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## **RESEARCH OF POSSIBLE SYNTHESIS OF ALKYD-STYRENE RESINS IN BELARUS**

The article covers possible methods of synthesis of alkyd-styrene resins, formulations and areas of application this film-formers in paint production. The article summarises the main regularities that can can help to get in the future alkyd-styrene resins of home production. Possibility of production alkyd-styrene resins at the chemical companies Republic of Belarus opens wide prospects for creation quick and naturally drying paint materials which will reduce the dependence on imports for Belarusian companies, provide energy savings and correspondingly increase the competitive ability of products of companies by cheapening of coatings preparation processes.

**Introduction.** Currently conventional semifinished alkyd lacquers dominate in the CIS market among the modified film formers, but in recent years due to the constant rise in energy prices there appeared a demand for paint-and-lacquer materials (PLM) that reduce energy costs in the preparation of varnish-and-paint coatings.

Energy intensity degradation of coatings production can be achieved using natural hardening capable of quick forming of coating (drying time – no more than 30 minutes).

Alkyd-styrene resins are a particular class of film-forming materials which are prepared based on these natural quick drying paints.

Styrenated alkyd resins in comparison with unmodified styrene resins have some advantages: faster drying, increased resistance to water and chemicals, their films are less susceptible to brightness reversion. The disadvantages of such resins include the occurrence of defects during drying and solvent resistance reduction that often result in "blistering" (delamination) during application of a secondary layer. The defect of the upper layers of alkyd-styrene resins is their low endurance, especially to scratching, so they are mainly used in undercoating [1].

This binding group is used in formula of anticorrosion paints for painting ships, equipment, equalizing compositions and fillings, both one and multi-layer coatings in natural and hot drying [2].

Alkyd-styrene resins are widely represented currently by foreign manufacturers, but the Republic of Belarus does not produce them though the Belarusian enterprises widely developed the production of coatings based on such film-forming agents. For example, in 2008 JSC "Lakokraska", Lida, started producing of accelerated drying prime coating "Fastpraym" based on alkyd-styrene resin with the addition of pigment and additive suspension [3]. This primer is already in great demand among consumers (total output since the technical conditions were about 100 t) due to its numerous advantages: short drying in natural conditions and good opacity and adhesion, corrosion resistance and high resistance to mineral oils and diesel fuel.

In this regard, there is a constant demand for the film former at the Belarusian enterprises that they have to meet at the expense of purchase of raw materials in the international market. Therefore, the quality and competitiveness of domestic coatings are influenced by such factors as irregular supply, oxidation and loss of raw materials while transporting, as well as the high cost compared with other domestic film formers.

Thus, the creation of alkyd-styrene resins in the Republic of Belarus is one of the urgent problems that can both reduce the dependence on import of Belarusian enterprises and provide energy saving respectively increasing the competitiveness of market at the expense of the cost reduction in coloring process.

**Main part.** To produce domestic alkyd-styrene resins satisfying the requirements of world standards, we analyzed the scientific literature on the preparation, recipes, as well as the possibilities of application of alkyd-styrene resins in paint-and-varnish production.

Such properties of polystyrene as good coloring maintained unchangable for a long time, the exceptional water resistance, resistance to alkalis and high dielectric properties contributed to intensive search for methods of applying it as a filming. Unfortunately, polystyrene has a number of significant shortcomings along with the good properties.

One of the ways of the removal of shortcomings is to combine its positive properties with substance properties which are film-forming themselves.

Getting styrenated drying oils and alkyd resins created the fourth important group of synthetic products for accelerated drying for decorative and protective coatings in addition to phenolic resins, alkyd resins and nitrocellulose [4]. According to the literature, styrene was polymerized with dehydrated oils in the early 1940s for the first time. The first patent for a alkyd resins, modified by styrene was obtained in the UK in 1942 [1].

Styrenated alkyds appeared in commercial quantities in 1948 and occupied a certain place in the fast-drying coatings and hot air drying. Large production capacity of styrene and its relatively low price stimulated its implementation in coating manufacture [5].

The basis of obtaining alkyd-styrene copolymer process is styrene copolymerization reaction with the double bonds of the fatty acid residues of oils [6].

The copolymerization reaction is characteristic for vinyl group. Styrene can be readily copolymerized by any conventional methods: in block or in solution. The process of copolymerization is carried out by heating for 20 hours or more at about 140°C.

Xylene is usually used for copolymerization in the solution. The reaction rate and the amount of the resulting product are changed when xylene is replaced by other solvents. For example, the reaction time increases from 23 to 49 h when xylene is replaced by dipentene. The products obtained from solution dipentene are better aligned but they dry slower. Therefore it is recommended to replace only a part of xylene by dipentene [5].

Limiting styrene content is about 40%. Loading it in large quantities deteriorates compatibility with other film formers as well as it makes worse solubility in solvents; moreover, obtained coatings are brittle. Optimum performance is observed at a ratio of styrene : oil = 40 : 60.

As is known [7], the molecular weight of the polymer depends on a number of factors - the concentration of monomers, temperature, initiator catalyst content and chain regulators in the reaction mixture, etc. As a means of reducing the concentration of monomer may be its slow introduction (droplet method) into heated to a certain temperature of the oil, fatty acid oils or alkyd resins. At the same time the decrease in molecular weight will be determined not only by a low concentration of monomer, but also high content of  $\alpha$ -methylene groups per particle injected styrene that, in addition, will provide polystyrene chains, significantly enriched with oil component. High concentration of  $\alpha$ -methylene groups eliminates the necessity for the introduction of specific growth regulator of circuit. The method of the slow introduction of the monomer allows to obtain homogeneous products with a higher styrene content (up to 70% in the reaction mixture) than conventional mortar method, although a high styrene content (50-70% in the mixture), significantly increases viscosity and reduces elasticity and shock resistance of the films.

