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A scientific session of the Physical Sciences Division of the Russian Academy of Sciences (RAS) was held on May 30, 2007 in the conference hall of the P N Lebedev Physical Institute, RAS. The following reports were presented at the session:

(1) **Aksenov V L** (Russian Research Centre ‘Kurchatov Institute’, Moscow) “Neutron reflectometry of layered magnetic systems”;

(2) **Avdeev M V** (Joint Institute for Nuclear Research, Dubna, Moscow region) “Structural features of magnetic fluids.”

An abridged version of the second report is given below.

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Structural features of magnetic fluids

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1. Introduction

Magnetic fluids, or ferrofluids, constitute liquid dispersions of magnetic nanoparticles with characteristic sizes of about 10 nm, which corresponds to a single-domain state in such particles and determines the superparamagnetic behavior of the given systems [1]. To balance the dipole–dipole attraction between the magnetic particles, they are coated with a stabilizing layer, in particular, consisting of surface-active substances, or surfactants (Fig. 1). If the stability parameter (the ratio of the attraction energy to the repulsion energy) in the magnetic fluid is of order unity or greater, an external magnetic field acting on the fluid may induce specific aggregation of the nanoparticles into linear chains aligned with the field. Such aggregation dramatically changes all the properties of magnetic fluids, making them anisotropic in relation to the direction of the magnetic field. This, in turn, is the cause of many unusual effects in magnetic fluids [1]. Of interest are also certain surface effects related to the reaction of the free surface of a magnetic fluid to an external magnetic field [1]. Magnetic fluids are widely used in technical devices [1, 2]. Their medical and biological applications are also being actively developed [3–5].

The present report gives an idea of the most recent results of research into the structure of various types of magnetic fluids on the nanometer level (1–100 nm) that uses small-

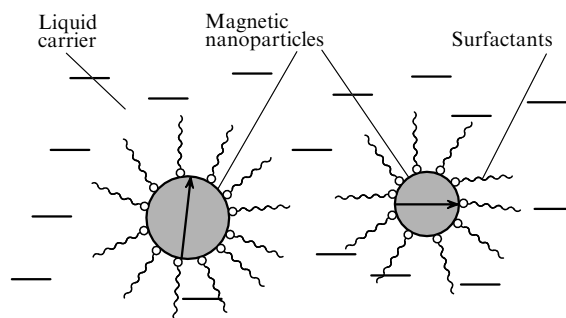


Figure 1. A rough picture depicting the structure of magnetic fluids. The characteristic size of the magnetic nanoparticles coated with a surfactant corresponds to their single-domain state of magnetization denoted by heavy arrows inside the nanoparticles.

angle neutron scattering (SANS). The goal of such structural studies is to determine and control the parameters of the atomic and magnetic structures of nanoparticles in magnetic fluids. In this connection, I will touch on the problem of determining the nonmagnetic layer in magnetic nanoparticles and peculiarities of stabilizing magnetic fluids. Another class of structural problems is related to the description and control of the interaction between the particles.

2. Subject and method of research

Here, we specify the two main types of magnetic fluid, the results of investigation of which are reflected in this report.

Stabilization of magnetic particles in organic nonpolar fluids is done via chemisorption of surfactants on the surface of the particles. A classical surfactant used for this purpose is oleic acid ($C_{18}H_{34}O_2$), unsaturated monocarboxylic acid. The fairly large length (1.8 nm) of the molecule and the fact that it is bent in the middle due to a double bond ensure non-charge (steric) repulsion of the shells belonging to different particles.

In polar fluid bases (including water), the interaction between the lyophilic head of the surfactant and the fluid competes with the adsorption of the surfactant’s head at the surface of the magnetic particles. This interaction is avoided by employing double steric stabilization, where atop the particles with one surfactant layer described above a second surfactant layer is formed due to its physical adsorption on the first layer. This requires an excess of the second surfactant in the solution.

