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**ORGANIC PIGMENTS SURFACE MODIFICATION BY ISOBUTYL VINYL ETHER  
COPOLYMERS UNDER THE ACTION OF ULTRASONIC**

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**Abstract**

For aqueous disperse systems colloidal stabilization of the organic copper phthalocyanine pigment (CuPc), there were used series of homo- and block copolymers of methyl vinyl (MVE) and isobutyl vinyl (IBVE) ethers with a well-characterized molecular structure. It is shown that ultrasonic treatment of disperse systems is of great importance for their stabilization. The results are highly dependent on the hydrophilic-hydrophobic polymers and their structure balance. The temperature dependence of polymethylvinyl ether solubility makes it possible to control polymer stabilizers surface activity.

**Keywords**

Pigments – stability – particulates – copolymers – Ultrasonic

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## Introduction

For aqueous disperse systems colloidal stabilization of the organic copper phthalocyanine pigment (CuPc), there were used series of homo- and block copolymers of methyl vinyl (MVE) and isobutyl vinyl (IBVE) ethers with a well-characterized molecular structure<sup>1</sup>. It is shown that ultrasonic treatment of disperse systems is of great importance for their stabilization<sup>2</sup>. The results are highly dependent on the hydrophilic-hydrophobic polymers and their structure balance<sup>3</sup>. The temperature dependence of polymethylvinyl ether solubility makes it possible to control polymer stabilizers surface activity<sup>4</sup>.

## Results and discussion

There used three block copolymers series of MVE as a hydrophilic fragment and IBVE as a hydrophobic fragment<sup>5</sup>. The blocks' size, their location, polymers' molecular weight characteristics and other parameters varied systematically in a fairly wide range<sup>6</sup>. The reason for choosing PMVE and PIBVE block copolymers as the stabilizer rather than statistical copolymers was the previously published results, which compared the emulsifying effect of statistical and block copolymers<sup>7</sup>. It was concluded that the use of the statistical copolymers PMVE and PIBVE does not allow to get a stable emulsion of either direct or reverse type<sup>8</sup>. In addition, polymethylvinyl ether (PMVE) is a thermosensitive ("smart") polymer, the hydrophilic-hydrophobic balance of which is largely dependent on temperature<sup>9</sup>. The transition temperature value (LCST is low critical solution temperature) is 34 °C, and below this temperature the polymer shows hydrophilic and higher hydrophobic properties<sup>10</sup>.

<sup>1</sup> V. V.; Nigmatzyanov; V. A.; Pogodin; L. N. Rabinskiy and 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.

<sup>2</sup> M. N. Kirichenko; N. A. Bulychev; L. L. Chaikov; M. A. Kazaryan and 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*, num 10614 (2018): OM.

<sup>3</sup> V. Y. Gidasov; O. A. Moskalenko and 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.

<sup>4</sup> V. F. Formalev; S. A. Kolesnik and 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.

<sup>5</sup> V. Y. Gidasov and 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.

<sup>6</sup> M. N. Kirichenko; L. L. Chaikov; I. S. Burkhanov; N. A. Bulychev and 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*, num 11322 (2019): 1E.

<sup>7</sup> L. N. Rabinskiy and 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.

<sup>8</sup> N. S. Severina, "Software complex for solving the different tasks of physical gas dynamics", *Periodico Tche Quimica*, Vol: 16 num 32 (2019): 424-436.

<sup>9</sup> V. A. Pogodin, L. N. Rabinskiy and 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.

<sup>10</sup> A. V. Ivanov; V. N. Nikiforov; S. V. Shevchenko; V. Yu.; Timoshenko; V. V. Pryadun; N. A. Bulychev; A. B. Bychenko and M. A. Kazaryan, "Properties of Metal Oxide Nanoparticles Prepared

The following polymers' series were used:

1) Homopolymers

PMVE 95,  $M_n = 5600$  ( $K = M_w/M_n = 1.09$ )

PIBVE 22,  $M_n = 2300$  (1.07)

2) Two-block copolymers

PMVE 36 – PIBVE 54,  $M_n = 7500$  (1.07)

PMVE 43 – PIBVE 10,  $M_n = 3500$  (1.13)

