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RESEARCH OF TECHNICAL PROPERTIES OF SILICON CONTAINING ANTIPYRENS FOR PRACTICAL APPLICATION IN THE PRODUCTION OF WATER-DISPERSION COATINGS

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Abstract
In the modern world, widely used water-borne paints and varnishes form coatings with high resistance to the action of water and chemicals, good electrical insulation, physico-mechanical and decorative properties. Everyone knows they are used for painting radio and electrical products, in the automotive industry, agricultural machinery, in the production of household products and residential buildings.The article discusses the technical justification of research results for the practical use of silicon-containing flame retardants in the production technology of fire-retardant water-dispersion paint. The regulations and an effective technological scheme for the production of AP-1 silicon-containing oligomeric flame retardant and the technology for producing fire-retardant water-dispersion paint coating have been developed.The test method for modified fire-retardant water-dispersion coatings based on polyacrylic dispersion was carried out according to standard methods "Determination of the combustibility of fire retardants for wood" in accordance with GOST 16363-98, which includes methods for determining the combustibility, flammability of coatings, obtained in collaboration with Laboratory Institute of Fire Safety of the Ministry of Emergencies of the Republic of Uzbekistan.Based on the data presented, based on the results of studies on the modification of the properties of fire-retardant water-dispersion coatings with silicon-containing oligomeric flame retardants, new fire-resistant water-dispersion coatings with high physicomechanical, heat-resistant, and operational properties have been developed.The developed technological regulations and the condition for obtaining coatings by the modified proposed flame retardants proposed by the authors apply to the production of fire-retardant water-dispersion paints, which are a suspension of pigments and fillers in an aqueous dispersion of synthetic polymers with the addition of AP-4 flame retardant and various auxiliary substances according to TU 6.12-78-2000 , TU 6.12-28-96, produced by mixing components in mixers and dispersing pastes in bead mills.

Key words: *silicon-containing, flame retardants, fire retardancy, water dispersion paint, modification, oligomers, fire resistance, physical and mechanical, heat resistance, operation.*

Water-borne paints and varnishes form coatings with high resistance to water and chemicals, good electrical insulation, physico-mechanical and decorative properties. They are used for coloring products of radio and electrical engineering, in the automotive industry, agricultural engineering, in the production of household products and residential buildings [1].

Based on the data obtained according to the results of studies on the modification of the properties of fire-retardant water-dispersion coatings with silicon-containing oligomeric flame retardants, new fire-resistant water-dispersion coatings with high physicomechanical, heat-resistant and operational properties have been developed.

The technological scheme for producing silicon-containing oligomeric flame retardant AP-1 and the technology for producing fire-retardant water-dispersion paint coating [2] have been developed.

Modified fire-retardant water-dispersion paint coatings based on polyacrylic dispersion were tested, the standard methods were used to determine the combustibility of fire retardants for wood in accordance with GOST 16363-98, which includes methods for determining the combustibility, flammability of coatings obtained in collaboration with the Institute of Fire Safety laboratory Ministry of Internal Affairs of the Republic of Uzbekistan.

Fire-retardant water-dispersion paints based on a polyacrylic base modified with silicon-containing oligomeric flame retardants AP-4 were obtained and the combustibility group of the oligomeric flame retardant AP-4 was determined.

The proposed developed technological scheme for the production of oligomeric flame retardant AP-4 based on the interaction of sodium metasilicate with sodium tetraborate in a glycerol solution is shown in Fig. 1.

To obtain a flame retardant, a 1M, 2M aqueous solution of sodium tetraborate and technical glycerin are immersed in a 4P reactor with a stirrer, a jacket and a reflux condenser. Raise the temperature to 80 ° C, constantly mixing the mass for 2-3 hours. Then the reaction mass is cooled in the refrigerator 5X to 293-298 K and transferred to the reactor 7P.

Sodium metasilicate 3M is added from the mercury and the reaction is continued for 6-7 hours at a temperature of 343 K. The resulting mass is evaporated in a dryer 9C and transferred to an intermediate tank 8E, after which the excess reagent residue is sent to a 6E tank. The resulting mass is washed with water to get rid of the solution of glycerol and alkalis, then re-evaporated in a dryer 10C. The resulting target product is sent to the warehouse.