The main method in the studies being discussed is SANS. A SANS experiment involves analyzing the elastic scattering of neutrons by inhomogeneities with sizes varying from 1 to 100 nm. The differential scattering cross section of neutrons is a function of the scattering vector $\mathbf{q} = \mathbf{k} - \mathbf{k}_0$, or the difference between the wave vectors of the incident (\mathbf{k}_0) and

scattered (\mathbf{k}) beams. When the scattering on magnetic nanoparticles takes place, the nuclear component is augmented by magnetic scattering, which is the result of the interaction between the magnetic moments of the neutron and the particles. When the nanoparticle concentration is low (the volume fraction φ_m of the magnetic substances in the magnetic fluid is less than 3%), the scattering on the magnetic fluid is equivalent to scattering by individual particles. When no magnetic field is applied to the sample, the magnetic moments of the nanoparticles in the magnetic fluid are misoriented, with the result that both scattering components are isotropic in relation to the direction of vector \mathbf{q} and, therefore, the cross section depends solely on the length of this vector, $q = (4\pi/\lambda) \sin(\theta/2)$, where λ is the neutron's wavelength, and θ is the scattering angle. For the cross section we have the following relationship:

$$\frac{d\sigma}{d\Omega}(q) \approx F_N^2(q) + \frac{2}{3} F_M^2(q), \quad (1)$$

where F_N and F_M are the respective nuclear and magnetic scattering amplitudes, or the Fourier transforms of the density distributions of the nuclear and magnetic scattering lengths inside a single nanoparticle. The magnetic scattering length depends on the angle between the scattering vector and the direction of the particle magnetic moment. When the moments are distributed at random, the respective averaging produces the factor 2/3 in formula (1). An important feature that makes SANS so useful is the possibility of varying the contrast in the system. The method is based on analyzing the changes in scattering inflicted by variations in the contrast, $\Delta\rho = \langle\rho\rangle - \rho_s$, the difference between the average scattering length densities of the particle and solvent. The most recent possibilities of employing contrast variations in magnetic fluids are demonstrated in Refs [6–11].

The nuclear and magnetic scattering components can be directly separated by using small-angle scattering of polarized neutrons [12, 13]. When the system undergoes magnetization and the incident neutron beam is polarized along (–) or against (+) the direction of the magnetizing field, the scattering cross section becomes anisotropic. For the two polarization states of the neutrons we have

$$I^+(q, \varphi) = F_N^2(q) + (F_M^2(q) - 2PF_N(q)F_M(q)) \sin^2 \varphi, \quad (2a)$$

$$I^-(q, \varphi) = F_N^2(q) + (F_M^2(q) + 2PF_N(q)F_M(q)) \sin^2 \varphi, \quad (2b)$$

where φ is the radial angle in the scattering intensity distribution in the plane (perpendicular to the neutron beam) of the detector behind the sample, and P is the relative degree of polarization of the neutron beam. Using formulas (2) to analyze the scattering anisotropy, we can single out the nuclear and magnetic scattering form factors F_N^2 and F_M^2 and directly extract information about the scattering amplitudes in the form of the product $F_N(q)F_M(q)$. Worth mentioning here are two aspects in applying this method to magnetic fluids. First, the extracted information refers to the structure of a magnetic fluid in an external magnetic field, which can differ from the magnetic-fluid structure in the absence of such a field. Second, the magnetic particles in a magnetic fluid usually exhibit a broad polydispersity, with the result that expressions (1) and (2) must be averaged over the respective size distribution function, which hinders data interpretation [in particular, this is true of the interference term $F_N(q)F_M(q)$].

It is also possible to perform complete polarization analysis which takes into account the variation in polarization under scattering. Aksel'rod et al. [14] and Mériquet et al. [15] did this in their experiments in which the nuclear and magnetic components of scattering in magnetic fluids were separated. On the one hand, this experimental technique is much more complicated than the one described in the present report and requires much more time in the experiments to gather sufficient statistics. On the other hand, it can be used when dealing with unmagnetized samples. The characteristic features of the structure of the magnetic-fluid particles can be determined from what is known as the integral parameters of SANS curves. For instance, the radius R_g of particle gyration furnishes averaged information about the density distribution of the scattering lengths inside the particles. It can be obtained from the Guinier approximation at small q 's ($qR_g < 1$):

$$I(q) \approx I(0) \exp\left(-\frac{1}{3} R_g^2 q^2\right), \quad (2)$$

where $I(0)$ is the forward-scattering intensity. The radius of gyration varies with changing the contrast in the system. When one has to deal with a polydisperse or magnetic system, interpreting the given parameter becomes more complicated, since the parameter carries additional information about the size distribution function and the magnetic component of scattering [10]. As the nanoparticle concentration in the magnetic fluid rises, interparticle interference effects come into play. We will discuss some examples of qualitative and quantitative analysis of such effects in Section 5.

