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**NEW APPROACHES TO STABILIZE AQUEOUS SOOT SUSPENSIONS
IN THE FIELD OF ULTRASOUND**

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Abstract

In this work, we investigated the possibility of stabilization of aqueous low- and highly concentrated dispersed soot systems by diphilic high-molecular compounds. Based on the results, it can be concluded that the presence of hydrophobic fragments in the macromolecule chain plays a very significant role and positively affects the results of stabilization of aqueous soot dispersions. Studies have shown that the use of a stabilizer in combination with ultrasonic treatment of dispersed systems makes it possible to obtain stable homogeneous finely dispersed pastes. It was noted that a strong increase in the time and intensity of ultrasound processing of the system does not lead to a significant improvement in the results.

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Introduction

Soot (carbon black) is very widely used in the rubber and synthetic rubber industries¹. The range of fields of its application is very wide: from use as a pigment for coloring various products to filling rubber products². While making soot-filled rubbers, 10-25% aqueous suspension of soot is preliminarily prepared³. Dispersion is carried out in an apparatus with a stirrer⁴. However, soot has a hydrophobic surface and is not wetted by water⁵. Therefore, it is interesting to investigate the possibility of modifying the surface of it in order to obtain aqueous dispersions and pastes with high aggregative and sedimentation stability⁶.

In this work, we investigated the possibility of stabilization of aqueous low- and highly concentrated dispersed soot systems by diphilic high-molecular compounds⁷. The reason for choosing such substances as a surfactant is the need to create a diphilic adsorption layer on the surface of soot particles that impedes the processes of aggregation and coagulation of particles due to the adsorption-solvate and structural-mechanical stability factor of colloidal systems⁸. However, in a number of studies, as well as in the course of preliminary experiments, it was shown that a high-intensity mechanical effect on a dispersed system, in particular, processing the system in an ultrasound field, is an effective method of activating surfactant adsorption on the surface of pigments⁹.

The dispersing effect of the ultrasonic field on colloidal systems is well known¹⁰. It is based on the phenomenon of cavitation, which occurs in a liquid as a result of a local

¹ A. A. Asratyan; S. A. Ambrozevich; O. S. Andrienko; N. A. Bulychev; A. G. Grigoryants; M. A. Kazaryan; S. M. Kazaryan; N. A. Lyabin; R. G. Mkhitarian; G. A. Tonoyan; I. N. Shiganov y V. I. Sachkov, "Comparative analysis of parameters of pulsed copper vapour laser and known types of technological lasers", Proceedings of SPIE Vol: 10614 num 02 (2018).

² N. A. Bulychev; M. A. Kazaryan; A. S. Averyushkin; M. N. Kirichenko; A. R. Zakharyan y A. A. Chernov, "Dynamic characteristics of electric discharge in liquid under ultrasonic cavitation", Proceedings of SPIE, Vol: 10614 num 14 (2018).

³ N. A. Bulychev; M. A. Kazaryan; E. S. Gridneva; E. N. Murav'ev; V. F. Solinov; K. K. Koshelev; O. K. Kosheleva; V. I. Sachkov y S. G. Chen, "Plasma discharge with bulk glow in the liquid phase exposed to ultrasound", Bulletin of the Lebedev Physical Institute, Vol: 39 num 7 (2012): 214-220.

⁴ V. F. Formalev; É. M. Kartashov y S. A. Kolesnik, "Simulation of Nonequilibrium Heat Transfer in an Anisotropic Semispace Under the Action of a Point Heat Source", Journal of Engineering Physics and Thermophysics, Vol: 92 num 6 (2019): 1537-1547.

⁵ I. N. Borovik; E. A. Strokach y N. S. Severina, "Influence of the turbulent Prandtl number on numerical simulation reaction flow", AIP Conference Proceedings, Vol: 2181 num 1 (2019): 20-29.

