Investigation of Thermophysical Properties of Nanomagnetite-based Polymer Materials

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Abstract—In this work, samples of magnetite nanoparticles are synthesized using various synthetic methods. It is shown that of the existing methods of magnetite synthesis, the most widespread is the method of coprecipitation of Fe^{2+} and Fe^{3+} salts. Less common, but of interest due to the slower and more controlled kinetics of particle nucleation, is the Fe^{2+} oxidation method. However, magnetite is unstable and under external influences its phase transition to maghemite can occur, which leads to a change in magnetic characteristics and a change in biological responses. After analyzing the results of the study of samples by the XRD method, the following conclusions can be drawn: The magnetite phase was identified by the characteristic peaks in the diffraction patterns for all samples, except for the sample with EDTA, which may be a consequence of an insufficient amount of the introduced oxidant, or poor diffusion of the oxidant to iron ions due to the formed chelate complex. It can be concluded that magnetite is a promising material for its use in industry and medicine.

Keywords—Nanoparticles, magnetite, polymers, mechanical properties.

I. INTRODUCTION

CURRENTLY, much attention is paid to the field of nanotechnology, which is one of the most promising and rapidly developing. A large number of works are underway to study the use of magnetic nanoparticles in various fields, including industry and medicine [1]-[12]. Most of the applied magnetic nanoparticles are iron oxide nanoparticles, namely magnetite Fe_3O_4. Among the methods for obtaining magnetite

nanoparticles, liquid-phase methods are mainly used, which are much easier to perform and require less energy and economic costs [13]-[24]. Among them, the most common method: the method of coprecipitation of iron salts (II, III). The advantages of the method include the rapidity of synthesis due to the rapid nucleation of nanoparticles, the ease of synthesis due to one-stage mixing of salts in an alkaline medium, a high yield of the target product due to the complete conversion of the starting components, as well as the possibility of differentiation of the morphology and composition of nanoparticles at the synthesis stage [25]-[41]. However, despite the indicated advantages, this method also has a number of disadvantages. The main problems in the rapid nucleation of nanoparticles are the problem of separation of nucleation and growth of nanoparticles, as well as the problem of the need to control after nucleation over the growth processes of monodisperse nanoparticles, which are reflected in the uniformity of nanoparticle size distribution.

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 [42]-[68]. 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.

There are several ways to synthesize composite nanoparticles [25]-[41].

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]-[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.

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

Nanoparticles, even with a very low volumetric content (less than 1%), are contained in such a fragment in a very large amount, and it is impossible to model their effect at this scale level. For example, a cubic fragment of a 1 μm matrix contains more than thousand nanoparticles for a given volumetric content. Therefore, in particular, the nano-modified binder is white, while the usual binder is yellow. To model such materials, it is necessary to resort to multiscale approaches and to carry out a consistent determination of effective properties at various scale levels. This task is greatly simplified if the properties of the nanomodified matrix are known from experiments. The missing characteristic is Poisson's ratio, which can be approximately taken unchanged, or estimated on the basis of analytical calculations using the found value of the "effective" volumetric content of the filler, which was done. Further, it suffices to numerically solve the averaging problem on a representative fragment containing only nanoparticles.

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]-[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].

II. Synthesis of the Magnetite Nanoparticles

One of the main characteristics that magnetite nanoparticles used in immunoassay and clinical diagnostics should possess today is a narrow nanoparticle size distribution. To obtain monodisperse magnetite particles, it is necessary to separate the process of ion nucleation in solution and the growth of particle crystals. Thus, the coprecipitation method allows one to obtain nanoparticles with a rather wide size distribution (coefficient of variation of sizes > 20%).

Table 1. List of obtained samples of magnetite nanoparticles.

Sample	Sample description
M - E	Synthesis of magnetite from FeSO4·7H2O and EDTA at room temperature
M - X	Synthesis of magnetite from FeCl2
M - CM	Synthesis of magnetite from $FeSO_4 \cdot (NH_4)_2 SO_4 \cdot 6H_2O$
M - C	Synthesis of magnetite from FeCl3·6H2O и FeCl2·4H2O

Table 1 gives the overview of the synthesized samples of nanoparticles with different conditions of synthesis.

III. RESULTS AND DISCUSSION

To study the phase and microstructural characteristics, an X-ray phase analysis was carried out. 3 samples were studied: M - E, M - CM, M - C. The sizes of the regions of coherent scattering of X-rays corresponding to the sizes of nanoparticles were determined by the Scherrer formula.

