Methods For Optimizing the Content of Vocs To Create Environmentally Friendly Materials For Protective Coatings

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Abstract. In order to successfully reduce the emission of toxic organic solvents harmful to human health and the environment during the application and curing of paints and varnishes (VPM), ways to create environmentally friendly organically soluble VPM "SB" and water-dispersed VPM "WB" are proposed. The influence of the values of dry residues in solutions of organic oligomers and the density of the components of pigmented materials on the values of the content of volatile organic compounds (VOC) for different types of materials has been studied. Mathematical analysis of situations with the addition and reduction of the amount of solvents allowed to set the limit values of the amount of solvents in varnishes for "SB" materials with different densities and VOC = 300 g/l. The influence of volumetric concentration of pigments, dry residue of varnishes and density of pigments and fillers on the VOC content in materials has been studied. The ratios of VPM components have been optimized and formulations of environmentally friendly materials have been developed.

1 Introduction

A promising way to develop the chemistry and technology of paints and varnishes [1-6] is the creation of innovative resource and energy-saving technologies for environmentally friendly materials, which include three groups of materials. The first group is materials with a low content of volatile organic compounds: white spirit, solvent, xylene, etc. The second group includes powder paints, which consist of pigments, fillers, plasticizers and polymer powders. The third group of materials consists of water-dispersion paints and varnishes, the market of which is developing at a very high rate.

Currently, the majority of paints and varnishes available on the market are made up of water-based materials. Most of the paints and varnishes available nowadays are water-based materials. The environmental safety of these products is determined by the level of volatile organic compounds present in them. To ensure compliance with safety regulations, the Technical Regulation was adopted in Ukraine, as per the order of the Ministry of Economic Development and Trade. This regulation sets limits on the level of volatile organic compounds allowed for different types of materials.

The Order on approval of the Technical Regulation on limiting emissions of volatile organic compounds due to the use of organic solvents in paints and varnishes for buildings and repair of wheeled vehicles puts into effect the Technical Regulation gradually, at the first stage less stringent requirements are fixed compounds should be significantly reduced. The technical regulation is based on Directive 2004/42/EC of the European Parliament and of the Council of 21 April 2004 on the limitation of emissions of volatile organic compounds due to the use of organic solvents in certain paints and varnishes and vehicle recycling products and amending the Directive 1999/13 / EC. The terms used in the Technical Regulation refer to the VOC content – the weight of volatile organic compounds per unit volume, expressed in grams per liter, g/l, in the formulation of the material in its ready-to-use state. The Technical Regulations provide information on water-dispersion paints (type

"WB"), the viscosity of which is regulated by the addition of water; organosoluble paints (type "SB"), the viscosity of which is regulated by the addition of organic solvent.

The traditional approach to producing environmentally friendly materials involves researching and developing materials with a high content of non-volatile substances, usually equal to or greater than 80%. In order to obtain oligomers for varnishes with a high content of non-volatile substances and low viscosity, it is essential to synthesize new film-forming substances with lower molecular weight, a narrow molecular weight distribution, and the use of flexibilizers that are highly effective at reducing the viscosity of solvents, as well as reactive solvents. The influence of various factors influencing the VOC values, namely the density of the pigment part and the reduction of the content of volatile organic compounds (VOC) in organic oligomers for pigmented materials, is also considered.

2 Materials and methods

The following materials and intermediates were used as raw materials for obtaining enamel with a low VOC content: varnish – Long oil alkyd resins Crestakyd 10-0504, Crestakyd 10-1019 (produced by Scott Bader, Croatia), pigment – titanium dioxide (of (produced in China), desiccants – siccatives Co, Mg, Ba (produced by Venator, UK), solvent – white spirit (145-200) (produced by Matrapac®WS, Germany), filler – Calcium carbonate brand Andcarb CT-2x (of oproduction LKB Company, Türkiye).

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The expediency of selecting pigments and fillers with different densities has been studied, and the limits of increasing and decreasing the solvent content to obtain materials with specified VOC values have been determined. The analysis possibilities are presented, along with the formulations for pigmented materials with set VOC values, which can be utilized for decorative and protective coatings on products made of metal, wood, and plastic, both for outdoor and indoor use.