3. Determining the nonmagnetic surface layer in magnetic nanoparticles

Experiments with magnetic nanoparticles show that their specific magnetization is decreased as the nanoparticles get smaller. It is assumed that the reason for this effect is the formation of a nonmagnetic layer on the nanoparticle surface due to the canting of the spin angular momenta of atoms from the direction of domain magnetization at the interface. The hypothesis that such a layer exists explains in a natural way the results of various experiments, including the analysis of magnetization [16–19], the Mössbauer effect [20, 21], the structural diffraction of polarized neutrons [22], etc., which yield estimates of the thickness of the given layer in the range from 0.1 to 0.8 nm. The spin deflections discussed here were corroborated in theoretical calculations [23]. However, no direct experimental observations of a nonmagnetic surface layer in magnetic nanoparticles have been reported. I believe that, in studying powders of magnetic nanoparticles used in most experiments, the effects of magnetic interaction between particles, including quantum effects when the particles come into contact, are very important. In view of this, the purest experiment is apparently the one involving weakly concentrated colloidal solutions (liquid or solid) of magnetic nanoparticles.

In our first attempts to observe such a layer by SANS, we utilized magnetic fluids prepared by industrial techniques [24–26]. Nanomagnetite was fabricated by the reaction of the condensation of trivalent and bivalent iron salts and was dispersed in organic nonpolar fluids (benzene, cyclohexane, and decahydronaphthalene). Oleic acid (OA) or myristic acid (MA) was used for stabilization. The magnetic nanoparticles prepared in this manner in a magnetic fluid exhibit a rather broad size distribution function which has the logarithmically

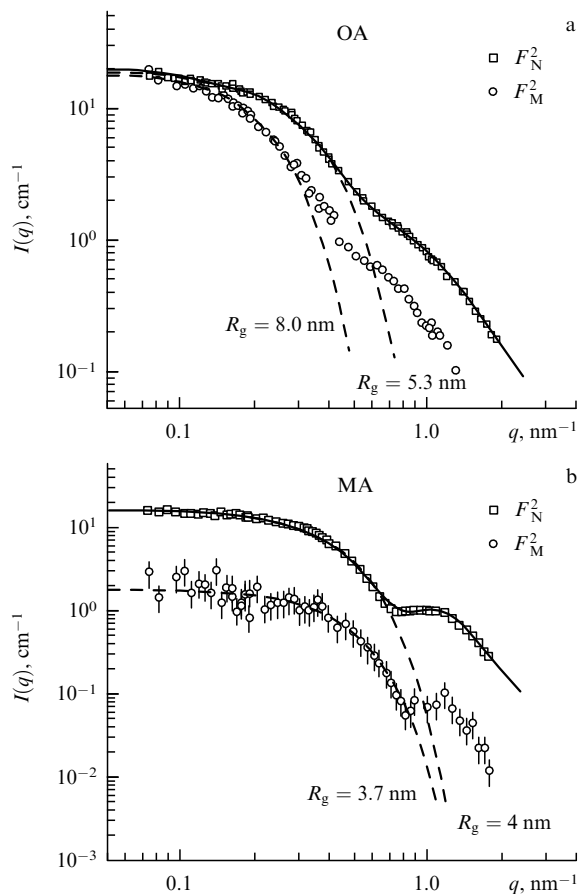


Figure 2. Separated nuclear and magnetic components of neutron scattering according to the data on the polarized neutron scattering for magnetic fluids based on deuterated cyclohexane with magnetite covered with oleic acid (OA) and myristic acid (MA): $\varphi_m = 3\%$, and $H = 2.5$ T [12]. Solid curves conform to fitting by the 'spherical core-shell' model with allowance for polydispersity (3), and dashed curves correspond to the Guinier approximation (2) and are labelled with the observed radius of gyration.

normal form

$$D_N(R) = \frac{1}{\sqrt{2\pi}SR} \exp\left[-\frac{\ln^2(R/R_0)}{2S^2}\right], \quad (3)$$

where the parameters R_0 and S are the distribution's mean radius and variance, respectively. Stabilization via OA and MA yields different functions (3). The effect will be discussed in Section 4.

Figure 2 displays the form factors of nuclear and magnetic scattering obtained from the data on scattering of polarized neutrons [12]. The nuclear-scattering curves are fitted well by the 'spherical core-shell' model which reflects the concept of particle structure. The model gives an estimate for the parameters of the size distribution function of the magnetic particles (see Table 1) and the surfactant-layer thickness $h \approx 0.14$ nm which is the same for the two magnetic fluids. The results are in full agreement with the data on contrast variation in the absence of a magnetic field, which supports the idea that the nuclear structure of a magnetic fluid remains unchanged when a magnetic field is applied to the sample.