⁶ V. Y. Gidasov y N. S. Severina, "Modeling of detonation of metal-gas combustible mixtures in high-speed flow behind a shock wave", High Temperature, Vol: 57 num 4 (2019): 514-524.

⁷ M. N. Kirichenko; N. A. Bulychev; L. L. Chaikov; M. A. Kazaryan y A. V. Masalov, "Effect of iron oxide nanoparticles on the blood coagulation according to light scattering data", Proceedings of SPIE, Vol: 10614 num 2C (2018).

⁸ V. Y. Gidasov; V. K. Golubev y N. S. Severina, "A software package for simulation of unsteady Flows of the reacting gas in the channel. Bulletin of the South Ural State University, Series: Mathematical Modelling", Programming and Computer Software, Vol: 9 num 3 (2016): 94-104.

⁹ N. S. Severina, "Software complex for solving the different tasks of physical gas dynamics", Periodico Tche Quimica, Vol:16 num 32 (2019): 424-436.

¹⁰ L. N. Rabinskiy y S. A. Sitnikov, "Development of technologies for obtaining composite material based on silicone binder for its further use in space electric rocket engines", Periodico Tche Quimica, Vol: 15 num 1 (2018): 390-395.

decrease in pressure during the passage of an acoustic wave of high intensity¹¹. The cavitation bubbles formed, moving to a region with a higher pressure, collapse, emitting a shock wave¹². However, in order to prevent subsequent coagulation of particles, the effect of ultrasound should be fixed by the creation of protective layers of the stabilizer¹³.

Stabilization of aqueous soot suspensions using water-soluble copolymers

In order to select optimal surfactants for stabilization of aqueous soot suspensions, colloidal stability studies of 0.1% and 12% aqueous soot suspensions in the presence of high molecular weight surfactants were carried out¹⁴. As a result, optimal stabilizers for soot – water systems were selected, and the concentrations of these stabilizers were determined¹⁵.

As surfactants, industrially produced ionogenic (1-4) and nonionic (5) water-soluble stabilizers were used:

- 1) copolymer of polyacrylic ester and sodium polyacrylate (commercial name is Tegodispers 750 W);
- 2) copolymer of polyacrylic ester and ammonium polyacrylate (commercial name is Coatex P 90);
- 3) copolymer of polyacrylic acid and sodium polyacrylate (commercial name is Dispex N 40);
- 4) copolymer of polyacrylic acid and ammonium polyacrylate (commercial name is Dispex A 40);
- 5) ethylhydroxyethyl cellulose (nonionic, water soluble and environmentally friendly polymer).

¹¹ Yu. O. Kirilina; I. V. Bakeeva; N. A. Bulychev y V. P. Zubov, "Organic-inorganic hybrid hydrogels based on linear poly(N-vinylpyrrolidone) and products of hydrolytic polycondensation of tetramethoxysilane", Polymer Science Series B, Vol: 51 num 3-4 (2009): 135.

¹² N. Bulychev; W. Van Camp; B. Dervaux; Y. Kirilina; K. Dirnberger; T. Schauer; V. Zubov; F. E. Du Prez y C. D. Eisenbach, "Comparative Study of the Solid-Liquid Interface Behaviour of Amphiphilic Block and Block-like Copolymers", Macromolecular Chemistry and Physics, Vol: 210 (2009): 287-298.

¹³ N. A. Bulychev y M. A. Kazaryan, "Optical Properties of Zinc Oxide Nanoparticles Synthesized in Plasma Discharge in Liquid under Ultrasonic Cavitation", Proceedings of SPIE, Vol: 11322 (2019): 219.

¹⁴ A. S. Averyushkin; A. N. Baranov; N. A. Bulychev; M. A. Kazaryan; A. D. Kudryavtseva; M. A. Stokov; N. V. Tcherniega y K. I. Zemskov, "Stimulated low-frequency Raman scattering in aqueous suspension of nanoparticles", Proceedings of SPIE, Vol: 10614 num 0K (2018).

