Modification of carbonyl iron particles by carboxyl-containing polydimethylsiloxanes

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The possibility of preparing magnetic particles with the "core—shell" structure was demonstrated. The procedure is based on the treatment of carbonyl iron particles with polydimethylsiloxanes (PDMS) containing terminal or distributed over the chain carboxyl groups attached *via* the alkyl or aryl spacer. The presence of carboxyl-containing PDMS grafted to the surface of carbonyl iron particles was revealed by electron microscopy. Magnetorheological fluids and magnetic elastomers containing magnetic particles with the "core—shell" structure were obtained, and their rheological properties were studied.

Key words: carbonyl iron, polydimethylsiloxane, carboxyl-containing polymers, magnetic particles, magnetic fluids, magnetic elastometers.

The preparation of magnetic fluids and magnetic elastomers capable of changing their physical mechanical and electromagnetic properties in wide ranges under the action of external magnetic fields is a very urgent trend in the modern chemistry. Interest in these materials is evoked by numerous unusual physical phenomena observed under the action of external magnetic fields and also by a broad scope of their possible applications.[1,2] For example, magnetic fluids find use in the equipment providing the control of vibrations or transfer of effort (for instance, in vibroinsulators, dampers, and brake gears).[3,4] The technological approaches to the use of magnetic elastomers in various devices were described in the review.[5]

Prevention of magnetic particle aggregation is the common urgent task for the preparation of magnetic fluids in which magnetic particles are dispersed in the liquid nonmagnetic medium and for magnetic elastomers in which elastomer serves as a matrix. To solve this problem, a technological method is developed for the deposition onto the particle surface of a protection layer compatible with the matrix with the purpose of formation of the "core—shell" structure. Poly(methyl methacrylate) (PMMA),[6—9] copoly(styrene-ethylene-ethylenepropylene-styrene),[10] polyfluorostyrene,[11,12] and poly(*N*-isopropylacrylamide)[13] are grafted on the surface of magnetic particles. The magnetic fluids and elastomers based on polydimethylsiloxane (PDMS) can be used in biomedicine due to biological inertness of this polymer.[14]

The oxide film on the surface of carbonyl iron (CI) particles affects their ability to modification. It is known that such organic ligands as carboxylic acids can be bound to iron oxides.[15] In addition, a covalent bond can be formed between iron and carboxyl group.

The purpose of this work is to elaborate the method of modification of carbonyl iron by carboxyl-containing siloxane oligomers and to evaluate its efficiency by measuring the main rheological parameters of magnetofilled compositions based on DMS.

Experimental

Materials. Carbonyl iron (R20 trade mark, Vekton), toluene (analytical grade), sodium hydroxide (reagent grade, Khimmed), ethanol (Medkhimprom), hydrogen peroxide (37%, Khimmed), 1,1,3,3,5,5,7,7-octamethylcyclotetrasiloxane (D_4) (ABCR), 1,1,3,3-tetramethyldisiloxane (Sigma-Aldrich), α,ω-divinyldimethylsiloxane (DVK-5 trade mark, Penta-91), polymethylhydrosiloxane (Penta-804 trade mark, Penta-91), sulfonic cation-

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exchange resin (CT-175 trade mark, Purolite), and platinum(0)-1,3-divinyl-1,1,3,3-tetramethyldisiloxane complex (2% solution in xylene, Karstedt´s catalyst, Sigma-Aldrich) were used as received.

Methods. Carbonyl iron particles were dispersed in a toluene solution in an ultrasonic bath (model PSB-4035-05m) with a power of 450 W.

Samples were treated on a Centrifuge 5804 R centrifuge (Eppendorf) at a rotation rate of 11 000 rpm for 10 min.

The microstructure of particles was studied by scanning transmission electron microscopy (STEM) on a Titan 80-300 microscope (FEI, USA) equipped with a corrector of spherical aberrations, a high-angle annular dark-field (HAADF) detector (Fischione, USA), and an energy dispersive X-ray spectroscopy (EDXS) system (USA). The study was conducted at an accelerating voltage of 300 kV. The sample was prepared by the deposition of an alcoholic solution with modified CI particles on the copper grid followed by drying at ~20 °C.

The rheological properties of the obtained magnetopolymeric compositions were studied on an MCR 302 rotation rheometer (Anton Paar, Austria) equipped with a magnetic cell (plane—plane type measuring unit). The viscosity of the magnetic liquids was measured in the mode of a constant shear rate in a range of 0.1—100 s^{-1}. Dynamic mechanical tests were carried out in the dynamic mode of shear oscillations in the range of linear viscoelastic behavior (strain amplitude 1%) in a frequency range of 0.01—1000 Hz. All measurements were conducted at 20±0.5 °C. The rheological characteristics were measured in various magnetic fields to study the magnetorheological response of materials. The uniform magnetic field directed perpendicularly to the shear plane was generated by an electromagnet, the current value in which was controlled in the range from 0 to 5 A. According to the specification of the instrument, the magnetic flux density in an empty magnetic cell increased linearly with an increase in the current reaching the value of 1 T at the maximum current of 5 A.

^{1}H NMR spectra were recorded on a Bruker WP-250 SY spectrometer using $CDCl_3$ as a solvent (ACD LABS program).

