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ВЛИЯНИЕ ВЯЗКОСТИ ДИСПЕРСИОННОЙ СРЕДЫ НА ЭЛЕКТРОРЕОЛОГИЧЕСКОЕ ПОВЕДЕНИЕ СУСПЕНЗИЙ ГАЛЛУАЗИТА В ПОЛИДИМЕТИЛСИЛОКСАНЕ

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Исследовано электрореологическое поведение суспензий галлуазита в полидиметилсилоксане с кинематической вязкостью 50, 100 и 400 сСт, концентрация наполнителя составляла 4 и 8 масс.%. Исследуемые суспензии являются электрореологическими жидкостями: под действием электрического поля все образцы проявляют вязкоупругие свойства, и значения предела текучести существенно возрастают. Изменение реологического поведения суспензий связано с образованием протяженных колончатых структур из частиц наполнителя вдоль силовых линий электрического поля. В работе изучена зависимость интенсивности электрореологического эффекта от напряженности электрического поля, концентрации наполнителя и вязкости дисперсионной среды. Показано, что значения предела текучести увеличиваются с возрастанием напряженности электрического поля и концентрации наполнителя. Выявлено, что в рамках точности эксперимента значения предела текучести не зависят от вязкости дисперсионной среды при фиксированной концентрации и напряженности электрического поля. Также была оценена седиментационная устойчивость суспензий. Скорость осаждения частиц галлуазита ниже в суспензиях на основе более вязких масел, что в первом приближении согласуется с формулой Стокса. Величина равновесного седиментационного отношения – высоты коллоидной фазы к общей высоте столба жидкости, зависит от концентрации наполнителя и оказывается выше для суспензий с большим содержанием частиц. Выявлен не монотонный характер зависимости относительной эффективности суспензий от вязкости дисперсионной среды для 8 масс.% суспензий. Показано, что комбинация трех параметров: вязкости дисперсионной среды, концентрации наполнителя и напряженности электрического поля позволяет получать электрореологические жидкости с заданными, прогнозируемыми свойствами.

Ключевые слова: электрореологическая жидкость, галлуазит, вязкость, предел текучести

EFFECT OF THE DISPERSION MEDIUM VISCOSITY ON THE ELECTRORHEOLOGICAL BEHAVIOR OF HALLOYSITE SUSPENSIONS IN POLYDIMETHYLSILOXANE

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The electrorheological behavior of halloysite suspensions in polydimethylsiloxane with kinematic viscosities of 50, 100, and 400 cSt was studied. The filler concentration was 4 and 8 wt%. The suspensions under study are electrorheological fluids. All samples revealed viscoelastic properties under electric field and the yield stress values significantly increased. The rheological behavior of suspensions are changed due to the extended columnar structures formation from the filler particles along with the electric field. In present study, the dependence of the electrorheological effect intensity on the electric field strength, concentration of the filler and the viscosity of the dispersion medium was obtained. It was shown that the values of the yield stress increase with electric field strength and filler concentration. The values of the yield stress are independent on the viscosity of the dispersion medium at a fixed concentration and electric field in the framework of experimental accuracy. The sedimentation stability of the suspensions was evaluated as well. The sedimentation rate of halloysite particles was lower in suspensions based on more viscous oils, which in rough estimation correlates with the Stokes equation. The level of the final sedimentation ratio, i.e. the height of the colloidal phase to the total height of the liquid, depends on the concentration of the filler. Thus, the higher the particles concentration, the higher the sedimentation ratio. The non-monotonic dependence of the relative efficiency of the suspensions on the viscosity dispersion medium for 8 wt% suspensions was revealed. It was shown that a combination of three parameters, namely the dispersion medium viscosity, the filler concentration and the electric field strength, makes it possible to obtain electrorheological fluids with specified, predictable properties.

Key words: electrorheological fluid, halloysite, viscosity, yield stress

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INTRODUCTION

Electrorheological fluids (ERFs) are suspensions of a solid dispersed phase with a high dielectric constant in a non-polar dispersion medium. The rheological behavior of ERFs reversibly changes under electric field resulting in an increase in viscosity up to several orders of magnitude or the yield stress. Thus, the fluid reveals elastic properties like a solid body [1]. ERFs are called "smart" materials due to the fast and reversible change in properties, as well as the ability to control the characteristics by external stimuli. The phenomenon of a reversible increase in the viscosity of dispersed systems under electric field is the electrorheological (ER) effect. Along with other "smart" materials, ERFs are widely used in technology in various devices such as shock absorbers, control valves, dampers and in microfluidics, robotics, sensors, etc [2]. The modern applications of the ER effect include sensors for rapid plague diagnostics and nanodiaphragms with adjustable aperture [3, 4]. The several qualities are essential to the wide practical application of ERFs, namely significant change of rheological properties under electric field, low conductivity, chemical, aggregate and thermal stability. Moreover, they should be non-abrasive and environmentally friendly. The fabrication of ERFs with the specified properties requires fundamental research of the ER effect associated with the study of the influence of many factors. Thus, electric field strength [5], temperature [6], concentration [7], size and morphology [8, 9], as well as the electrophysical characteristics of the filler particles [10, 11], their modifications [12, 13] and orientation under electric field [14, 15], the presence of additives [16,17] and the viscosity of the dispersion medium [18-21] affect the ER effect.

