SEDIMENTATION CONSTANT OF MAGNETORHEOLOGICAL LIQUIDS

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An original procedure and a measuring device for evaluating the sedimentation constant of magnetorheological liquids are developed. The informativeness of the proposed approach and its adaptability to streamlined production are demonstrated in experiments with specimens of liquids with variation of the concentration and particle size of the disperse phase and the viscosity of the dispersion medium.

Introduction. The problem of sedimentation stability is one of the most complicated tasks solved in the process of production of magnetorheological liquids (MRLs) that are a suspension of ferromagnetic noncolloidal particles ($\rho \approx 7.5 \text{ g/cm}^3$) in a carrier dispersion medium ($\rho_0 \approx 1 \text{ g/cm}^3$). As a rule, ensuring the sedimentation stability of such compositions boils down to a substantial reduction in the rate of particle sedimentation, for example, by introducing into the composition surfactants that prevent agglomeration of particles and progressive sedimentation of agglomerates. Use has also been made of a method consisting in imparting plastic thixotropic properties to the carrier liquid [1] by dispersion of a solid colloidal stabilizer in it. In the process of synthesis of a composition of one type or another, reliable quantitative information on the stability of the final product is of great importance. Methods of measuring the rate of particle sedimentation that are based on visual observation of the movement of the boundary of suspended particles in the carrier medium [2] are most widely used.

However, this methodology does not allow performance of observations in nontransparent media and has intricacies connected with optical measurements using centrifuges, which are necessary in the process of studying the stability of slowly settling suspensions.

The indicated drawbacks can be overcome if use is made of the special feature of an MRL that consists in the fact that its composition has a ferromagnetic disperse phase and, therefore, the magnetic permeability of the liquid depends strongly on the concentration of the particles. The process of sedimentation will be accompanied by departure of ferromagnetic particles from the top layer of the liquid and a reduction in its permeability. Thus, the rate of change of the magnetic permeability of the liquid's top layer can serve as a measure of the rate of particle sedimentation. It is precisely this approach that was used in the proposed rapidmethod of evaluating an MRL's sedimentation stability.

Procedure and Measuring Device. It is common practice to evaluate the intensity of sedimentation of solid particles in a liquid carrier medium by the sedimentation constant, which is the ratio S = u/g [3]. The svedberg (1 Sb = 10^{-13} sec) is taken as the unit of measurements of S. For actual compositions the constant is rather large, and therefore it is more convenient to measure it in megasvedbergs (1 MSb = 10^{-7} sec).

During the sedimentation of a particle in a liquid a frictional force $F_{\rm fr}$ appears, which is written as the Stokes law $F_{\rm fr} = 6\pi\eta au$ in the case of laminar flow of a viscous medium about micron spherical particles (and it is exactly such a situation that is characteristic of the case in question). After balancing the forces of friction and gravity the particle moves uniformly with the velocity $u = 2(\rho - \rho_0)a^2g/9\eta$. Thus, the sedimentation constant equals

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$$S = 2 (\rho - \rho_0) a^2 / 9\eta .$$
 (1)

Since sedimentation under gravity is a slow process in many cases, to intensify it use is usually made of centrifugal force, which is created by means of a centrifuge. In this case the force that causes sedimentation of the particles is the centrifugal force $F_c = \frac{4}{3}\pi(\rho - \rho_0)a^3\Omega^2 R$. It is more convenient to write the equilibrium between $F_{\rm fr}$ and F_c that occurs in sedimentation in the following manner:

$$6\pi \,\eta a dR/dt = \frac{4}{3} \,\pi \left(\rho - \rho_0\right) a^3 \,\Omega^2 R \,, \tag{2}$$

where dR/dt is the sedimentation rate.

Relation (2) holds if one can disregard gravity (provided $F_c >> F_g$) and the influence of the diffusion of the disperse phase, which is easily attainable under actual conditions. By separating the variables in relation (2) and integrating it between the limits from the initial radius R_0 to R and, accordingly, from t = 0 to t, we will obtain $S = \frac{\ln(R/R_0)}{\Omega^2 t}$. Taking into account the fact that $\Omega = \pi n/30$ and $x = R - R_0$ (x is the distance cov-

ered by the particles in the time t), we obtain a relation for determining the sedimentation constant:

$$S = \frac{900 \ln (1 + x/R_0)}{\pi^2 n^2 t} \,. \tag{3}$$

Thus, the main task to be solved in the process of measuring the sedimentation constant consists in on-line monitoring of the distance covered by the settling particles. To this end, use is made of a method based on measuring the inductance of a solenoid enclosing a small portion of the MRL column in its upper part.

It is known that the inductance of a solenoid in alternating current is determined by the following expression:

$$L = \frac{m^2 \mu_0 \,\mu W}{l} = L_0 \,\mu \,, \tag{4}$$

where μ is the relative magnetic permeability of the specimen enclosed by the solenoid.

It is seen from (4) that the solenoid's inductance is sensitive to the magnetic properties of the medium located inside it.