GPC analysis was conducted on a chromatographic system consisting of a STAIER c.2 high-pressure pump (Akvilon, Russia), a Smartline RI 2300 refractometer (KNAUER, Germany), and a JETSTREAM 2 PLUS thermostat (KNAUER, Germany) at 40±0.1 °C using a toluene—THF (2%) system as an eluent and a flow rate of 1.0 mL min^{-1}. Columns 300×7.8 mm and sorbent Phenogel (5 μm, pore size from 10^3 to 10^5 Å) (Phenomenex, USA) were used.

The interfacial angle for a water droplet was measured on a KRUSSEasyDropStandard instrument with the DSAv 1.90.0.14 software. The volume of the studied droplets was 1 μL.

Telehelic PDMS containing the terminal fragment of undecenoic acid and PDMS with fragments of undecenoic acid distributed along the chain were synthesized according to an earlier described procedure.[16]

The synthesis of PDMS containing fragments of benzoic acid distributed along the chain was conducted using a known procedure.[17]

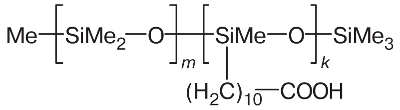
Grafting of carboxyl-containing PDMS to CI particles. Particles of CI (1 g) and toluene (50 mL) were loaded into a 500-mL round-bottom one-neck flask. The flask was placed in an ultrasonic bath. The particles were dispersed for 10 min, carboxyl-containing PDMS (0.3 g) was added, and the mixture was stirred for 16 h at 110 °C. Then the mixture was centrifuged, and the solution was decanted from settled particles. Toluene (70 mL) was added to the precipitate, and the mixture was stirred washing off PDMS molecule that were not covalently bound. Then the solution was again centrifuged and decanted from settled particles. Washing was repeated three times. The obtained particles were dried for 5 h at 50 °C (1 Torr).

Etching of the CI particle surface with alkali. Carbonyl iron (1 g) and a saturated solution of NaOH in ethanol (20 mL) were loaded into a 100-mL round-bottom one-neck flask. The flask was placed in an ultrasonic bath, and the particles were dispersed for 10 min. Then the mixture was stirred for 5 h at 78 °C. The mixture was centrifuged, and the solution was decanted from settled particles. Ethanol (70 mL) was added to the particles, and the mixture was stirred washing off NaOH residues. Then the mixture was centrifuged, and the solution was decanted from settled particles. Washing was repeated three times. The obtained particles were dried for 5 h at 50 °C (1 Torr).

Etching of the CI particle surface with hydrogen peroxide. Carbonyl iron particles (3 g) and hydrogen peroxide (10 mL) were loaded into a 100-mL round-bottom one-neck flask. The mixture was stirred for 0.5 h at 20 °C. Then the mixture was centrifuged, and the solution was decanted from settled particles. The particles were again dissolved in ethanol (70 mL), and the solution was stirred washing from hydrogen peroxide residues. Then the mixture was centrifuged, and the solution was decanted from settled particles. Washing was repeated three times.

Results and Discussion

Studies were carried out for three types of PDMS: **I** with fragments of undecenoic acid (U) statistically distributed along the chain, **II** with the terminal U fragment, and **III** with fragments of benzoic acid statistically distributed along the chain. The synthesis and properties of these copolymers were described previously.[16,17] The characteristics of the carboxyl-containing PDMS used in this work are presented in Table 1. Five modified samples (**1**—**5**) were studied.

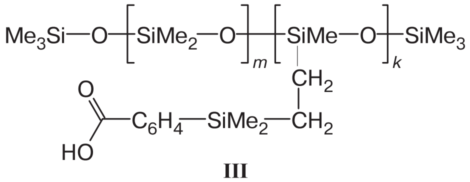


$$\mathrm{Me{-}[SiMe_2{-}O]_m{-}[SiMe(\underset{(H_2C)_{10}-COOH}{|}){-}O]_k{-}SiMe_3}$$

I

$$\mathrm{HOOC{-}(CH_2)_{10}{-}[SiMe_2{-}O]_n{-}SiMe_2{-}(CH_2)_{10}{-}COOH}$$

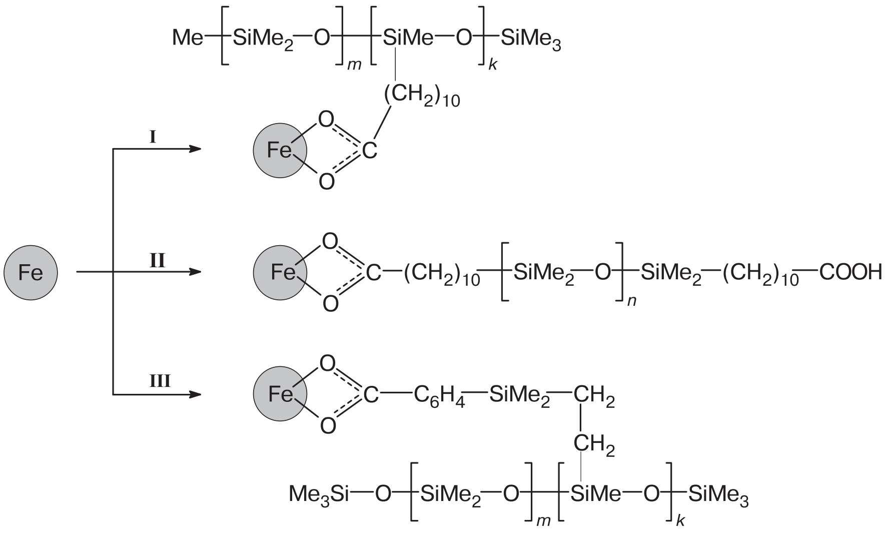
II



III

Sample **1** was obtained by the interaction of the CI particles with PDMS **I** containing U fragments statistically

