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The Results of Research of Magnetic Fluid Nanoparticles by Microscopy and X-ray Methods

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This is a researching of dispersed, elemental and phase composition of magnetic fluids. Samples of magnetic fluids based on kerosene and vacuum oil are the objects of study. As the researching methods, we used scanning electron microscopy and atomic force microscopy as well as X-ray diffraction. There is a comparative analysis of data from these methods.

Keywords: Magnetic nanoparticles, Magnetic fluid, Scanning probe microscopy, Scanning electron and transmission electron microscopy, X-ray diffraction method, Particle size distribution curve.

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Nanodispersed magnetic fluid (MF) is a smart-material, because its physical properties could be controlled and managed [1]. Particularly, special interest is magnetic and rheological properties of such colloidal system, which strongly depend on the particle size distribution. Therefore, this work devoted to the study of nanoparticles of magnetic fluid is actual and has practical significance [2, 3].

Microscopy is one of the best-known research methods to determine ferroparticle size distribution in colloidal solutions. The atomic force, scanning electron and transmission electron microscopy are the most suitable types for such tasks. However, each of these methods has its own advantages and disadvantages, so it seems appropriate to carry out a complex research using various methods.

For this research, we used Fe_3O_4 magnetic fluids based on kerosene (MF1 – MF5) and mineral oil (MF-6). Samples MF1 – MF4 are from NIPI Gaspererabotka (Krasnodar, Russia) and samples MF5 – MF6 were synthesized in Scientific-Educational Centre 'SWSU - ICMM UB RAS' by method of chemical condensation [4, 5]. The physical parameters of the samples are shown in Table 1.

Scanning electron microscopy allows to estimate the sizes and the structural and morphological characteristics of nanoparticles agglomerates in low vacuum mode. Moreover, it is possible to determine the composition of elements in nanoparticles and agglomerates.

This research was carried out in The Centre for collective use of scientific equipment "Diagnosis of the structure and properties of nanomaterials", Belgorod State National Research University using scanning electron microscope Quanta 200 3D. Preparation of magnetic fluid samples was carried out as follows.

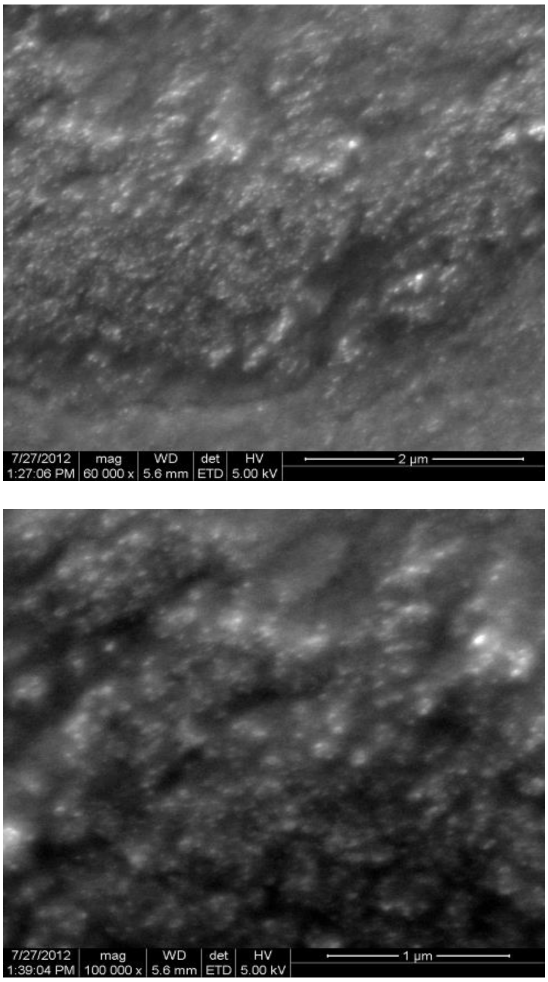


Fig. 1 – Electron microscope scans of magnetic nanoparticles

Table 1 – Parameters of magnetic fluids

Sample	Medium	Density ρ [kg/m^3]	Concentration of solid state φ [%]	Magnetization saturation M_S [kA/m]	Initial magnetic susceptibility χ
MF1	kerosene	1315	12	45.8	3.4
MF2	kerosene	1934	26	90.7	9.7
MF3	kerosene	886	1.94	9.7	0.52
MF4	kerosene	814	0.32	5	0.25
MF5	kerosene	1222	9.5	–	–
MF6	mineral hydrocarbon oil	1052	–	10.7	0.26

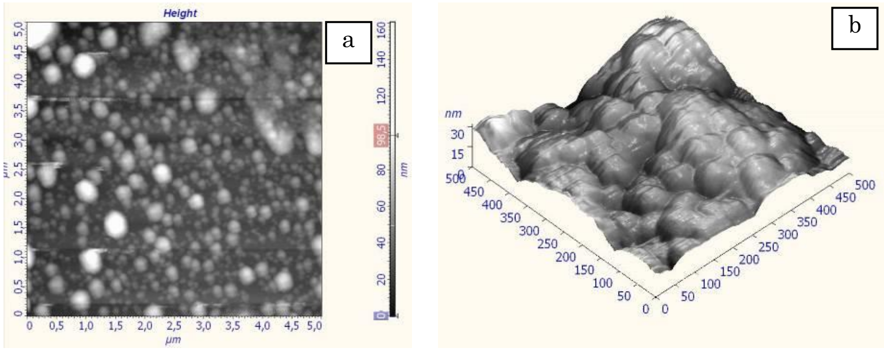
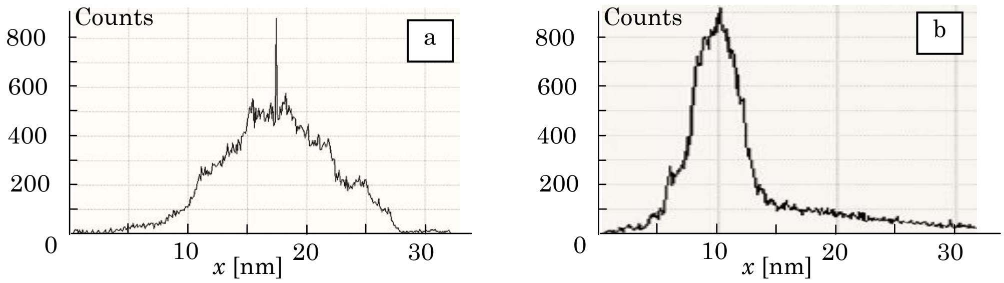


