



Thermal diffusion of magnetic nanoparticles in ferrocolloids: Experiments on particle separation in vertical columns

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Abstract

Experiments on nonstationary separation of nanometer-sized Fe_3O_4 particles of hydrocarbon-based ferrocolloids in a flat vertical thermal diffusion column are performed. By using a modified separation theory which accounts for an one-dimensional mixed (thermal and concentration) convection in the column, the Soret coefficient of magnetic nanoparticles are calculated. It is shown that particles are transferred in the direction of decreasing temperature. The thermal diffusion ratio α_T for magnetite particles suspended in tetradecane reaches the value $\alpha_T \approx -20$. Neither a dependence of α_T on temperature nor any changes in particle-size distribution curves during the separation are observed.

Keywords: Thermal diffusion; Magnetic fluids; Colloids; Natural convection; Separation

1. Introduction

Transport properties of magnetic particles play a significant role in the problem of long-term stability of magnetic fluids. Usually, in the research of a colloidal stability attention is paid only to the gravitational sedimentation and to the magnetophoretic separation of ferroparticles in the colloid. Nevertheless, it may not be excluded that in many magnetofluid devices, for example, in high-speed rotary seals or in loudspeaker cooling systems in which high-temperature gradients are present, the long-term stability of the magnetic

fluid may be effected also by a thermophoretic nanoparticle transfer. Some experiments, e.g. those of magnetic nanoparticle grating in optical interference bands (the forced Rayleigh scattering experiment [1]), indirectly indicate a high thermophoretic mobility of particles in colloidal dispersions [2]. In the present paper, original results of macroscopic experiments on particle separation in a thermal diffusion column and results of evaluation of a thermal diffusion coefficient of nanoparticles in magnetic colloids are presented.

2. The column theory

An attempt to measure the particle thermomagneto-phoretic mobility in ferrocolloids was made

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already in 1983 [3]. The investigations were based on non-stationary particle separation measurements in a vertical thermal diffusion column. The obtained results were interpreted by using a simple theory [4] according to which the convection in vertical column is caused by a thermogravitational buoyancy force only. When the mass transfer in magnetic colloids is considered, it is necessary to take into account in the column theory both the thermal as well as the concentration buoyancy forces because even a small variation of particle concentration during their thermophoretic transfer causes an additional density change comparable with the effect of fluid thermal expansion [5].

A sketch of a thermal diffusion column is shown in Fig. 1. According to Ref. [5], the distribution of vertical flow velocity $v = v_z(x)$ across the non-isothermic fluid layer between two vertical infinite plates of different temperatures T_δ and T_0 for the complex natural convection caused by both the thermal and the concentration buoyancy forces may be written in the following nondimensional form:

$$v = \frac{v_z \delta}{v} = x(1-x) \times \left[\frac{Gr_T}{12}(2x-1) + \frac{Gr_\phi}{k} \left(\frac{6}{\exp k - 1} + 3 - \frac{6}{k} \right) \right] + \frac{Gr_\phi}{k} \left(\frac{\exp kx - 1}{\exp k - 1} - x \right). \quad (1)$$

Here δ is the width of channel (δ is used also as a scale for non-dimensional coordinate x) and v is the kinematic viscosity of fluid, the vertical coordinate z is oriented vertically in direction opposite to the vector of acceleration constant of gravity g . The non-dimensional separation parameter $k = \alpha_T \Delta T / T$ contains a thermal diffusion ratio $\alpha_T = TD_T / D$ (D_T and D are coefficients of thermal diffusion and of Brownian diffusion of colloidal particles) and a temperature difference $\Delta T = T_\delta - T_0 > 0$. The thermal and the concentration Grashoff numbers $Gr_T = g\beta_T \Delta T \delta^3 / v^2$ and $Gr_\phi = g\beta_\phi \phi_0 d^3 / v^2$ include coefficients $\beta_T = -1/\rho(\partial\rho/\partial T)$ and $\beta_\phi = 1/\rho(\partial\rho/\partial\phi)$ which characterize a dependence of fluid density ρ on temperature T and on particle volume con-