¹⁵ N. A. Bulychev; M. A. Kazaryan; L. S. Lepnev; A. S. Averyushkin; M. N. Kirichenko; A. R. Zakharyan y A. A. Chernov, "Luminescent properties of nanoparticles synthesized in electric discharge in liquid under ultrasonic cavitation", Proceedings of SPIE, Vol: 10614 num 13 (2018).

If there is no polymeric surfactants, soot particles are not wetted by water and float on its surface¹⁶. The introduction of polymer stabilizers without machining the dispersion practically does not allow obtaining stable dispersed systems¹⁷. The use of mechanical mixing or ultrasonic treatment in the absence of polymers also does not lead to the formation of homogeneous dispersed systems¹⁸. However, in the presence of polymers, the image changes qualitatively. With the combined effects of polymer stabilizers and ultrasonic treatment, it is possible to obtain highly stable dispersed systems¹⁹.

The stability of disperse systems was studied by sedimentation²⁰. As a criterion for assessing colloidal stability, the semi-sedimentation time of the solid phase was chosen²¹. Table 1 presents data on the effect of polymers on the colloidal stability of pigment suspensions²².

Stabilizer	Half-life of the solid phase, days
Tegodispers 750 W	90
Coatex P 90	10
Dispex N 40	1
Dispex A 40	0.5
Ethylhydroxyethyl cellulose	3

Table 1
Colloidal stability of aqueous soot dispersions stabilized
by high molecular weight surfactants

As we can see from the Table 1, the colloidal stability of the obtained dispersed systems substantially depends on the nature of the polymer²³. The strongest effect was obtained when using Tegodispers 750 W as a stabilizer²⁴.

¹⁶ V. F. Formalev; S. A. Kolesnik y B. A. Garibyan, "Mathematical modeling of heat transfer in anisotropic plate with internal sinks", Computational Mechanics and Modern Applied Software Systems (CMMASS'2019) AIP Conf. Proc., Vol: 2181 num 020003 (2019).

¹⁷ M. N. Kirichenko; L. L. Chaikov; I. S. Burkhanov; N. A. Bulychev y M. A. Kazaryan, "Interaction of aluminum oxide nanoparticles with human blood plasma thrombin (according to light scattering)", Proceedings of SPIE, Vol: 11322 num 1Y (2019).

¹⁸ V. N. Nikiforov; N. A. Bulychev y V. V. Rzhhevskii, "Elastic properties of HTSC ceramics", Bulletin of the Lebedev Physical Institute, Vol: 43 num 2 (2016): 74-79.

¹⁹ Yu. V. Ioni; S. V. Tkachev; N. A. Bulychev y S. P. Gubin, "Preparation of Finely Dispersed Nanographite", Inorganic Materials, Vol: 47 num 6, (2011): 597-602.

²⁰ K. V. Pushkin; S. D. Sevruk; N. S. Okorokova y A. A. Farmakovskaya, "The most efficient corrosion inhibitors for aluminum anode of electrochemical cell used as a controlled hydrogen generator", Periodico Tche Quimica, Vol: 15 num 1 (2018): 414-425.

²¹ V. Y. Gidasov; O. A. Moskalenko y N. S. Severina, "Numerical Study of the Influence of Water Droplets on the Structure of a Detonation Wave in a Hydrogen–Air Fuel Mixture", High Temperature, Vol: 56 num 5 (2018): 751-757.

²² A. V. Ivanov; V. N. Nikiforov; S. V. Shevchenko; V. Yu. Timoshenko; V. V. Pryadun; N. A. Bulychev; A. B. Bychenko y M. A. Kazaryan, "Properties of Metal Oxide Nanoparticles Prepared by Plasma Discharge in Water with Ultrasonic Cavitation", International Journal of Nanotechnology, Vol: 14 num 7/8 (2017): 618-626.