Scheme 1



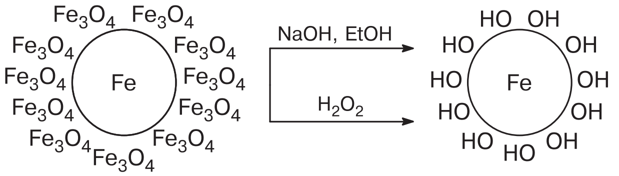
Reagents and conditions: toluene, 110 °C.

distributed along the chain. According to the electron microscopy data, a heterogeneous shell is observed on the surface of the CI particles (Fig. 1, *a*). The EDXS method showed that the shell contains silicon and carbon atoms (see Fig. 1, *d*), which makes it possible to identify the material of the shell as grafted PDMS.

Sample **2** was prepared by the interaction of the CI particles with telehelic PDMS oligomer **II** containing terminal U fragment, and sample **3** was obtained by the interaction with PDMS **III** containing benzoic acid fragments statistically distributed along the chain. Figures 1, *b* and *c* demonstrate the nonuniform distribution of the PDMS layer on the surface of spherical particles.

We studied the influence of pretreatment (etching) of the iron particle surface on the degree of grafting of the PDMS shell. For this purpose, the PDMS oligomer with a high content of carboxyl-containing units distributed along the chain (PDMS-S-U-4) was used. The CI particles were pretreated with a sodium hydroxide solution in ethanol or a solution of hydrogen peroxide and then modified by oligomer PDMS-C-U-4 (samples **4** or **5**, respectively). The pretreatment of the CI particles is presented in Scheme 2.

Scheme 2



The STEM images of the obtained particles are shown in Fig. 2. Agglomeration is observed for the particles sub-

Table 1. Characteristics of carboxyl-containing PDMS

Designation	Code of polymer*	M_n**	M_w/M_n**	Content of modifying units (mol.%) (^{1}H NMR)
I	PDMS-S-U-4	4300	2.8	5.3
II	PDMS-T-U-5	5500	3.3	3.3
III	PDMS-S-B-4	4300	—***	8.0

* Designations in the code of polymer: position of modifying units (T means telehelic, C means that fragments are distributed along the copolymer chain, and U and B designate carboxyl-containing fragments (undecenoic and benzoic acids, respectively); 4 or 5 is molecular weight ($\times 10^{-3}$).

** Determined by GPC relative to polystyrene standards; M_n and M_w are number-average and weight-average molecular weights.

*** The copolymer is poorly soluble in toluene, and only the number-average weight of the initial PDMS with siliconhydride groups is presented for this copolymer.