The influence of the dispersion medium viscosity on the ER effect was studied by several groups of researchers for various fillers and media. However, the number of such studies is limited [18-21]. In some studies, an increase in the ER effect with an increase in oil viscosity was noted [18, 19], in others the weakening of the effect occurs [20, 21]. The each group hypothesizes explanation of their results.

N. Ma and X. Dong suggested that oil viscosity affects the ER effect in two ways. On the one hand, the wettability of higher viscosity oils on filler particles is lower than that of lower viscosity ones, which in turn leads to inhomogeneous dispersion of the filler and decreases the ER effect. On the other hand, oil macromolecules promote particles agglomeration and the formation of columnar structures and result in increase of the ER effect [19]. It is well known that the viscosity of polydimethylsiloxane (PDMS) rises with the chain length of its macromolecules. Therefore, the effect of agglomeration may be stronger than the effect of deteriorating wettability for some fillers with increasing oil viscosity.

Currently, a significant influence of the particle shape on the ER effect has been shown [8, 9, 22]. Suspensions filled by particles with a high aspect ratio exhibit a significant ER activity even at a low concentration, due to the percolation network formation at fillings of a few weight percent. Layered aluminosilicates are one of such fillers.

Aluminosilicate particles reveal an ER effect and are an inexpensive natural raw material. That is why it attracts the attention of researchers [23, 24]. Halloysite is one of the promising aluminosilicate fillers. Halloysite refers to the kaolinite group and consists of two layers. One of them is silicon-oxygen tetrahedra and another is oxygen octahedra with a central aluminum atom [25]. The morphology of halloysite depends on the natural conditions of formation, but most often, the mineral is found in the form of multilayer nanotubes with a cavity inside. Halloysite suspensions in PDMS reveal an ER effect [26]. Moreover, a small amount of water adsorbed on the filler surface becomes fundamentally important for the ERF operation and the magnitude of the ER effect [27]. However, the effect of the medium viscosity on the ER behavior of suspensions filled by particles with a high aspect ratio has not been studied. In the presented study, the influence of PDMS viscosity on the ER behavior of halloysite suspensions of various concentrations was investigated. The dependence of the ER effect on the rheological properties of the dispersion medium is an urgent task and will make it possible to better comprehend the mechanism of the ER effect in order to produce materials with predetermined properties, and promote the expansion of the areas of ERF application.

MATERIALS AND METHODS

Halloysite nanotubes (Sigma Aldrich, USA) were used as a filler for ERFs. The sizes of nanotubes range from 50 nm to 2 μm in length and from 20 to 300 nm in diameter. The typical aspect ratio is 5-6. The walls of the nanotube consist from 11 to 20 layers with an interplanar distance of 0.81 nm [27].

PDMS of various molecular weights (brands PMS 50, PMS 100, PMS 400, Penta Junior, Russia) were used as a dispersion medium. The declared kinematic viscosities are 50, 100 and 400 cSt, respectively. The molecular weight characteristics were determined by the size-exclusion chromatography. The molecular weight and polydispersity index are 5.8, 10.7, 20.9 kDa and 2.0, 1.8, 2.0 respectively.

Halloysite powder was dried under vacuum at 60 °C for 24 h with following increasing the temperature up to 80 °C for 2 h to increase the stability of the

ER response and reduce the conductivity of the suspensions. Suspensions filled up to 4 and 8 wt% were obtained by mixing halloysite powder with PDMS of the selected brand. Homogenization was carried out using an MR Hei-Tec magnetic stirrer (Heidolph, Germany) at a rate of 400 rpm for seven days. The sonication was performed for 30 min using an ultrasonic bath UZV-4.0/1 TTTs (RMD) (150 W, 35 kHz) (Sapfir LLC, Russia) before each measurement. The reliability of the results was achieved by studying three independent series of samples for each concentration and type of dispersion medium.