PMVE 55 – PIBVE 10,  $M_n = 4200$  (1.2)

PMVE 83 – PIBVE 8,  $M_n = 5700$  (1.15)

3) Three-block copolymers

PMVE 50 – PIBVE 20 – PMVE 50,  $M_n = 7800$  (1.18)

PMVE 62 – PIBVE 10 – PMVE 62,  $M_n = 8200$  (1.22)

PIBVE 26 – PMVE 40 – PIBVE 26,  $M_n = 7600$  (1.25)

PIBVE 22 – PMVE 75 – PIBVE 22,  $M_n = 8800$  (1.35)

Using these block copolymers as surfactants, the studies of 0.1% colloidal stability of aqueous dispersed CuPc systems were undertaken as well as the contact angles of CuPc surface wetting with water were measured to determine the surface modification degree with these polymers<sup>11</sup>. All used block copolymers are soluble in ethyl acetate and, with the exception of the most hydrophilic PMVE 95 and PMVE 83 – PIBVE 8, are insoluble in water, therefore, to comply with the uniform conditions for aqueous disperse systems preparation, the copolymers were pre-solubilized in ethyl acetate with a 5% polymer concentration, and then introduced into the aqueous phase together with pigment (phase ratio = 1:10)<sup>12</sup>. Special experiments have shown that the ethyl acetate presence in the system does not affect the polymers' stabilizing ability, but allows more efficient polymer stabilizers' delivery to the organic pigments surface through the aqueous phase<sup>13</sup>. Thus, after dispersions' ultrasonic two-minute treatment a direct type microemulsion was formed, and the pigment particles were in the microemulsion's droplets<sup>14</sup>. All the polymers used at this stage in combination with the ultrasonic treatment use as a dispersing effect make it possible to obtain disperse systems<sup>15</sup>.

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by Plasma Discharge in Water with Ultrasonic Cavitation", *International Journal of Nanotechnology*, Vol: 14 num 7/8 (2017): 618-626.

<sup>11</sup> K. V. Pushkin; S. D. Sevruk; N. S. Okorokova and 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.

<sup>12</sup> V. Y. Gidasov; V. K. Golubev and 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.

<sup>13</sup> V. F. Formalev; S. A. Kolesnik and 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.*, num 2181 (2019): 020003.

<sup>14</sup> N. A. Bulychev; E. L. Kuznetsova; V. V. Bodryshev and 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.

<sup>15</sup> 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 and 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.

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Studies of the disperse systems' colloidal stability were carried out by the sedimentation method, the solid phase's semi-sedimentation time was chosen as assessing stability criteria <sup>16</sup>. The results are presented in Table 1.

<u>Polymer</u>	Solid phase's semi-sedimentation time, days
PMVE 95	2
PIBVE 22	3
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

Table 1

Solid phase's half-sedimentation time in CuPc disperse systems for different polymers

From the Table 1 it is seen that the homopolymers PMVE and PIBVE do not allow to obtain stable aqueous CuPc disperse systems <sup>17</sup>. For most dispersions in the interval from 2 to 20 hours rapid sedimentation and destruction of the microemulsion is observed, after 10 days pigment particles complete sedimentation is observed <sup>18</sup>. For two-block copolymers, the CuPc and colloidal carbon aqueous dispersed systems colloidal stability depends on the polymer hydrophilic-hydrophobic balance <sup>19</sup>.

The data presented for CuPc dispersions allow to conclude that two-block copolymers PMVE and PIBVE with a ratio of hydrophilic and hydrophobic blocks from 5 to 7 will be the most optimal stabilizers providing long-term disperse systems stability <sup>20</sup>.

Analyzing the data on the colloidal stability of CuPc disperse systems obtained using three-block copolymers, it can be concluded that the order of the hydrophilic and hydrophobic blocks arrangement in a macromolecule plays a key role <sup>21</sup>. Obviously, only

<sup>16</sup> Y. A. Dyakov; M. A. Kazaryan; M. G. Golubkov; D. P. Gubanova; N. A. Bulychev and S. M. Kazaryan, "Laser-induced dissociation processes of protonated glucose: dehydration reactions vs cross-ring dissociation", Proceedings of SPIE, num 10614 (2018): 17.

