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**THE EFFECT OF SURFACE MECHANICAL ACTIVATION OF INORGANIC PIGMENTS
ON THE STABILITY OF THEIR AQUEOUS DISPERSIONS IN THE PRESENCE OF ETHYL
HYDROXYETHYL CELLULOSE**

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Abstract

The stability of aqueous dispersions of inorganic pigments TiO_2 and Fe_2O_3 in the presence of a non-toxic water-soluble polymer (ethylhydroxyethyl cellulose) with two methods of mechanically activating the surface of the pigments processing them in a disintegrator and in ultrasound field was studied. If there is no polymer stabilizer, intense exposure to aqueous dispersions of pigments leads to their rapid coagulation. If there is ethylhydroxyethyl cellulose, a small stabilizing effect is observed, which is significantly enhanced when processing dispersions in a disintegrator and in an ultrasonic field. Moreover, the stability of pigment dispersions depends not very much on the method of mechanically activating their surface, but on the duration of intense exposure.

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Introduction

In recent years, there is great scientific and practical interest of environmentally friendly aqueous dispersions of pigments widely used in the paint and varnish and printing industries¹. An important problem for dispersed systems is the problem of their stability². Surfactants are usually used to stabilize dispersions: with low molecular weight and polymeric ones³.

A number of studies have shown that some water-soluble polymers (such as polyvinyl alcohol, polyvinylpyrrolidone, polyvinylcaprolactam, cellulose derivatives) can be used as stabilizers for aqueous disperse systems⁴. In this case, the polymers are reversibly and irreversibly adsorbed on the surface of solid particles, forming protective adsorption-solvate layers⁵. This method of stabilization of disperse systems is commonly called adsorption-chemical modification⁶.

Recently, works that show that, to obtain highly dispersed stable systems, it is effective to use mechanochemical modification along with the adsorption-chemical modification of the surface of the solid phase (mechanochemical modification) have appeared⁷. In a number of studies, the method of mechanochemical modification (mechanical action during processing in various types of mills) was used to obtain dispersions of iron, chromium and lead oxides in organic solvents in the presence of polymers⁸. The authors conclude that an increase in the rate of adsorption of the polymer from the solvent on the surface of metal oxides during mechanical action due to structural changes in the surface layer of the solid phase⁹.

¹ 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.

² 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.

³ 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).

⁴ 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.

⁵ 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.

⁶ 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.

⁷ 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.

⁸ 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).

⁹ 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.

It was previously shown that treatment in an ultrasonic field of aqueous dispersions of titanium dioxide in the presence of water-soluble polymers leads to a significant increase in their stability¹⁰. It is assumed that during the process of mechanical activation in the presence of a polymer stabilizer, two processes simultaneously occur: disaggregation of pigment agglomerates and the formation of protective adsorption-solvate layers of the polymer on the “freshly formed”, and therefore “active” surface of the solid phase¹¹. The result is highly dispersed and stable systems¹².

However, these assumptions have not yet been experimentally confirmed, and the mechanism of mechanical activation processes remains unclear¹³.

In this work, there was set the task to obtain highly dispersed stable aqueous pigment systems TiO₂ and Fe₂O₃ in the presence of polymer stabilizer ethylhydroxyethyl cellulose as a result of mechanical action on them in two ways: by processing them in an ultrasonic field and in a disintegrator¹⁴.

Experimental part

Objects of study

Pigment Fe₂O₃ of Bayferrox company; pigment surface is treated with aluminum and silicon oxides¹⁵.

Pigment TiO₂ RN-56 of Kronos company; pigment surface is treated with oxides of aluminum, silicon and zirconium to reduce photosensitivity¹⁶.

A non-toxic water-soluble polymer, ethylhydroxyethyl cellulose, with a molecular weight of 60 000, was used as a stabilizer of aqueous pigment dispersions¹⁷.

¹⁰ 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.

¹¹ 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).

¹² 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).

¹³ 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.

¹⁴ 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).

¹⁵ 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.

¹⁶ 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.

Research methods

The sedimentation behavior of the pigments was investigated as follows: weighed portion of the pigment was mixed with an aqueous dispersion medium¹⁸. The dispersion was placed in a glass cylinder with divisions and then, over time, the lowering of the interface between the solid phase and the pure dispersion medium was monitored¹⁹. ml from sedimentation time (min., logarithmic coordinates)²⁰; The average particle size of the fillers and pigments was evaluated on the device Coulter – N – 4 – particle size analyzer²¹; Mechanical activation by ultrasonic treatment was carried out on an ultrasonic generator UZDN-2 with a frequency of 22 kHz and intensity 30W/cm², processing time ranged from 2 to 30 minutes²².