The rheological behavior of suspensions without and under electric field were studied by rotational viscometry on a Physica MCR 501 rheometer (Anton Paar GmbH, Germany) in a measuring system of Searle-type two coaxial cylinders. The electrical potential supplied from DC high voltage source FuG HCP 14 – 12500 MOD (FuG Elektronik GmbH, Germany) to an inner cylinder isolated from the main device by a ceramic gasket. The cell volume was 20 ml and the gap was 1 mm. The electric field strength was varied in the range of 0-5 kV/mm. The flow curves were obtained in the controlled shear stress (CSS) mode in the stress range from 0.01 to 300 Pa with the shear rate measurement range limited to 100 s^{-1}.

Optical microscopy images without and under electric field were obtained using an Axio50 Imager.M2m optical polarizing microscope (Carl Zeiss AB, Sweden). The electric potential was supplied from a high voltage source IVNR 15/1 (±) (Plazon, Russia), the operating voltage range was up to 10 kV.

Halloysite precipitates over time and a phase separation appears. The suspensions were transferred in test tubes protected from mechanical influences to assess the sedimentation stability and the dependence of the sedimentation ratio R (the ratio of the height of the colloid phase to the total height of the liquid) on time was measured.

RESULTS AND DISCUSSION

PDMS exhibits Newtonian behavior. The viscosity of the oils is constant in whole studied shear rate range and the shear stress increases linearly with shear rate. When halloysite is added to PDMS, the rheological behavior of the suspensions changes. The fluids exhibit elastic properties, and the yield stress is observed. Such behavior can be approximated by the Bingham model:

$$\tau = \tau_0 + \eta_p \dot{\gamma}, \qquad (1)$$

where τ is shear stress, τ_0 is yield stress, $\dot{\gamma}$ is shear rate, η_p is plastic viscosity, the coefficient takes the value of viscosity at high shear rates.

The yield stress indicates the formation of a weak percolation network from interacting particles. The yield stress values increase with filler concentration. However, the plastic viscosity of the suspensions changes insignificantly, the flow curves at high shear rates have a similar slope. Figure 1 shows the flow curves for PMS 100 and halloysite suspensions based on it at various concentrations as an example.

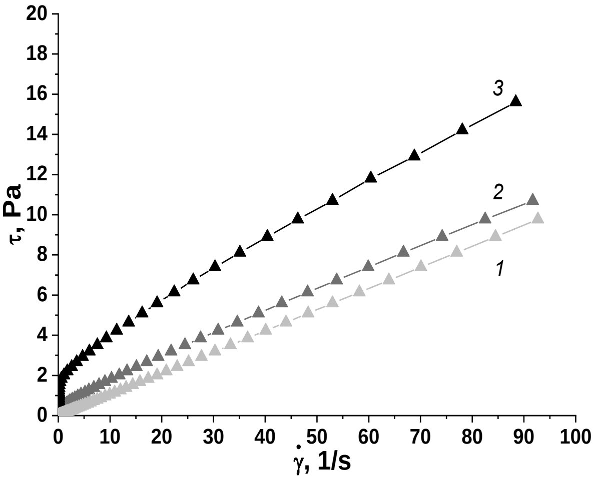

Fig. 1. Flow curves of PMS 100 (1) and halloysite suspensions with concentration of 4 wt% (2) and 8 wt% (3)

Рис. 1. Кривые течения ПМС 100 (1) и суспензий галлуазита с концентрациями 4 масс.% (2) и 8 масс.%. (3)

The electric field has no influence on the rheological behavior of PDMS, which confirms the high insulating properties of the oils, but it critically affects the behavior of halloysite suspensions. Thus, halloysite particles polarize and form columnar structures under electric field resulting in significant increase of the yield stress (Fig. 2). An independent series of experiments was carried out on identically prepared samples to obtain reliable results for the yield stress values. A noticeable increase in the yield stress for all studied samples is observed at electric field strengths above 3 kV/mm. Therefore, further analysis of the dispersion medium viscosity effect on the ER behavior of suspensions provided in the range of electric fields of 3-5 kV/mm.

The yield stress was defined as the maximum value of the shear stress at zero shear rate. Since the shear stress during the experiment takes discrete values, the yield stress can be found with an error determined by the difference between two nearest values. The results are summarized in Fig. 3.

One can note that the yield stress increase with electric field, as well as with halloysite concentration for all studied oils. However, the dispersion medium viscosity does not affect the values of the yield stress within the experimental accuracy. The obtained result can be explained by the low efficiency of both factors of the medium viscosity influence on the ER proposed by N. Ma and X. Dong [19]. The length of the PMS 400 molecules is small compared to the halloysite particle in order to strengthen the columnar structure due to the adhesion of one molecule between two particles. On the other hand, the low wetting of halloysite by more viscous oil can lead to inhomogeneous dispersion of the filler, but does not qualitatively change the shape of the agglomerate. Thus, the strength of the structure formed under electric field remains.

