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Article

Investigation of the Main Operational Properties of Polymer-Based Composites with the Content of Modified Mineral Filler

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Abstract: The article presents the results of research work devoted to the study of composite materials on polymer binder with the content of modified mineral filler - water-dispersion paint and varnish materials on acrylic polymer with modified filler - diatomite. The aim of the study is to obtain polymer-based composites with modified diatomite (water-dispersion paint on acrylic binder) with improved performance properties. Methods: determination of porosity, adhesion, elasticity, hiding power of the control composition; determination of porosity, adhesion, elasticity, hiding power of the obtained composites with the use of modified filler; investigation of the influence of radiation in the infrared radiation (IR) region of the spectrum of the paint coating surface using FLIRB620 thermal imager. As a result of work it is noticed that modification of water dispersions by silica activated diatomite promotes elimination of the main disadvantages of materials and coatings on the basis of acrylate binder - low water resistance and low physical and mechanical characteristics, introduction of modified diatomite into water emulsion paint on acrylate basis does not lead to deterioration of the main operational characteristics of paint and varnish coatings - porosity, adhesion, elasticity and covering. When analyzing the effect of emissivity in the IR region of the spectrum of the paint coating surface, a significant effect of the quantitative content of modified diatomite on the reduction of emissivity of paint coatings in the IR region of the spectrum was established and it was shown that a significant effect of the reduction of emissivity of paint coatings in the IR region of the spectrum is provided by the introduction of modified diatomite in the amount of 40 wt.h. into the mixture. The influence of the time of full-scale tests in the investigated small range regardless of the quantitative composition of modified diatomite on the performance characteristics of the paintwork was established.

Keywords: acrylate binder; modified filler; adhesion; coatings; covering; porosity

1. Introduction

The deteriorating environmental situation in the world causes a tightening of requirements not only for various industries, but also for the materials they produce, as even newly developed products are not always environmentally friendly for humans and the environment. Among such materials, protective polymer coatings play an important role. It is known [1–3] that in many industries paint and varnish materials based on organic solvents, which are toxic to humans, are used, which leads to the search for an alternative method of protection of structures and buildings. One such option is the use of water-based paints and varnishes - water-dispersion.

Creation of environmentally friendly polymer protective composite materials resistant to external factors is one of the priority directions of researchers. It is known that currently the most

common and available way to solve this problem is the use of water-based filled acrylic compositions [4–8].

Modifying target additives and natural fillers play an important role in obtaining protective composite coatings [9,10]. In this regard, the aim of this work was to study the influence of mineral fillers of different nature and shape of particles on the physical-mechanical and operational properties of coatings (Pc), for the protection of concrete and metal surfaces.

There are large reserves of diatomite in Kazakhstan, which makes it possible to use them in paint and varnish formulations. According to the data of geological prospecting works, siliceous rocks within the Aktobe region of the Republic of Kazakhstan have an extensive distribution.

The aim of the research work is to obtain polymer-based composites with modified diatomite (water-dispersion paint on acrylic binder) with improved performance properties and low cost.

Scientific novelty in obtaining water-dispersion paint and varnish composition on acrylic base with diatomite content; estimation of economic efficiency of this technology in comparison with existing analogues.

2. Materials and Methods

2.1. Materials

The following materials were used as the object of the study.

Filler: Diatomite is a loose or weakly cemented siliceous sedimentary rock composed mainly of the shells of diatom algae and is white, grey or pinkish in color. In the natural state diatomite's have a large uniformly distributed porosity, reaching 80-85%. Silica in diatomite's is in amorphous state and makes up 78-95%.

Binder: Akremos-115A aqueous dispersion is a copolymer of styrene and acrylic monomers obtained by emulsion method. It is used as a binder to produce colors that are suitable for exterior and interior painting. The composition "Akremos-115A" does not contain organic solvents and belongs to low hazardous substances (4th class of hazard) [11–13].