The magnetic-scattering component indicates the presence of complex correlations between the magnetic moments of the particles [12, 13]. Figure 2 shows that the

Table 1

Sample	R_0		δ	S	
	Scattering	Magnetization		Scattering	Magnetization
OA	3.4 nm	2.7 nm	0.7 nm	0.38	0.37
MA	2.4 nm	2.3 nm	0.1 nm	0.28	0.28

correlation length exceeds the nuclear size of the magnetic particles. Probably, the reason lies in the effect of the dipole-dipole interaction on the orientation of the magnetic moments of the particles even when their concentration is low ($\varphi_m \sim 1\%$) and in the presence of an external magnetic field. I believe that an important factor is the strong polydispersity of the magnetic particles. At the same time, the positions of the particles proper at the given concentrations may be considered to be weakly correlated. With a further decrease in particle concentration for the purpose of reducing magnetic correlations, special study is required, since the detected scattering dramatically diminishes and, hence, so does the accuracy of the experiment. What is interesting in the data on magnetic scattering is that the result of the addition of the nuclear and magnetic components in the presence of a magnetic field in accordance with Eqn (1) is in good agreement with the scattering curve obtained in the absence of a magnetic field. This means that the quantitative characteristics of the magnetic correlations that occur in the absence of a magnetic field are retained when such a field reaches saturation. At present we are studying the possibility of implementing such correlations in magnetic fluids.

Thus, the dipole-dipole interaction in magnetic fields makes it more difficult to directly determine the magnetic size of the nanoparticles by small-angle scattering of polarized neutrons and to compare the results with the similar nuclear size. Nevertheless, the nuclear size determined with high accuracy in the given experiments can be compared with the results obtained through other methods, say, by analyzing magnetization which yields information about the magnetic size. Such comparisons are presented in Table 1. The magnetization curves of the fluids investigated were processed in the Langevin approximation with allowance for polydispersity of type (3). The difference between the average sizes obtained by two methods gives an estimate for the thickness δ of the nonmagnetic layer. An important conclusion that can be drawn from the data listed in Table 1 is that the thickness of this layer depends on particle size: the larger the particle, the greater the difference between its nuclear and magnetic sizes.

4. Stabilization of magnetic fluids by monocarboxylic acids

As noted in Section 2, the strong stabilizing properties of oleic acid in preparing magnetic fluids are related to the bend in the middle of its molecule. This follows from the fact that the saturated analog of oleic acid, viz. stearic acid ($C_{18}H_{36}O_2$), is a very weak stabilizer. A full explanation of this difference in stabilization efficiency of the two acids has yet to be found [27]. Recently, as a result of structural investigations of changes in the structure of magnetic fluids inflicted by deviations from optimum composition [12], it has been discovered that the short monocarboxylic acids, namely,

myristic acid ($C_{14}H_{28}O_2$) and lauric acid ($C_{12}H_{24}O_2$), manifest a much higher effectiveness of stabilizing nanomagnetite in organic nonpolar bases than stearic acid, but the parameters of the size distribution function of the stabilized magnetite particles (mean radius and variance) are much smaller than those in the case of oleic acid. This has actually been demonstrated by Fig. 2 and Table 1 which give the results of an analysis of the nuclear scattering component in a benzene-based magnetic fluids. Similar results have been obtained for cyclohexane- and decahydronaphthalene-based magnetic fluids. Here, the thickness of the surfactant layer practically coincides for the two types of acids in all bases. Thus, we may conclude that the stabilization efficiency of monocarboxylic acids is to a great extent related to the elastic properties of the adsorption layer, the properties determining (according to the energy condition) the stabilized distribution function over the magnetite sizes in the magnetic fluid. While oleic acid is the most effective stabilizer of nanomagnetite (fabricated as a result of a condensation reaction) within a broad range of particle radii from 1 to 100 nm, the short saturated monocarboxylic acids (the myristic and lauric acids) stabilize this interval only partially, dispersing only a fraction of the small particles into the fluid.

The discovered structural features of the magnetic fluids in question explain the difference in their magnetorheological properties [26]. The jump in viscosity induced by an external magnetic field in a magnetic fluid with oleic acid amounts to about 30%, while in a magnetic fluid with myristic or lauric acid it is much smaller, reaching only 10%. The stronger effect in the first case is due to the larger particles affecting the fluid's flow in the magnetic field. Along with it, the absolute values of the viscosity of the magnetic fluid with myristic or lauric acid, $\eta \sim 0.083$ Pa s, is actually ten times lower than that of the magnetic fluids with oleic acid, $\eta \sim 0.775$ Pa s, which is caused by the larger volume fraction of small particles in the former and a narrower size distribution of particles.