Despite the fact that the magnitude of the ER effect (yield stress) does not depend on the dispersion medium viscosity, the efficiency of fluids, namely the contrast transition from viscous behavior to elastic ones, turned out to be different. Thus, the intensity of the ER effect can be expressed by its relative efficiency

$$E_{Rel.} = \frac{\tau_E - \tau_0}{\tau_0} \quad (2)$$

where τ_E is the yield stress under electric field, τ_0 is the yield stress without electric field. The relative efficiency values of the studied fluids are presented in Table.

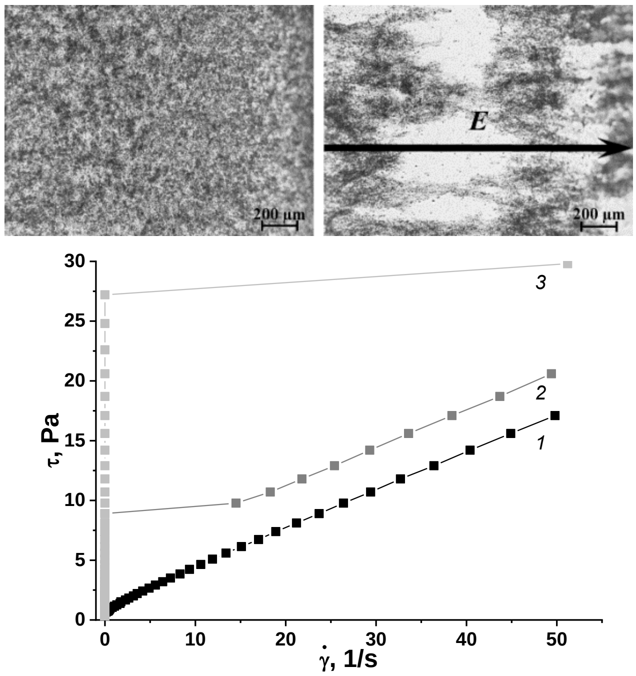

Fig. 2. Flow curves of halloysite suspensions in PMS 400 with concentration of 4 wt% without (1) and under electric field of 3 kV/mm (2) and 5 kV/mm (3). The inset shows optical images of 1 wt% halloysite suspensions in PMS 100 without and under electric field of 1 kV/mm. The columnar structures are clearly seen under electric field action

Рис. 2. Кривые течения 4 масс.% суспензии галлуазита в ПМС 400 вне (1), и под действием электрического поля напряжённостью 3 (2) и 5 кВ/мм (3). На вставке приведены оптические фотографии 1 масс.% суспензии галлуазита в ПМС 100 вне и под действием электрического поля напряженностью 1 кВ/мм. Видно формирование колончатых структур под действием электрического поля

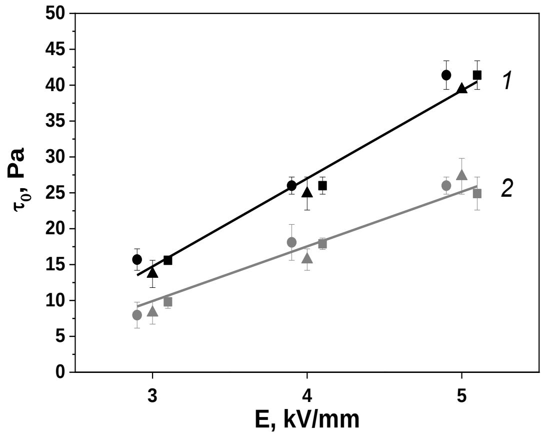

Fig. 3. The yield stress of halloysite suspensions in PMS 50 (●), PMS 100 (▲), PMS 400 (■) at concentration of 8 wt% (1) and 4 wt% (2) under various electric fields. For clarity, the values of the yield stress for suspensions based on oils of different viscosities at the selected electric field strength are shifted along the abscissa axis

Рис. 3. Зависимость предела текучести от напряженности электрического поля 8 масс.% (1) и 4 масс.% (2) суспензий галлуазита в ПМС 50 (●), ПМС 100 (▲), ПМС 400 (■). Для наглядности значения предела текучести для суспензий на основе масел разной вязкости при выбранном значении напряженности электрического поля сдвинуты вдоль оси абсцисс