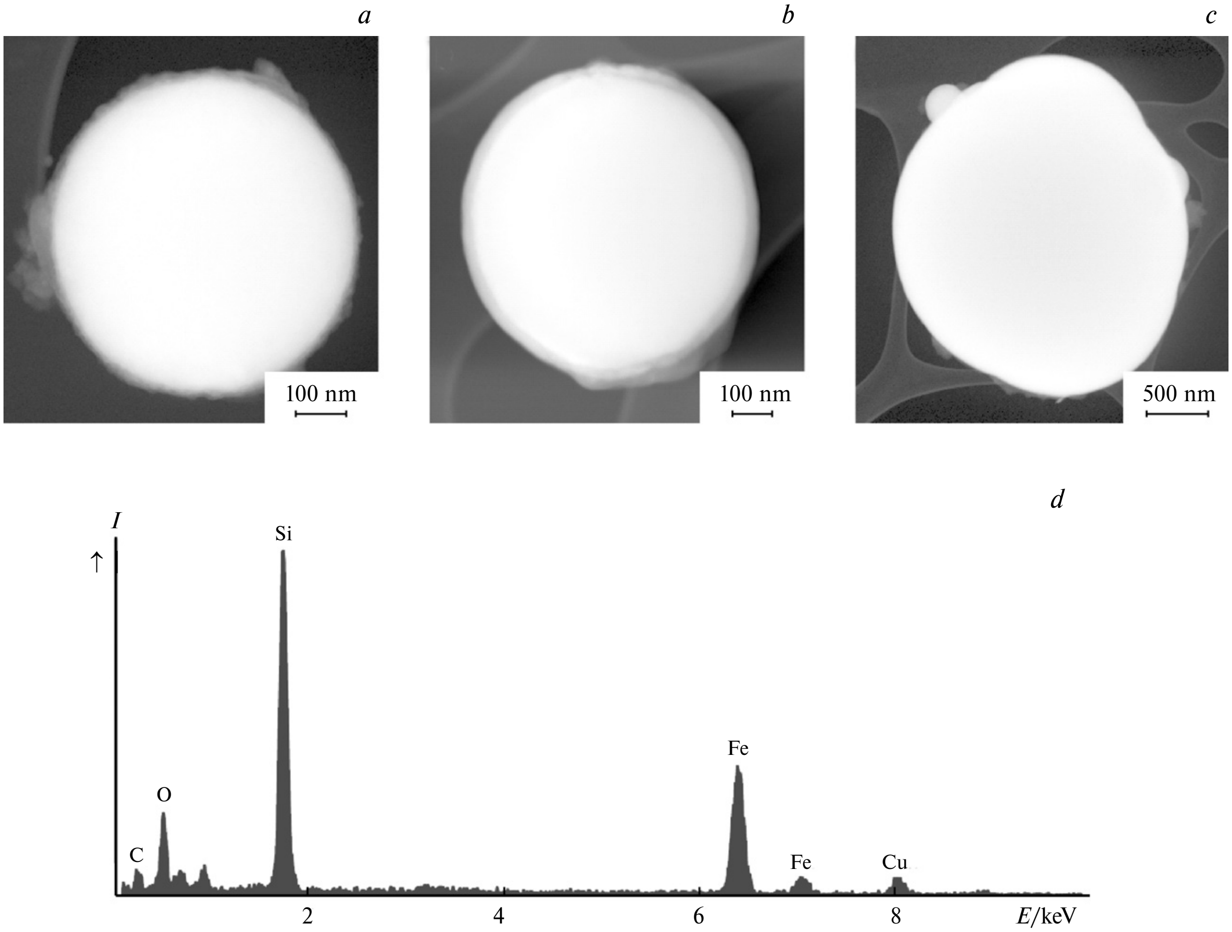


Fig. 1. STEM images of samples **1** (*a*), **2** (*b*), and **3** (*c*) and the EDXS spectrum of the PDMS film (*d*).

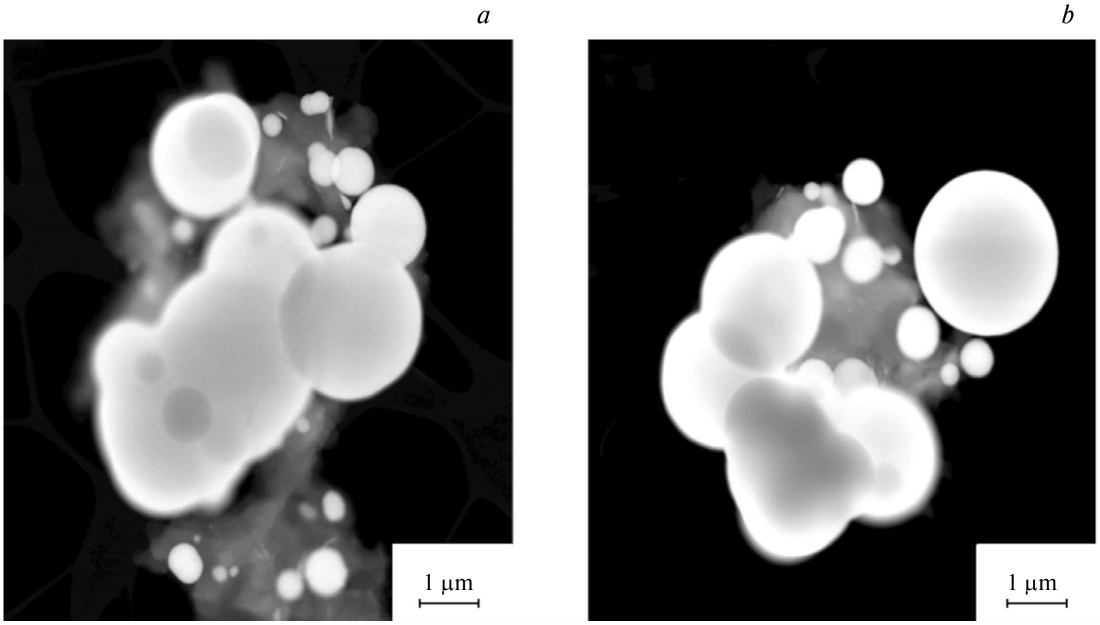


Fig. 2. STEM images of samples **4** (*a*) and **5** (*b*).

jected to both the first and second type of surface pretreatment. It is possible that the etching of the surface increases the activity of the surface layer, therefore several particles can interact with the polymer chain, leading to agglomeration.

Thus, chemical activation of the surface requires to select conditions to avoid undesirable processes. Possibly, the use of monofunctional carbonyl-containing PDMS will enable one to form a shell on the iron particle surface that would not result in aggregation.

In this work, sample **1** obtained by the treatment of iron with the PDMS-S-U-4 polymer without preliminary activation (etching) of the surface was used for the further study of sedimentation, rheology of magnetic and elastomers, and water contact angle on the surface of magnetic elastomers.

A suspension of CI particles (30 wt.%) in PDMS (DVK-5 trade mark) was prepared to study the influence of the shell on the sedimentation of the particles. For better dispersion, a mixture was stirred and then treated in an ultrasonic bath for 30 min. According to the qualitative estimation, the sedimentation rate of the particles is approximately the same, and the first stratification appear in 24 h.