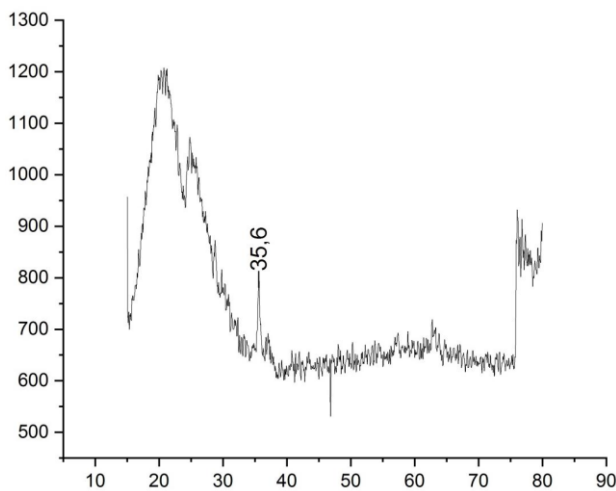Consider the diffraction pattern of the M - E sample shown in Figure 1.

Fig. 1 Diffraction pattern of sample M - C in coordinates intensity – two theta.

Sample M - E was obtained by oxidation of Fe^{2+} from a complex with EDTA at room temperature. The diffractogram for this sample shows one peak ($2\theta = 35$), which is characteristic of the magnetite phase, but the other peaks are absent. On this basis, it can be said that the crystal structure of magnetite was not formed during the synthesis. This may be due to an insufficient amount of an oxidizing agent, or to a weak diffusion of an oxidizing agent to Fe^{2+} ions due to a too strong steric barrier formed by EDTA.

The diffractogram of the M - C sample obtained by coprecipitation of iron (II, III) chlorides is shown in Figure 2.

The peaks in the diffractogram of the sample were identified as belonging to the magnetite phase.
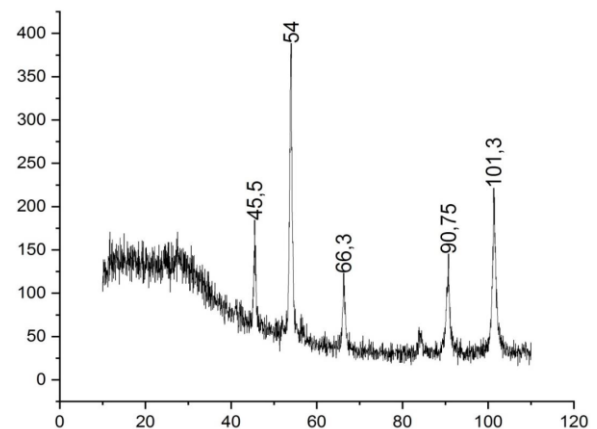

Fig. 2 Diffraction pattern of sample M – C in coordinates intensity – two theta.

XRD results for sample M - C and calculations of the stoichiometric composition showed that the sample is dominated by the magnetite phase (93%).

Also, for sample M - C, according to the results of XRD, a histogram of the particle size distribution was plotted, shown in Figure 3. Analyzing the histogram of the particle size distribution, it can be concluded that the particles are unevenly distributed, that is, there is a polymodal distribution.
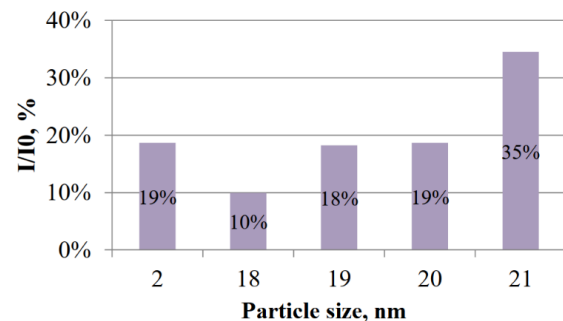

Fig. 3 Histogram of particle size distribution in sample M - C.

The diffractogram of sample M - X is shown in Fig. 4, it clearly shows resonance peaks characteristic of magnetite.
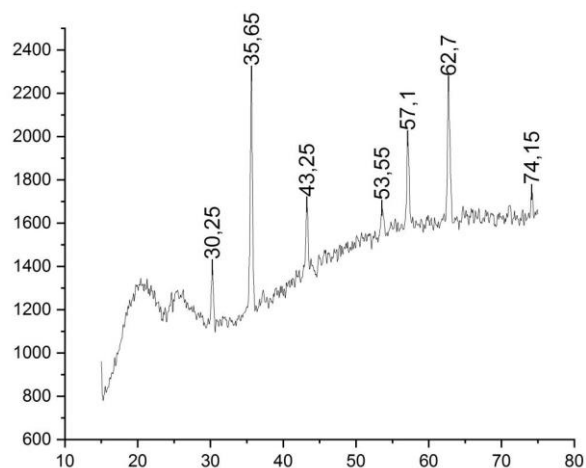
Fig. 4 Diffraction pattern of sample M – X in coordinates intensity – two theta.
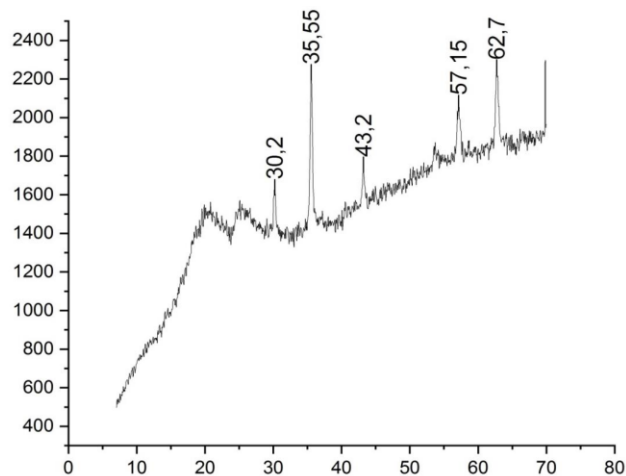

Fig. 6 Diffraction pattern of sample M – CM in coordinates intensity – two theta.