Table

The relative efficiency of ERFs

***Таблица*. Относительная эффективность ЭРЖ**

Conc., wt%	*E*, kV/mm	$E_{rel.}$, rel. un.		
		PMS 50	PMS 100	PMS 400
4	3	50	27	18
	4	90	55	37
	5	140	83	57
8	3	15	8	18
	4	27	14	31
	5	43	23	50

One can see that the relative efficiency of all suspensions increases with electric field strength and higher at lower fillings. Nevertheless, a feature is observed in the values of the relative efficiency for suspensions of various concentrations at different viscosities of the dispersion medium. Thus, for 4 wt% samples the relative efficiency of fluids decreases with an increase in oil viscosity. With an increase in concentration up to 8 wt% the dependence of the relative efficiency on the medium viscosity reveal extremum (minimum for PMS 100). It is obvious that suspensions based on different oils exhibit the same values of the yield stress under electric field, but different without the electric stimuli. The yield stress without electric field of suspensions filled up to 4 wt% increases with PDMS viscosity from 50 cSt to 400 cSt. The percolation network formed by particles breaks and immediately relaxes at low shear rates. The movement of particles in a more viscous medium requires more stress. However, suspensions filled up to 8 wt% based on

PMS 400 have lower yield stress than fluids based on PMS 50 and PMS 100. Such feature is probably associated with a lower quality of halloysite dispersion in a more viscous oil with increasing concentration and leads to a decrease in the strength of the percolation network. Thus, at a filler concentration of 4 wt%, the relative efficiency is higher for suspensions based on less viscous oils. However, for 8 wt% fluids the quality of filler dispersion plays a crucial role for producing the ERFs with predetermined properties.

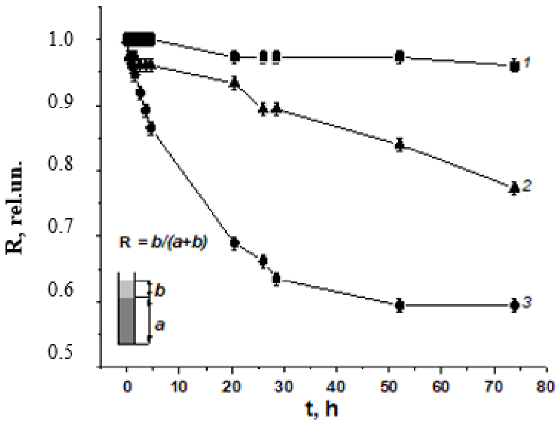

Fig. 4. Sedimentation stability of 4 wt% halloysite suspensions in PMS 400 (1), PMS 100 (2), PMS 50 (3)
Рис. 4. Седиментационная устойчивость 4 масс.% суспензий галлуазита в ПМС 400 (1), ПМС 100 (2), ПМС 50 (3)

Sedimentation stability is an important characteristic of ERF. The viscosity of the dispersion media significantly affects the stability of the suspensions. Fig. 4 shows the analysis of the sedimentation stability for 4 wt% halloysite suspensions based on various PDMS as an example. The sedimentation ratio decreases in time, reaching a constant value. The slower the sedimentation, the more stable the suspension. According to the Stokes equation, the sedimentation rate is lower in more viscous dispersion media:

$$V = \frac{2}{9}\frac{r^2 g(\rho_p - \rho_f)}{\eta} \qquad (3)$$

where V is settling velocity, r is the radius of a spherical particle, g is the gravitational field strength, ρ_p and ρ_f are the mass densities of the particles and the fluid, respectively, and η is dispersion media viscosity.

The Stokes equation determines the settling rate of spherical non-interacting particles. Halloysite particles have a high aspect ratio, and their interaction cannot be neglected at the studied concentrations. However, the inverse proportionality of the settling rate to viscosity is observed for the halloysite suspensions as well (Fig. 4). The sedimentation ratio is leveling after the formation of an equilibrium colloidal structure. The final sedimentation ratio is obviously the greater, the higher the filler concentration.

CONCLUSIONS

The plastic viscosity of halloysite suspensions in PDMS filled up to 8 wt% changes slightly. The yield stress of suspensions increases with the filler concentration and electric field strength in the whole studied range. PDMS viscosity in the range from 50 cSt to 400 cSt does not significantly affects the ER effect, nevertheless, it increases the sedimentation stability of suspensions. It is shown that the relative efficiency of the ERFs depend on the quality of the filler dispersion and on the strength of the native percolation network. Thus, by varying several parameters such as the viscosity of the dispersion medium, the concentration of the filler, and the strength of the electric field, one can obtain ERF with different efficiency and predetermined predictable properties.

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The authors declare the absence a conflict of interest warranting disclosure in this article.

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