Two magnetic fluids containing 80% CI and 20% DVK-5 were prepared to study the influence of modification of the magnetic particles on the properties of the polymeric composites derived from them. The initial untreated CI particles were used in the first composition whereas iron particles modified by PDMS-S-U-4 without preliminary etching of the iron surface were used in the second composition.

The dependence of the viscosity on the shear rate for the obtained magnetic fluids are presented in Fig. 3. The viscosity was measured after thorough stirring of the compositions in different magnetic fields, which were varied by changing the electromagnetic current.

Figure 3 shows that without magnetic field the viscosity of the magnetic fluid with the CI particles modified by PDMS-C-U-4 differs strongly from the viscosity of the sample with the untreated CI particles. This sample demonstrates the behavior close to that of the Newtonian fluid: the viscosity of this composition depends weakly on the shear rate. On the contrary, the magnetic fluids based on the modified CI particles exhibit the non-Newtonian behavior in the whole studied range of shear rates. The deviation from the Newtonian character of the flow has previously been observed[18,19] for the magnetic fluids based on hyperbranched polycarbosilanes and untreated CI particles with the weight content from 72% and higher, which was explained by a high content of the magnetic filler at which the magnetic particles interact with each other to form a three-dimensional network. Possibly, a more pronounced non-Newtonian flow of the magnetic fluid based on the modified CI particles is related to an effective increase in the particle size due to the formation of an additional layer of the polymer on the surface or due to the adhesion of several particles into large aggregates. Particle structuring along the shear direction should be expected for high shear rates. In the indicated regime, the fluids mainly contributes to the viscosity rather than the particles and, hence, the viscosities of different magnetic fluids are approximately equal in this range of shear rates.

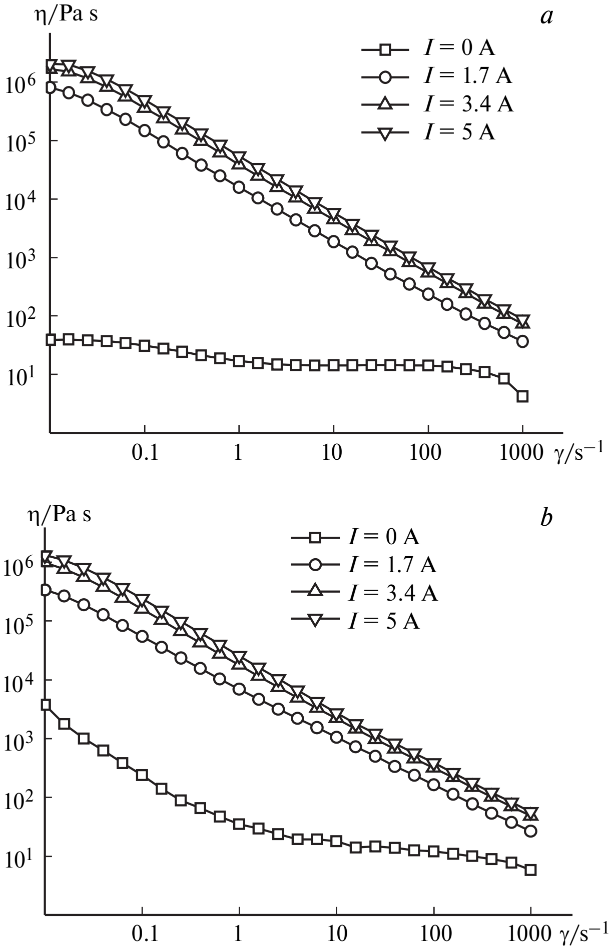


Fig. 3. Viscosity *vs* shear rate of magnetic liquids based on the initial iron particles (*a*) and CI (*b*) modified by PDMS-S-U-4. Measurements were conducted in different magnetic fields (at different electromagnetic currents).

Magnetic field application results in a sharp increase in the viscosity of the magnetic fluid. The character of the dependences of the viscosity on the shear rate is the same for both samples, and the viscosity values themselves in the maximum magnetic field are close. The magnetic fluid based on the unmodified CI particles demonstrates a much higher increase in the viscosity in the magnetic field: the magnetorheological effect (relative increase in viscosity) in the range of low shear rates is more than 4.5 orders of magnitude. The modification of the iron particles by PDMS-S-U-4 leads to a decrease in the relative change in the viscosity because of the high viscosity values of the corresponding magnetic fluid in the absence of magnetic field.

We also studied the relative efficiency of using magnetic particles with different treatments of the surface in the elastomeric composites. The following composition for the matrix was prepared for obtaining magnetic elastomers: 4.98 g of DVK-5, 0.1405 g of α,ω-dihydridooligodimethylsiloxane, and 0.0536 g of PMDS with methylhydrosiloxane units distributed along the chain.

The composition for vulcanization was mixed in tetrafluoroethylene bowl, which was loaded with the composition for matrix and CI particles. The mixture was stirred, Karstedt's catalyst was added (in a ratio of 1 μL per 1 g of the mixture), and the obtained mixture was again thoroughly stirred. The mixture was kept for 0.5 h at ~20 °C and then for 1 h at 100 °C.