The XRD results show that the phase composition of stoichiometric magnetite is even lower than that of the M - C sample, which may be due to the insufficient amount of the introduced oxidant. This results in the lack of the oxygen in the reacting system and so affects the stoichiometry of the magnetite.

On the histogram of the particle size distribution plotted for the M - X sample, shown in Figure 5, you can see a large percentage of particles of practically the same size 25-26 nm, that is, the distribution is bimodal.
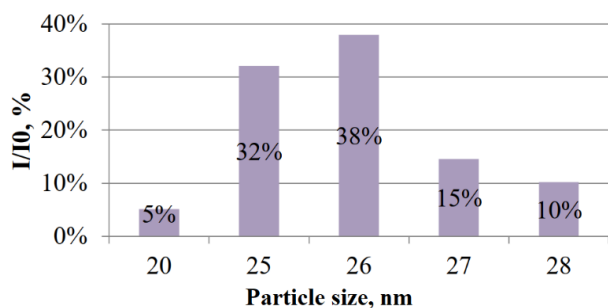
According to the XRD data for the M - CM sample, it can be noted that even less percentage of stoichiometric magnetite (88%) is observed in the composition of particles than in the previous two samples, which may be a consequence of either an insufficient amount of the introduced oxidant, or hindered diffusion of the oxidant to Fe^{2+} ions due to the steric structure of the Mohr salt chelate complex.

Based on these data, a histogram of the particle size distribution was built, shown in Figure 7. More than half of the particles were of the same size equal to 23 nm, that is, a monomodal distribution was observed.
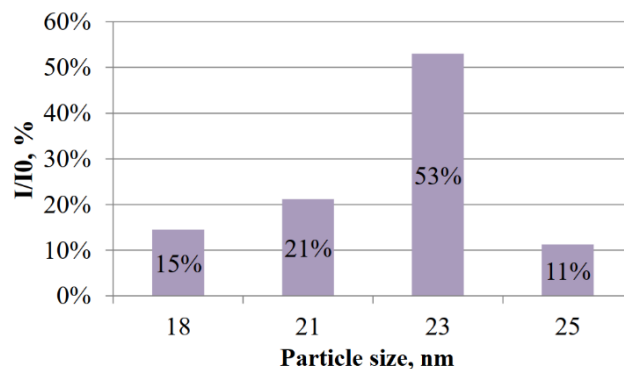

Fig. 5 Histogram of the distribution of particles in the sample M-X.

Fig. 7 Histogram of the particles distribution in the sample M-CM.

The M - CM sample was also studied by XRD. Its diffractogram is shown in Figure 6, in which the resonance peaks are identified as corresponding to the magnetite phase.

Thus, the synthesis by the oxidation of iron ions from a complex with a higher binding constant, compared to the binding constant of $FeCl_2$, actually allows one to obtain particles with a monomodal size distribution. This result demonstrates that the synthesis of magnetite from $FeSO_4 \cdot (NH_4)_2 SO_4 \cdot 6H_2 O$ if favorable in view of homogeneity and allows to obtain high quality nanoparticles.

IV. CONCLUSION

After analyzing the results of the study of samples by the XRD method, the following conclusions can be drawn: The magnetite phase was identified by the characteristic peaks in the diffraction patterns for all samples, except for the sample

with EDTA, which may be a consequence of an insufficient amount of the introduced oxidant, or poor diffusion of the oxidant to iron ions due to the formed chelate complex; the closest to stoichiometric magnetite was the calculated composition of sample M - C, equal to 93%, for sample M - X this figure was 91%, and for sample M - CM 88%; sample M - C had a polymodal particle size distribution, sample M - X bimodal, and sample M - CM monomodal, which may indicate the effect of the binding force (complexation constant values) on the kinetics of nanoparticle formation, that is, on the dispersion of the resulting particles by size.

This opens new prospective for tailored fabrication of polymer nanocomposites with desired structure and properties. Further research can be aimed toward the development of nanocomposites with advanced mechanical properties.

To determine the structure of the obtained nanocomposite, the method of IR spectroscopy was used. IR spectroscopy allows first to get evidences of the interaction between nanoparticles and polymer matrix and, second, get the information about the structure of the resulting nanocomposite. For the details of the IR characterization of the polymer nanocomposites, it is referred to the literature [53]-[55].

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