The polymer dispersion "Arakril ADC 777" is an aqueous dispersion of styrene copolymer and acrylic acid esters with high dry residue, obtained by emulsion method. Dispersion of high quality styrene-acrylic copolymer in water, specially formulated for the production of general purpose paints, with high to medium pigment volume concentration (PVC). The product contains finer copolymer particles (less than 0.1 μm) compared to standard grades of styrene-acrylic dispersions.

Pigment: rutile P-02 grade titanium dioxide (mass fraction of rutile titanium dioxide - 95%). Titanium dioxide is a white dense powder, insoluble under normal conditions in alkalis, solutions and acids.

2.2. Methods

Sampling was carried out according to GOST 9980.2 (GOST – Government standard) [14]. To prepare the middle sample for research according to GOST 28196 item 4.2 [15], the film was removed from the surface of the material, then the test material was thoroughly mixed to a homogeneous state.

It is allowed to determine the color and appearance of the film on the samples after testing by the indicator "Coverage of dried film" according to GOST 28196 item 4.3 [15]. The covering power of the dried film can be determined by two methods, namely:

1. Plotting the material on a laboratory chart;
2. Application on a glass plate according to GOST 8784 section 1 [16].

To identify this characteristic, method No.1 was used: the test sample, 250 μm thick, was applied to the laboratory card on the left side using a rakley in a single layer; on the right is the control sample. After drying, visual comparison of the tested sample with the control sample was carried out for one hour under natural daylight in a room at air temperature (20 $\pm$ 2) °C.

Determination of the covering power of the coating according to method No. 2 consists in applying layers of coating material on a glass plate (glass for photographic plates of size 9 x 12-1.2) until the contours of a black-and-white contrasting plate or a checkerboard placed under the glass

plate become invisible. To determine the hiding power, the coating material is diluted to the working viscosity. On a glass plate prepared according to GOST 8832 and weighed to the fourth decimal place, one or two layers of coating material are applied. A glass plate with the coating material is placed on a contrasting plate and observed in diffused daylight whether the white and black fields are translucent.

The dynamic viscosity of the resulting paint will be measured using a Haake VT 24 rotary viscometer.

Test procedure: The material sample is thoroughly mixed to a homogeneous mass, avoiding the formation of air bubbles. The tests are carried out at air temperature (20±2) °C. The pin of the rotating body is immersed in a plastic cup with the sample to be tested up to the mark, then the motor is started. After one minute, a reading of the measured value is taken from the scale of the Haake viscometer. After the measurement is completed, the rotating body pin is gently rinsed with water to prevent damage to the rotating body pin. Each test shall be carried out twice and the test result will be taken as the arithmetic mean.

To determine the pH range, we used a pH-meter with a glass electrode, measurement error not more than 0.1, a plastic beaker (volume 240 ml, height 8.0 cm, upper diameter 6.7 cm, lower diameter 5.2 cm), distilled water according to GOST 6709 [17]. All tests are carried out in accordance with GOST 52020 item 9.4 [18].

Studies to determine the mass fraction of non-volatile substances are carried out in accordance with GOST 31939 [19].

Density is determined according to GOST R 53654.1 [20], adhesion of coating is tested according to GOST 31149 [21], degree of grinding according to GOST 31973-2013 [22], resistance to static impact of water according to GOST 9.403, method A [23], resistance to vapour permeability according to GOST 25898 [24], conditional light resistance of coating according to GOST 21903 [25].

Evaluation of the appearance of coatings was carried out according to GOST 6992-68 [26]. The adhesion of paint coatings was determined by the lattice notch method [21]. The adhesion value (%) is judged by the number of squares of the coating that have not separated. If adhesion is good, the coating material should not lag anywhere on the metal. If adhesion is poor, the film will come away from the metal almost over the entire lattice. The elasticity of the films characterises their bending strength. It was determined using the elasticity scale according to GOST 9.074-77 [27].

3. Results

3.1. Results of Experimental Research

The results of studies of diatomites of Utesai, Zhalpak and Kirgiz deposits of the Aktobe region of the Republic of Kazakhstan.