Two types of samples of magnetic elastomers were prepared: based on the modified and unmodified CI particles. The filling with CI was 80 wt.% in both cases. The results of studying the rheological properties of the obtained magnetic elastomers are presented in Figs 4—6. Figure 4 demonstrates the behavior of the real and imaginary parts of the dynamic modulus of the samples (storage modulus and loss modulus, respectively) at different shear strain frequencies at a fixed shear amplitude of 0.1%. The dependences of the corresponding values on the shear amplitude at the fixed frequency of mechanical action are presented in Fig. 5. The storage modulus characterizes the elastic properties of the material and determines the amount of the accumulated energy during mechanical loading, whereas the loss modulus shows what amount of the energy is scattered in the strain cycle in the form of heat and quantitatively determines the viscous properties of the material. It is seen that the values of moduli of both samples increase with an increase in the magnetic field intensity, which is characteristic of the magnetic elastomers.[20—24]

The value of magnetorheological effect can be estimated as the relative change in the storage modulus $\Delta G'$ of the material in the region of linear viscoelasticity in which it is independent of the strain value.

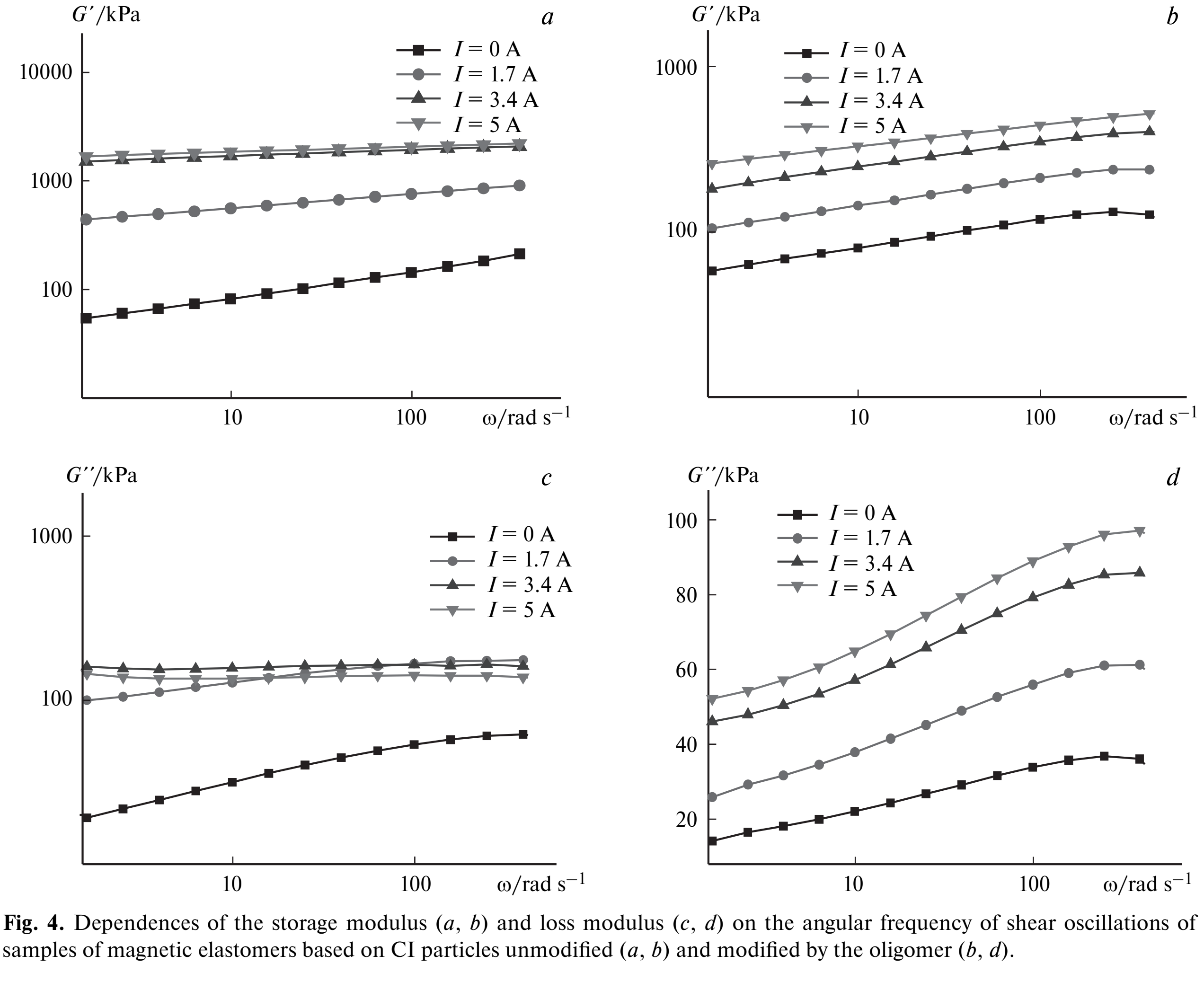


Fig. 4. Dependences of the storage modulus (*a*, *b*) and loss modulus (*c*, *d*) on the angular frequency of shear oscillations of the samples of magnetic elastomers based on CI particles unmodified (*a*, *b*) and modified by the oligomer (*b*, *d*).

