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**MODIFICATION OF THE SURFACE OF CARBON BLACK WITH VINYL ETHER COPOLYMERS
UNDER ULTRASONIC TREATMENT**

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Abstract

In this work, we investigated the possibility of stabilizing aqueous dispersed carbon systems with vinyl ether copolymers. It was shown that the presence of hydrophobic fragments in the macromolecule chain plays a significant role and positively affects the results of stabilization of aqueous dispersions of carbon particles. Studies have shown that the use of a stabilizer in combination with ultrasonic treatment of dispersed systems allows obtaining stable homogeneous highly dispersed suspensions. 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.

Modification of the surface of carbon black with vinyl ether copolymers under ultrasonic treatment pág. 554

Keywords

Carbon – Stability – Particles – Copolymers - Ultrasound

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Introduction

Obtaining aqueous dispersions of soot of high stability is of undoubted scientific and practical interest¹. Recently, scientific studies have been carried out aimed at increasing the stability and dispersion of suspensions of various pigments using high and low molecular weight surfactants in combination with mechanical activation methods; in particular, processing suspensions in an ultrasound field². In a number of studies, there were results on the preparation of stable aqueous dispersed systems of nanoparticles using high molecular weight compounds³.

The results show the possibility of using polymer modifiers of soot surface to obtain stable aqueous disperse systems⁴. However, the use of industrially produced polymers for these studies, such as Tegodispers 750 W, Coatex P 90, ethylhydroxyethyl cellulose, Dispex N 40, Dispex A 40, etc., has certain difficulties⁵. Commercially available polymers have a very wide molar mass distribution, are not always uniform in their composition and molecular mass characteristics and their composition and structure may partially vary⁶. Therefore, despite a number of indisputable advantages associated with their use, the study of the mechanism of the stabilizing effect on the example of such polymers is unlikely to provide comprehensive and correct information⁷.

Stabilization of dispersed carbon systems by diphilic copolymers

To obtain more systematic information on the relation between the structure of a diphilic polymer and its surface-active properties, a series of new, specially synthesized copolymers with a given molecular structure and regularly varying parameters was used⁸.

¹ 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).

² 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.

³ 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).

⁴ V. Y. Gidaspov; 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.

⁵ 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.

⁶ N. S. Severina, "Software complex for solving the different tasks of physical gas dynamics", Periodico Tche Quimica, Vol:16 num 32 (2019): 424-436.

⁷ 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.

⁸ K. V. Pushkin; S. D. Sevruck; 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.

Four types of diphyll copolymers of methyl vinyl ether (PMVE) as the hydrophilic fragment and isobutyl vinyl (PIBVE) or octadecyl vinyl ether (PODVE) as the hydrophobic fragment were investigated⁹. These copolymers were synthesized by the method of continuous cationic polymerization on new catalytic systems¹⁰. The size of blocks, their location, molecular weight characteristics of the polymers and other parameters systematically varied over a fairly wide range¹¹. A series of statistical copolymers was also used for comparison¹². For all copolymers used, the number average molecular weight was in the range from 5 000 to 8 000, and the degree of polydispersity was from 1.05 to 1.3)¹³. The following types of polymers were used (number in the copolymer formula shows the degree of polymerization of this monomer — number of units in the block)¹⁴:

- 1) Homopolymers:
 - PMVE 95;
 - PIBVE 22.
- 2) Statistical copolymers:
 - PMVE 30 – st – PIBVE 70;
 - PMVE 50 – st – PIBVE 50;
 - PMVE 80 – st – PIBVE 20.
- 3) Two-block copolymers:
 - PMVE 36 – PIBVE 54;
 - PMVE 43 – PIBVE 10;
 - PMVE 55 – PIBVE 10;
 - PMVE 83 – PIBVE 8.
- 4) Three-block copolymers:
 - PMVE 50 – PIBVE 20 – PMVE 50;
 - PMVE 62 – PIBVE 10 – PMVE 62;
 - PIBVE 26 – PMVE 40 – PIBVE 26;
 - PIBVE 22 – PMVE 75 – PIBVE 22;
 - PODVE 4 – PMVE 168 – PODVE 4;
 - PMVE 48 – PODVE 11 – PMVE 48;
 - PMVE 28 – PODVE 10 – PMVE 28.