An environmentally friendly enamel with a VOC content of 300 g/l was obtained using a bead mill with a stirrer shaft speed of 2000 rpm, glass beads with ball diameters of 2-3 mm. The dispersion period limited to the time required to achieve a dispersion degree of 25 μm as measured to the Klin device according to ISO 1524. The enamel was applied to glass surfaces with dimensions of 90x120 mm and having a thickness of 1.2 mm to determine the conditional hardness of the coatings.

The drying time determined according to ISO 9117, while the elasticity of the film during bending (ISO 1519) was tested on plates made of black hot-rolled tin with dimensions of 70x150 mm and a thickness of 0.25-0.28 mm. The impact strength of the film and the adhesive strength (ISO 6272-2 and ISO 1519) were tested on plates made of body steel grades 08kp and 08ps, measuring 150x70 mm and having a thickness of 0.8-0.9mm. The coatings were applied to the surfaces using an applicator, and the thickness of the coating layer was 25 μm, as measured by a thickness gauge according to ISO 2808.

3 Discussion

The concept of the influence of component density on VOC values is important. The maximum VOC content of paints used for interior/exterior work on wood, metal, or plastic should be 150/130 g/L for type "WB" and 400/300 g/L for type "SB" before and after 01.01.24, respectively. The VOC content of non-volatile substances in "SB" paints and varnishes is determined by the following formula:

$$VOC = 10 \cdot d_e \cdot (100 - NV) \quad (1)$$

where d_e – density of the material, kg/m^3; NV – content of non-volatile substances in the material, %.

The density of the material is calculated by the formula:

$$d_e = \frac{\sum m_i}{\sum \frac{m_i}{d_i}}, \quad (2)$$

where m_i – weight of the i-th component, kg; d_i – density of the i-th component, kg/m^3.

For water-dispersion paints "WB", the main volatile component of which is water, the VOC content will depend on the prescription number of components that contain organic volatile compounds (mainly coalescents). If this content is predetermined in weight percent n (VOC), wt. %, the calculation equation takes the form:

$$VOC = 10 \cdot d_e \cdot n(VOC) \quad (3)$$

By analyzing equations (1) and (3), we can see that an increase in the content of non-volatile substances in varnishes and a decrease in the density of the material for materials "SB" creates the possibility of reducing the content of VOCs. For "WB" materials, creating special dispersions without coalescents in the composition of water-dispersion VPM can lead to a decrease in the parameter n(VOC) and VOC. Increasing the content of non-volatile substances NV in "SB" materials can potentially reduce the amount of VOCs. As for "WB" materials, a promising direction is the development of special dispersions without coalescents in the composition of water-dispersion VPM. This leads to a decrease in the parameters n(VOC) and VOC. Previous studies [7, 8] have shown that the use of glass microspheres in the pigmented part of enamel significantly reduces the density of raw materials. Glass microspheres were introduced in the market of raw materials for paints and varnishes specifically to create low VOC content materials.

In this analysis, we will examine the formulations of materials containing fillers such as barite, calcium carbonate, Aerosil, and pigment titanium (IV) oxide. Table 1 shows the formulations used.

When considering the impact of component density on the volatile substance content in pigmented materials, we found that replacing calcium carbonate with barium sulfate led to an increase in enamel density from 1175 kg/m^3 to 1195 kg/m^3 and an increase in VOC from 415.83 g/l to 422.9 g/l. On the other hand, replacing calcium carbonate with 3% of Aerosil reduced the enamel density from 1175 kg/m^3 to 1164 kg/m^3 and decreased the VOC content from 415.83 g/l to 411.9 g/l. When 5% of Aerosil was used instead of calcium carbonate, the enamel density decreased to 1069 kg/m^3, and the VOC content decreased to 378.32 g/l.

Table 1. Formulations of VPM "SB" with different density of filler

The name of the component	d_i [kg/m^3]	Recipe, m [kg]			
		№ 1	№2	№3	№4
1. Varnish NV=53%	1100	66.47	66.47	66.47	66.47
2. Titanium (IV) oxide	4200	19.46	19.46	16.46	13.88
3. Calcium carbonate	2700	9.92	–	9.92	10.50
4. Aerosil	2000	–	–	3.00	5.00
5. Barytes	4500	–	9.92	–	–
6. White spirit	790	3.48	3.48	3.48	3.48
7. Desiccant	800	0.67	0.67	0.67	0.67
Together		100	100	100	100
Contents VOC [g/l]		415.83	422.9	411.9	378.32
Density, d_e [kg/m^3]		1175	1195	1164	1069
Enamel, NV [%]		64.61	64.61	64.61	64.61