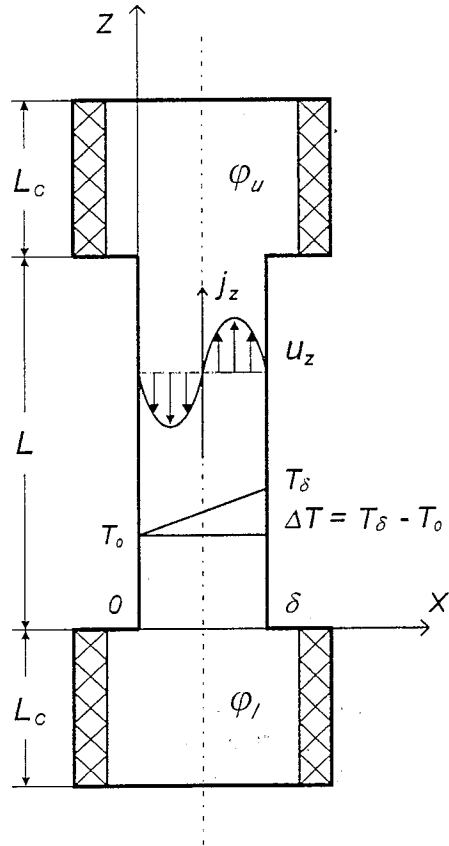


Fig. 1. Principal sketch of the thermal diffusion column.

centration ϕ , ϕ_0 is the mean volume concentration of particles. The expression (1) corresponds to a linear distribution of temperature across the channel ($T = T_0 + \Delta T x$) and to a steady particle concentration profile

$$\frac{\phi}{\phi_0} = \frac{k \exp kx}{\exp k - 1} \quad (2)$$

caused by thermal diffusive particle transfer along the temperature gradient $dT/dx = \Delta T/\delta$ in diluted colloids for which the one-dimensional particle flux, according to Ref. [4], is written in the form

$$j_x = -\rho D \frac{d\phi}{dx} + \rho D_T \phi \frac{dT}{dx} = -\rho D \left(\frac{d\phi}{dx} - \frac{\alpha_T}{T} \phi \frac{dT}{dx} \right). \quad (3)$$

The velocity of nanoparticles in gravitational sedimentation in colloids is considerably less than the velocity of convective transport of particles. Therefore, the mean vertical particle flux in channel may be written in the form of

$$j_z = \rho \int_0^1 v_z(x) \varphi(x) dx. \quad (4)$$

Taking into account both the velocity profile (1) and the concentration distribution (2) from (4) it follows:

$$\begin{aligned} \frac{j_z \delta}{\rho v \varphi_0} = J_z = \frac{Gr_T}{k} \left[\frac{1}{12} + \frac{1}{k^2} - \frac{\exp k + 1}{2k(\exp k - 1)} \right] \\ + \frac{Gr_\phi}{k} \left[\left(\frac{6}{\exp k - 1} + 3 - \frac{6}{k} \right) \right. \\ \left. \times \left(\frac{\exp k + 1}{\exp k - 1} - \frac{2}{k} \right) \frac{1}{k} + \frac{1}{k} - \frac{\exp k + 1}{2(\exp k - 1)} \right]. \end{aligned} \quad (5)$$

Fig. 2 represents the function $J_z/Gr_T = f(k)$ graphically. For small values of parameter k ($k < \pm 1$) instead expression (5), a more simple relation may be used:

$$J_z = \frac{k}{6!} (Gr_T - kGr_\phi). \quad (6)$$

When molecular liquids are considered, the effect of concentration buoyancy force on the convection in a channel usually may be neglected, $kGr_\phi \ll Gr_T$. In magnetic colloids there are transferred particles of density ρ_p considerably higher than that of carrier liquid ρ_0 , therefore both terms in Eq. (6) have to be taken into account. If the convection in channel is caused mostly by a concentration buoyancy force, $Gr_\phi \gg Gr_T$ (it may happen when colloids of high particle concentration are measured), the particle flux is directed toward the vector \mathbf{g} independently of the sign of parameter k . The direction of particle thermophoresis may be detected from separation measurements in diluted colloids provided the thermogravitation mechanism of convection prevails, $Gr \gg Gr_\phi$.

