SiO_2 nanoparticles using the example of magnetite nanoparticles modified with 3-aminopropyltriethoxysilane.

2. Study of the sorption properties of magnetite

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) [31],[32],[33], [34],[35],[36],[37],[38],[39],[40],[41],[42],[43],[44],[45],[46], [47].

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.

In order to derive the equation of the adsorption isotherm, a number of simplifications are introduced. All the places where the adsorbed particles are fixed are the same, and adsorption on one of them does not affect the state of the other. The interaction between the adsorbed particles is negligible. The adsorption layer is monomolecular, i.e. it consists of one layer of molecules. In this case, the bond of the adsorbate with the adsorbent is sufficiently strong, which excludes the movement

of the adsorption complex along the surface of the adsorbent (localized adsorption).

Active (well absorbing) adsorbents have a very large specific surface area. For example, the absorbers most often used for scientific purposes and industrial practice - activated carbon, silica gel, zeolites – have s_0 up to several hundred and even thousands of square meters per 1 g.

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 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].

The effect of HA adsorption on the charge of Fe_3O_4, Fe_3O_4-APTES (argon) and Fe_3O_4- APTES (air) particles at different pH was studied by adding HA until complete coverage of the nanoparticle surface was achieved (until a plateau appeared). At low HA concentrations, complete coverage of the nanoparticle surface does not occur. According to [38], HA has a negative charge over the entire investigated pH range 3-10. The adsorption of polyanionic HA on Fe_3O_4 NPs causes an inversion of the zeta potential in the acidic region; negative charges on NPs gradually become dominant. Reaching a negatively charged surface over the entire pH range (3-10) indicates complete coverage of the HA surface. The maximum negative zeta potential that can be achieved with increasing HA concentration is -40 mV. In the case of incomplete coverage of magnetite nanoparticles with humic acids, the value of the ξ-potential of the samples is in the range of + 20 mV <ξ <-20 mV at pH <7-8, which indicates the instability of the nanoparticle system. Upon reaching full HA coverage, the zeta potential becomes ξ> -20 mV over the entire pH range.

3. Aggregate stability of Fe_3O_4-APTES nanoparticles

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 introduction of HA of various concentrations into a system with various nanoparticle samples led to a change in the sign of the ξ potential and its increase to -20 mV. As can be seen from the data in Fig. 1, the value of the ξ-potential of Fe_3O_4-APTES nanoparticles decreases to zero (that is, the surface charge of the nanoparticles is completely neutralized) upon adding a certain concentration of HA. With an increase in the concentration of adsorbed HA, the ξ-potential of the particles increases to a certain value and then reaches a plateau due to the probable saturation of the NP surface (binding of all surface Fe_3O_4-APTES groups). The adsorption capacity of NP samples with respect to HA significantly exceeds the point of compensation of the electrostatic charge (ξ = 0 mV), which indicates that not only electrostatic bonds are involved in the binding of HA to NPs.

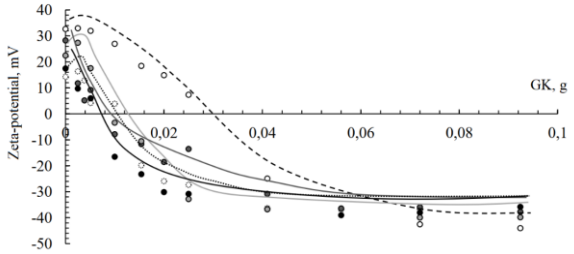

Fig. 1 Influence of HA concentration on NP charge and aggregate stability of dispersions (pH ~ 5, KCl = 0.01 M).

Without HA or with the addition of a low HA concentration (up to 0.015 g/g NPs), Fe_3O_4 and Fe_3O_4-APTES (argon) nanoparticles aggregate and, as a result, settle, which is associated with a low value of the ξ potential due to a low charge density. At the same time, samples of Fe_3O_4-APTES (I-D-M), Fe_3O_4-APTES (air), and Fe_3O_4-APTES (I-D-Us) nanoparticles remain stable at pH ~ 5 without adding HA. With the addition of 0-0.002 g of HA per 1 g of NPs, the samples of Fe_3O_4-APTES (air) and Fe_3O_4-APTES (I-D-Us) become unstable, aggregate and precipitate. However, the Fe_3O_4-APTES (I-D-M) sample does not aggregate even at a low HA concentration (up to 0.02 g/g) and becomes stable with the addition of 0.038 g/g HA, probably due to the initial higher intrinsic surface charge.

Influence of HA on the hydrodynamic diameter and polydispersity coefficient of nanoparticles. The average

hydrodynamic diameter Dh of nanoparticles depends on the pH value. Before the introduction of HA in the region of the isoelectric point (5 <pH <9), NPs have an average hydrodynamic diameter from 300 to 700 nm (Fig. 2). The pH at which NPs have the maximum size fully correlates with the IEP pH value.