Table 2. Formulations of VPM "WB" [5] with different filler density

The name of the component	d_i [kg/m^3]	Recipe, *m* [kg]		
		№ 1	№2	№3
1. Aqueous dispersion NV =50%	1000	60	60	60
2. Titanium (IV) oxide	4100	25	–	22
3. Calcium carbonate	2700	–	25	–
4. Glass microspheres	125	–	–	3
5. Water+VOC	1000	12+3	12+3	12+3
Together		100	100	100
Contents VOC [g/l]		37.0	35.6	28.0
Density, d_e [kg/m^3]		1233	1187	933
Enamel, NV [%]		55	55	55

In the formulations of VPM "WB" [7] the use of fillers with lower density also leads to a decrease in density and VOC content in materials.

Thus, the above results of calculations indicate a significant decrease in VOC with a decrease in the density of pigments and fillers for pigmented paintwork materials of groups "SB" and "WB".

Analysis of the formulation of pigmented material. According to the given formulas (1), (2) calculations of indicators of pigmented materials according to the recipe № 1 in Table 1 are carried out. VCP, CVCP, density and content of non-volatile substances in enamel.

$$CVCP = \frac{1}{1+0,01\cdot M_I \dfrac{d_n}{d_o}}\cdot 100\%,$$

where *CVCP* – critical volume concentration of pigments; M_I – oil capacity of a mixture of pigments, $M_I = \frac{\sum M_{Ii}P_i}{\sum P_i}$; M_{Ii}.– oil pigment capacity, g/100g; P_i – the proportion of pigment in the pigment part; d_n – density of pigments, kg/m^3; d_o – the density of the oligomer base, kg/m^3;
VCP – volume concentration of pigments; $VCP = (V_p + V_f)/(V_p + V_f + V_o)$, where V_p,, V_f, V_o – volumes of pigment, filler and oligomer, respectively.

The characteristics of the pigmented material and the source components are shown in Table 3.

Table 3. Characteristics of pigmented material

The name of the component	Amount [kg]	Density [kg/m^3]
1. Varnish, NV =53±2%	66.47	1100(basis)
2. Titanium (IV) oxide	19.46	4200
3. Calcium carbonate	9.92	2700
4. White spirit	3.48	790
5. Desiccant	0.67	800
Together	100.00	
NV varnish [%]	53.00	
NV enamel [%]	64.64	
Contents VOC [g/l]	413.70	
VCP enamel [%]	21.00	
CVCP [%]	52.00	

Thus, the analysis of the characteristics of the enamel, which are shown in Table 3, indicates the need to adjust the enamel formulation to reduce the VOC content in order to comply with the Technical Regulations, namely 400 g/l and 300 g/l before and after 01/01/2024, respectively.

The concept of reducing the solvent content. In the case of reducing the amount of solvent x_i, the VOC value is calculated by the formula:

$$VOC = \left(\frac{100 - x_i - NV}{100 / d_e - x_i / d_s} \right) \tag{4}$$

The first derivative of the expression allows you to determine the conditions for increasing VOC (5), decreasing VOC (6) and the presence or absence of an extremum (7, 8). In the case under consideration, there is no extremum:

$$1 - \frac{d_s}{d_e} - \frac{NV}{100} > 0, \tag{5}$$

$$1 - \frac{d_s}{d_e} - \frac{NV}{100} < 0, \tag{6}$$

$$1 - \frac{d_s}{d_e} - \frac{NV}{100} = 0, \tag{7}$$

$$d_e = \frac{d_s}{1 - \frac{NV}{100}} \quad . \tag{8}$$

Consideration of the situation with a decrease in the amount of solvent is necessary to establish the concentration ranges of VOC content in organic oligomers.

There are two possible ways to solve the problem of obtaining VPM that meet these requirements:

1. Synthesis or use of varnishes or resins with low VOC content [9-12].
2. Increasing the content of non-volatile substances in varnishes, which are in the range of modern leading industrial productions [13-16]. To reduce the VOC content in the enamel without changing the components and their density, you can increase the content of non-volatile substances in the varnish. Theoretical calculations allowed to obtain enamel formulations with different VOC content when changing NV varnishes from 53% to 100%. The results of the calculations are presented in Tables 4 and 5.