The samples of investigated diatomites were finely dispersed powders of greyish-yellowish color, obtained by grinding of slightly cemented rocks of corresponding deposits. The particle size distribution of the dispersed material prepared in this way is shown in Table 1.

Table 1. Particle size distribution of materials.

Size, microns	Particle content of deposit diatomite, %		
	Utesai deposit	Zhalpak deposit	Kirgiz deposit
0.260	0.890	2.885	1.026
0.291	5.770	13.138	6.178
0.325	13.407	20.307	13.444
0.362	23.774	27.364	23.075
0.404	23.191	23.215	22.481
0.451	14.573	9.292	14.311
0.504	9.551	3.373	9.565
0.563	4.028	0.051	4.418
0.628	1.319	0	1.780

0.701	1.497	0.062	1.712
0.783	0.963	0.106	1.074
0.834	0.499	0.039	0.556
0.975	0.192	0.004	0.210
1.089	0.087	0	0.095

Measurements were carried out using a Shimadzu SALD-3101 fine particle size analyzer with a measurement range of 50 nm to 3 mm. The analysis of the size distribution of natural diatomite particles showed that 99% of their total amount has linear sizes: 0.260 - 0.783 μm (Utesai deposit); 0.260 - 0.504 μm (Zhalpak deposit); 0.260 - 0.789 μm (Kyrgyz deposit). Particle size distributions are close to Gaussian - the most probable values of particle size for dispersed material of Utesai, Zhalpak and Kyrgyz deposits are close to each other and lie in the interval $\sim 0.350 - 0.410 \mu\text{m}$. Thus, the powders of the studied diatomite's are ultra-disperse granular systems consisting mainly of submicrometre-sized particles.

Figure 1 shows electron microscopy images of natural diatomite powders. Remains of shell flaps of diatom algae with regular channels of $\sim 300\text{-}500 \text{ nm}$ diameter, which constitute the life support system of unicellular algae, as well as various in shape and size fragments of dispersed material, are clearly visible. A characteristic feature of granular systems is their significant porosity, reaching even for natural fossil rocks, 70% and more. Free space between particles, as well as inhomogeneities of amorphous silica particles themselves in the form of pores, channels, cracks of nano- and submicrometre sizes form a developed pore structure of different scale levels, which determines many properties of mineral powders.

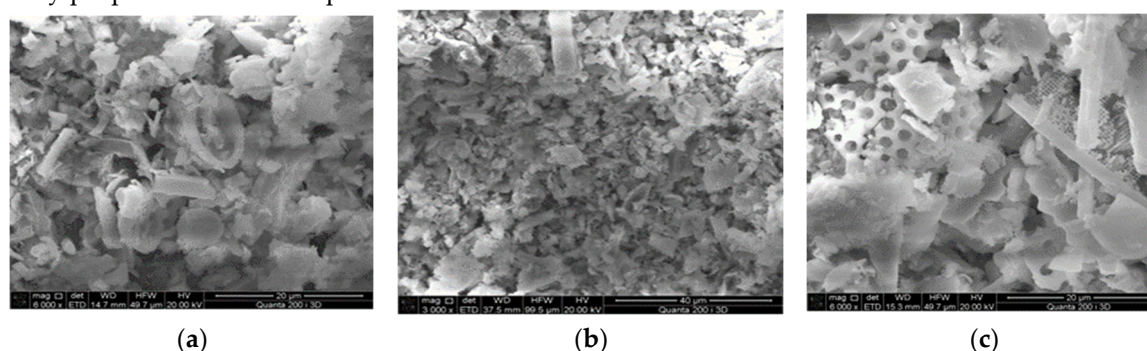


Figure 1. Microphotographs of natural diatomite powders from: (a) Utesai, (b) Zhalpak and (c) Kyrgyz deposits.