According to Ref. [4], for $k \ll 1$ the mass transfer equation of thermal diffusion column may be writ-

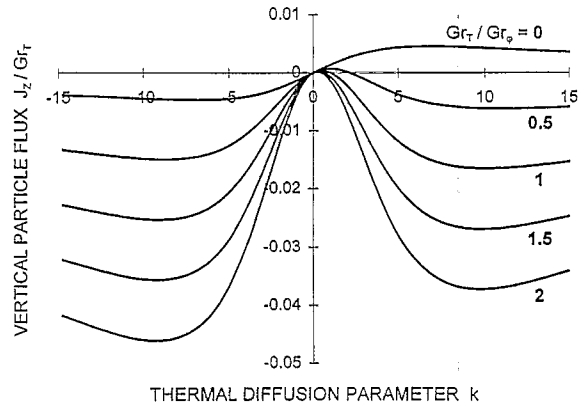


Fig. 2. The vertical stationary convective flux of particles caused by their thermal diffusion across the channel (5).

ten in the following dimensionless form:

$$\frac{\partial \varphi}{\partial \tau} = \frac{D_c}{D} \frac{\partial^2 \varphi}{\partial z^2} - \sigma \frac{\partial \varphi}{\partial z}. \quad (7)$$

Here $\varphi = \varphi(z)$ is the particle mean volume concentration across the channel, $\tau = Dt/\delta^2$ is the non-dimensional time, D_c is the 'convective' diffusion coefficient which for small k is equal to

$$D_c = D \left[1 + \frac{Sc^2}{9!} (Gr_T - kGr_\phi)^2 \right], \quad (8)$$

and σ is the nondimensional particle separation parameter which with respect to relation (6) may be written in the form of

$$\sigma = \frac{kSc}{6!} (Gr_T - kGr_\phi) \quad (9)$$

($Sc = v/D$ is the Schmidt number of colloidal particles).

The analytical solution of Eq. (7) gives the following asymptotic dependencies which may be used to calculate a thermal diffusion coefficient from the measurements of particle separation in a column [3]:

(1) In the initial stage ($\tau < 1$) of separation the concentration difference in upper (φ_u) and lower (φ_l) containers (see Fig. 1) follow a linear dependence

$$N = \frac{\Delta \varphi}{\varphi_0} = \frac{\varphi_u - \varphi_l}{\varphi_0} = 2 \frac{\delta}{L_c} \sigma, \quad \tau = 2 \frac{L}{L_c} \alpha Fo, \quad (10)$$

$$\alpha = \frac{\sigma LD}{\delta D_c}, \quad \text{Fo} = \frac{D_c t}{L^2} = \frac{D_c \delta^2}{DL^2} \tau.$$

(2) In the steady regime of separation ($\tau \gg 1$)

$$N = \frac{3\alpha(1 - \exp(-\alpha))}{2\alpha + (1 - \alpha)(1 - \exp(-\alpha))} \approx \alpha. \quad (11)$$

Here $L = V/S$ is the height of the separation channel (V and S are its volume and cross-sectional area), $L_c = V_c/S$ is the effective length of the upper and lower separation chambers (it is assumed that both chambers are of equal volume $V_u = V_l = V_c$), Fo and α are the 'convective' diffusion Fourier number and the generalized separation parameter of the column.

In the intermediate interval of times ($\text{Fo} \approx 1$) the concentration difference N develops according to a square-root dependence [3]

$$\frac{\Delta\varphi}{\varphi_0} = C \frac{L}{L_c} \alpha \sqrt{\text{Fo}}. \quad (12)$$

The coefficient C slightly depends on the ratio L/L_c . For devices having approximately equal volumes of the active column V and of the upper and lower containers V_c , from calculation results reported in Ref. [3] it is found that the value of $C \approx 0.37$.