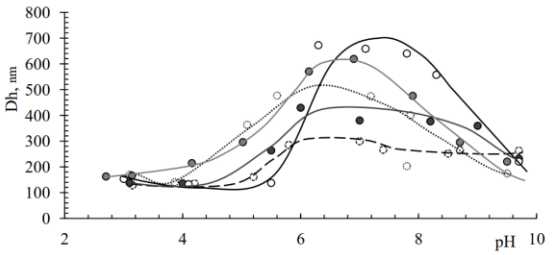

Fig. 2 Change in the average hydrodynamic diameter of nanoparticles at different pH values.

In the range of pH <5 and pH> 9, the average hydrodynamic size is 100–400 nm. The values of the hydrodynamic diameter in the region of the isoelectric point increase in the series: Fe_3O_4- APTES (argon) < Fe_3O_4- APTES (I-D-US) < Fe_3O_4 < Fe_3O_4- APTES (air) < Fe_3O_4- APTES (I-D-M). According to the data obtained, magnetic particles with an APTES shell have a hydrodynamic diameter of 33.9 nm and a polydispersity index of 0.512. The suspension remains stable for more than 3 months at room temperature. The small hydrodynamic diameter of nanoparticles may indicate the complete polymerization of APTES (the formation of a Si-O-Si bond and the absence of OH groups on the surface). In our case, for Fe_3O_4-APTES (air) and Fe_3O_4-APTES (I-D-M) nanoparticles, it can be assumed that large Dh values are associated with incomplete polymerization of silanes.

The hydrodynamic size of other Fe_3O_4-APTES (air) and Fe_3O_4-APTES (I-D-M) samples correlates with the zeta potential: NPs have the largest sizes, which leads to the sorption of a larger amount of HA due to a larger number of sites for the sorption of both HA COO groups and OH groups on the surface of nanoparticles.

The value of the polydispersity index (PI) also depends on the position of the IEP. In the IEP region (pH ~ 6-7), the PI value is lower than for other pH regions. For example, in the case of Fe_3O_4 at pH 5-7, the IP values = 0.23-0.27 indicate a more uniform distribution of nanoparticles in size, compared to that at pH> 8 and pH <5, for which the IP varies from 0.31 to 0.43. With the addition of HA, the average hydrodynamic particle size Dh also depends not only on the position of the IEP, but also on the concentration of HA, which shift the IEP to the left into the acidic region. For all NPs, the average hydrodynamic size decreases with an increase in the HA concentration and, accordingly, the degree of coverage of the NP surface. So, if the surface of NPs is not completely covered with humic acids, then Dh depends on pH. With full HA coverage, the average hydrodynamic size does not depend on pH and remains constant - 130-150 nm.

The probability functions of the distribution of hyperfine parameters for high-temperature Mössbauer spectra are different for different samples. Thus, for a sample of native particles, the field distribution has the form of a predominantly asymmetric bimodal peak, with maxima localized in the region of high fields, and an extended "wing" extending into low fields. In the sample of coated particles (with the same predominant asymmetric bimodal peak), this wing is slightly more resolved - three local maxima are clearly manifested on it. Before the beginning of this "wing", two weak "satellites" are observed for both samples in the region of 255 and 300 kOe. In the sample of oxidized particles, in contrast to their predecessors, there is practically no "fine structure" on the dependence of the probability of the distribution of magnetic fields - the distribution has the form of a noticeably wider unimodal peak, with the weighted average shifted to lower fields. This indicates a much wider size distribution of the sample particles (variants of the local environment). It is obvious that the reason for the distortions of the relaxation nature in the Mössbauer spectra of the samples is the small sizes of the iron-containing domains, which allows us to consider the description of the spectra within the framework of the model of multilevel superparamagnetic relaxation.

An examination of the data obtained makes it possible to show that systematic changes are observed in the experimental spectra, which are similar at different pH values (Fig. 2). This fact can give us a conclusion about the stability of nanoparticles modified by the APTES. 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 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.

4. Conclusion

Thus, it has been shown that changes in the synthesis conditions of nanoparticles of one component composition Fe_3O_4-APTES (argon) and Fe_3O_4-APTES (air) (in an inert medium and in an air atmosphere, respectively) lead to a change in the charge of the particle surface and a subsequent change in the sorption properties in relation to HA. A decisive role in the study of surface properties is played by the purification from low-molecular-weight impurities that can screen the surface of nanoparticles or bind with an indifferent electrolyte, because the presence of low-molecular-weight electrolytes can shift the double electric layer at the surface of the particles. The stage of dispersive post-preparation of samples is also important for the correct determination of the sorption capacity and hydrodynamic diameter of particles. The

important finding is that the preparation of the dispersion on a magnetic stirrer leads to an increase in the sorption capacity of the sample as compared to ultrasonic dispersion, which causes irreversible destruction of the core-shell nanoparticle structure due to an increase in temperature and pressure in the cavities. The last fact needs to be further investigated in the following studies in order to optimize the parameters of ultrasonic action. The data obtained opens the prospective for tailored fabrication of functional nanocomposites.

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