

Temperature Dependence of Particle Size Distribution in Transformer Oil-Based Ferrofluid

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Abstract The temperature dependence of the particle size distribution (PSD) of a transformer oil-based ferrofluid was studied using an ultrasound method. The measurements of the ultrasound velocity and attenuation were carried out in the absence of an external magnetic field as a function of the volume concentration of magnetite particles at temperatures ranging from 10 °C to 80 °C. The experimental results of ultrasound measurements were analyzed within the framework of the Vinogradov–Isakovich theory which takes into account contributions to acoustical parameters due to friction and heat exchange between magnetic particles and the surrounding carrier liquid. From the best fit of the experimental results and theoretical predictions, the parameters characterizing the PSD at different temperatures were determined. In order to analyze ultrasonic data, the density and viscosity of ferrofluid samples and the transformer oil were also measured.

Keywords Magnetic fluid · Nanoparticles · Particle size distribution · Velocity and absorption of ultrasound

1 Introduction

Ferrofluids are colloidal suspensions of nano-sized magnetic particles dispersed in a carrier liquid and stabilized by the surfactant layer that produces entropic (steric) repulsion preventing agglomeration of particles. Ferrofluids exhibit special properties that make them suited for many technical applications. Experimental research is now focused on using magnetic particles of nanometer size to improve the thermal and

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dielectric properties of transformer oils. Typically, transformer oils have a low thermal conductivity, and therefore they exhibit rather poor cooling behavior. To improve removal of heat generated by the large power transformers, mineral oils with suspended magnetic nanoparticles can be used [1]. The decrease of the saturation magnetization with temperature and the presence of a non-uniform magnetic field in the transformer cause the ferrofluid close to the winding to be less attracted in comparison to the ferrofluid in the cooler region of the transformer. This additional convective flow of the ferrofluid under the influence of an ac magnetic field enhances the fluid circulation within the transformer windings and thus reduces their temperature. This can increase the transformer capacity to withstand lightning impulses, while minimizing the effect of moisture in typical insulating fluids [2].

In order to optimize the parameters of the transformer-oil-based ferrofluids, it is necessary to study their physical, magnetic, and rheological characteristics. One of the most important properties of magnetic colloidal dispersions is the particle (or aggregate) size distribution (PSD) which may influence their magnetic, rheological, thermal, and elastic properties. Among the methods commonly used to measure the PSD function and study its properties are ultrasonic techniques which are recognized as very promising from an industrial point of view [3]. The usefulness of ultrasonic methods lies in their relative simplicity and non-invasive nature. Basically, the application of these methods to study colloidal suspensions involves measuring the velocity and the attenuation of ultrasound as a function of particles concentration, temperature, and frequency of the wave. In practice, to evaluate ultrasonic data within a framework of the chosen theoretical model, it is necessary to determine a number of thermophysical properties of both the phases.

In this article, the velocity and the attenuation of ultrasound in a transformer oil-based magnetic fluid were measured within a temperature range of 10 °C to 80 °C for eight samples with different concentrations of the dispersed phase. Additionally, the density and viscosity of the studied samples were determined. Assuming a log-normal particle size distribution (PSD), the parameters of the PSD were determined by fitting the expressions for viscous and thermal losses derived by Vinogradov and Isakovich [4] to the ultrasonic attenuation data.

2 Materials and Methods

Ferrofluids composed of transformer oil ITO 100 (inhibited transformer oil) and Fe_3O_4 particles coated with oleic acid were prepared at the Institute of Experimental Physics, Slovak Academy of Sciences in Košice. Magnetic particles were obtained by chemical precipitation of ferrous and ferric salts in an alkali medium. From the magnetization curve obtained using the vibrating sample magnetometer (VSM) method, the volume concentration of magnetite particles, $\phi_{m,1} = 6.6\%$, and their mean diameter, $\langle d \rangle = 10.27$ nm, were determined [5]. Samples with eight different volume concentrations of magnetite particles have been prepared. The initial ferrofluid FF was further diluted with transformer oil in the following proportions: FF:2 ($\phi_{m,2} = 3.3\%$), FF:4 ($\phi_{m,3} = 1.65\%$), FF:8 ($\phi_{m,4} = 0.82\%$), FF:16 ($\phi_{m,5} = 0.41\%$), FF:32 ($\phi_{m,6} = 0.2\%$), FF:64 ($\phi_{m,7} = 0.1\%$), and FF:128 ($\phi_{m,8} = 0.05\%$). This method of

preparation of the samples should not affect the particle size as these kinds of ferrofluids have been shown to be stable for long periods. In other words, the measured PSD should be independent of the volume concentration of the sample.