The linear dependence (10) corresponds to the steady concentration profile (2) which is valid only if $\tau > 0.4$ [6]. In the initial stage of separation, $\tau < 0.1$, instead of stationary law (6), an approximately linear dependence of vertical mass flux on time is valid [7]. Besides, because in this region the concentration nonhomogeneities across the channel are not developed yet, we may assume the influence of a concentration buoyancy force on convection velocity being neglected. It may be shown that, if also $\text{Fo} < 0.1$, instead of formula (10), a square dependence of particle concentration difference on time is awaited:

$$\frac{\Delta\varphi}{\varphi_0} \approx 0.0063 kSc \text{Gr}_T \frac{\delta}{L_c} \tau^2. \quad (13)$$

Usually, in real separation devices, the column width is varied in the interval approximately from

$\delta = 0.5 \text{ mm}$ to $\delta = 1 \text{ mm}$ and the thermal as well as the concentration Grashoff numbers may reach values up to 10^0 – 10^2 . Besides, for colloidal particles the Schmidt number Sc values are very high ($Sc > 10^4$). Therefore, a significant multiplication effect of particle separation in thermal diffusion columns takes place. The relaxation time of particle separation in column depends on the ratio L/L_c . If $L \approx L_c$, the steady regime is awaited to be reached when $\text{Fo} \approx 1$. This value corresponds to a real time interval of one to several hours.

3. Ferrofluid samples and measurement technique

The separation experiments are performed by using a vertical flat column of width $\delta = 0.5 \text{ mm}$ and of height $L = 85 \text{ mm}$. The heated and cooled walls (copper plates of polished surfaces) are connected with two precise thermostats, the temperature difference $T_\delta - T_0$ is varied in the interval of 2–20°C. In order to lower the temperature nonhomogeneities caused by an external heat exchange, the upper and lower containers of equal volume ($V_c \approx 1 \text{ cm}^3$, $L/L_c \approx 1.2$) as well as the outside walls of channel are made from a plastics of low heat conductivity and of low thermal expansion.

The particle concentration in upper and in lower chambers during the separation process are detected from measurements of resonance frequency of an LC oscillator, the inductance coils of which are mounted inside the containers. The optimum oscillation frequency has to be chosen taking into account a compromise between the sensitivity of the particle concentration measurements and the errors caused by a relaxation of colloid magnetization. This frequency f was verified experimentally by measuring the Q -factor of the coil. For given ferrofluid samples the oscillation frequency should be chosen between 70 and 85 kHz approximately. A special LC-oscillator of high stability was designed [8]. The calibration curve for each magnetic fluid is measured separately. For colloids of moderate particle concentration used in thermal diffusion experiments the function $f = f(\varphi)$ may be regarded linear (see Fig. 3). Such a linearity reported also in Ref. [9], obviously, is expected only for colloids of

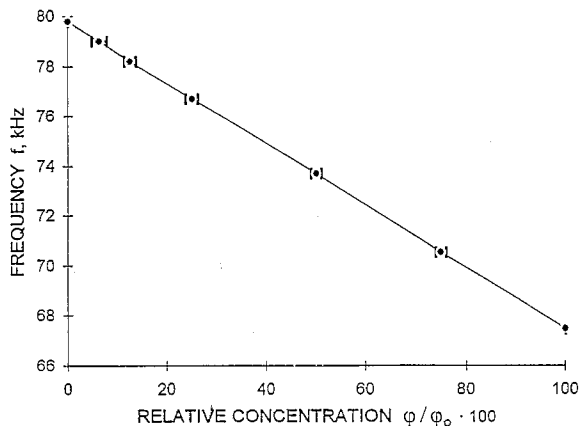


Fig. 3. The resonance frequency f of LC-oscillator versus particle relative concentration (here ϕ_0 is the initial volume concentration of ferrofluid sample TD-5).

low initial magnetic susceptibility $\chi \ll 1$. For Langevin-type monodisperse colloids containing non-interacting subdomain particles of magnetic moments m , this susceptibility is proportional to the particle concentration $n = \phi/V_p$ (V_p is the particle volume):

$$\chi = \frac{\mu_0 m^2}{3k_B T} n. \quad (14)$$

Here μ_0 is the magnetic constant and k_B is the Boltzmann constant; the magnetic moment m is proportional to the particle volume and to the saturation magnetization of particle material M_s , $m = M_s V_p$.