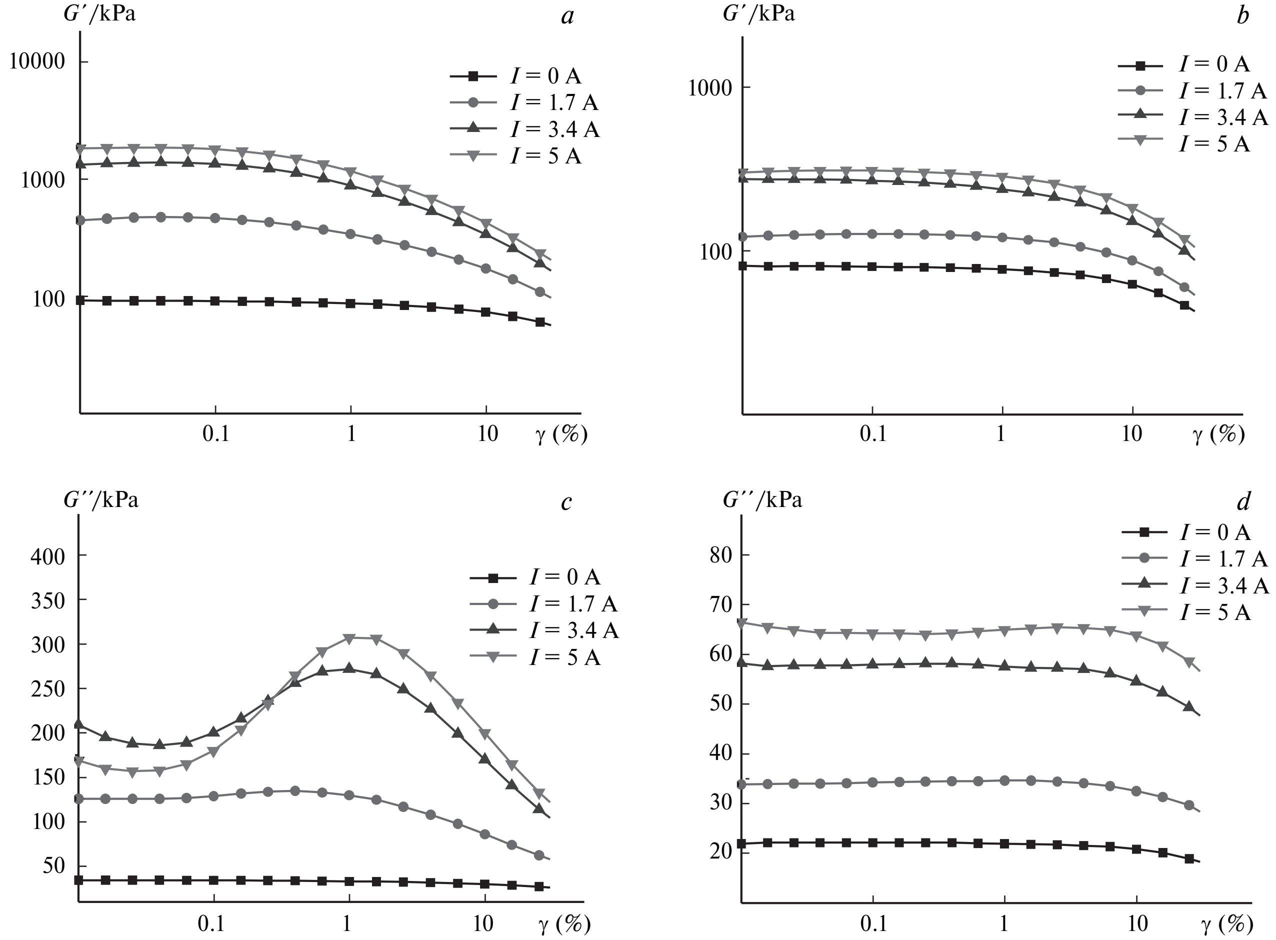


Fig. 5. Dependences of the storage modulus (*a*, *b*) and loss modulus (*c*, *d*) on the strain amplitude (γ) of the samples of magnetic elastomers based on CI particles unmodified (*a*, *c*) and modified by the oligomer (*b*, *d*).