²³ V. A. Pogodin; L. N. Rabinskiy y S. A. Sitnikov, "3D Printing of Components for the Gas-Discharge Chamber of Electric Rocket Engines", Russian Engineering Research, Vol: 39 num 9 (2019): 797-799.

²⁴ I. S. Burkhanov; L. L. Chaikov; N. A. Bulychev; M. A. Kazaryan y V. I. Krasovskii, "Nanoscale metal oxide particles produced in the plasma discharge in the liquid phase upon exposure to

The dispersed system does not stratify for 3 months or more²⁵. The particle size of soot is about 1 µm. Polymers Dispex N 40 and Dispex A 40 show significantly worse results, nonionic polymer ethylhydroxyethyl cellulose and Coatex P 90 are in the middle of this interval²⁶.

These results are in good agreement with the data on the values of the contact angles obtained for soot samples treated with these high molecular weight surfactants (Table 2)²⁷.

Stabilizer	Wetting angle, deg.
Without polymer	124
Tegodispers 750 W	0
Coatex P 90	110
Dispex N 40	116
Dispex A 40	119
Ethylhydroxyethyl cellulose	106

Table 2
Values of water contact wetting angles of soot surfaces treated with high molecular weight surfactants

The table shows that in the absence of polymers deposited on the soot surface, the value of the contact angle is quite large and clearly indicates the hydrophobicity of the pigment surface²⁸. The difference in the wetting angle of the clean soot surface from the values obtained in the presence of polymers indicates the formation of surface layers during ultrasonic treatment²⁹. It also follows from the table that only Tegodispers 750 W, adsorbing on the surface of soot particles, can effectively wet it with water³⁰.

Analyzing the Tables 1 and 2, we can conclude that only polymer Tegodispers 750 W (copolymer of polyacrylic ester and sodium polyacrylate) shows good results³¹. This can

ultrasonic cavitation. 2. Sizes and stability. Dynamic light scattering study”, Bulletin of the Lebedev Physical Institute, Vol:41 num 10 (2014): 297-304.

²⁵ V. F. Formalev y S. A. Kolesnik, “Heat Transfer in a Half-Space with Transversal Anisotropy Under the Action of a Lumped Heat Source”, Journal of Engineering Physics and Thermophysics, Vol: 92 num 1 (2019): 52-59.

²⁶ V. Y. Gidasov y N. S. Severina, “Numerical Simulation of the Detonation of a Propane-Air Mixture, Taking Irreversible Chemical Reactions into Account”, High Temperature, Vol 55 num 5 (2017): 777–781.

²⁷ M. N. Kirichenko; L. L. Chaikov; I. S. Burkhanov; N. A. Bulychev y M. A. Kazaryan, “Effect of the pH of iron oxide nanoparticles solution on the rate of fibrin gel formation (according to light scattering data)”, Proceedings of SPIE, Vol: 11322 num 1E (2019).

²⁸ A. V. Rudnev; N. G. Vanifatova; T. G. Dzherayan; E. V. Lazareva y N. A. Bulychev, “Study of stability and dispersion composition of calcium hydroxyapatite in aqueous suspensions by capillary zone electrophoresis”, Russian Journal of Analytical Chemistry, Vol: 68 num 8 (2013): 700.

²⁹ M. N. Kirichenko; N. A. Bulychev; L. L. Chaikov; M. A. Kazaryan y A. V. Masalov, “Effect of iron oxide nanoparticles on the concentration-versus-sizes relation of proteins in the blood plasma and serum, and in model solutions”, Proceedings of SPIE, Vol: 10614 num OM (2018).

³⁰ V. F. Formalev y S. A. Kolesnik, “On Thermal Solitons during Wave Heat Transfer in Restricted Areas”, High Temperature, Vol: 57 num 4 (2019): 498-502.