Fig. 2 – Morphology of surface with magnetic nanoparticles



Amount of sampling		65636
Max		32 nm
Average	17.4 nm	

Amount of sampling		65535
Max		81 nm
Average	13.6 nm	

Fig. 3 – Nanoparticle size distribution

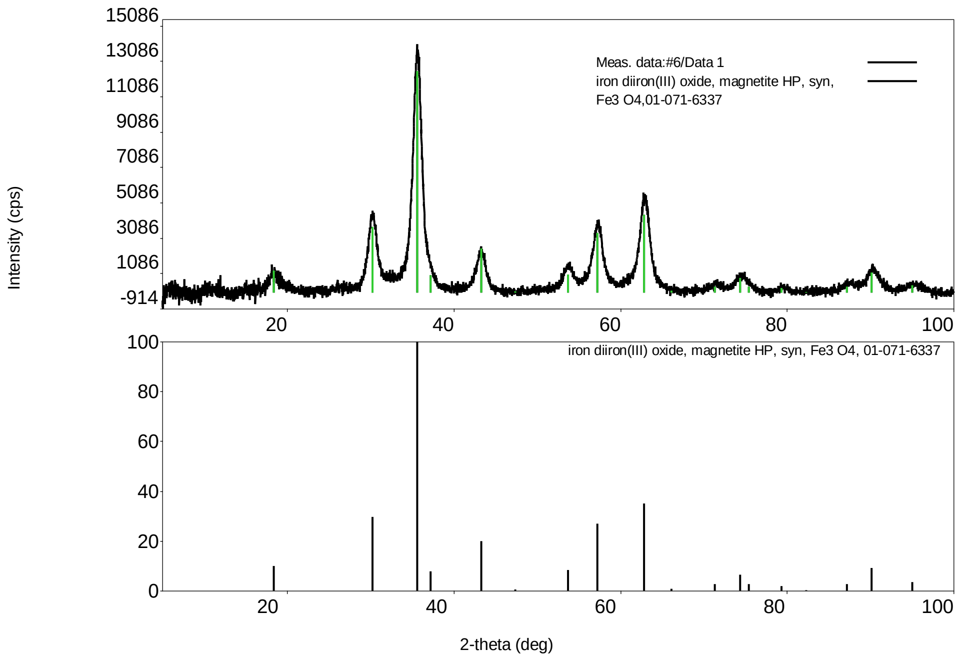


Fig. 4 – X-ray spectrum of MF6

We covered a clean glass surface with a thin layer of the solid phase extracted from the diluted magnetic fluid. Then this sample was being dried in an oven for 2 hours with protection from dust and other contaminants.

The obtained electron microscope scans (Fig. 1) allow to make only a rough size estimation of magnetic nanoparticles, but they are not clear enough for the statistical processing to get particle size distribution curves.

We also studied the composition of elements in nanoparticles of samples MF1-MF6 using Quanta 200 3D. Analyzing the obtained data for all samples, we can conclude that their main components are iron compounds with oxygen. However, the functionality of Quanta 200 3D does not allow us to get an answer if these oxides are magnetite or not.

We researched magnetic nanoparticle size distribution by atomic force microscopy using a scanning laboratory based on Integra platform. Fig. 2a shows the results for surface morphology in 2D for sample MF1. Based on analysis of 2D images and a profile of scanned surface we obtained 3D-representation of nanoparticles – see Fig. 2b.

Processed scans of scanning probe microscope by special software and algorithms, we investigated nanoparticle size distribution of the samples. Fig. 3 shows the results for MF1 (a) and for MF5 (b).

Analyzing the curves of particle size distribution, we conclude that the average particle size in the sample MF1 (from NIPI Gazpererabotka) is 17 nm, and in the sample MF5 (synthesized in SWSU) it is 14 nm. However, the range of diameter values in the sample MF5 is larger than in MF1, which indicates the higher polydispersity of MF5 comparing with MF1.

To determine the specific type of iron compound with oxygen in the solid phase of magnetic fluid, dried powders of samples MF1, MF2, MF5 and MF6 were examined using X-ray diffractometer Ultima. Fig. 4 presentes the results for sample MF6.

Based on the experimental data for all of the samples (synthesized both in industrial conditions and in the SWSU) we can state that the magnetic phase is magnetite.

Thus, comparatively analyzing data, which were obtained for MF samples by various ways, we can conclude that all of the studied samples belong to the same class, are stable and satisfy contemporary requirements to magnetic fluid.

Our research showed that in addition to indirect methods (magnetogranulometric, acoustogranulometric [6], and others), which allow to determine the parameters of MF magnetic nanoparticles, it is appropriate to study the MF structure using microscopic methods. We propose to use advanced analytical equipment - scanning probe and electron microscopy, and X-ray diffraction methods to analyze disperse, elemental and phase composition of magnetic fluids.

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