

The cover features a central image of a person standing on a dark rock in a snowy, mountainous landscape. The sky is dark with a vibrant green aurora borealis. The background is overlaid with several diagonal, semi-transparent bands in shades of teal and brown. The title 'REVISTA INCLUSIONES' is centered in large, white, bold, sans-serif capital letters.

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**ULTRASONIC TREATMENT IMPACT ON THE STABILITY OF AQUEOUS DISPERSIONS
OF INORGANIC AND ORGANIC PIGMENTS IN THE PRESENCE OF SURFACTANTS**

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Abstract

The colloidal stability of aqueous dispersions of the inorganic pigment Fe_2O_3 (red) and the organic pigment of copper phthalocyanine CuPc (blue), which were treated in an ultrasonic field in the presence of water-soluble polymers and surfactants, was studied. In the presence of ethyl hydroxyethyl cellulose (EHEC), ultrasonic treatment leads to a significant increase in the stability of aqueous Fe_2O_3 dispersions, while the sizes of the pigment particles decrease from $1 \mu\text{m}$ to $0.3 \mu\text{m}$, and the particle size distribution becomes narrower. For aqueous dispersions of CuPc, mechanochemical modification of the surface removes the wettability problem and obtain intensely colored stable aqueous dispersions of this pigment.

Keywords

Pigments – stability – particles – Mechanical activation – Ultrasound

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Introduction

Aqueous dispersions of the inorganic pigment Fe_2O_3 (red) and the organic pigment copper phthalocyanine CuPc (blue) are widely used in the printing and varnish and paint and coatings industries¹. Therefore, the stabilization of aqueous environmentally friendly dispersions of these pigments is an important task from a scientific and practical point of view².

It was previously shown that the stability of aqueous dispersions of inorganic and organic pigments can be increased in the presence of water-soluble polymers³. It was also found that the stability of aqueous dispersions of titanium dioxide can be significantly increased if they are subjected to ultrasonic treatment⁴.

The aim of this work was to obtain stable aqueous dispersions of the inorganic pigment Fe_2O_3 and the organic pigment CuPc in the presence of water-soluble polymers and surfactants under conditions of processing them in an ultrasonic field⁵.

Experimental part

EHEC with a molecular weight of 60 000 was used as a stabilizer of aqueous dispersions of Fe_2O_3 (red)⁶.

To stabilize aqueous dispersions of CuPc, polyvinyl alcohol (PVA) was used with an acetate group content of 2% and 17% and a molecular weight of 10 000, as well as an anionic dispersant NF⁷. To study the sedimentation behavior of the dispersion of the pigment Fe_2O_3 0.25% was mixed with 10 ml of water or an aqueous solution of EHEC⁸. The dispersion was subjected to pre-dispersion for 15 minutes using a laboratory stirrer (700 rpm), subjected to ultrasonic treatment and placed in glass cylinders with divisions and

¹ Yu. V. Ioni; S. V. Tkachev; N. A. Bulychev & S. P. Gubin, "Preparation of Finely Dispersed Nanographite. Inorganic Materials", Vol: 47 num 6 (2011): 597-602.

² 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 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.

³ N. Bulychev; W. Van Camp; B. Dervaux; Y. Kirilina; K. Dirnberger; T. Schauer; V. Zubov; F. E. Du Prez & C. D. Eisenbach, "Comparative Study of the Solid-Liquid Interface Behaviour of Amphiphilic Block and Block-like Copolymers", Macromolecular Chemistry and Physics, num 210 (2009): 287-298.

⁴ I. S. Burkhanov; L. L. Chaikov; N. A. Bulychev; M. A. Kazaryan & 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.

⁵ A. V. Rudnev; N. G. Vanifatova; T. G. Dzherayan; E. V. Lazareva & 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.

⁶ N. A. Bulychev; E. L. Kuznetsova; V. V. Bodryshev & 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.

⁷ Yu. O. Kirilina; I. V. Bakeeva; N. A. Bulychev & 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).

⁸ V. N. Nikiforov; N. A. Bulychev & V. V. Rzhetskii, "Elastic properties of HTSC ceramics", Bulletin of the Lebedev Physical Institute, Vol: 43 num 2 (2016): 74-79.

monitored the movement of the interface between the solid phase and the pure dispersed medium⁹.

The average particle size of Fe₂O₃ was evaluated on a Coulter-N-4-particle size analyzer¹⁰.

An ultrasound generator “UZDN-2” with a frequency of 22 kHz and a maximum intensity of 30 W / cm² was used for ultrasonic treatment¹¹.

The optical density of the liquid phase of CuPc was measured calorimetrically after keeping a 0.1% dispersion of CuPc in a stabilizer solution for 1 hour and separating the precipitate by filtration¹². The aging time was understood as the time during which the optical density of the liquid phase approached a constant value¹³. This time was approximately 1 hour. The optical density was measured at a wavelength of 364 nm and with a temperature of 18 °C¹⁴.