³¹ V. V. Nigmatzyanov; V. A. Pogodin; L. N. Rabinskiy y S. A. Sitnikov, “The polymer-ceramic material for the manufacture of gas discharge chamber for the electric rocket engine”, Periodico Tche Quimica, Vol: 16 num 33 (2019): 801-808.

be explained by the presence of hydrophobic fragments of esters of acrylic acid in it, which have an affinity for hydrophobic surfaces and are well adsorbed on the soot surface, as well as a strongly dissociated carboxyl group, which actively interacts with the aqueous phase³².

Comparing the effect of Tegodispers 750 W and Coatex P 90, which have the same structure and differ only in the nature of the positive ion (Na^+ and NH_4^+), it can be concluded that the nature of the positively charged ion affects the effectiveness of the stabilizing effect of surfactants³³. The difference in the values of the contact angles of wetting also indicates a significant role of the counterion in the modification of the surface of organic pigments³⁴.

The presence of carboxyl groups in the H-form in the macromolecule structure negatively affects the stabilization of the system by Dispex N 40 and Dispex A 40 polymers³⁵. The nonionic stabilizer ethylhydroxyethyl cellulose is in the intermediate position³⁶. Based on the above results, it can be concluded that the presence of hydrophobic fragments in the macromolecule chain plays a very significant role and positively affects the results of stabilization of aqueous soot dispersions³⁷. Next, we conducted studies stabilize 12% of aqueous dispersed soot systems (pastes), which can be used to fill various products³⁸. The experiments showed that the high molecular weight stabilizers used in the previous stage, even when using ultrasonic treatment as a mechanical effect, do not give a positive effect³⁹. The exception is only Tegodispers 750 W, when using which it was possible to obtain relatively stable dispersed systems, but its concentration in this case turned out to be quite high and reached 10%⁴⁰. In this regard,

³² A. S. Averyushkin; A. N. Baranov; N. A. Bulychev; A. I. Erokhin y M. A. Kazaryan, "Ag nanoparticles suspensions for stimulated Rayleigh backscattering of single frequency 0.5 micron pulsed laser radiation", Proceedings of SPIE, Vol: 10614 num 1L (2018).

³³ N. A. Bulychev; A. I. Erokhin y M. A. Kazaryan, "A Comparative Study of Anti-Stokes Shift under Stimulated Rayleigh-Mie Scattering in Suspensions of Ag Nanoparticles Obtained in Plasma Discharge in Liquid under Ultrasonic Cavitation", Proceedings of SPIE, Vol: 11322 num 2G (2019).

³⁴ N. A. Bulychev; E. L. Kuznetsova; V. V. Bodryshev y L. N. Rabinskiy, "Nanotechnological Aspects of Temperature-Dependent Decomposition of Polymer Solutions", Nanoscience and Technology: An International Journal, Vol: 9 num 2 (2018): 91-97.

³⁵ V. F. Formalev; S. A. Kolesnik y B. A. Garibyan, "Heat transfer with absorption in anisotropic thermal Protection of high-temperature products. Herald of the Bauman Moscow State Technical University", Series Natural Sciences, Vol: 86 num 5 (2019): 35-49.

³⁶ V. F. Formalev; S. A. Kolesnik; E. L. Kuznetsova y L. N. Rabinskiy, "Origination and propagation of temperature solitons with wave heat transfer in the bounded area during additive technological processes", Periodico Tche Quimica, Vol: 16 num 33 (2019): 505-515.

³⁷ V. V. Bodryshev; A. V. Babaytsev y L. N. Rabinskiy, "Investigation of Processes of Deformation of Plastic Materials with the Help of Digital Image Processing", Periodico Tche Quimica, Vol: 16 num 33 (2019): 865-876.

³⁸ N. A. Bulychev; M. I. Danilkin; N. Yu. Vereshchagina y M. A. Kazaryan, "Luminescent Properties of ZnO Nanoparticles Doped by W Obtained in Plasma Discharge in Liquid under Ultrasonic Cavitation", Proceedings of SPIE, Vol: 11322 num 1S (2019).