<sup>17</sup> I. S. Burkhanov; L. L. Chaikov; N. A. Bulychev; M. A. Kazaryan and 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.

<sup>18</sup> V. F. Formalev and S. A. Kolesnik, "On Thermal Solitons during Wave Heat Transfer in Restricted Areas", High Temperature, Vol: 57 num 4 (2019): 498-502.

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

<sup>20</sup> Yu. O. Kirilina; I. V. Bakeeva; N. A. Bulychev and 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.

<sup>21</sup> A. V. Rudnev; N. G. Vanifatova; T. G. Dzherayan; E. V. Lazareva and 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.

when the hydrophilic PMVE block (even if not very big) is located in the macromolecule center and the hydrophobic PIBVE blocks are located at the edges, this polymer has a good stabilizing effect on CuPc dispersions<sup>22</sup>. At the same time, the presence of hydrophobic PIBVE block in the macromolecule center, even not very long, does not allow one to obtain stable disperse systems<sup>23</sup>. In both cases, an insignificant role of the extreme blocks and their length should be noted<sup>24</sup>.

For a series of homo- and copolymers, the CuPc surface contact angles were also measured<sup>25</sup>. These data is presented in Table 2.

Polymer	Contact angle value, degree
Without a polymer	132
PMVE 95	24
PIBVE 22	118
PMVE 36 – PIBVE 54	122
PMVE 43 – PIBVE 10	114
PMVE 55 – PIBVE 10	78
PMVE 83 – PIBVE 8	59
PMVE 50 – PIBVE 20 – PMVE 50	119
PMVE 62 – PIBVE 10 – PMVE 62	86
PIBVE 26 – PMVE 40 – PIBVE 26	117
PIBVE 22 – PMVE 75 – PIBVE 22	83

Table 2.

The of contact angles' values of CuPc surface water wetting treated with homo- and copolymers<sup>26</sup>

Table 2 shows that the contact angle of the pigment surface substantially depends on the copolymer's structure<sup>27</sup>. The difference between the contact angles' values of pigment's clean surface wetting from the values obtained in the presence of polymers indicates the polymers' surface layers formation during ultrasonic treatment<sup>28</sup>. The table also shows that the presence of adsorbed copolymers 3 and 4 for two-block and 2 and 4

<sup>22</sup> N. Bulychev; W. Van Camp; B. Dervaux; Y. Kirilina; K. Dirnberger; T. Schauer; V. Zubov; F. E. Du Prez and 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.

<sup>23</sup> V. F. Formalev; É. M. Kartashov and 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.

<sup>24</sup> V. F. Formalev; S. A. Kolesnik; E. L. Kuznetsova and 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.

<sup>25</sup> V. F. Formalev and 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 num1 (2019): 52-59.

<sup>26</sup> M. N. Kirichenko; N. A. Bulychev; L. L. Chaikov; M. A. Kazaryan and A. V. Masalov, "Effect of iron oxide nanoparticles on the blood coagulation according to light scattering data", *Proceedings of SPIE*, num 10614 (2018): 2C.

<sup>27</sup> M. N. Kirichenko; L. L. Chaikov; I. S. Burkhanov; N. A. Bulychev and M. A. Kazaryan, "Interaction of aluminum oxide nanoparticles with human blood plasma thrombin (according to light scattering)", *Proceedings of SPIE*, num 11322 (2019): 1Y.

<sup>28</sup> V. Y. Gidaspov and 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.

for three-block on the pigment surface allows them to be wetted with water<sup>29</sup>. This data is consistent with the study results of CuPc dispersions colloidal stability<sup>30</sup>.

Assumptions about the adsorption method of polymer stabilizers on the organic pigments surface can be expressed as follows<sup>31</sup>. The hydrophobic PIBVE blocks (PODVE) are adsorbed on the organic pigment surface due to hydrophobic interaction, and the associated hydrophilic PMVE blocks are turned into the aqueous phase<sup>32</sup>.

In this case, the central hydrophilic block “covers up” the hydrophobic blocks lying on the pigment surface, thereby providing a two-layer diphilic structure<sup>33</sup>. In the case of 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 system<sup>34</sup>.