5. The interaction of particles

The upper bound on the volume magnetic fraction in highly stable magnetic fluids is about 25%. This restriction is caused by the existence of a surfactant fraction in the magnetic fluid and by the interaction between the particles. The particle interaction also determines the response of the system to an external magnetic field. Hence, describing the interaction potential in a magnetic fluid is an important goal in fabricating a magnetic fluid with controllable properties. Generally speaking, this problem is a complex one because of the polydispersity of the particles and the dependence of the magnetic interaction on the relative orientation of the particles. A qualitative estimate of the nature of such an interaction is demonstrated in Fig. 3 where the concentration dependences [28, 29] of the SANS curves are compared for two types of magnetic fluids: a nonpolar magnetic fluid with a single stabilizing surfactant layer (benzene), and a polar magnetic fluid with a surfactant bilayer (pentanol). These fluids are highly stable at magnetic material concentrations up to about 25% [25]. The neutron scattering was recorded for samples based on ordinary undeuterated fluids. In this case, the scattering on the surfactant layer is almost fully matched and only the magnetic particles can be seen. In view of the strong contrast between the magnetic material and the solvent, the magnetic scattering can be ignored, i.e., the

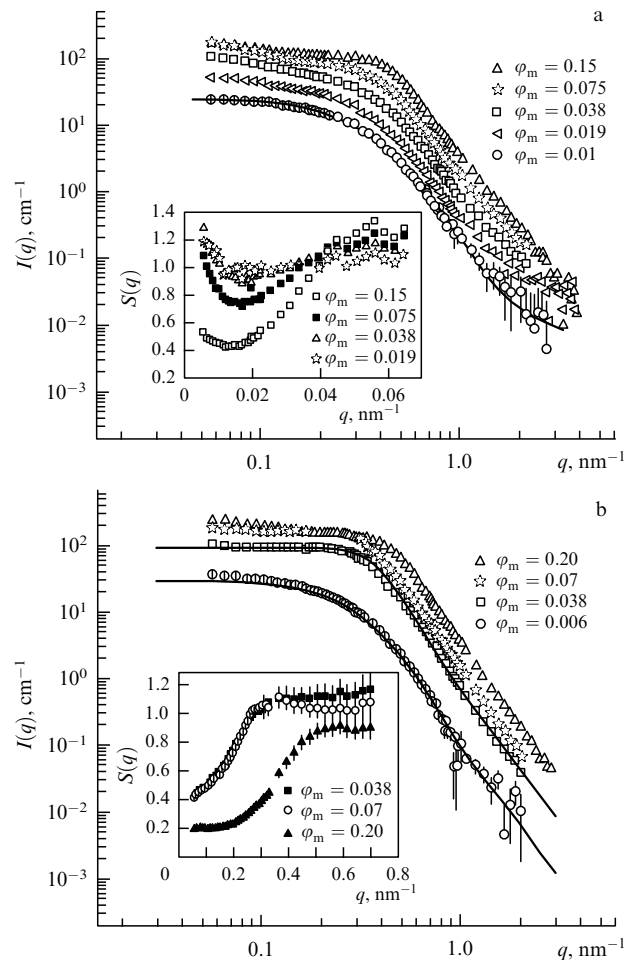


Figure 3. SANS curves representing functions of the magnetite nanoparticle concentration for magnetic fluids based on (a) an organic nonpolar solvent (benzene), and (b) a polar solvent (pentanol) with double stabilization by oleic and dodecylbenzene sulfonic acids. Solid curves depict minimum-concentration curves corresponding to fitting by the ‘noninteracting spheres’ model with allowance for polydispersity (3). The solid curve for a pentanol-based magnetic fluid with $\phi_m = 0.038$ corresponds to fitting by the ‘rigid polydisperse spheres’ model with the use of the Vrij formalism [30, 31]. The insets show the effective structure-factors — the result of dividing the scattering curve by the minimum-concentration curve.