Results and discussion

Ultrasonic treatment impact on the stability of aqueous dispersions of Fe₂O₃

Ultrasonic treatment of aqueous dispersions of the pigment Fe₂O₃ in the presence of EHEC strongly affects their sedimentation stability and the average particle sizes of the corresponding dispersions¹⁵. In the absence of polymer, the treatment of dispersions in an ultrasonic field leads to their rapid coagulation, and the particle sizes are more or less 1 - 2 microns¹⁶. This is probably due to the appearance of a freshly formed surface with uncompensated charges on the surface of Fe₂O₃ during ultrasonic treatment¹⁷. The system

⁹ A. V. Ivanov; V. N. Nikiforov; S. V. Shevchenko; V. Yu. Timoshenko; v. v. Pryadun; N. A. Bulychev; A. B. Bychenko & 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.

¹⁰ N. A. Bulychev; M. A. Kazaryan; A. Ethiraj & 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 2 (2018): 263-266.

¹¹ N. A. Bulychev & M. A. Kazaryan, Optical Properties of Zinc Oxide Nanoparticles Synthesized in Plasma Discharge in Liquid under Ultrasonic Cavitation. Proceedings of SPIE, 11322, 219 (2019).

¹² N. A. Bulychev; M. I. Danilkin; N. Yu. Vereshchagina & M. A. Kazaryan, Luminescent Properties of ZnO Nanoparticles Doped by W Obtained in Plasma Discharge in Liquid under Ultrasonic Cavitation. Proceedings of SPIE, 11322, 1S. (2019).

¹³ N. A. Bulychev; A. I. Erokhin & 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, 11322, 2G. (2019).

¹⁴ N. A. Bulychev & M. A. Kazaryan, Application of Optical Spectroscopy for Study of Hydrogen Synthesis in Plasma Discharge in Liquid under Ultrasonic Cavitation. Proceedings of SPIE, 11322, 1A. (2019).

¹⁵ M. N. Kirichenko; L. L. Chaikov; I. S. Burkhanov; N. A. Bulychev & M. A. Kazaryan, Interaction of aluminum oxide nanoparticles with human blood plasma thrombin (according to light scattering). Proceedings of SPIE, 11322, 1Y. (2019).

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¹⁷ I. N. Borovik; E. A. Strokach & N. S., Influence of the turbulent Prandtl number on numerical simulation reaction flow. AIP Conference Proceedings, 2181(1), 020029. (2019).

tends to reduce surface energy, which leads to a sharp decrease in the stability of dispersions¹⁸.

The presence of EHEC in the system increases the stability of the dispersions, and the particle size decreases to 0.6 microns¹⁹. This is due to the formation of protective adsorption-solvate layers of the polymer on the surface of the pigment²⁰.

During ultrasonic treatment of dispersions in the presence of EHEC, the stability of dispersions increases significantly (approximately 100 times)²¹. In this case, the particle size decreases almost to the initial particle size of the used pigment Fe₂O₃ (0.3 μm)²².

Thus, ultrasonic treatment of aqueous dispersions of Fe₂O₃ pigment in the presence of a polymer leads to a significant increase in their stability, which is associated with the disaggregation of pigment particles²³.

However, it should be noted that polymers can be degraded during ultrasonic treatment²⁴. This fact can significantly affect the efficiency of the method of mechanochemical surface modification²⁵. The sedimentation stability of Fe₂O₃ pigment dispersions in an aqueous EHEC solution was studied at various durations of ultrasonic treatment. The optimal processing time to obtain stable dispersions of this pigment is 2 minutes²⁶. An increase in the time of ultrasonic exposure leads to a significant decrease in the stability of systems. It can be assumed that EHEC, being an insufficiently flexible polymer, undergoes mechanical destruction, which weakens its stabilizing impact²⁷.

¹⁸ N. A. Bulychev; M. A. Kazaryan; L. S. Lepnev; A. S. Averyushkin; M. N. Kirichenko; A. R. Zakharyan & A. A. Chernov, Luminescent properties of nanoparticles synthesized in electric discharge in liquid under ultrasonic cavitation. Proceedings of SPIE, 10614, 13. (2018).

¹⁹ N. S. Severina, Software complex for solving the different tasks of physical gas dynamics. Periodico Tche Quimica, Vol: 16 num 32 (2019): 424-436.

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²¹ M. N. Kirichenko; N. A. Bulychev; L. L. Chaikov; M. A. Kazaryan & A. V. Masalov, Effect of iron oxide nanoparticles on the blood coagulation according to light scattering data. Proceedings of SPIE, 10614, 2C. (2018).

²² M. N. Kirichenko; N. A. Bulychev; L. L. Chaikov; M. A. Kazaryan & 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, 10614, 0M. (2018).

²³ N. A. Bulychev; M. A. Kazaryan; A. D. Kudryavtseva; M. V. Kuznetsova; T. F. Limonova; N. V. Tcherniega & K. I. Zemskov, Anti-Stokes luminescence in nanoscale systems. Proceedings of SPIE, 10614, 0N. (2018).