The ultrasonic measurements were carried out using the ResoScan (Germany) ultrasonic device which measures the ultrasonic velocity, c , and attenuation, α/f^2 , of the sample, where f is the frequency of the ultrasonic wave. The ResoScan system is based on the resonance method [6]. A sample cell with a path length of 7.0 mm constitutes the ultrasonic resonator in which a standing wave is set up. During the initialization, a frequency range of 7 MHz to 9 MHz is scanned. From the resonance peaks, the ultrasonic velocity and attenuation are calculated. The temperature control of the sample is provided by a Peltier thermostat which operates in the 5 °C to 80 °C temperature range with an uncertainty of 10 mK. The resolution of the ultrasonic velocity is $0.001 \text{ m} \cdot \text{s}^{-1}$, and the repeatability of the absolute velocity after automatic reinitialization is $\pm 0.01 \text{ m} \cdot \text{s}^{-1}$. The relative error of the ultrasonic attenuation coefficient is calculated from the series of repeated measurements and is better than 5%.

The shear viscosity coefficient was measured using a cone-and-plate Brookfield DV II+ viscometer within the 15 °C to 50 °C temperature range with a temperature control uncertainty of 0.2 °C. The viscometer was operated at a rotational speed of 100 rpm using cone number CP42, recommended for the viscosity range of $0.3 \text{ mPa} \cdot \text{s}$ to $6000 \text{ mPa} \cdot \text{s}$. The viscosity measurement was repeated ten times, and the result is reported as an average with a standard deviation of $0.02 \text{ mPa} \cdot \text{s}$.

The density was measured using a DMA-38 oscillating U-tube density meter from Anton Paar that measures sample density values accurately to $1 \text{ kg} \cdot \text{m}^{-3}$ in the temperature range of 15 °C to 40 °C.

3 Results and Discussion

The density of the ferrofluid samples and transformer oil were measured within the 15 °C to 40 °C temperature range, and the results of measurements are shown in Fig. 1. The variation of density with temperature for all samples studied obeys a linear relation,

$$\rho = a + bT, \quad (1)$$

where T is expressed in K and ρ in $\text{kg} \cdot \text{m}^{-3}$. The values of coefficients a and b obtained by fitting Eq. 1 to the density data are listed in Table 1.

Figure 2 shows the flow curve, and the temperature dependence of the viscosity is presented in the inset for the transformer oil (carrier liquid). The shear stress versus shear rate dependence is linear presenting a Newtonian-like fluid behavior; thus, within the temperature range of the measurements, the transformer oil viscosity dependence on temperature can be described by the well-known Arrhenius formula,

$$\eta = 0.00012 \exp\left(\frac{3343}{T}\right), \quad (2)$$

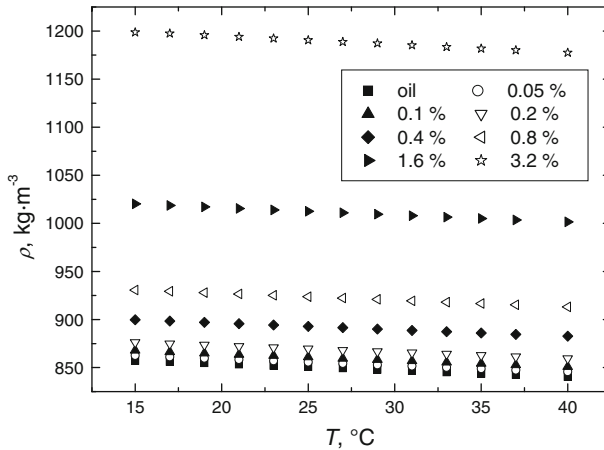


Fig. 1 Temperature dependencies of density for FF samples and mineral oil ITO100

Table 1 Values of parameters a , b obtained from fitting Eq. 1 to the temperature dependence of density for transformer oil-based ferrofluid samples