Table 4. Parameters of enamel formulations with different VOC values

Parameters	Recipe №1	Recipe №2	Recipe №3
NV varnish [%]	53	56	70
d varnish [kg/m^3]	929	940	980
d enamel [kg/m^3]	1170	1180	1230
NV enamel [%]	64.64	66.63	75.94
VOC [g/l]	415.5	395.33	296.76
VCP [%]	21	20	16
1. Varnish	66.47	66.47	66.47
2. Titanium (IV) oxide	19.46	19.46	19.46
3. Calcium carbonate	9.92	9.92	9.92
4. White spirit	3.48	3.48	3.48
5. Desiccant	0.67	0.67	0.67
Together	100.00	100.00	100.00

Table 5 presents the results of theoretical calculations of enamel parameters using varnishes with different content of non-volatile substances to obtain enamels with different VOC content.

It should be noted that enamels with VOC = 395.33 g/l and VOC = 296.76 g/l can be obtained using varnishes with dry residues of 56% and 70%, respectively.

Table 5. Parameters of enamel formulations with different VOC values

NV varnish [%]	d_e enamel [kg/m^3]	d_i varnish [kg/m^3]	NV enamel [%]	VOC [g/l]	VCP [%]	VCP/CVCP [con.un]
53	1170	929	64.64	415.50	21.0	0.400
56	1180	940	66.63	395.30	20.0	0.380
60	1200	950	69.29	367.97	19.0	0.360
65	1220	970	72.61	332.88	17.0	0.320
70	1230	980	75.94	296.76	16.0	0.310
75	1250	1000	79.26	259.57	15.0	0.290
80	1270	1020	82.58	221.26	14.5	0.285
85	1290	1040	85.91	181.78	14.0	0.270
90	1310	1060	89.23	141.07	13.5	0.250
95	1330	1080	92.55	99.08	13.0	0.245

Figures 1 and 2 depict the graphical dependencies of the change in VOC content with respect to the decrease in the amount of solvent xi for enamel formulations with specified parameters.

It is necessary to consider the situation where the amount of solvent is decreased to establish the concentration ranges of the VOC content in organic oligomers to obtain environmentally friendly materials that meet regulatory requirements.

The dependencies shown in Fig. 1 enable the determination that to obtain an environmentally friendly material with VOC = 300 g/l for enamels with densities of 1170, 1180, 1200, 1220, and 1230 kg/m^3, it is necessary to retain 16, 13, 9, 5, and 1 g of solvent, respectively.

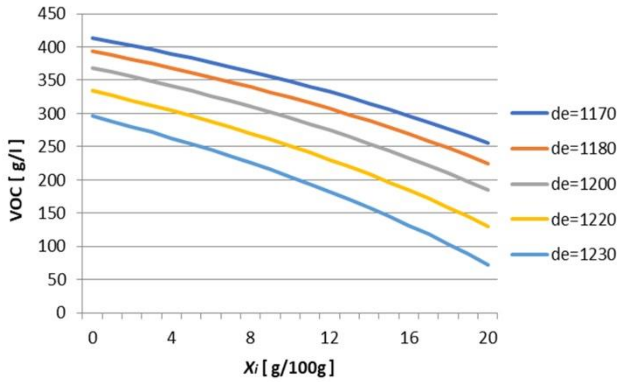

Fig. 1. Dependence of VOC values on the reduction of the amount of solvent x_i for formulations № 1-5 of Table №5 for d_e = 1170; 1180; 1200; 1220; 1230 [kg/m^3]

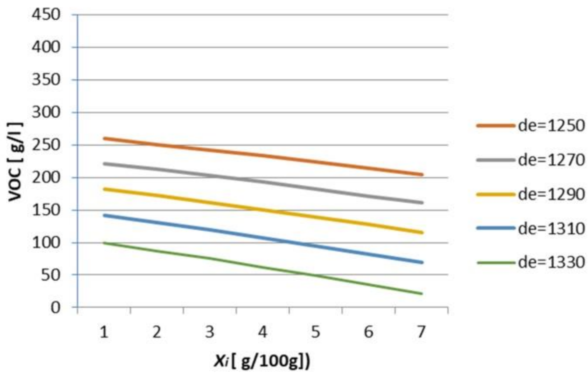

Fig. 2. Dependence of VOC values on the reduction of the amount of solvent x_i for recipes № 6-10 of the Table №5 for d_e = 1250; 1270; 1290; 1310; 1330 [kg/m^3]

On Fig. 2 shows the dependences of VOC on x_i for compositions of materials with a density of 1250 to 1330 kg/m^3 that meet the requirements of the Technical Regulations and on the basis of which absolutely environmentally friendly compositions can be obtained.