⁹ 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.

¹⁰ 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.

¹¹ 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.

¹² 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).

¹³ 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.

¹⁴ 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 ultrasonic cavitation. 2. Sizes and stability. Dynamic light scattering study", Bulletin of the Lebedev Physical Institute, Vol:41 num 10 (2014): 297-304.

Using these copolymers as surfactants, colloidal stability studies of 0.1% aqueous dispersed soot systems were carried out, and the contact angles of water soot surface were measured to determine the degree of surface modification with these polymers¹⁵.

All copolymers used are soluble in ethyl acetate and, with the exception of the most hydrophilic PMVE 95 and PMVE 83 – PIBVE 8, are insoluble in water¹⁶. Therefore, to comply with the identical conditions for the preparation of aqueous disperse systems, the copolymers were previously dissolved in ethyl acetate (polymer concentration is 5%), and then introduced into the aqueous phase together with the pigment (phase ratio = 1:10)¹⁷.

Special experiments have shown that the presence of ethyl acetate in the system does not affect the stabilizing ability of polymers, but allows more efficient delivery of polymer stabilizers to the surface of organic pigments through the aqueous phase¹⁸.

Thus, after the ultrasonic treatment of the dispersions, direct type microemulsion was formed, and the pigment particles were in droplets of the microemulsion¹⁹. All polymers used at this stage in combination with the use of ultrasonic treatment as a dispersing effect make it possible to obtain disperse systems²⁰. At the same time, due to the low molecular weight, there is no danger of the destruction of these polymers in the ultrasonic field²¹.

The results of the study of colloidal stability by sedimentation are presented in the Table 1²². As a criterion for assessing the colloidal stability of dispersed systems, the half-life of the solid phase was chosen²³.

¹⁵ 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.

¹⁶ 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.

¹⁷ 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).

¹⁸ 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.

¹⁹ 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).

²⁰ 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.

²¹ 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.

²² 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.

Stabilizer	Half-life of the solid phase, days
PMVE 95	0,5
PIBVE 22	1,5
PMVE 30 – st – PIBVE 70	0,2
PMVE 50 – st – PIBVE 50	0,3
PMVE 80 – st – PIBVE 20	0,4
PMVE 36 – PIBVE 54	4
PMVE 43 – PIBVE 10	15
PMVE 55 – PIBVE 10	70
PMVE 83 – PIBVE 8	30
PMVE 50 – PIBVE 20 – PMVE 50	8
PMVE 62 – PIBVE 10 – PMVE 62	15
PIBVE 26 – PMVE 40 – PIBVE 26	30
PIBVE 22 – PMVE 75 – PIBVE 22	90
PODVE 4 – PMVE 168 – PODVE 4	90
PMVE 48 – PODVE 11 – PMVE 48	7
PMVE 28 – PODVE 10 – PMVE 28	1

Table 1
Colloidal stability of aqueous soot dispersions stabilized with copolymers of polymethyl vinyl ether

We can see in the Table 1 that the homopolymers PMVE 95 and PIBVE 22 do not allow obtaining stable aqueous dispersed soot systems²⁴. Within 10-30 hours, rapid sedimentation and destruction of the microemulsion is observed, after 2 days complete subsidence of pigment particles is observed²⁵. The small difference between the effects observed for PMVE 95 and PIBVE 22 can be explained by the fact that PMVE 95, being a water-soluble polymer, is poorly fixed on the hydrophobic surface of soot and, unlike the hydrophobic PIBVE 22, passes into the aqueous phase, thereby promoting particle coagulation²⁶. For two-block copolymers, the colloidal stability of aqueous dispersed soot systems depends on the hydrophilic-hydrophobic balance of the polymer²⁷. The dependence of the stability of a disperse system on the structure of the stabilizer can be represented as the dependence of half-life of the solid phase on the ratio (in the number of units) of the hydrophilic and hydrophobic parts of the macromolecule²⁸. From the analysis of the data it follows that from the point of view of colloidal stability of aqueous

²³ 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).