Sample	a ($\text{kg} \cdot \text{m}^{-3}$)	b ($\text{kg} \cdot \text{m}^{-3} \cdot \text{K}^{-1}$)
Mineral oil	1056	-0.689
$\phi_{m,1} = 6.60\%$	1831	-1.027
$\phi_{m,2} = 3.30\%$	1445	-0.857
$\phi_{m,3} = 1.65\%$	1237	-0.754
$\phi_{m,4} = 0.82\%$	1129	-0.689
$\phi_{m,5} = 0.41\%$	1100	-0.697
$\phi_{m,6} = 0.20\%$	1069	-0.670
$\phi_{m,7} = 0.10\%$	1062	-0.676
$\phi_{m,8} = 0.05\%$	1061	-0.689

where η is the dynamic viscosity in $\text{mPa} \cdot \text{s}$ and T is the temperature in K.

The results of the ultrasonic velocity measurements are shown in Fig. 3. The obtained results are in qualitative agreement with simple macroscopic theories that predict a parabolic relationship between the ultrasonic velocity in a colloidal mixture and the volume concentration of the continuous phase [7].

Figure 4 shows the experimental results of ultrasonic attenuation, αf^{-2} , as a function of magnetite particle concentration, ϕ_m , for temperatures in the range of 10 °C to 80 °C. For all temperatures the ultrasonic attenuation increases with increasing volume concentration of the magnetite particles.

To calculate the PSD function from ultrasonic attenuation data, information about the various physical parameters of the solid particles and liquid dispersion medium is required. The studied ferrofluid was considered as composed of magnetite/oleic acid aggregates of density ρ_a , dispersed in transformer oil of density ρ_f . The aggregate density, ρ_a , and oleic acid volume concentration, ϕ_{sm} , were calculated from the formulas,

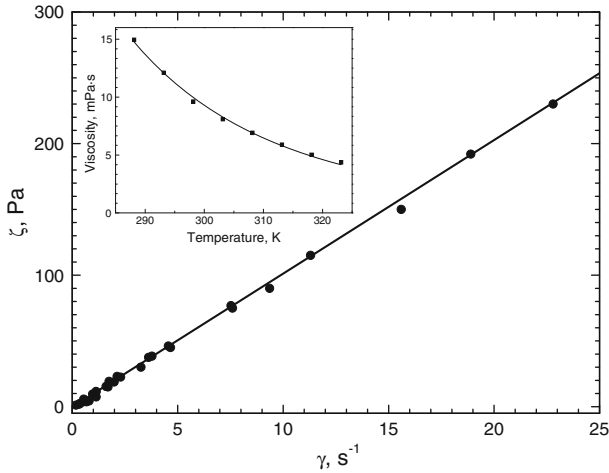


Fig. 2 Shear stress versus shear rate for transformer oil ITO 100. *Inset* shows the dependence of the viscosity of transformer oil on the temperature

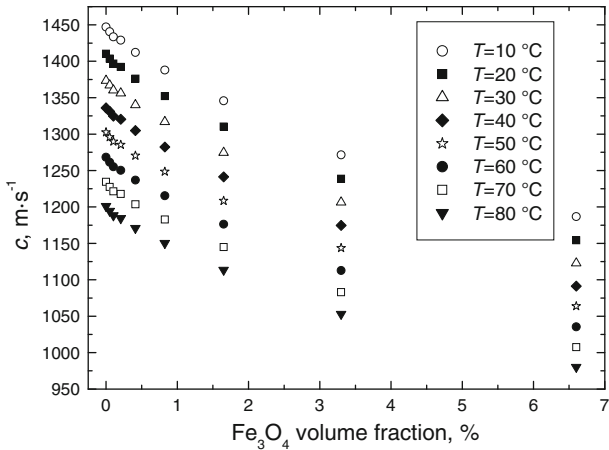


Fig. 3 Ultrasonic wave velocity as a function of magnetite (Fe_3O_4) volume concentration