The processes occurring during heating of diatomite's were investigated by thermogravimetric analysis [28]. Figure 2 shows the results of thermal analysis of diatomite samples from Utesai, Zhalpak and Kyrgyz deposits. The thermogravimetric analysis (TG) curve shows three stages of mass loss of the samples during the heating process. The first of these begins immediately after the start of heating and continues until about 350 $^{\circ}\text{C}$. This partially dehydrates the natural diatomites and reduces the mass of the samples by about 6%. At further heating in the temperature range 400 – 600 $^{\circ}\text{C}$ along with continuing dehydration of small-scale pores and pore channels of amorphous silicon dioxide particles, there is an intensive burning of organic material residues in flaps and shell fragments of diatom algae, as well as other impurities of organogenic origin. These processes are recorded on the thermogravimetric analysis diagram as an endothermic peak around 500 $^{\circ}\text{C}$. The mass loss at this stage is approximately 2.5 %.

Heating of dispersed material from 600 to 1000 $^{\circ}\text{C}$ is also accompanied by a decrease in the mass of diatomite samples. The minimum mass loss is observed in diatomite of Zhalpak deposit, the maximum - in diatomite of Kyrgyz deposit. In total during the heating cycle diatomite of Utesai deposit loses $\sim 11\%$, Zhalpak $\sim 9\%$, Kyrgyz $\sim 13\%$ of the initial mass.

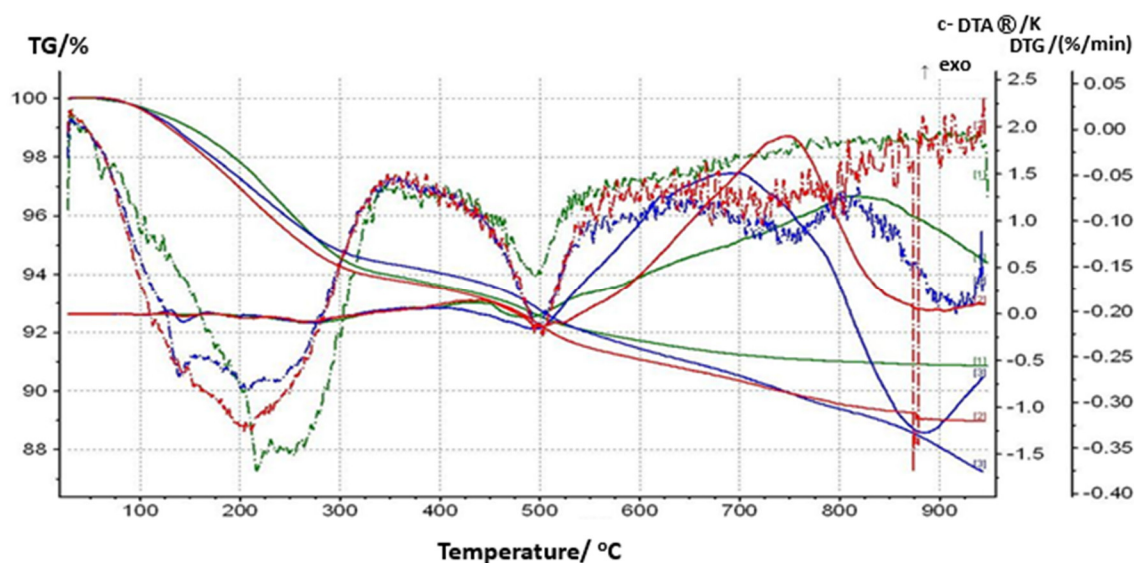


Figure 2. Data of thermogravimetric analysis of natural diatomite samples: 1 - Zhalpak; 2 - Utesai; 3 - Kyrgyz deposits.

The average molecular weight of the acrylic polymer was determined by cryoscopy method [29]. The M value for the acrylic film former is 1250, and for the polyether siloxane copolymer and sodium, polyacrylate is 1120 and 1190, respectively.