Let us consider a long cylindrical specimen of an MRL, a small part of which is located inside the measuring solenoid. The upper end of the specimen coincides with the beginning of the solenoid's turns. All MRL particles are of the same size, and their sedimentation is not constrained by the lower layers. Since the process of sedimentation is accompanied by removal of ferromagnetic particles from the solenoid's active zone and accordingly by a reduction in the average value of μ over the volume, the inductance will fall, the decrease being proportional to the number of particles leaving the zone. In other words, uniform movement of the column of particles in the carrier medium occurs. Since the nonmagnetic carrier medium of the MRL has $\mu \approx 1$, the process is modeled by the situation where the "core," which possesses a certain magnetic permeability μ , is removed from the coil. In this case the solenoid's inductance is determined by the expression $L = L_0(\mu + \frac{x}{l}(1-\mu))$. As a result, the distance covered by the particles during the time of action of the centrifugal force is calculated from the current value of the inductance of the solenoid *L* on the basis of the following relation:

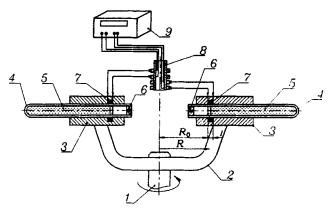


Fig. 1. Diagram of the measuring device.

$$x = l \frac{(L_{\max} - L)}{(L_{\max} - L_0)},$$
(5)

where L_{max} is the solenoid's inductance before the beginning of the process of sedimentation.

Furthermore, the method allows evaluation of the yield stress of the dispersion medium of the MRL in the case where stabilization is carried out by introducing special additives to impart plastic thixotropic properties to the dispersion medium. It is known [1] that the limiting shear stress (or yield stress) τ_0 that keeps a spherical particle in the dispersion medium in a motionless state in the centrifugal-force field is determined by the relation $\tau_0 = \pi a (\rho - \rho_0) \Omega^2 R_0 / 7.5$ or

$$t_0 = \frac{\pi^3 a \left(\rho - \rho_0\right) R_0 n_*^2}{6750},\tag{6}$$

where n_* is the limiting value of the centrifuge shaft's rotational velocity, before reaching which the MRL did not stratify.

A diagram of a set up that realizes the procedure proposed for determining the MRL sedimentation constant is given in Fig. 1. On the rotor 1 of the centrifuge a balance beam 2 with rings 3 is mounted, in which test tubes 4 filled with MRL specimens 5 are installed. Stoppers 6 are inserted into the test tubes from their open end to prevent evaporation of the carrier medium in the process of the experiment. In the rings measuring solenoids 7 fitting the test tubes' walls are fixed. The solenoid coil leads are connected to the inductance meter 9 through the rotating current collector 8. The setup allows measurement of the sedimentation constant simultaneously for two MRL specimens. In testing one specimen the opposite test tube with a liquid must be installed for balancing.

To determine the sedimentation constant for constant rotational velocity of the shaft, the solenoid's inductance is measured in a certain time interval, and S is calculated using relation (3).

When the yield stress τ_0 of the dispersion medium is evaluated, the shaft's revolutions are continuously increased to the point where the inductance begins to drop, and a reading of n_* is taken, which is used to determine the yield stress in accordance with expression (6).

Measurement Results and Discussion. The method and the device were tested in experiments aimed at determining the influence of the particle size, the concentration of the ferromagnetic disperse phase, and the carrier-liquid viscosity on the MRL sedimentation stability. Specimens of the liquid with a volume of 30 ml were produced by hand by thorough (for 2 h) "grinding" of the components in a mortar mill. Ferromagnetic carbonyl iron powder was used as the disperse phase. Three series of experiments were conducted. In the first series the specimen tested was a stabilized iron suspension in mineral oil. Similar compositions are used in magnetorheological devices of electrohydraulics [4]. The volume concentration of the disperse phase was C =25%, and the mean size of the particles varied from specimen to specimen from 1.17 to 23 μ m. In the second

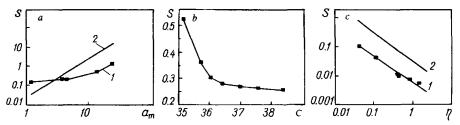


Fig. 2. Sedimentation constant vs. mean size of the particles of the ferromagnetic disperse phase (a) [1) experiment; 2) calculation by (1)], its volume concentration (b), and viscosity of the carrier liquid (c) [1) experiment; 2) calculation by (1)]. S, MSb; a_m , μm ; C, %; η , Pa·sec.

series aqueous suspensions used in the technology of glass polishing [5] were tested. Particles with $a_m = 3.5 \mu m$ whose content by volume varied from 35 to 39% were used. In the third series experiments were conducted with suspensions of particles ($a_m = 3.5 \mu m$, C = 36%) in a water-glycerin mixture with different viscosities in the range of 0.44–1.44 Pa·sec.

Measurement results are presented in Fig. 2 as graphs of the sedimentation constant versus the size of the particles, their concentration, and the viscosity of the carrier liquid, respectively.