$$\rho_a = \frac{\rho_m \phi_m + \rho_s \phi_{sm}}{\phi_m + \phi_{sm}}, \tag{3}$$

$$\phi_{sm} = \frac{V_m}{V_s} \phi_m = \left[\frac{3\delta}{\langle R \rangle} + \frac{\delta^2(3\langle R \rangle + \delta)}{\langle R \rangle^3} \right] \phi_m, \tag{4}$$

where $\langle R \rangle = 5 \text{ nm}$ is the average radius of a magnetite particle and $\delta = 2 \text{ nm}$ is the thickness of the surfactant layer. The aggregates volume concentration is then given by

$$\phi_a = \phi_m + \phi_{sm}. \tag{5}$$

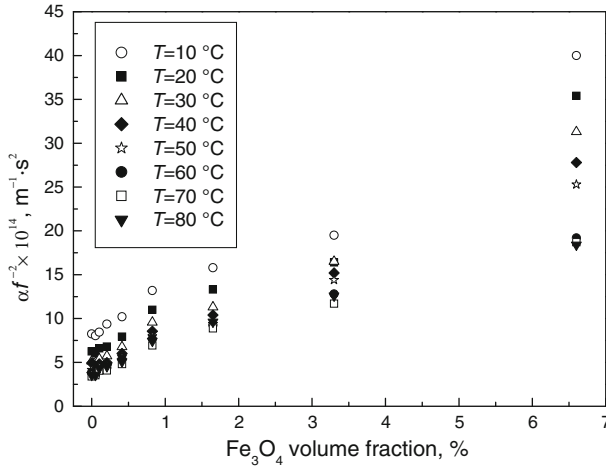


Fig. 4 Ultrasonic wave attenuation as a function of magnetite (Fe₃O₄) volume concentration

The temperature dependencies of the density of oleic acid, ρ_s [8], and of magnetite, ρ_m [9], are given by

$$\rho_s = 1092 - 0.66503T \tag{6}$$

and

$$\rho_m = 5053.3 + 1.5137T - 0.00542T^2 + 5.89226 \times 10^{-6}T^3, \tag{7}$$

where T is expressed in K, while ρ_s and ρ_m are expressed in $\text{kg} \cdot \text{m}^{-3}$.

From the temperature dependencies of the density given in Eqs. 1, 6, and 7, the values of the thermal expansion coefficient for the transformer oil, γ_f , magnetite, γ_m , and oleic acid, γ_s , were determined by calculating the derivative,

$$\gamma = -\frac{1}{\rho} \frac{d\rho}{dT}. \tag{8}$$

The thermal expansion coefficient of aggregates composed of a magnetite particle and oleic acid layer was determined from the formula,

$$\gamma_a = \psi_m \gamma_m + (1 - \psi_m) \gamma_s, \tag{9}$$

where $\psi_m = \phi_m / (\phi_m + \phi_{sm})$ is the volume fraction of magnetite particles in the magnetite–oleic acid binary system.

The coefficient of thermal conductivity, λ_a , and heat capacity, C_a , at constant pressure were determined from the following relations assuming the additivity rule:

$$\lambda_a = x_m \lambda_m + (1 - x_m) \lambda_s, \tag{10}$$

$$C_a = x_m C_m + (1 - x_m) C_s, \tag{11}$$

Table 2 Values of volume concentrations of aggregates composed of magnetite particles and surfactant layers

ϕ_m	ϕ_{sm}	ϕ_a
0.00052	0.00091	0.00143
0.00103	0.00179	0.00282
0.00206	0.00359	0.00565
0.00412	0.00720	0.01132
0.00825	0.01437	0.02262
0.01650	0.02877	0.04527
0.03300	0.05755	0.09055
0.06600	0.11510	0.18110