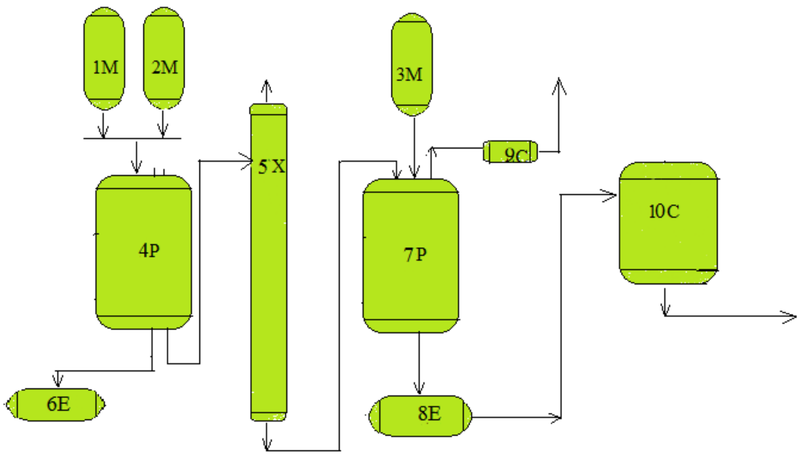

Fig.1. -Schematic diagram of the production of oligomeric flame retardant AP-4 based on sodium metasilicate with sodium tetraborate:

1M, 2M, 3M - measuring devices for reagents; 4P, 7P - reactors; 5X-refrigerator; 6E-collection; 8E - intermediate capacity; 9C - dryer; 10C - the evaporator and the capacity of the target product

The results of the analysis of industrial technology for the production of water-dispersible fire-retardant paint under industrial conditions (Fig.-1) revealed the conditions for obtaining the modified oligomeric flame retardant AP-4 by the interaction of sodium metasilicate with sodium tetraborate in glycerol solution during the production of water-dispersible fire retardant paints for internal work.

The main principle of obtaining fire-retardant water-dispersion paint is to attach a flame retardant during preparation of the material obtained in advance by the interaction of sodium metasilicate with stearic acid and a urea adduct with phosphoric acid.

Despite the complexity of the formulation of water-dispersion paints, the technology for their manufacture is simple and fundamentally no different from the technology for the production of enamels and primers containing organic solvents.

Prior to the main stages of the technological process, preparatory work is preliminarily carried out, in which the preparation of the constituent components is carried out according to the corresponding recipe below.

The technological process for the production of water-dispersion paints for internal work is carried out in stages:

1. Reception, preparation and dosage of raw materials, preparation of semi-finished product.

2. Preparation of pigment paste (milling pigments with semi-finished product) and its dispersion.

3. Compilation of paint, mixing a water-dispersion binder with pigment pastes;

4. Typing of paint, tinting, filtering and packaging of paint, curing equipment.

When accepting, preparing and dosing raw materials, all raw materials are subject to mandatory verification of their quality indicators before being used in production. Accepted raw materials are stored in a warehouse and supplied to the workshop based on daily requirements in accordance with the production plan. The thickener, the sodium salt of carboxy methyl cellulose, is the most common water-soluble cellulose ether, which is used as a 10% aqueous solution, is produced in Namangan by the SPS Carbonam plant. The thickener is prepared on the basis of the daily requirement, according to the following recipe: sodium salt of carboxy methyl cellulose-10%, water-90%. Its preparation is carried out in a bowl, by stirring on a wall-mounted mixer at a speed of rotation of the mixer of 300-500 rpm, until a homogeneous mass is obtained (sample in bulk on glass). To accelerate the dissolution process, 25% ammonia solution is used. Dosing of raw materials according to the routing is carried out by weight. Water, acrylic dispersion and dry components are weighed on the balance. Dosing of additives is made from containers for storing additives. Preparation of water-dispersion paint based on acrylic dispersion, prefabricated mixture, an aqueous solution of excipients, emulsifiers, dispersants.

Instead of the auxiliary substance, an OP-7 wetting agent was used, which is the product of processing a mixture of mono- and dialkylphenols with ethylene oxide, which meets the requirements of GOST 8433-81.

The process of preparing a semi-finished paint for interior work is carried out in a tank with a capacity of 3500 l using a rotary dissolver for mixing. The semi-finished product is a solution of special additives.

Before starting the loading of raw materials, check: - the cleanliness and serviceability of the equipment and communications of the entire production line;

- shutoff valves;
- serviceability of instrumentation;
- the availability of a set of raw materials in the required quantity.

Then the prescription amount of water is loaded into the tank, the dissolver stirrer is turned on, and the components previously weighed in the prescription quantities are loaded (at low revolutions of the stirrer) in the following sequence - sodium polyphosphate dispersant, excipient OP-7. Mixing of the contents is carried out at a speed of rotation of the mixer 400-500 rpm until complete dissolution and combination of the components for 20 minutes The container must be closed during operation. The preparedness of the semi-finished product is determined by the homogeneity of the sample when pouring onto glass.

Preparation of pigment paste for dispersion is as follows. In a container with a semi-finished product with a working stirrer (300-400 rpm) and local suction turned on, titanium dioxide, chalk, and AP-4 flame retardant are loaded according to the formulation, as a result, a suspension is formed.