³⁹ N. A. Bulychev; M. A. Kazaryan; A. D. Kudryavtseva; M. V. Kuznetsova; T. F. Limonova; N. V. Tcherniega y K. I. Zemskov, "Anti-Stokes luminescence in nanoscale systems", Proceedings of SPIE, Vol: 10614 num 0N (2018).

⁴⁰ Y. A. Dyakov; M. A. Kazaryan; M. G. Golubkov; D. P. Gubanova; N. A. Bulychev y S. M. Kazaryan, "Laser-induced dissociation processes of protonated glucose: dehydration reactions vs cross-ring dissociation", Proceedings of SPIE, Vol: 10614 num 17 (2018).

the industrially produced anionic stabilizer NF was studied. The use of this stabilizer in combination with ultrasonic treatment of dispersed systems allows obtaining stable homogeneous finely dispersed pastes⁴¹. The colloidal stability of the obtained disperse systems was studied by the sedimentation method; in addition, the average particle size of soot was evaluated as one of the main criteria for assessing the quality of a disperse system⁴².

Three formulations were chosen for the preparation of the studied pastes: using NF stabilizer, using Tegodispers 750 W polymer stabilizer and using them together. The results of the study of colloidal stability and analysis of particle sizes of soot dispersions are shown in the Table 3.

Stabilizer	Half-life of the solid phase, days	Average particle size, μm
Tegodispers 750 W (10%)	10	5 – 6
NF (5% mass)	30	1 – 2
NF (3% mass) + Tegodispers 750 W (5%)	30	1 – 2

Table 3

Colloidal stability and average particle size of aqueous soot dispersions stabilized by various surfactants

From the data presented in the table, it follows that the use of NF stabilizer and mixed formulation allows obtaining sufficiently stable finely dispersed pastes. It was noted that an increase in the time and intensity of ultrasound processing of the system does not lead to a significant improvement in the results.

It was also interesting to find out how the concentration of NF stabilizer affects the stability and particle size of soot pastes and whether it is possible to establish a certain optimal concentration that will provide satisfactory values of these parameters. Therefore, experiments were conducted with different concentrations of NF. The results are presented in the Table 4.

Concentration of NF stabilizer, %	Half-life of the solid phase, days	Average particle size, μm
1	2	6 – 8
2	10	5 – 6
3	20	3 – 4
5	30	1 – 2
10	35	1 – 1.5

Table 4

Colloidal stability and average particle size of aqueous soot dispersions, depending on the concentration of NF stabilizer

⁴¹ N. A. Bulychev y M. A. Kazaryan, "Application of Optical Spectroscopy for Study of Hydrogen Synthesis in Plasma Discharge in Liquid under Ultrasonic Cavitation", Proceedings of SPIE, Vol: 11322 num 1A (2019).

⁴² N. A. Bulychev; M. A. Kazaryan; A. Ethiraj y L. L. Chaikov, "Plasma Discharge in Liquid Phase Media under Ultrasonic Cavitation as a Technique for Synthesizing Gaseous Hydrogen", Bulletin of the Lebedev Physical Institute, Vol: 45 num 9 (2018): 263-266.

The table shows that the average particle size of the dispersions and their stability depend on the concentration of the stabilizer, therefore, in each case, the required concentration can be determined depending on the requirements for the resulting dispersed system.

Conclusions

Thus, as a result of the studies, the high efficiency of the proposed method for modifying the surface of a hydrophobic organic pigment, carbon black, consisting in the combined action of polymer or low molecular weight surfactants and mechanical activation of the soot surface in the ultrasound field is shown. A number of industrially produced polymer surfactants have been selected, which have an optimal structure and composition, provide high stability for low concentrated aqueous dispersed soot systems, and also show the possibility of stabilizing concentrated suspensions with a low molecular weight anionic stabilizer NF.

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