Table 3 Values of densities ρ_a , coefficients of thermal expansion, γ_a and γ_f , coefficients of thermal conductivity, λ_a and λ_f [10], and heat capacities, C_a and C_f [10], for magnetite/oleic acid aggregates and carrier liquid

T (°C)	ρ_a ($\text{kg} \cdot \text{m}^{-3}$)	$\gamma_a \times 10^6$ (K^{-1})	$\gamma_f \times 10^6$ (K^{-1})	λ_a ($\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$)	λ_f ($\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$)	C_a ($\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$)	C_f ($\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}$)
10	2462	511	801	45.29	0.130	929	1786
20	2458	517	807	45.37	0.129	949	1821
30	2453	523	814	45.44	0.127	967	1856
40	2448	529	820	45.52	0.126	985	1891
50	2443	535	827	45.59	0.124	1001	1926
60	2439	541	834	45.67	0.123	1019	1961
70	2434	547	841	45.74	0.122	1033	1996
80	2429	552	848	45.82	0.120	1047	2031

where $x_m = \rho_m \phi_m / (\rho_m \phi_m + \rho_s \phi_{sm})$ is the mass concentration of magnetite particles in the magnetite–oleic acid binary system. The values of the magnetite thermal conductivity, λ_m , the oleic acid thermal conductivity λ_s , the magnetite thermal capacity at constant pressure C_m , and the oleic acid heat capacity at constant pressure C_s , were taken from [4]. The thermophysical parameters characterizing the magnetite/surfactant aggregates and carrier liquid used in analyzing ultrasonic attenuation data are listed in Tables 2 and 3.

The ultrasonic attenuation results were analyzed by fitting the theoretical model proposed by Vinogradov and Isakovich [4] to the experimental data for the temperature range of 20 °C to 80 °C, with the parameters of the PSD function treated as fitting variables. The interaction between the acoustic wave and the suspended particles of nanometer size leads to additional attenuation of the sound compared to that in the carrier liquid (α_0). Three mechanisms of this interaction associated with differences in density, thermal properties, and compressibility between phases can be distinguished: visco-inertial absorption (α_η), thermal absorption (α_T), and scattering losses (α_S) [3]. For ultrasound frequencies below 100 MHz, the scattering effect becomes negligible

for particles below 1 μm . Also for small kd , i.e., for wavelengths larger than the particle size, viscous and thermal components of ultrasound absorption are additive. This allows us to consider these effects separately and to neglect their possible couplings. Thus, the overall ultrasonic attenuation per frequency squared in the magnetic liquid can be conveniently expressed as the sum of these three contributions,

$$\frac{\alpha}{f^2} = \frac{\alpha_\eta}{f^2} + \frac{\alpha_T}{f^2} + \frac{\alpha_0}{f^2}. \quad (12)$$

The expressions for $\alpha_\eta \cdot f^{-2}$ and $\alpha_T \cdot f^{-2}$ for a polydisperse ferrofluid, with a log-normal distribution function,

$$p(R) = \frac{1}{\sqrt{2\pi}\sigma(R - R_0)} \exp\left(-\frac{[\ln(R - R_0) - m]^2}{2\sigma^2}\right) \quad (13)$$

describing the probability of having a particle of radius R , were given by Vinogradov–Isakovitch [4]. Here, m is the logarithmic mean, σ is the logarithmic standard deviation, and $R_0 = 2$ nm is the minimal radius of the magnetite particle. The mean m and standard deviation σ which give the best agreement between the measured and predicted ultrasonic attenuation can be found by a least-squares analysis.