The composition and structural characteristics of the samples were established by IR spectroscopy [30]. Free carboxyl groups were observed in the IR spectra of the acrylic film former, as evidenced by the presence of absorption peaks characteristic of hydroxyl groups ($3650\text{--}3200$) in the 3358 cm^{-1} region and carbonyl groups ($1800\text{ cm}^{-1}\text{--}1650\text{ cm}^{-1}$) in the 1728 cm^{-1} region Figure 3. The shift to the shorter wavelength region of the spectrum of the carbonyl group proves the alkyl structure of the hydrocarbon skeleton connected to the carboxyl group ($1725\text{ cm}^{-1}\text{--}1700\text{ cm}^{-1}$).

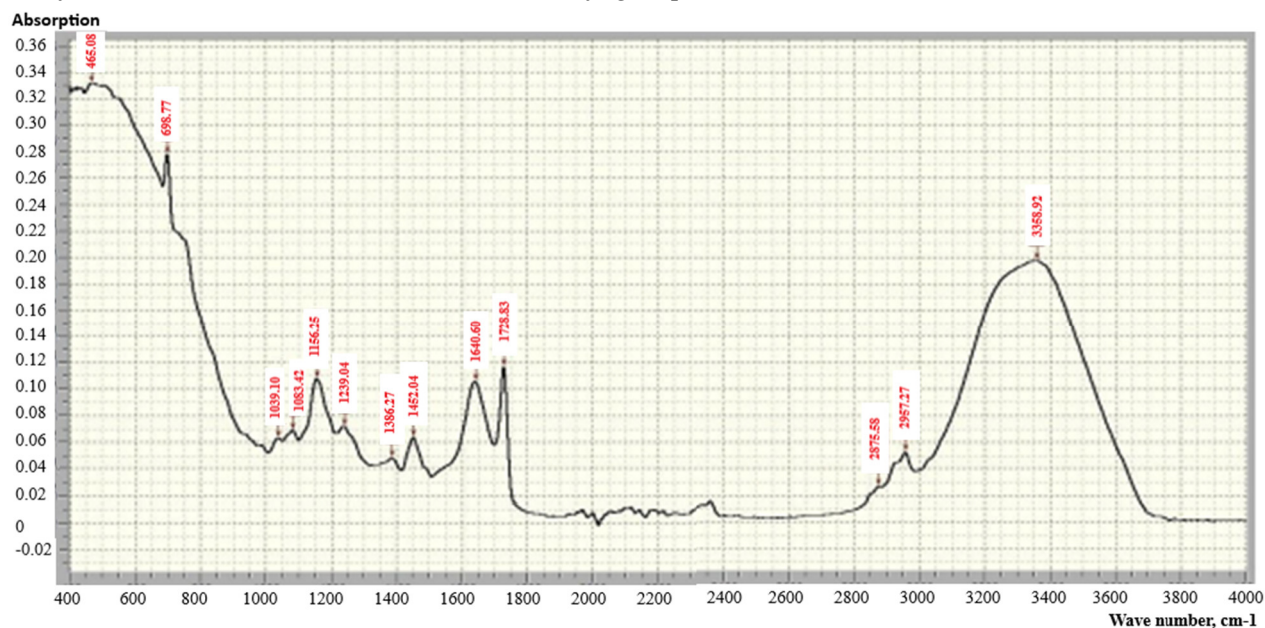


Figure 3. IR spectra of acrylic polymer samples [30].

Water-dispersion paint is a finishing paint and varnish composition, the main component of which are synthetic polymers, often resin or oil. The solvent of the paint is water, which is why the material is considered environmentally friendly, harmless and fireproof. The peculiarity of finishing

walls with water-dispersion paint is that in a few hours after opening the container with paint from the surface water evaporates, and solid particles form a dense impermeable layer, resistant to moisture and fire. This material is produced as a thick suspension, which can be diluted with water to the required consistency if necessary. Water-dispersion paint can be white in colour, or it can be colored in a variety of shades. Traditionally, the following components are present in the composition of such paint:

- 1. Film-forming materials (binder) are the base of the paint, which creates a polymer film on the surface that is impervious to water and resistant to abrasion.
- 2. Dyes (pigments) - components that give the paint a certain shade and are responsible for the decorative qualities of the material.
- 3. Fillers - they are responsible for the operational and technical features of the paint. These components are calcite, talc, dolomite, copper, marble chips, etc.
- 4. Special additives (additives) give water-dispersion paint additional properties, for example, are responsible for the speed of drying of the composition on the surface; accelerate the process of dispersion of colorants, etc.