The suspension is mixed for 20-30 minutes. After sampling, the stirrer speed is gradually increased to 1500 rpm over a period of 10 minutes. At this speed, the process of dispersion of the pigment paste takes place for 45-60 minutes, after which the required degree of milling (50 μm) is checked for 1 hour, the further process of dispersion is carried out in a bead mill with water cooling. Before applying the paste for dispersion, the bead mill is checked for cleanliness and serviceability. Before dispersing, water is supplied to the jacket of the bead mill container. In the process of dispersion, it is necessary to ensure that the temperature of the paste at the outlet of the bead mill does not exceed 323 K. An emulsifier and a dispersant are introduced into the mixer. After a short 5-10 minute stirring, load the remaining components of the semi-finished product (stabilizer, antiseptic, etc.), and then the whole mixture is stirred for 2 hours. By adding water, the solids content is adjusted to 20-22%.

Thus obtained semi-finished product is loaded into the kneading machine 4; pigment, flame retardant AP-1, is also added there. The quality of the paste of each pigment obtained separately with a pre-selected amount of the semi-finished product in accordance with its moisture capacity depends on the nature of the pigment and the weight ratio of the semi-finished pigment, which is 80:20. A batch is prepared for 60-90 minutes until a homogeneous mass is obtained, which is then transferred to a paint-spraying machine-6. Typically, two passes of the paste through a three-roll paint machine can be enough for a paste with a maximum particle size of 30-45 microns (80-100 microns are often enough for technical specifications).

In the mixer 3, equipped with a paddle mixer, which rotates at a speed of 60-70 rpm, load the prescription amount filtered through a mesh or gauze latex from the tank 1 through the measuring device 2. Then, with stirring, pigment paste is added. Mixing the components in the reverse order is unacceptable, since usually leads to the appearance of significant quantities of coagulum.

After mixing the latex with pigment paste and AP-4 flame retardant, the paint is checked for uniformity (lack of inclusion of large particles) and the standard for color. If necessary, tinting is done with pastes previously diluted with latex in a ratio of 1: 1. In addition, the paint is analyzed for solid content, after which it is filtered and pumped to the receiver-typifier-9, from where after checking all the indicators of the paint, it is poured into a container through a weight or volumetric dispenser-10.

Thus, fire-resistant water-dispersion paint coatings have been developed, which are a dispersed system consisting of heterofunctional silicon-containing flame retardants, obtained on the basis of local raw materials and simplified technology widely used in the production of building materials, including coatings.

Development of production technology and technical conditions for the production of fire-retardant water-dispersion paint based on AP-4 flame retardant.

The technology for preparing waterborne paints and varnishes is similar to that used in the preparation of traditional paints and varnishes containing organic solvents. Dispersing equipment are bead mills. To reduce foaming, pigments are dispersed in minimal amounts of a film former and water, obtaining a highly viscous paste into which the rest of the binder and other components are introduced. The commodity form of water-borne paints and varnishes is a suspension containing 30-80% (by weight) of non-volatile substances. Water-borne paints and varnishes are applied to the fat-free and phosphated surface of ferrous and non-ferrous metals, as well as to wood, plastics, etc. The main application methods are electrodeposition, spraying, spraying, dipping [3].

Paint and varnish materials for external surfaces are exposed to sunlight, humidity, dryness, heat and cold. The combined effects of UV radiation, temperature and humidity quickly destroy organic polymers. External manifestations of such defects are a noticeable loss of gloss and chalking.

The developed technological regulations proposed by the authors and the condition for obtaining coatings with the modified proposed flame retardants apply to the production of fire-retardant water-dispersion paints, which are a suspension of pigments and fillers in an aqueous dispersion of synthetic polymers with the addition of AP-4 flame retardant and various auxiliary substances according to TC 6.12-78-2000 , TC 6.12-28-96, produced by mixing components in mixers and dispersing pastes in bead mills.As a result, the above technology contributes to the production of a new fire-retardant water-dispersion paint with a flame retardant AP-4 modification of an organosilicon oligomeric composition based on sodium metasilicate (liquid glass can be used in production) with sodium tetraborate. Properties of paint before introduction (before modification) - combustibility index Group III refers to combustible materials according to GOST 16363-98 Interstate standard “Fire-retardant products for wood. Methods for determining fire retardant properties.

"The properties of the paint after modification with flame retardant are fire-resistant according to GOST 16363-98, the combustibility index, which is group II, and the paint belongs to G2-low-combustible, with moderate smoke-forming ability and moderate in toxicity of combustion products. These indicators are permissible for the use of paint in places of evacuation on any substrates [4].

The characteristics of the products are the main indicators of the developed technology, offered in two types of paint. Paints are intended for exterior “WDPA-112” and interior, “WDPA -229” works, for painting buildings and structures on brick, concrete, wood, plastered surfaces. Paint is made in various colors. Technical requirements for the production of water-dispersible fire retardant paint are presented in table 1.