Additional measurements of an ohmic resistance of both induction coils are performed to control the temperature in separation chambers. By using precise cooling and heating thermostats, a high stability of average temperature (approximately $\pm 0.5^\circ\text{C}$) during the separation experiment is achieved.

Experiments are performed employing a tetradecane-based ferrofluid sample (TD-5) containing chemically coprecipitated magnetite nanoparticles. The colloid is stabilized by using oleic acid as a surfactant. The volume fraction of magnetic phase $\phi_0 = nV_p$ is calculated from the saturation magnetization of the sample M_s (ϕ_m) and from the fluid density (ϕ_ρ). Assuming that the M_s of particle

material is equal to that of bulk magnetite ($M_s = 480 \text{ kA/m}$) the value $\phi_m = 2.2 \times 10^{-2}$ is found, whereas from density measurements it follows a higher value $\phi_\rho = 3.7 \times 10^{-2}$. Such difference between 'magnetic' and 'physical' sizes of ferroparticles is typical for magnetic fluids and usually it is interpreted to be caused by a nonmagnetic surface layer of ultra-small particles. The mean particle magnetic moment measured by a magnetogrulometry technique [10] is: $m = 1.4 \times 10^{-19} \text{ A m}^2$. Such value of m with respect to saturation magnetization of magnetite corresponds to the mean 'magnetic' diameter of particles $d = 7.1 \times 10^{-9} \text{ m}$. This value is used to calculate the particle Brownian diffusion coefficient by using a formula $D = k_B T / (3\pi\eta d)$. The dynamic viscosity of colloid $\eta = 4.6 \times 10^{-3} \text{ Ns/m}^2$ as well as its thermal expansion coefficient $\beta_T = 8.3 \times 10^{-4} \text{ 1/K}$ are detected experimentally, whereas the coefficient $\beta_\phi = 5.9$ is calculated by using the known values of the density of magnetite (ρ_p) and of the carrier liquid (ρ_0): $\beta_\phi \approx \rho_p/\rho_0 - 1$.

4. Results and discussions

From results presented in Fig. 2 and from expressions (9)–(12), it follows that in the situation when convection in the column is effected by a concentration buoyancy force, two values of the thermal diffusion parameter k of different sign from nonstationary particle separation curves may be calculated. Moreover, if the concentration buoyancy force in a convection channel prevails, particles are transferred from the upper chamber to the lower one independent of the direction of a particle thermal diffusion transfer across the channel. In order to clarify this uncertainty, a special series of experiments by using ferrocolloids of different particle concentration are performed. Fig. 4 represents the curves of nonstationary concentration changes in both separation chambers obtained by using a ferrofluid of low magnetization (sample TD-5 diluted to 1:20). Since the influence of particle concentration changes on convection in this case is relatively small, we may conclude that the particle accumulation in lower chamber means that the value of k is negative, particles are transferred

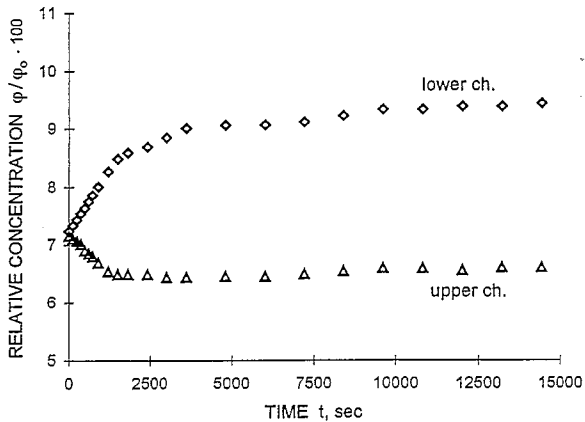


Fig. 4. The particle concentration dependence on time in both chambers, sample TD-5, dilution 1 : 14.