The concept of adding solvent content. VOC is measured in ready-to-use material. Before applying paintwork materials, it is necessary to prepare it, add a solvent to bring it to working viscosity, depending on the chosen application method. Therefore, it is advisable to consider the case of increasing the amount of solvent when diluting pigmented materials.

In the case of increasing the amount of solvent, the VOC value is calculated by the formula:

$$VOC = \left(\frac{100 + x_i - NV}{100 / d_e + x_i / d_s} \right). \tag{9}$$

Obviously, it is advisable to dilute the paintwork material and bring it to a working viscosity in order to maintain low VOC values with a solvent with a lower density.

The first derivative of the expression allows you to determine the conditions for increasing VOC (10), decreasing VOC (11) and the presence or absence of an extremum (12, 13). In the case under consideration, there is no extremum:

$$\frac{d_s}{d_e} + \frac{NV}{100} - 1 > 0, \tag{10}$$

$$\frac{d_s}{d_e} + \frac{NV}{100} - 1 < 0, \tag{11}$$

$$\frac{d_s}{d_e} + \frac{NV}{100} - 1 = 0, \tag{12}$$

$$d_e = \frac{d_s}{1 - \frac{NV}{100}} \tag{13}$$

In Fig. 3, 4 the dependences of the change in the VOC content on the increase in the amount of solvent x_i for enamel formulations with the specified parameters are given.

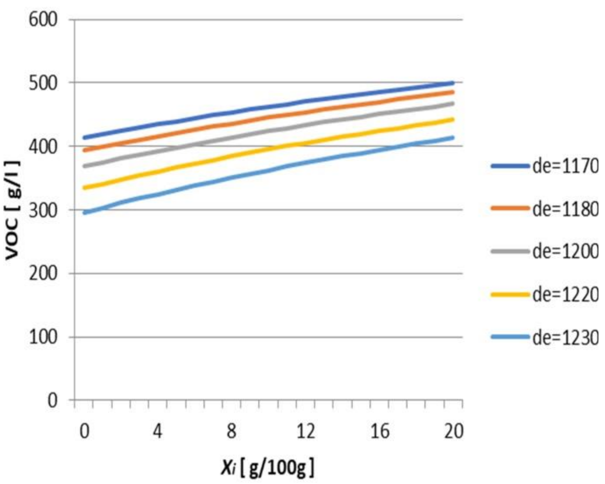

Fig. 3. Dependence of VOC values on the increase in the amount of solvent *xi* for recipes № 1-5 of Table №5 for d_e=1170; 1180; 1200; 1220; 1230 [kg/m^3]

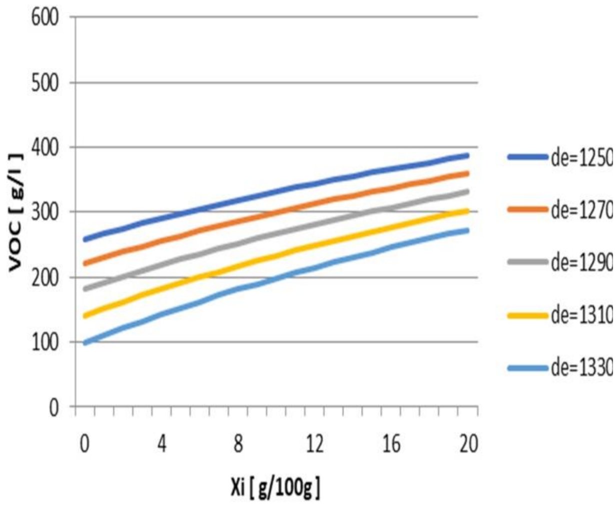

Fig. 4. Dependence of VOC values on the increase in the amount of solvent *xi* for recipes № 6-10 Table №5 for d_e=1250; 1270; 1290; 1310; 1330 [kg/m^3]

The results presented in Fig. 3 indicate the impossibility of using compositions with a density from 1170 to 1220 kg/m^3 to obtain environmentally friendly materials when adding a solvent to the liquid medium of the material, since any increase in the amount of solvent will lead to VOCs of more than 300 g/l.