According to the model proposed by Vinogradov–Isakovitch [4], the contributions to absorption of ultrasound due to visco-inertial and thermal processes are given by

$$\frac{\alpha_\eta}{f^2} = \frac{K\pi\phi_a}{c} \int_{R_{\min}}^{R_{\max}} f(R)R^2 \times \frac{Q(R, f) + S(R, f)q_3\tau_V f}{Q(R, f)^2 + W(R, f)} dR + \frac{2\pi^2\tau_V}{c}, \quad (14)$$

where

$$\begin{aligned} K &= \frac{4\pi(1 - \phi_a)(\rho_a - \rho_f)^2}{9\rho\eta_f}, \quad Q(R, f) = 1 + R\sqrt{q_1 f}, \\ S(R, f) &= R\sqrt{q_1 f} + \frac{2}{9}q_1 R^2 f + q_2 R^2 f, \\ W(R, f) &= q_1 R^2 f + \frac{4}{9}R^3 \sqrt{(q_1 f)^3} + 2q_2 R^3 \sqrt{q_1 f}, \\ q_1 &= \frac{\pi\rho_f}{\eta_f}, \quad q_2 = \frac{4\pi(1 - \phi_a)\rho_a\rho_f}{9\rho\eta_f}, \\ q_3 &= \frac{4\pi\rho_a^2}{(1 - \phi_a)(\rho_a - \rho_f)^2}, \quad \tau_V = \frac{\alpha_f}{f^2} \frac{\rho_f c_f^3}{2\pi^2 \rho c^2}, \end{aligned}$$

and

$$\frac{\alpha_T}{f^2} = -\frac{\phi_a}{f^2} \int_{R_{\min}}^{R_{\max}} f(R) \text{Im} \left[-\frac{j\lambda_a \lambda_f [Z_a - \tanh(Z_a)](Z_f + 1)}{\lambda_a [Z_a - \tanh(Z_a)] + \lambda_f (Z_f + 1) \tanh(Z_a)} \right] dR, \quad (15)$$

where

$$Z = \frac{3\rho Tc}{2R^2} \left(\frac{\gamma_a}{\rho_a C_a} - \frac{\gamma_f}{\rho_f C_f} \right)^2,$$

$$Z_a = (1 + j)R\sqrt{\pi f \rho_a C_a \lambda_a^{-1}}, \quad j = \sqrt{-1},$$

$$Z_f = (1 + j)R\sqrt{\pi f \rho_f C_f \lambda_f^{-1}}.$$

The subscripts a and f refer to the properties of the magnetite/oleic acid aggregates and the transformer oil, respectively.

The results of the ultrasonic attenuation data analysis for all temperatures are shown in Fig. 5. The solid lines represent the total absorption calculated from Eqs. 14 and 15 with the PSD parameter values m and σ obtained from the “best-fit” procedure whereas dotted lines represent contributions to the attenuation attributed to the viscous and heat exchange losses. The error bars represent the deviations of experimental data from the fitted theoretical curves. A systematic decrease in the accuracy of the fits with increasing temperature can be observed. This can be due to worse estimation of parameters, especially heat capacities, for higher temperatures. In some cases their values had to be extrapolated outside the range of available experimental data points. The PSD parameters for all temperatures are listed in Table 4. It is also seen from Fig. 5 that the viscous losses dominate in ultrasonic attenuation for higher values of volume concentrations of magnetite particles ϕ_m . However, the role of thermal losses in ultrasound attenuation substantially increases for higher temperatures. This means that a neglect of heat losses, especially at higher temperatures and in more concentrated samples, can lead to improper values of the PSD parameters.

Figure 6 shows the log-normal PSD obtained by the ultrasonic method for various temperatures in the range of 20 °C to 80 °C. It is seen from the figure that the amount of larger particles decreases with temperature. The effect of temperature on the PSD can be due to: (1) thermal expansion of particles and (2) break down of the aggregates composed of two or more magnetite particles. The first factor leads to an increase of the amount of larger particles and is rather small. This is not the case in our measurements. On the other hand, if the magnetic dipole–dipole attractive interaction exceeds the thermal energy and steric repulsion, an aggregation of magnetic particles will take place. These aggregates will collapse at elevated temperatures leading to a decrease of the amount of larger structures.