An increase in the size of the particles undoubtedly decreases the MRL stability (Fig. 2a). The graph shows that in terms of stability a size of the order of 1-4 μ m is preferable since as a_m decreases, the magnetorheological effect, which is the main user property, of the MRL falls linearly [6]. We note the divergence in the character of the experimental dependence $S = f(a_m)$ (curve 1) and the dependence calculated by (1) (curve 2) in the region of relatively small a_m . It is probably due to the fact that fine particles are united into agglomerates despite the adopted measures aimed at their disintegration. What is meant here is the use of surfactants and mechanical grinding in a mill. For larger particles this problem does not arise and the character of the experimental dependence coincides with the calculated one. The calculated and measured data differ significantly in absolute value. This is due to the fact that, unlike the "ideal" conditions of unconstrained particle sedimentation characteristic of very dilute suspensions, in the experiments use was made of rather concentrated compositions in which such conditions were not satisfied.

An increase in the volume concentration of carbonyl iron in the MRL composition decreases the sedimentation constant (Fig. 2b), in connection with which more concentrated compositions are preferable from the viewpoint of solving the problem of stability.

An increase in the viscosity of the carrier medium stabilizes the MRL (Fig. 2c). The calculated (curve 2) and experimental (curve 1) dependences $S = f(\eta)$ are practically equidistant while the reason for the discrepancy in the absolute values of S is the same – a high concentration of the particles.

The experimental procedure presented above was employed to determine the plastic properties of the dispersion medium of an MRL, which are characterized by the yield stress. For this purpose a specimen was synthesized (C = 10%) that contained solid colloidal SiO₂ particles in its composition. At rest the colloid forms a spatial grid in the volume of the carrier liquid under the action of van der Waals forces; the grid keeps large ferroparticles from sedimenting. The presence of this microstructure and its strength are judged from τ_0 . Even a small shear breaks down the grid, liquefying the MRL to an acceptable level. Thus, the colloidal stabilizer, preventing sedimentation of the particles of the disperse phase at rest, neither increases substantially the viscosity of the composition as a whole in flow nor keeps the ferromagnetic particles from approaching each other under the action of a magnetic field, which is required for manifestation of a magnetorheological effect. One traditionally determines τ_0 in a viscosimetric experiment from an analysis of the flow curve $\tau = f(\dot{\gamma})$. Here, by extrapolating the curve in the direction of a decrease in the rate of shear until it intersects the axis of ordinates at $\gamma = 0$ one obtains the required value τ_0 . Based on this approach a yield stress of 2.0 Pa is obtained for the synthesized specimen. The sedimentation method of measurement gave a similar value of $\tau_0 = 2.1$ Pa, which indicates its reliability and applicability.

CONCLUSIONS

1. The experiments showed that the method proposed is applicable for on-line determination of the sedimentation constant of actual MRLs that are used in different technological processes. It makes it possible to evaluate, in the synthesis stage, the sedimentation stability of the compositions developed and to introduce corrections into the formula for the liquids.

2. The procedure described assumes free (not constrained by neighboring particles) motion of particles of the same size. In actual MRLs, there is a size distribution of the particles; in moderately concentrated (1% < C < 10%) and concentrated ($C \ge 10\%$) compositions, particle sedimentation is certainly constrained. In this connection, the measurement error for the absolute value of the true sedimentation constant is rather large. However, in developing MRLs and investigating their properties evaluation of the sedimentation stability of actual working compositions rather than model experimental conditions is of prime interest. From this viewpoint, the rapid procedure proposed is very informative and adaptable to streamlined production.

NOTATION

ρ, density of the particles of the disperse phase, g/cm³; ρ₀, density of the dispersion medium, g/cm³; u, sedimentation rate of the particles, m/sec; g = 9.8, acceleration of gravity, m/sec²; S, sedimentation constant, Sb; $F_{\rm fr}$, frictional force, N; F_g , gravity force; F_c , centrifugal force, N; η, dynamic viscosity of the liquid, Pa·sec; a, radius of a particle, m; $a_{\rm m}$, mean radius of a particle, m; Ω, angular velocity, sec⁻¹; R, current radius of rotation, m; R_0 , initial radius of rotation, m; n, rotational velocity of the centrifuge shaft, rpm; n_* , limiting value of the rotational velocity of the centrifuge shaft, rpm; t, time, sec; x, distance traversed by the sedimenting particles, m; L, inductance of the solenoid, H; L_0 , inductance of the "empty" solenoid, H; $L_{\rm max}$, inductance of the "filled" solenoid, H; m, number of turns of the solenoid coil; μ, relative magnetic permeability, H/m; $\mu_0 = 1.256 \cdot 10^{-6}$, magnetic permeability of vacuum, H/m; W, cross-sectional area of the turns of the solenoid coil, m²; l, solenoid length, m; τ_0 , yield stress, Pa; C, volume concentration, %; γ , rate of shear, sec⁻¹. Subscripts: fr, friction; g, gravity; c, centrifugal; m, mean.

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