across the channel toward decreasing temperatures. As is seen from the results presented in Fig. 4, in the first 1000 s of separation the concentration difference develops nearly linearly. Using Eq. (10) and neglecting the effect of concentration changes on the convection, a value $k = -0.23$ is obtained. The account for a real situation ($Gr_\phi/Gr_T = 0.3$) leads to a practically equivalent result $k = -0.22$. In region $t > 12000$ s the separation curves reach a saturation regime. From Eq. (11) an approximately four times higher value of the parameter k is calculated. Usually, such difference in calculation results is considered to be due to some disturbances of one-dimensional convection in the channel [4]. We suspect that in our case a more important reason for divergence of results is the uncertainty of some physical properties and the polydispersity of colloid, (for example, the diffusion coefficient of nanoparticles D is calculated by using a mean 'mag-

netic' diameter detected from the magnetization measurements).

Two series of separation experiments are performed by using the liquid TD-5. In the first series, A, particle concentration is varied by dilution of the initial sample, whereas in the second one, B, the temperature difference ΔT is changed. In all experiments the average temperature is kept constant ($(T_0 + T_\delta)/2 = 27^\circ\text{C}$). The level of dilution and the ΔT for both series are shown in Table 1.

In some cases the particle concentration during the experiment is also controlled by magnetization measurements of a ferrofluid sample before and after the separation experiment. As an example, in Fig. 5 are presented magnetization curves for one of the experiments. The particle concentration calculated from saturation magnetization usually is in a good agreement with the results obtained from resonance frequency measurements. Fig. 6 represents the distribution curves of particle magnetic moments which are calculated from the magnetization curves shown in Fig. 5 by using a regularization technique [10]. It is seen that there are no noticeable changes in distribution curves during the particle separation. This allows us to conclude that the thermal diffusion mobility of magnetic nanoparticles in chemically stabilized colloid does not depend on particle size.

The results of particle separation measurements are presented in Figs. 7 and 8. Dots are plotted in dimensionless coordinates defined above. The value of parameter k of these coordinates is calculated by using the square-root law (12) for each experiment separately. As is seen, the dots in both figures do not fit to an universal dependence predicted by theory [4]. Only those experiments which correspond to a low particle concentration or to

Table 1
The conditions of experiments and the calculated values α_T

Series	A				B				
	1	2	3	4	6	7	8	9	10
Number	1	2	3	4	6	7	8	9	10
Dilution	1 : 10	1 : 5	1 : 3.3	1 : 2.5	1 : 4	1 : 4	1 : 4	1 : 4	1 : 4
ΔT ($^\circ\text{C}$)	10	10	10	10	2	6	10	14	18
α_T , calculated from (12)	-19	-16.5	-19.5	-19	-19	-20.5	-28	-27.5	-24.5
α_T , calculated from (13)	—	-20	-22	—	—	-22	-31	-27	-21

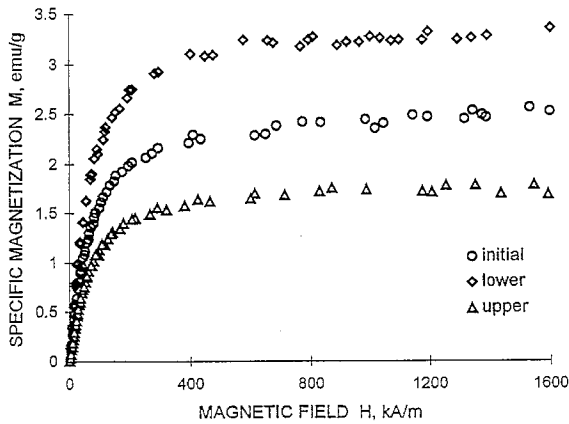


Fig. 5. The magnetization curves of ferrofluid sample TD-5 (experiment 8, dilution 1 : 4, $\Delta T = 10^\circ\text{C}$) before (\circ) and after the separation in the column for 10 000 s (\triangle in upper chamber, \diamond in lower one).