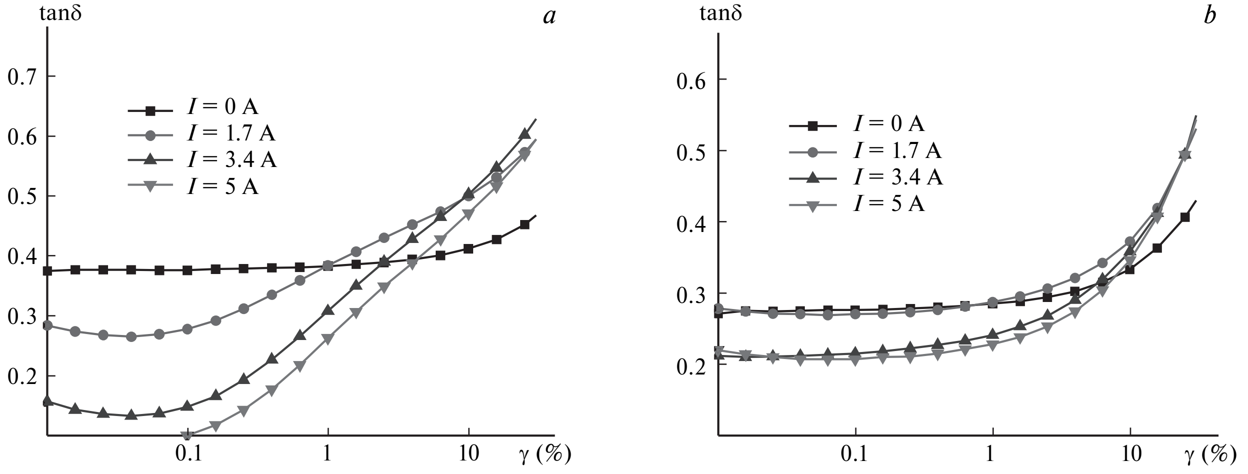


Fig. 6. Dependences of the loss factor (tanδ) on the strain amplitude (γ) for magnetic elastomers based on iron unmodified (*a*) and modified by the oligomer (*b*).

$$\Delta G' = (G'(I_{\max}) - G'(I = 0))/(G'(I = 0)), \qquad (1)$$

where $G'(I_{\max})$ is the value of storage modulus at the maximum current strength of the electromagnet, and $G'(I = 0)$ is the value of storage modulus in the absence of magnetic field.

The magnetorheological effect for the matrix with the unmodified CI particles $\Delta G'$ is 20 kPa at a shear oscillation frequency of 10 rad s^{-1}, and the magnetic response of the composite based on the modified CI particles is somewhat lower ($\Delta G' \approx 4$ kPa). It is most likely that the magnetorheological effect of the sample with the modified CI particles decreases because of a decrease in the effective magnetic permeability of the particles. The magnetic elastomers obtained[11] from the CI particles covered by fluorinated polystyrene in the silicone matrix manifested a similar tendency.

It follows from the amplitude dependences of the components of the dynamic modulus presented in Fig. 5 that the material obtained is characterized by the pronounced Payne effect: a decrease in the storage modulus with an increase in the strain amplitude and the maximum of the loss modulus in the region of strains corresponding to a decrease in G'. It is known that the Payne effect is a specific feature of the behavior of the stres-strain dependence of filled rubbers.[25] This effect is attributed, in particular, to the decomposition of aggregates of particles under strong strains. In the magnetic elastomers, the Payne effect is much more pronounced under the applied magnetic field when the magnetic particles begin to interact strongly with each other.[22,24,26]

The behavior of the magnetic elastomers based on the CI particles unmodified and modified by oligosiloxane is qualitatively the same, but quantitative distinctions are observed. Indeed, it is seen from Fig. 5 than in the obtained magnetic elastomers the Payne effect is enhanced in the applied magnetic field. However, its value, which can be estimated by Eq. (2), differs for two samples.

$$\Delta G' = (G'(\gamma_{\max}) - G'(\gamma_0))/G'(\gamma_0), \qquad (2)$$

where $G'(\gamma_{\max})$ is the value of storage modulus at the maximum strain amplitude, and $G'(\gamma_0)$ is the value of storage modulus in the region of linear viscoelasticity.

This value is 23 and 8 kPa in the magnetic field of the maximum intensity for the matrices with the untreated and treated CI particles, respectively. It should be mentioned that the region of linear viscoelasticity in which the storage modulus is approximately constant is broader for the sample containing CI particles with the modified surface. It can be assumed that the polymer—polymer interaction is stronger than the iron—polymer interaction and, hence, the treated CI particles are better compatible with the polymeric matrix. According to this, the Payne effect in the magnetic field is less pronounced and the region of linear behavior becomes broader. Thus, the modified CI can be used for the preparation of more resistant to strain devices based on magnetic elastomers. Similar results were obtained by the study of the magnetorheological elastomers based in the CI particles with the PMMA shell.[9]Substantial quantitative differences are observed for the dependences of the loss factor $\tan\delta$ for the modified and unmodified samples (determined as the ratio of the loss modulus to the storage modulus $\tan\delta = G''/G'$) on the magnetic field intensity. Figure 6 shows that the value of $\tan(\delta)$ in the absence of magnetic field is lower for the sample with the modified CI particles, whereas its value in the maximum field is higher. Thus, in spite of significant changes of the moduli themselves with increasing magnetic field, their ratio changes weakly from 0.28 to 0.21. The variation of $\tan(\delta)$ for the samples with the unmodified CI particles is broader: from 0.38 without magnetic field to 0.09 in the maximum field.

The water contact angles were studied for the magnetic elastomer filled with the CI particles modified by PDMS-S-U-4 without magnetic field application and in the magnetic field. The angle was 120±2° on the average during the whole measurement time without field application, and the contact angle was 116±2° in the field. Thus, the contact angle values almost coincide with allowance for the measurement inaccuracy of the instrument.

To conclude, the treatment of the particles with PDMS oligomers containing carboxyl groups is an efficient method for the modification of magnetic particles to impart them a high affinity to the polymeric matrix. The system is sensitive to the modification procedure and to the choice of the modifying agent. The further optimization of these factors will prevent particle aggregation. Thus, the proposed approach is promising for the preparation of magnetic particles with the "core—shell" structure and with controlled properties.

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