²⁴ A. S. Averyushkin; A. N. Baranov; N. A. Bulychev; M. A. Kazaryan; A. D. Kudryavtseva; M. A. Stokov; N. V. Tcherniega & K. I. Zemskov, Stimulated low-frequency Raman scattering in aqueous suspension of nanoparticles. Proceedings of SPIE, 10614, 0K. (2018).

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²⁶ A. S. Averyushkin; A. N. Baranov; N. A. Bulychev; A. I. Erokhin & M. A. Kazaryan, Ag nanoparticles suspensions for stimulated Rayleigh backscattering of single frequency 0.5 micron pulsed laser radiation. Proceedings of SPIE, 10614, 1L. (2018).

²⁷ Y. A. Dyakov; M. A. Kazaryan; M. G. Golubkov; D. P. Gubanova; N. A. Bulychev & S. M. Kazaryan, Laser-induced dissociation processes of protonated glucose: dehydration reactions vs cross-ring dissociation. Proceedings of SPIE, 10614, 17. (2018).

Experiments were performed showing the particle size distribution of aqueous dispersions of the Fe_2O_3 pigment in the presence of EHEC in size before and after ultrasonic treatment²⁸. It is shown that, under ultrasonic treatment of dispersions, the distribution curve narrows significantly, which indicates that a dispersion of Fe_2O_3 with a more uniform structure is obtained.

Ultrasonic treatment impact on the stability of aqueous dispersions of the organic pigment of copper phthalocyanine

For blue pigment (copper phthalocyanine), which is widely used in the printing and varnish industries to obtain intensely colored aqueous and non-aqueous systems, it is important to solve not only the problems of improving the wettability of its surface, but also the transition of the pigment to the environment²⁹.

It was previously shown that compounds that can solve these problems are PVA samples with a high residual content of acetate groups and a low molecular weight³⁰. Optimum parameters are 17% of acetate groups and molecular weight of 10 000 – 11 000³¹. Subsequent experiments showed that the NF dispersant also has the same effect, which, despite its good solubility in water, has powerful hydrophobic groups³². The use of ultrasonic treatment for aqueous dispersions of CuPc in the presence of these substances provides an additional improvement in wetting the surface of the pigment³³.

Experiments were carried out to measure the optical density of an aqueous dispersion of CuPc with PVA, with a separated solid phase, after ultrasonic treatment and without it³⁴. It was found that when using PVA with a content of acetate groups of 2%, without ultrasound treatment, the staining of the solution is insignificant, as evidenced by the low optical density. An increase in the content of acetate groups in PVA to 17% and without ultrasound treatment leads to an increase in the optical density³⁵. Experiments with

²⁸ V. Y. Gidasov & 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.

²⁹ 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 & V. I. Sachkov, Comparative analysis of parameters of pulsed copper vapour laser and known types of technological lasers. *Proceedings of SPIE*, 10614, 02. (2018)

³⁰ V. A. Pogodin; L. N. Rabinskiy & 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.

³¹ V. Y. Gidasov; O. A. Moskalenko & 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.

³² V. V. Nigmatzyanov; V. A. Pogodin; L. N. Rabinskiy & S. A. Sitnikov, "The polymer-ceramic material for the manufacture of gas discharge chamber for the electric rocket engine", *Periodico Tche Quimica*, num 16 Vol: 33 (2019): 801-808.

³³ L. N. Rabinskiy & 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.

³⁴ V. F. Formalev; S. A. Kolesnik & 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.

³⁵ V. F. Formalev; S. A. Kolesnik & 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.*, 2181, 020003. (2016).

ultrasonic treatment of aqueous dispersions of CuPc indicate a high efficiency of exposure to the properties of the pigment surface (possibly, it is activated due to the appearance of defective areas) in the presence of even 2% acetate groups in PVA³⁶. But a particularly intense staining of the aqueous medium in blue is observed in the presence of PVA with a content of acetate groups of 17% under ultrasonic treatment³⁷.

This is confirmed by the results of measuring the optical density of an aqueous dispersion of CuPc with a separated solid phase in the presence of a NF dispersant without and after ultrasonic treatment³⁸. And in this case, the positive effect of ultrasonic treatment is clearly manifested to increase the intensity of staining of aqueous CuPc solutions³⁹. Based on these experiments, it can be assumed that the surface of organic pigments in the ultrasound field undergoes structural rearrangements⁴⁰. The adsorption of these surfactants is intensified on the formed active sites on the surface of CuPc in the presence of the corresponding substances (PVA and NF dispersant)⁴¹. Thus, mechanochemical modification of the surface of organic pigments using ultrasonic treatment will help in the future to solve important practical problems associated with the use of these pigments⁴².

Conclusions

Thus, it can be concluded that the ultrasonic treatment of aqueous dispersions of the inorganic pigment Fe₂O₃ and the organic pigment copper phthalocyanine CuPc with the use of polymer and low molecular weight surfactants is an effective method to increase their stability. Obviously, the use of this technology can significantly increase the efficiency of industrial processes associated with the use of pigments.

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