The main task of the research was to study the influence of mineral natural filler on the characteristics of polymer-based water-dispersion composite.

Table 2 presents a comprehensive study of the effect of the mineral additive diatomite in water-dispersion composite, designed for interior finishing works, on some standardized indicators of paint quality.

Table 2. Research results of diatomite introduction into water-dispersion composite.

Method of introducing diatomite into the composite	Quantity of diatomite, wt.h.	Coverage, g/m³	Viscosity, Pa·s	Dry residue,%	Density, g/cm³	
During the mixing process	1	20	170.3	11500	65.5	1.66
	2	10	165.7	12100	65.5	1.66
	3	40	181.3	11400	66.0	1.66
	4	13	168.5	11700	60	1.66
CaO+diatomite mixture	1	5	155.26	11980	65.5	1.66
Standardized parameters		170.5	10500- 11200	65.5	1.66	

The addition of diatomite to the composite was carried out directly during the mixing of the components in the dispersing unit. In this case, the study of composite properties was carried out in two stages: immediately after mixing and preparation of the composite, as well as after 2 months of composite storage. The latter study is due to the need to evaluate the influence of the degree of diatomite coagulation on the composite properties. The results of the research are presented in Table 3.

Table 3. Research results of diatomite incorporation into water-dispersion composite after 2 months.

Type of property test	Viscosity, Pa·s	Dry residue, %	Coverage, g/m ³	Density, g/cm ³
Immediately after adding diatomite to the composite	15780	65.5	175	1.66
After storing a sample of the prepared composite with diatomite	11890	65.0	175	1.65

Standardized parameters	10300	65.5	170.5	1.66
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4. Discussion

The introduction of diatomite into the water-dispersion composite did not particularly change the regulatory quality parameters such as density and dry residue. When diatomite is added, a significant increase in viscosity and hiding power of the composite is observed. At the same time, the influence of diatomite on these indicators is significantly higher, which is probably due to the different values of the wetting edge angle of these materials. The covering power as one of the main indicators of composite quality at introduction of diatomite increases very insignificantly and is practically within the normative limits. It should be noted that a slight increase in the viscosity of water-dispersion composites in the process of production while maintaining the other parameters is positive, because it allows to reduce the viscosity of the composite to dilute it with water to ensure the standardised quality, which, in turn, contributes to cheaper production and increased efficiency.

The addition of diatomite during the composite production process significantly increases its viscosity. However, after prolonged storage, the viscosity of the paint decreases to almost standard values. This effect can be favourably utilised when the composite is stored for long periods of time or transported long distances.

The conducted complex of researches has allowed defining the character of influence of the mineral natural filler introduced into water-dispersion composites on change of standard indicators of composites that gives the possibility of formation of a new complex of properties of water-dispersion composites (WDC) with simultaneous increase of efficiency of process of their industrial production.

Coatings and free films were obtained from the developed aqueous dispersion materials. Drying time of coatings up to degree 3 at temperature (20±2) °C is 1 h and does not change with the introduction of modified diatomite. The characteristics of the samples are summarized in Table 4.

Table 4. Physical-mechanical and operational properties of films and coatings based on compositions of polymer binder modified diatomite’s.