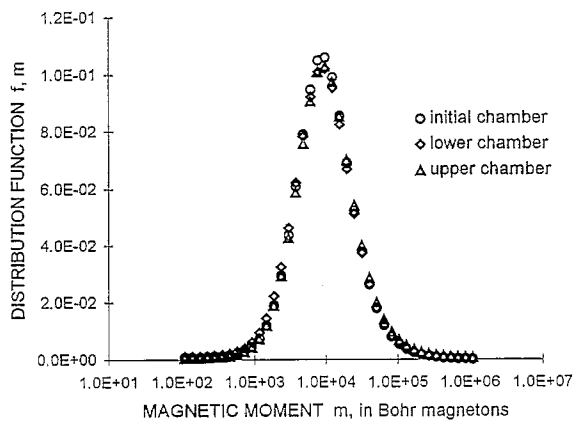


Fig. 6. The distribution curves of particle magnetic moments calculated from the magnetization curves presented in Fig. 5.

a small temperature gradient, indicate a linear development of the concentration difference on time. Increase of both, the ΔT or φ_0 , causes a considerable growth of the dispersion of dots in both figures and a broadening of time interval in which the concentration difference approaches a square dependence on time. We suspect that such divergence in results is caused by a nonstationarity of particle transfer across the channel. Therefore, we analyse the initial part of experimental curves by using the

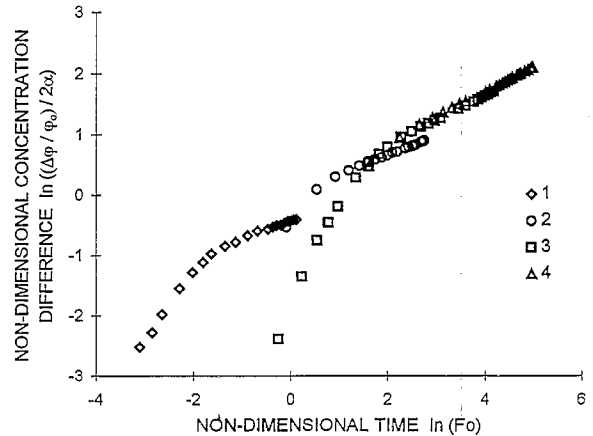


Fig. 7. The results of nonstationary particle separation measurements (Series A).

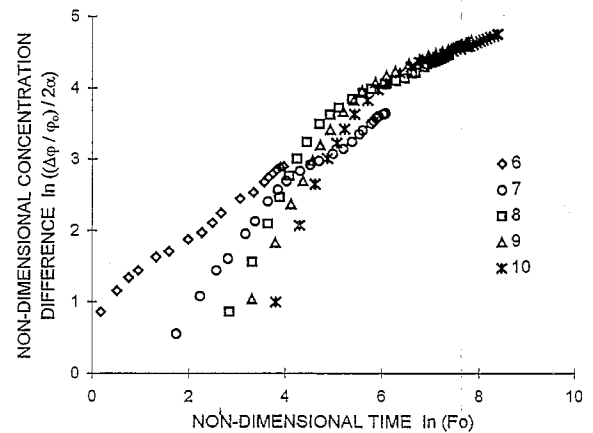


Fig. 8. The results of nonstationary particle separation measurements (Series B).

approximate expression (13). The calculated α_T values are given in Table 1. As is seen, there is a good agreement between these results and the results obtained from separation measurements in an intermediate interval of times by using expression (12). From calculation results it may be concluded that the thermal diffusion ratio α_T neither depends on particle concentration nor on temperature difference. The average value calculated from the initial part of separation curves is

$\alpha_T = -24 \pm 5$ whereas the analysis of measurements in the intermediate interval of times by using formula (12) gives a practically identical result, $\alpha_T = -21 \pm 4$.