The mean value M_ϕ , and root-mean-square deviation D_ϕ , of particle radius R can be determined from the expressions,

$$M_\phi = \exp(m + 0.5\sigma^2) + R_0, \quad (16)$$

$$D_\phi = \exp(2m + \sigma^2)(\exp \sigma - 1). \quad (17)$$

The values of M_ϕ and D_ϕ for all temperatures obtained from our analysis of ultrasonic attenuation data are listed in Table 4. The mean particle radius M_ϕ , evaluated from ultrasonic measurements, is the so-called “hydrodynamic” radius which is greater than the size of the magnetic core by a magnitude, $\delta_s + \delta_m$, where δ_s denotes the thick-

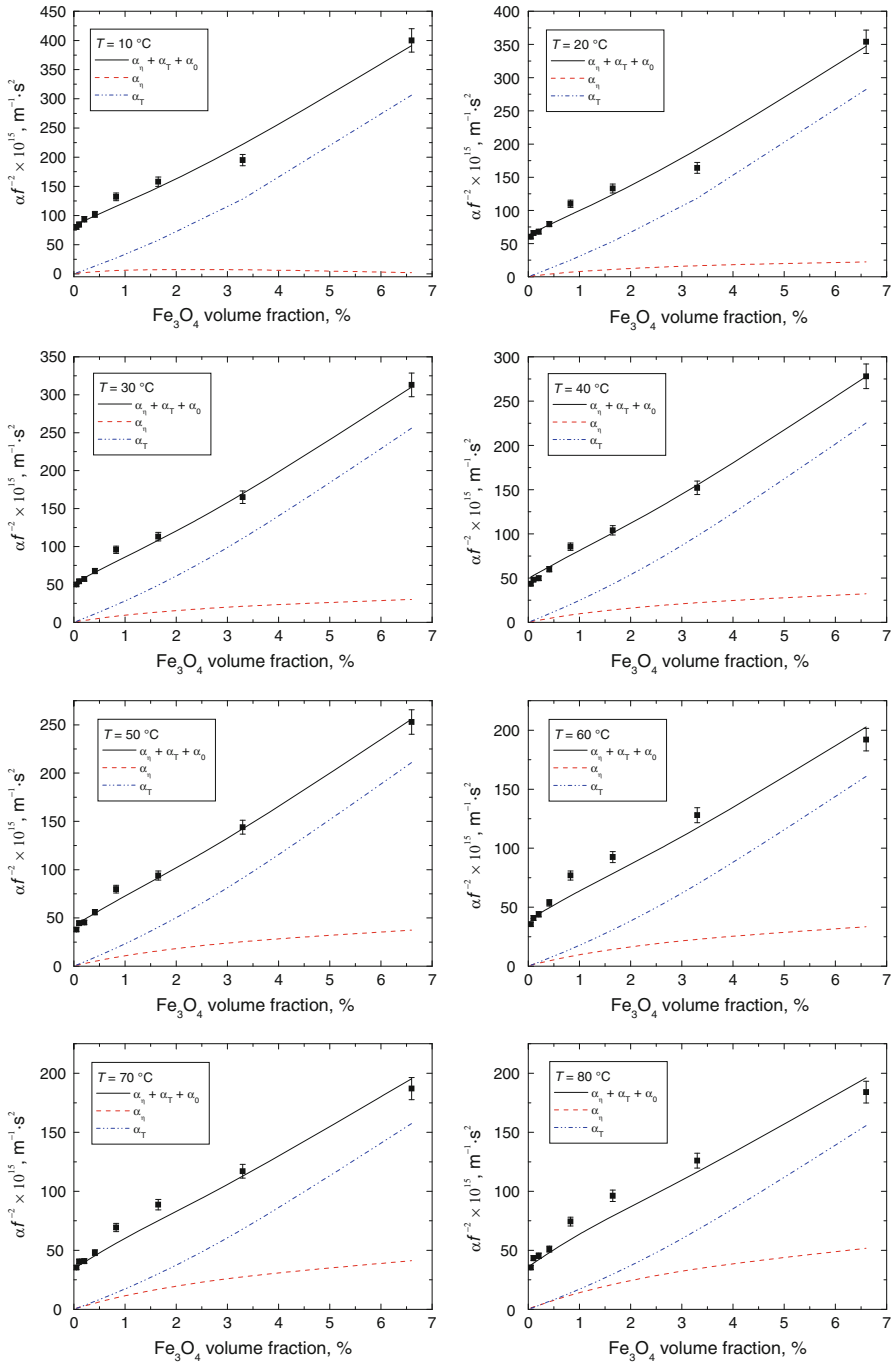


Fig. 5 Experimental and calculated results of the concentration dependence of ultrasonic wave attenuation for temperatures in the range of 20 °C to 80 °C