For mechanical activation, we also used a laboratory disintegrator manufactured by Disintegrator (Estonia) company; it has a passage type, containing five rows of discs with teeth that rotate towards each other at a speed of about 1000²³.

Results and discussion

Before proceeding to a direct discussion of the results, it is important to emphasize that in the studies there were used pigments, the surface of which was treated with oxides of aluminum, silicon and zirconium²⁴. This circumstance suggests the similarity of the properties of the studied pigment dispersions TiO₂ and Fe₂O₃²⁵.

¹⁷ 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.

¹⁸ 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).

¹⁹ 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); 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.

²⁰ 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).

²¹ 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).

²² 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).

²³ 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.

²⁴ 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.

²⁵ 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.

The results of a study of the effect of mechanical activation (ultrasonic treatment and processing in disintegrator (DI treatment) of aqueous pigment dispersions TiO_2 in the presence of ethylhydroxyethyl cellulose on their sedimentation stability, as well as on the average particle sizes of the corresponding dispersions, show that in the absence of polymer the treatment of dispersions in the ultrasound field and DI leads to their rapid coagulation, and the particle sizes are on average 1.0 - 1.1 μm ²⁶. This is apparently due to the formation on the surface TiO_2 during mechanical activation of a freshly formed surface with uncompensated charges²⁷. The system seeks to reduce surface energy, which leads to a sharp decrease in the stability of dispersions²⁸.

The presence of ethylhydroxyethyl cellulose in the system increases the stability of the dispersions, and the particle size decreases to 0.5 μm ²⁹. During mechanical activation (ultrasound or DI treatment) in the presence of ethylhydroxyethyl cellulose, the stability of dispersions increases significantly (approximately 100 times)³⁰. The particle size is thereby reduced to the original particle size of the titanium dioxide used³¹. A similar effect can be observed by analyzing the sedimentation stability of pigment dispersions Fe_2O_3 in water and in a solution of ethylhydroxyethyl cellulose, and data on particle sizes confirm a significant decrease in particle sizes of dispersions after their ultrasonic and di-processing³².

It should be noted that the stability of the systems obtained as a result of processing in an ultrasonic field is slightly higher than the stability of the same dispersions after their DI processing, but this difference is insignificant³³.

²⁶ 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; 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.

²⁷ 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).

²⁸ 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.

²⁹ 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.

³⁰ 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.

³¹ 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.

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; 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).

Thus, common to the studied aqueous dispersions of inorganic pigments TiO_2 and Fe_2O_3 is the disaggregation of pigment particles during mechanical activation, and in the presence of a polymer stabilizer, protective adsorption-solvate layers are formed, which apparently serves as the reason for the high activity of mechanical activation methods to increase the stability of disperse systems³⁴.

However, it should be noted that polymers can degrade during mechanical activation³⁵. This fact can significantly affect the effectiveness of the mechanochemical modification method³⁶.

In fact, in the study of sedimentation of pigment dispersions Fe_2O_3 in an aqueous solution of ethylhydroxyethyl cellulose for various durations of ultrasound treatment, it was found that the optimal processing time to obtain stable dispersions of this pigment is 2 minutes³⁷. An increase in the time of ultrasonic exposure leads to a significant decrease in the stability of systems. It can be assumed that ethylhydroxyethyl cellulose, being an insufficiently flexible polymer, undergoes mechanical destruction, which weakens its stabilizing effect³⁸.

Experimental results showing particle distribution of aqueous pigment dispersions Fe_2O_3 in the presence of ethylhydroxyethyl cellulose, in size before and after ultrasonic treatment, it is shown that, under ultrasonic treatment of dispersions, the distribution curve narrows significantly, which indicates that the dispersion Fe_2O_3 with a more uniform structure is obtained³⁹.

Conclusions

It was shown that ultrasonic treatment of aqueous dispersions of inorganic pigments leads to a significant narrowing of the particle size distribution curve, which indicates that dispersion with a more uniform structure is obtained. The spectral studies made it possible to conclude that the ethylhydroxyethyl cellulose is firmly sorbed on the surface of the pigments, and the intense effect leads to the activation of the adsorption of polymer molecules on the “freshly formed” surface of the pigments.

³⁴ 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; V. N. Nikiforov; N. A. Bulychev y V. V. Rzhetskii, “Elastic properties of HTSC ceramics”, *Bulletin of the Lebedev Physical Institute*, Vol: 43 num 2 (2016): 74-79.

³⁵ 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).

³⁶ 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.

³⁷ 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.

³⁸ N. S. Severina, “Software complex for solving the different tasks of physical gas dynamics”, *Periodico Tche Quimica*, Vol:16 num 32 (2019): 424-436.

³⁹ 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).

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