For compositions No. 6-10 Table. 5 with densities from 1250 to 1330 kg/m^3 environmentally friendly materials can be obtained by adding 5, 10, 15, 20 and 23 g of solvent, respectively. The dependences obtained in the work and presented on the graphs can be used to predict the content of volatile organic compounds in enamels based on the content of non-volatile substances in varnishes and enamels.

Figure 5 for VOC enamels that meet the Technical Regulations at different stages of implementation shows the dependences of enamel densities on the content of non-volatile substances.

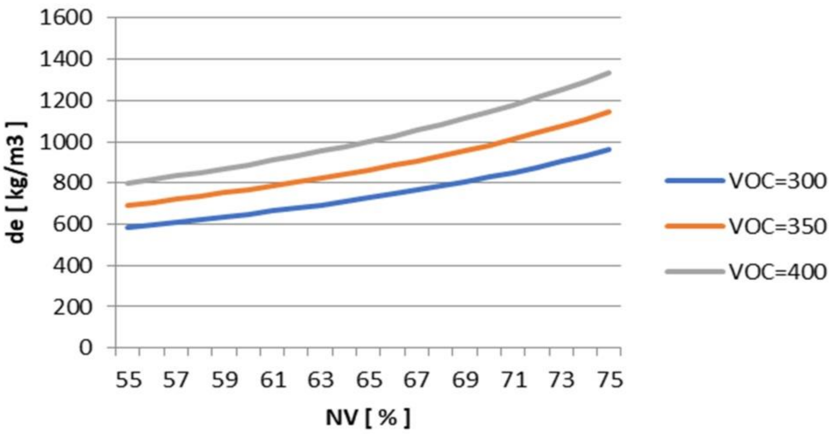

Fig. 5. Dependence of enamel density values on the increase in content non-volatile enamel substances for VOC=300 g/l, 350 g/l, 400 g/l

Figure 5 shows the area of existence of enamel with VOC=300 g/l in the density range from 586 to 961 kg/m^3. For enamel with VOC=350 g/l, the density varies in the range from 692 to 1145 kg/m^3. Enamel with VOC=400 g/l can be characterized by density values from 801 to 1336 kg/cm^3. For all the listed enamels, the content of NV varies from 55 to 75%.

On Fig. 6-8 show the results of studies of the influence on the content of volatile organic compounds of enamel solids of varnish and the ratio of volatile organic compounds in solids of enamel and VCP. In Fig.6, according to the dependence of VOC on the amount of dry residue of varnish, it can be established that enamel with VOC=300g/l contains varnish with HB=70%.

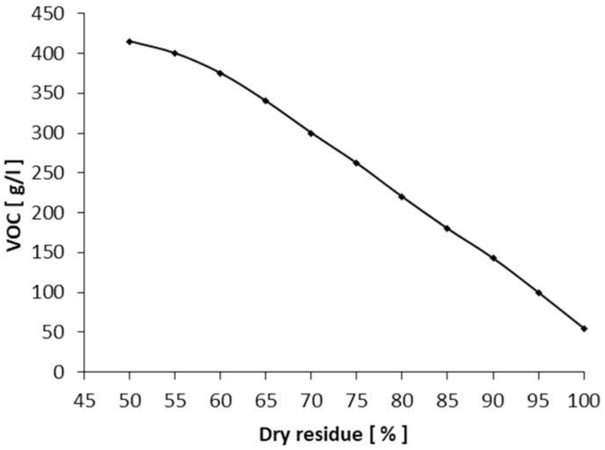

Fig. 6. Dependence of VOC on the value of the dry residue of the varnish

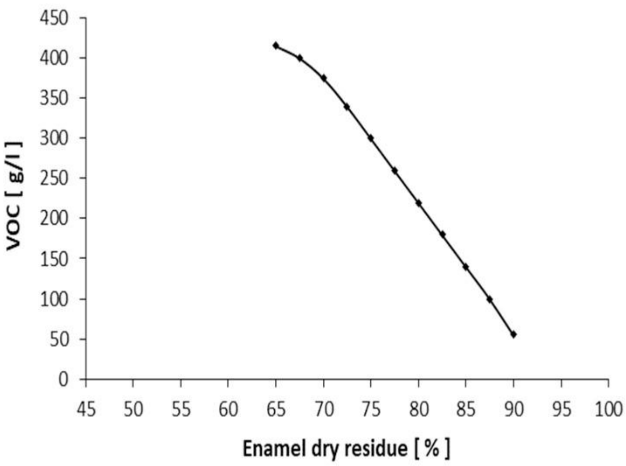

Fig. 7. Dependence of VOC on the value of dry enamel residue

According to the dependence of VOC on the value of the dry residue of enamel, shown in Fig. 7, it is possible to determine HB=75% of enamel with VOC=300g/l.