Table 4 Parameters m and σ of log-normal particle size distribution, mean value M_φ , and root-mean-square deviation D_φ of particle radius R calculated from ultrasonic data for different temperatures

T (°C)	$-m$	σ	$M_\varphi \times 10^9$ (m)	$D_\varphi \times 10^{18}$ (m ²)
10	18.79 ± 0.01	0.90 ± 0.01	12.4 ± 0.2	157 ± 9
20	19.00 ± 0.01	0.99 ± 0.01	11.1 ± 0.2	141 ± 8
30	19.25 ± 0.01	1.10 ± 0.01	10.0 ± 0.2	128 ± 8
40	19.41 ± 0.01	1.09 ± 0.01	8.7 ± 0.2	89 ± 7
50	19.56 ± 0.01	1.11 ± 0.01	7.9 ± 0.2	71 ± 4
60	19.89 ± 0.01	1.14 ± 0.01	6.4 ± 0.1	41 ± 3
70	20.03 ± 0.01	1.18 ± 0.01	6.0 ± 0.1	36 ± 2
80	20.27 ± 0.01	1.28 ± 0.01	5.6 ± 0.1	33 ± 2

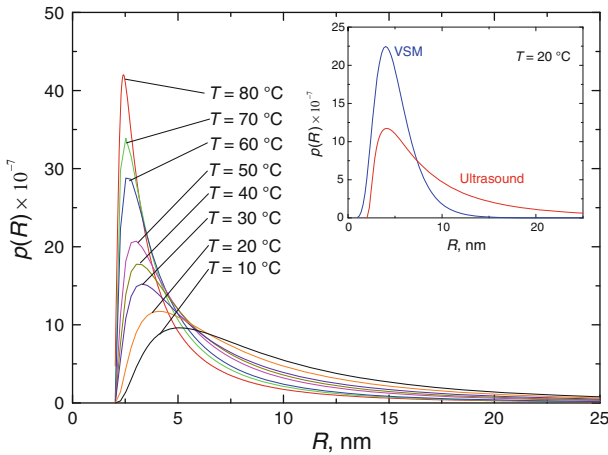


Fig. 6 Lognormal PSD functions obtained from ultrasonic measurements for different temperatures in the range of 20 °C to 80 °C. *Inset* shows comparisons of the PSDs obtained from VSM [5] and the ultrasound absorption data

ness of a protective surfactant layer and δ_m is the thickness of the magnetically inactive layer on the surface of the particles [11]. That is why magneto-granulometric analysis based on VSM measurements, which permits us to find only the size of the particle magnetic core, usually leads to lower values of the mean particle radius R and to the PSD with a smaller amount of larger particles. This can be seen in Fig. 6 where the PSDs obtained from VSM and ultrasound experiments are compared. Some differences in PSDs obtained by different techniques are the result of the chosen method. It means that the instruments based on different physical principles can yield slightly different PSDs [3, 12].

4 Conclusions

This article presents the results of the temperature dependence of the PSD in a transformer oil-based ferrofluid. To determine the PSD, the ultrasonic velocity and

attenuation were measured as a function of the volume fraction of the dispersed phase in the temperature range of 20 °C to 80 °C. The samples of different volume concentrations of magnetite were obtained from the original ferrofluid through subsequent dilution with the preparation solvent. The PSD obtained from ultrasound attenuation data exhibits a dependence on temperature. With an increase of temperature, the amount of larger structures decreases due to breaking down of aggregates consisting of two or more magnetite particles. The mean particle radius obtained from the ultrasonic measurements is larger than that obtained from the magnetization curve. This is because the ultrasonic method leads to a hydrodynamic radius which is greater than the size of the magnetic core by the surfactant and magnetically inactive layers.

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