Table 2 shows that the contact angle of the pigment surface substantially depends on the copolymer's structure<sup>35</sup>. The difference between the contact angles' values of pigment's clean surface wetting from the values obtained in the presence of polymers indicates the polymers' surface layers formation during ultrasonic treatment<sup>36</sup>. The table also shows that the presence of adsorbed copolymers 3 and 4 for two-block and 2 and 4 for three-block on the pigment surface allows them to be wetted with water<sup>37</sup>. This data is consistent with the study results of CuPc dispersions colloidal stability<sup>38</sup>.

<sup>29</sup> 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, and V. I. Sachkov, “Comparative analysis of parameters of pulsed copper vapour laser and known types of technological lasers”, Proceedings of SPIE, num 10614 (2018): 02.

<sup>30</sup> V. N. Nikiforov; N. A. Bulychev and V. V. Rzhetskii, “Elastic properties of HTSC ceramics”, Bulletin of the Lebedev Physical Institute, Vol:43 num 2, (2016): 74-79.

<sup>31</sup> N. A. Bulychev and M. A. Kazaryan, “Application of Optical Spectroscopy for Study of Hydrogen Synthesis in Plasma Discharge in Liquid under Ultrasonic Cavitation”, Proceedings of SPIE, num 11322 (2019): 1A.

<sup>32</sup> N. A. Bulychev; A. I. Erokhin and 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, num 11322 (2019): 2G.

<sup>33</sup> A. S. Averyushkin; A. N. Baranov; N. A. Bulychev; A. I. Erokhin and M. A. Kazaryan, “Ag nanoparticles suspensions for stimulated Rayleigh backscattering of single frequency 0.5 micron pulsed laser radiation”, Proceedings of SPIE, num 10614 (2018): 1L.

<sup>34</sup> N. A. Bulychev; M. A. Kazaryan; L. S. Lepnev; A. S. Averyushkin; M. N.; Kirichenko; A. R. Zakharyan and A. A. Chernov, A. A. “Luminescent properties of nanoparticles synthesized in electric discharge in liquid under ultrasonic cavitation”, Proceedings of SPIE, num 10614 (2018): 13.

<sup>35</sup> V. V. Bodryshev; A. V. Babaytsev and 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.

<sup>36</sup> N. A. Bulychev; M. A. Kazaryan; A. Ethiraj and 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.

<sup>37</sup> N. A. Bulychev; M. I. Danilkin; N. Yu. Vereshchagina and M. A. Kazaryan, “Luminescent Properties of ZnO Nanoparticles Doped by W Obtained in Plasma Discharge in Liquid under Ultrasonic Cavitation”, Proceedings of SPIE, num 11322 (2019) 1S.

<sup>38</sup> A. S. Averyushkin; A. N. Baranov; N. A. Bulychev; M. A. Kazaryan; A. D.; Kudryavtseva, M. A. Stokov; N. V. Tcherniega and K. I. Zemskov, “Stimulated low-frequency Raman scattering in aqueous suspension of nanoparticles”, Proceedings of SPIE, num 10614 (2018): 0K.

Assumptions about the adsorption method of polymer stabilizers on the organic pigments surface can be expressed as follows<sup>39</sup>. The hydrophobic PIBVE blocks (PODVE) are adsorbed on the organic pigment surface due to hydrophobic interaction, and the associated hydrophilic PMVE blocks are turned into the aqueous phase<sup>40</sup>.

In this case, the central hydrophilic block “covers up” the hydrophobic blocks lying on the pigment surface, thereby providing a two-layer diphilic structure<sup>41</sup>. In the case of 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 system<sup>42</sup>.

## Conclusions

As a result of the studies, it was shown that diphilic polymers are effective stabilizers of CuPc type hydrophobic organic pigments aqueous dispersions, which have the ability to be adsorbed on the surface of hydrophobic particles using their hydrophobic fragments and to hydrophilize the surface due to the hydrophilic fragments presence. The adsorption layers structure and the stabilizing effect strongly depend on the polymer structure, which is confirmed by data on colloidal stability and contact angles values of wetting. It was identified that in the case of nonionic PMVE + PIBVE stabilizers use, only block copolymers have stabilizing ability, and the efficiency strongly depends on the copolymers structure, length, number and location of blocks.

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