The VCP of enamel with VOC=300g/l corresponds to 0.17 (Fig. 8).

The data presented in Fig. 9 allow us to state the presence of enamels with VOCs from 200 to 300 g/l and from 300 to 400 g/l at an enamel density of 1270 to 1230 kg/m^3 and from 1350 to 1150 kg/m^3, respectively.

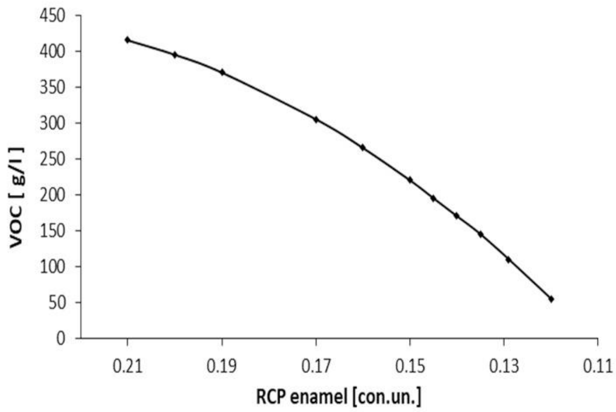

Fig. 8. Dependence of VOC on VCP enamel

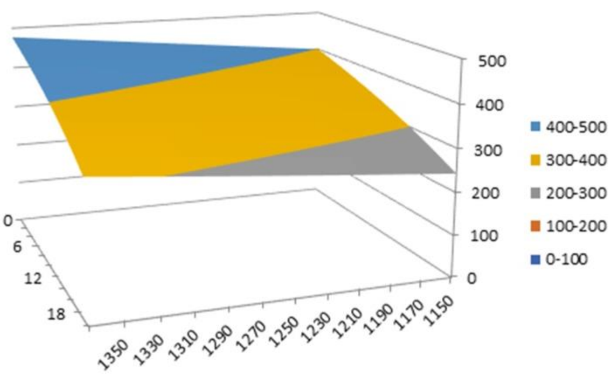

Fig. 9. Dependence of VOC on the value of the dry residue of the enamel

Experimental studies of the physical and mechanical properties of coatings based on recipe No. 3 of Table 4 are shown in Table 6. The obtained parameters correspond to the standard indicators.

Table 6. Physical and chemical properties of enamels (experimental and normalized)

Parameters	Experimental	Normalized
NV enamel [%]	76	65
Time natural drying [h]	24	24
Hardness [r. u.]	0.41	0.15
Adhesion [p]	0	0
The resistance of the coatings to the static action of water according [h]	24	24
The elasticity of the film in bending according [mm]	2	2
The impact resistance of the film [cm]	50	50

NV=100%, Crestakyd 10-1019 with NV=60% The content of Crestakyd 10-0504 in the total resins was 25%, and Crestakyd 10-1019 was 75%.

4 Conclusions

Thus, this work examines the effects of varnish and enamel dry residue, varnish and enamel density, volumetric concentration of VCP pigments, and the ratio of VCP to VCP on enamel VOC values. The results indicate that reducing the density of enamel, while maintaining the same dry residue value, is an effective method to reduce VOC content.

It has been established that an increase in the dry residue of enamel is the predominant factor determining the decrease in enamel VOC even with an increase in enamel density as a result of a decrease in the solvent content in the enamel composition.

The paper proposes methods for obtaining pigmented materials with a low VOC content, both by reducing the density of pigments and fillers, and by increasing the content of non-volatile substances of varnishes up to 56% for VOC 400 g/l and 70% for VOC 300 g/l.

The study presents material formulations with VOC values of 300 g/l and 400 g/l and determines the physical-mechanical and chemical properties of the coatings. The results show that the properties of the coatings meet the specified parameters.

Variants with the addition of solvents when diluting enamels before their use by selected methods and evaporating solvents to obtain environmentally friendly materials are considered, and the density intervals for the existence of enamels with a VOC content of 300 g/l are determined.

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