The second peculiarity of the experimental separation curves presented in Figs. 7 and 8 is the impossibility to reach a stationary regime even at $Fo > 10$ whereas the approximate theory [4] which is developed for fully developed convective flow in channel of infinite length, predicts a considerably shorter relaxation time of particle separation. Obviously, the theory should be corrected taking into account not only for a nonstationarity of concentration distribution across the channel but also for the two-dimensional character of free convection caused by the particle thermophoresis in channels of a finite length.

The phoretic transport of particles in dispersions is caused mostly by a slip velocity. Exact theories of small particle thermophoresis are developed only for gaseous suspensions of hard particles or droplets when the slip characteristics may be calculated by using existing gas theories. Some general ideas of theory of the slip velocity at a solid–liquid interface resulting from a tangential temperature gradient is given in Ref. [11]. Analyzing the enthalpy flux carried by forced convection of fluid across a porous barrier and applying Onsager's reciprocal theory for the slip velocity, the following expression for the slip velocity was obtained [12]:

$$v^s = - \left[\frac{2}{\eta T} \int_0^\infty y h(y) dy \right] \nabla T, \quad (15)$$

where y is the distance from the wall and $h(y)$ is the local excess specific enthalpy in the interfacial layer. If the solid surface is lyophilic, (the liquid phase is attracted at the molecular level) then $h(y) < 0$ and the slip velocity is directed toward higher temperatures. As a result, this theory predicts that free particles in colloids stabilized by surfactants would move in the opposite direction of decreasing temperatures. Unfortunately, there is currently no model from which to calculate $h(y)$.

In Ref. [13] some results of experiments on thermophoresis in liquid suspensions are reported. It is detected that micron-sized polystyrene-latex spheres suspended in water and hexane are trans-

ferred towards colder regions and the velocity of particle thermophoresis does not depend on particle size. Both these conclusions agree with the theory based on the slip-velocity phenomenon. In terms of our nomenclature the data [13] can be summarized in the form $\alpha_T/Sc = -0.13$. Remembering that $Sc > 10^4$, we may conclude that the corresponding value α_T is approximately two orders of magnitude higher than the mean value $\alpha_T = -22.5$ obtained in our experiments for nanometer sized particles in colloids. It is interesting to note that both results are several orders of magnitude less than the coefficient predicted from existing theories for thermal diffusion in gases. Nevertheless, the value of thermal diffusion ratio for nanoparticles in magnetic colloids calculated from our experiments are considerably higher than typical α_T values for molecular liquids and for solutions. Of course, from quantitative point of view our results may not be considered being absolutely precise because they are evaluated by using an approximate value of diffusion coefficient estimated from Stokes law for single Brownian particles suspended in viscous liquid. Nevertheless, the separation experiments indicate a very intensive thermophoresis of nanometer scaled particles in ferrofluids.

5. Conclusions

The particle separation measurements in a vertical thermal diffusion column indicate a high thermophoretic mobility of magnetic nanoparticles in ferrofluids. Colloidal particles stabilized in hydrocarbons by using surfactants are moving toward lower temperatures. Such direction of thermophoresis agrees with the predictions of thermodynamic theory accounting for a slip velocity of lyophilic solid–liquid boundaries. Some preliminary experiments performed by using the water-based ionic ferrofluids indicate an opposite direction of nanoparticle thermophoretic transfer. Obviously, the temperature nonsymmetry of a double-electric layer is responsible for the thermophoresis of charged particles.

According to existing thermal diffusion column theories the development of a concentration

difference in upper and lower chambers should be linear-dependent on time in the initial stage of separation. Most of the experiments, especially those for colloids of high particle concentration when strong temperature gradients are applied, indicate that the separation effect is nearly square-dependent on time. Such a law is predicted by numerical calculations as well as by a nonstationary concentration boundary layer analysis. Obviously, it is necessary to develop a more precise theory of separation in the column accounting for the two-dimensional character and for the nonstationarity of the mixed thermal and concentration-driven convection in the vertical column.

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