Table. 1.

Product Specifications

Name of indicators	Fireproof WDPA -112	Fireproof WDPA -229
	Norms	Norms
1. Paint film color White	Conforms to enterprise standards	Conforms to enterprise standards
2. Appearance	After drying, the paint should form a film with a smooth, uniform matte surface.	
3. White,% not less	84	84
4. Mass fraction of non-volatiles,% not less	45	50-57
5. The degree of milling, microns, not more	60	60
6. The viscosity according to a viscometer of type B3-246 with a nozzle diameter of 4 mm at a temperature of (20 ± 0.5) ° C, not less	40	40
7. pH of the paint	8-9	6-8
8. Hiding power in terms of dry film, g / m2, not more than: White	120	130
9. Washability of paint, g / m2, no more	2	3
10. Drying time to degree 3 at 20 ± 2, oC, h, no more	1	1
11. Frost resistance of paint, cycles, not less	2	-
12. Resistance of paint to the statistical effect of water, at (20 ± 2) ° C, h, at least	24	-

13. Flammability, fire resistance group	II	II

The raw materials used for production must comply with standard requirements [5-6]. In production, the preparation of fire-retardant water-dispersion paints akrilic (WDPA) must strictly comply with the formulations that use the following consumption rates for the production of 1 ton of products. Tables 2–3 show the consumption rate of the proposed fire retardant water-dispersion paints in two types of domestic use: internal and external.

Table. 2.

Fireproof WDPA internal TU 6.12-78-2000

№	Name	Unit. number	(Consumption rate)
1.	Acrylic emulsion 100%	kg	100
2.	Zinc white	kg	174
3.	Filler	kg	405
4.	Excipient	kg	25
5.	Polymer glue	kg	30
6.	Water	kg	300
7.	Flame retardant	kg	6
	Total	kg	1040

Table 3.

Fireproof WDPA external TU 6.12-28-96

№	Name	Unit. number	(Consumption rate)
1.	Acrylic emulsion 100%	kg	150
2.	Titanium dioxide	kg	114
3.	Filler	kg	375
4.	Excipient	kg	25
5.	Polymer glue	kg	30
6.	Water	kg	340
7.	Flame retardant	kg	6
	Total	kg	1040

In the event of a change in the recipe for consumption, a change in the quality of the product occurs or production stops. A decrease in some raw materials can lead to a decrease in viscosity, that is, dilution, or an increase in certain reagents, an increase in viscosity, that is, a thickening of paints that causes the devices to stop working.

The technological process for the production of paint is carried out according to the above technologies and consists of the following stages:

a) receipt and preparation of raw materials;
b) making batches in the dissolver;
c) dispersion of pastes in bead mills;
d) drawing up paints in mixers;
d) cleaning of paints;
e) discharge into containers and delivery of finished products.

The raw materials received at the enterprise are subject to control for qualitative and quantitative compliance with shipping documents. Raw materials are loaded into the dissolver and mixed until a homogeneous mass is obtained. The mixed homogeneous mass from the dissolver is fed to a bead mill for dispersion. Drawing up paints and setting them to a type is carried out in tinting mixers in accordance with loading formulations. The type of paint supplied from the mixer is passed through a cleaning filter. Ready paint from the drain mixers is poured into containers. Packaging, marking transportation and storage of VDAK is carried out by standard methods: Paint packaging - according to GOST 9980.3., Paint marking - according to GOST 9980.4., Transportation and storage according to GOST 9980.5.

Table. 4.

The specification for the main technological equipment

Name of equipment	Number	Material protection methods	Technical specifications
High-speed mixer-1.25m3 (dissolver)	To establish an enterprise	Steel 3	Working capacity -1.25 Electric motor-VAO-22 kW Speed-1500 rpm
MK-120 Bead Mill		painting	Working capacity -120 l Water Consumption-1.8-2.8 m3 Electric motor-VAO-30 kW
MPD-50 Bead Mills		Steel 3	Working capacity -50 l Water consumption - 1,5 m3 Electric motor-20 kW
Gear pumps		painting	DN 50 mm

It is necessary to have technological equipment and production control of VDAK according to the specification for the production of products given in table 4, otherwise it can lead to unnecessary economic costs [7].

The main advantages of water-borne paints and varnishes over traditional paints and varnishes: low content (or absence) of organic solvents, which leads to less fire and explosion hazard in the production and use of water-borne paints and varnishes, their harmlessness, as well as significant savings in organic solvents (200-400 kg per 1 t paintwork material); the possibility of applying to a damp surface, thereby eliminating the operation of drying (or blowing) after preparation for painting; reduction of energy consumption for ventilation of drying chambers.

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