Indicators	Values for composite with modified diatomite content,		
	% wt %.		
	0	1	2
Without pigment			
Modulus of elasticity, MPa	0.65±0.1	2.1±0.3	2.93±0.8
Tensile strength, MPa	5.0±0.7	5.15±0.4	4.9±0.4
Relative elongation at break, %	616±79	378±43	308±46
With pigment			
Modulus of elasticity, MPa	1.2 ±0.3	6.5±2.5	8.3±3.2
Tensile strength, MPa	2.7±0.4	2.9±0.3	3.1±0.3
Relative elongation at break, %	415.5±90	283±70	162±60
Hiding power.g/m², not more	81.3	83.5	83.0
Hardness. s.u,	0.21	0.23	0.25
Water absorption, %	77.4	66.5	32.4

It can be seen from the data in Table 4 that modification with activated diatomite provides a significant increase in the elastic modulus of the composite films without reducing the tensile strength. This indicates high adhesion strength at the interfacial boundary “polymer - modified diatomite” and, accordingly, good compatibility of modified diatomite and polymer matrix (acrylate binder). Adhesion strengthening of both pigment-free and pigmented developed coatings is provided. The introduction of modified diatomite into the sample with pigment does not lead to a decrease in the covering power of the paint material. Combined with the ability to control the

rheological properties of the dispersion during modification, this can help to reduce paint consumption during painting. Modified coatings with modified diatomite are characterized by increased hardness, this is presumably due to the ordering of polymer chains near the aluminosilicate surface, which is often observed in the case of good compatibility between the polymer and the highly dispersed filler [31], the polymer boundary layer formed is characterized by increased physical and mechanical properties compared to the polymer in the volume. Modification of coatings with diatomite allows to provide a significant reduction in water absorption of coatings (2.6 times when 2% of modified diatomite is introduced). This may also be due to the densification of the supramolecular structure of the polymer matrix, in addition, the introduction of ultrafine nanoparticles reduces the diffusion coefficient of water molecules in the composite [32]. The sharp decrease in the water absorption of the coating when increasing the modified diatomite content from 1 to 2% wt., % may be due to a change in the nanostructure of the composite; in [33], in this concentration range of modified diatomite in the polymer dispersion, a transition from an exfoliated morphology of the nanocomposite to an intercalated morphology characterized by a higher degree of particle ordering was observed.

Thus, modification of aqueous dispersions with silica-activated diatomite helps to eliminate the main disadvantages of materials and coatings based on acrylate binder - low water resistance and low physical and mechanical characteristics. Low cost of modified diatomite, simplicity of introduction into water-dispersion materials, possibility of regulation of rheological properties of dispersion and achieved improvement of characteristics of modified coatings determine the prospects of practical application of water-dispersion materials modified by activated diatomite, including acrylate polymer-based paints and varnishes.

Water-based paints based on modified fillers are attracting increased interest from consumers and manufacturers of paints and varnishes. With their high specific surface area, the granules of dispersed modified fillers create strong bonds between the components and fill the voids between them, forming a homogeneous and durable coating.

Dispersed silica granules are known to be hollow spheres that form the porous structure of the paint.

In this regard, we conducted a series of experiments:
- determination of porosity, adhesion, elasticity, covering power of the control composition;
- porosity, adhesion, elasticity, hiding power of modified filler.
The formulated amount of additive was introduced into a 200 ml glass beaker and stirred at room temperature using a magnetic stirrer for 5 min.

The experimental results are summarised in the tables of the first series of experiment 5-9.

Table 5. The influence of full-scale testing time on the performance characteristics of paint and varnish coatings (without the additive - modified diatomite).

Sample number, No.	Adhesion, %			Porosity, number of points per 40 cm²		
	Field test time, days					
	1	7	14	1	7	14
1	94	95	95	4	5	5
2	95	96	96	5	4	5
3	96	96	94	4	5	5
4	95	96	95	5	5	5
5	94	95	94	5	5	5

Table 6. The influence of full-scale testing time on the performance characteristics of paint and varnish coatings (with the additive content - modified diatomite (20 parts by weight)).