changes in the scattering curves with concentration are related solely to the spatial correlation of particles in the magnetic fluid. The SANS curves obtained are similar to X-ray scattering curves. Neutrons are used because of the possibility of attaining smaller values of q , which is important if we want to analyze the nature of the interaction. For both types of magnetic fluids at the lowest possible concentrations, the curves are described well by the model of polydisperse spheres with a size distribution of type (3), which corresponds to neutron scattering on magnetic particles. Although for polydisperse systems it is impossible to single out the structure-factor, the nature of the interaction potential can still be established from the effective structure-factor $S(q)$ — the ratio of the scattering curves to the form factor determined for the minimum concentration. The effective structure-factors for the magnetic fluids investigated are shown in the insets to Fig. 3. Their comparison demonstrates that there is an important difference between the two

magnetic fluids. In the nonpolar magnetic fluid, $S(q)$ qualitatively reflects the complex interaction potential describing attraction at large separations between particles and repulsion at their contacts. In the polar magnetic fluid, due to the larger surfactant shell, there is complete screening of attraction and $S(q)$ is close to the structure-factor of rigid noninteracting spheres with corresponding smoothing in polydispersity. Indeed, at moderate concentrations ($\varphi_m \sim 4\%$) in such a magnetic fluid, the scattering curves can be described by the Vrij formalism [30, 31] for rigid polydisperse spheres. Appropriate fitting (Fig. 3b) yields the characteristic interaction radius (the radius of the rigid spheres), which coincides with the full particle radius. The latter includes the radius of the magnetic core and the thickness of the surfactant layer. A comparison between the full radius and the magnetic core radius obtained from the analysis of a slightly concentrated sample was used [28] to estimate the thickness of the surfactant bilayer, $h \approx 2.3$ nm. This value is much smaller than the sum of surfactant lengths employed for forming a bilayer (2×1.8 nm = 3.6 nm), which points to a strong overlap of the surfactant sublayers. At high concentrations there is a strong reduction in the effective structure-factor (the inset to Fig. 3b). I believe that this is related to the possibility of overlapping the surfactant shells of different particles, when the particles move closer to each other. In this case, it is difficult to determine the volume fraction of the interacting particles. Thus, the volume fraction of the rigid particles is overvalued, which leads to reduction of the effective structure-factor.

The observed difference in the nature of the interparticle interaction explains the substantial difference in the magnetorheological properties of the two types of magnetic fluids [24, 25]. For instance, the pentanol-based magnetic fluid exhibits almost no magnetic-viscosity effect, i.e., an increase in viscosity when the fluid is placed in an external magnetic field. Such an effect is directly related to the formation of chain aggregates. The data on the interaction of the particles corroborates the fact that the formation of aggregates requires that there be a sufficient attractive component in the interaction potential, which exists in the nonpolar magnetic fluid. An additional surfactant layer screens attraction, and no chain aggregates are formed. Highly concentrated magnetic fluids with weak magnetic viscosity effect are used, for instance, in high-vacuum bearings, where any formation of aggregates is unwelcome.

The contribution of magnetic scattering becomes important when deuterated solvents are used to analyze the dependence of neutron scattering by magnetic fluids on particle concentration. SANS experiments in this case point to a very complicated form of the total structure-factor [29], since one has to deal here with a combination of the effective nuclear and magnetic structure-factors that is extremely difficult to interpret properly. Qualitatively, it can be said that the form of the effective magnetic structure-factor reflecting the presence of correlations in the orientations of the magnetic moments of the particles differs dramatically from the form of the nuclear structure-factor responsible for the correlations in the positions of the particles.

6. Conclusion

The SANS method has been used in structural studies on the nanolevel (1–100 nm) of the main types of highly stable magnetic fluids.

The various features of the nuclear and magnetic structures of the particles in slightly concentrated magnetic fluids based on nonpolar organic solvents have been investigated. It was found that the assumption about the presence of a nonmagnetic layer on the surface of the magnetic nanoparticles requires that the thickness of this layer depend on particle size. Complex and fairly strong correlations between the magnetic moments of the magnetic-fluid particles have been observed, and it was revealed that the correlations remain when an external magnetic field is applied to the sample. At the same time, the positions of the particles may be considered uncorrelated with a high accuracy. Structural studies have been used to demonstrate the possibility of stabilizing magnetic fluids by saturated monocarboxylic acids. Here, the high stabilization efficiency compared to classical stabilization by unsaturated oleic acid can be explained by the energy condition of stability (the elastic properties of the surfactant shell).

An analysis of the interaction between the particles done on the basis of SANS data has shown that magnetic fluids with single and double stabilization are very different in their properties. The model of rigid polydisperse spheres was for the first time successfully used in describing slightly concentrated magnetic fluids with a double surfactant shell, and additional structural information was gathered.

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