²⁴ 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).

²⁵ 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).

²⁶ 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.

²⁷ 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).

²⁸ 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.

dispersed soot systems, the optimal structure of the stabilizer is a structure with ratio of hydrophilic and hydrophobic blocks of order of 5:1 - 6:1²⁹. These data are consistent with data on the contact angles of soot surface treated with PMVE and PIBVE copolymers (Table 2)³⁰. Comparing them, we can conclude that in the case of the use of PMVE 55 - PIBVE 10 and PMVE 83 - PIBVE 8 polymers as surfactants, the best colloidal stability of dispersions corresponds to lower contact angles of contact, which indicates the formation of surface layers of these polymers with corresponding properties³¹.

Stabilizer	Wetting angle, deg.
Without polymer	124
PMVE 95	24
PIBVE 22	118
PMVE 36 – PIBVE 54	112
PMVE 43 – PIBVE 10	91
PMVE 55 – PIBVE 10	83
PMVE 83 – PIBVE 8	27
PMVE 50 – PIBVE 20 – PMVE 50	121
PMVE 62 – PIBVE 10 – PMVE 62	46
PIBVE 26 – PMVE 40 – PIBVE 26	118
PIBVE 22 – PMVE 75 – PIBVE 22	87

Table 2

Values of the contact angles of water wetting of soot surface treated with methyl vinyl ether copolymers

Analyzing the data on the colloidal stability of dispersed soot systems obtained using three-block copolymers, we can conclude that the order of the arrangement of hydrophilic and hydrophobic blocks in a macromolecule plays a key role³². Obviously, only when the hydrophilic PMVE block (even if it is not very big) is located in the center of the macromolecule, and the hydrophobic PIBVE blocks are located at the edges, this polymer has a good stabilizing effect on soot dispersions³³.

At the same time, the presence of a hydrophobic PIBVE block in the center of a macromolecule, even not very long, does not allow obtaining stable disperse systems³⁴. This effect is especially pronounced on the example of a very hydrophilic composition

²⁹ 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 Tchc Quimica*, Vol: 16 num 33 (2019): 505-515.

³⁰ 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).

³¹ 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).

³² 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).

³³ 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.

³⁴ 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.

PMVE 62 - PIBVE 10 - PMVE 62 copolymer, which, having a fairly pronounced affinity in the aqueous phase (contact angle is 46 deg.)³⁵. Nevertheless does not exhibit surface-active properties in investigated systems³⁶. In both cases, an insignificant role of the extreme blocks and their length should be noted³⁷. This is in good agreement with the results obtained for the three-block copolymers PMVE and PODVE, as it was mentioned above³⁸.

Assumptions about the method of adsorption of polymer stabilizers on the surface of organic pigments can be expressed as follows³⁹. Hydrophobic PIBVE blocks (PODVE) are adsorbed on the surface of the organic pigment due to hydrophobic interaction, and the associated hydrophilic PMVE blocks are turned into the aqueous phase⁴⁰.

In this case, the central hydrophilic block “covers” the hydrophobic blocks lying on the surface of the pigment, thereby providing a two-layer diphilic structure⁴¹. In the case of a central hydrophobic block, the hydrophilic edges freely extend into the aqueous phase without covering the central block, which in this case is in contact with water, which is energetically disadvantageous for the system⁴².

Conclusions

Thus, the results of the experiments show the possibility of stabilization of aqueous dispersed soot systems by block copolymers of methyl vinyl ether with simultaneous ultrasound exposure. It was also established that the surface-active properties of the studied copolymers strongly depend on their composition and structure. The optimal composition for two-block copolymers and the optimal structure for three-block copolymers were found; number of assumptions about the mechanism of the stabilizing effect of the studied block copolymers was made.

³⁵ 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).

³⁶ 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).

³⁷ 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.

³⁸ 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.

³⁹ 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.

⁴⁰ 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.

⁴¹ I. N. Borovik; E. A. Stokach 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.

⁴² 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).

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