Sample number, No.	Adhesion, %			Porosity, number of points per 40 cm²		
	Field test time, days					

	1	7	14	1	7	14
1	91	91	90	4	5	4
2	91	92	91	5	5	6
3	91	92	92	4	5	5
4	92	91	90	6	4	6
5	90	91	92	5	5	5

Table 7. The influence of full-scale testing time on the performance characteristics of paint and varnish coatings (with the additive content - modified diatomite (10 parts by mass)).

Sample number, No.	Adhesion, %			Porosity, number of points per 40 cm ²		
	Field test time, days					
	1	7	14	1	7	14
1	93	94	95	3	4	3
2	95	94	95	4	4	5
3	94	93	95	3	5	4
4	96	95	94	4	5	5
5	93	95	94	5	4	5

Table 8. The influence of full-scale testing time on the performance characteristics of paint and varnish coatings (with the additive content - modified diatomite (40 parts by weight)).

Sample number, No.	Adhesion, %			Porosity, number of points per 40 cm ²		
	Field test time, days					
	1	7	14	1	7	14
1	96	95	96	5	5	5
2	95	95	96	5	5	5
3	94	96	96	5	5	5
4	96	96	96	5	5	5
5	95	96	96	5	5	5

Table 9. The influence of full-scale testing time on the performance characteristics of paint and varnish coatings (with the additive content - modified diatomite (13 parts by weight)).

Sample number, No.	Adhesion, %			Porosity, number of points per 40 cm ²		
	Field test time, days					
	1	7	14	1	7	14
1	93	94	95	4	4	4
2	93	94	95	4	4	5
3	92	94	95	4	4	5
4	93	93	95	4	4	5
5	91	93	94	4	4	5

It has been established that the introduction of modified diatomite into acrylate-based water emulsion paint does not lead to deterioration of the main performance characteristics of paint and varnish coatings - porosity, adhesion, elasticity and hiding power. The influence of filler - modified diatomite on the performance characteristics of the paintwork is insignificant, at the level of experimental error.

The insignificant influence of the time of full-scale tests in the investigated small range regardless of the quantitative composition of the modified diatomite on the performance characteristics of the paint and varnish coatings was shown.

5. Conclusions

The results of the conducted research allowed us to formulate the following main conclusions:

1. Modification of aqueous dispersions with silica-activated diatomite helps to eliminate the main disadvantages of materials and coatings based on acrylate binder - low water resistance and low physical and mechanical characteristics. Low cost of modified diatomite, simplicity of introduction into water-dispersion materials, possibility of regulation of rheological properties of dispersion and achieved improvement of characteristics of modified coatings determine the prospects of practical application of water-dispersion materials modified by activated diatomite, including acrylate polymer-based paints and varnishes.

2. The introduction of diatomite into the water-dispersion composite did not particularly change the normative quality parameters such as density and dry residue.

3. When diatomite is added, a significant increase in viscosity and covering power of the composite is observed. At the same time, the influence of diatomite on these indicators is significantly higher, which is probably due to the different values of the wetting edge angle of these materials. The covering power as one of the main indicators of composite quality at introduction of diatomite increases very insignificantly and is practically within the normative limits. It should be noted that a slight increase in the viscosity of water-dispersion composites in the process of production while maintaining the other parameters is positive, because it allows to reduce the viscosity of the composite to dilute it with water to ensure the standardized quality, which, in turn, contributes to cheaper production and increased efficiency.

4. The addition of diatomite during the composite production process significantly increases its viscosity. However, after long-term storage, the viscosity of the paint decreases to almost standard values. This effect can be utilized positively for long-term storage or long distance transport of the composite.

The conducted complex of researches has allowed defining the character of influence of the mineral natural filler introduced into water-dispersion composites on change of standard indicators of composites that gives the possibility of formation of a new complex of WDC properties with simultaneous increase of efficiency of process of their industrial production.

Thus, the use of diatomite as a filler of water-dispersion composite in optimal ratios allows obtaining compositions with the required covering power and mass fraction of non-volatile substances, which confirms the feasibility of using mineral filler of Aktobe deposit for the production of water-dispersion paints.

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