A prerequisite to obtain a homogeneous reaction product is the initiator (3–4% by weight of monomer), which contributes not only to accelerate the reaction, but also to decrease molecular weight. As an initiator catalyst of copolymerization process it is better to use alkyl peroxides with relatively high decomposition temperature (tertiarybutyl peroxide). The use of peroxides with low decomposition temperature (lauryl peroxide, benzoyl peroxide, hydroperoxide of isopropylbenzene) gives a smaller effect [8].

Mixture consisting of styrene and  $\alpha$ -methyl styrene enables to obtain homogeneous light products with a large number of oil. The ratio between  $\alpha$ -methylstyrene and styrene is usually 3 : 7.  $\alpha$ -Methylstyrene is very helpful to get compatible products, moderating reaction and slowing entry styrene to copolymer.

Thus, one must regulate the quantitative ratios of the reactants in the copolymerization reaction, considering type and amount of initiator catalyst, temperature and solvent, if copolymerization is carried out in solution. Technically homogeneous products can be obtained when applied the same process conditions.

As the literary analysis showed, fundamentally different methods for alkyd-styrene copolymers are possible:

1) copolymerization of fatty acids of vegetable oils with styrene and further reacting of the obtained copolymer with other components of alkyd oligomers (phthalic anhydride, glycerol) by fatty acid method (method 1);

2) copolymerization of the styrene oil and the subsequent synthesis of the alkyd by the method of using the glyceride with oil copolymer (method 2);

3) copolymerization of monoglycerides with styrene and their further esterification (method 3);

4) copolymerization of preformed alkyd with styrene (method 4).

According to method 1 preparation of an alkyd-styrene resin can be represented as the simplified scheme shown in Fig. 1.

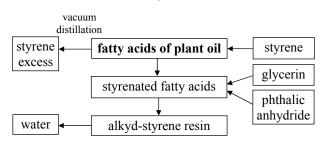


Fig. 1. The scheme for producing alkyd-styrene resin according to method 1

A number of papers concern studying the process of copolymerization of styrene with fatty acids of vegetable oils. In particular, the copolymerization of styrene with  $\alpha$ -eleostearic acid which has three conjugated bonds is under consideration. Tung oil is unique in content of eleostearic acid (80%). This research has made it possible to explain the mechanism of copolymers formation of styrene with fatty acids containing conjugated double bonds.

In this case, this reaction proceeds as in the copolymerization of styrene-butadiene: styrene-butadiene attachment occurs primarily in the 1,4-position, and chain growth of styrene via dienes [9].

Thus, the fatty acids with conjugated double bonds in the molecule (eleostearic, 9,11-linoleic) form two types of products with styrene – the true copolymers of high molecular weight (Fig. 2), and adducts of the Diels-Alder (diene synthesis) formed in the addition of styrene to fatty acid in a molar ratio of 1: 1 (Fig. 3) [8].

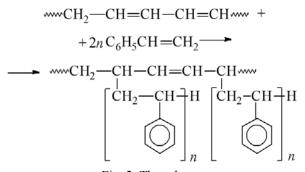


Fig. 2. The scheme for producing alkyd-styrene copolymers

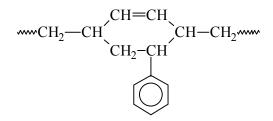


Fig. 3. The adduct of the Diels-Alder

This paper of Shneyderova V. V. deals with copolymerization of styrene with linoleic acid containing two isolated double bonds (9,12-linoleic acid), which is one of the main components of most drying and semidrying oils. Polymerization was accomplished in sealed nitrogen-filled glass ampoules at 150°C with benzoyl peroxide (1% of styrene content) and the molar ratios in the mixture of styrene and linoleic acid 94 : 6, 90 : 10, 86 : 14 and 80 : 20. This paper shows that increasing the molar proportion of fatty acid in the initial reaction mixture dramatically increases the percentage of low molecular weight copolymers of styrene with linoleic acid [10].

It is known [5] that the tung oil acids react with styrene slower than acid of oitisikov oil. De-

hydrated castor oil acids react with styrene with the greatest reaction rate. It is hard to explain, because tung oil has the greatest number and the greatest degree of conjugacy of double bonds. Perhaps the reason for this phenomenon is almost complete copolymerization of styrene with tung oil acids. Because of this, the polymerization of the styrene with the greatest reaction rate occurs to a lesser extent with formation of a certain amount of polystyrene. Since the reaction rate of polymerization styrene is higher than the reaction of copolymerization the styrene flow rate at a particular time must be greater when used dehydrated castor oil acids.

The formulation and technique [5] of obtaining styrene acids of dehydrated castor oil are known (Table 1).

Table 1

Formulation of styrene acids of dehydrated castor oil

Components	Component content, wt %
Acids of dehydrated castor oil	40.9
Styrene	59.1
Total	100.0

Note. Initiator - 3% benzoyl peroxide (from styrene content).

According to this recipe, dehydrated castor oil acids are heated in a flask with stirrer and cooler to 115°C; then when stirred, a mixture of styrene with a catalyst is added for an hour and raises the temperature during this time to about 145°C. This temperature was maintained for 30 minutes, after which the unreacted styrene is subjected to vacuum distillation.

When acids of dehydrated castor oil were styrenated it was established that if all components are all heated, rapid exothermal reaction occurs at 120°C. The product obtained is thus turbid and inhomogeneous representing an incompatible mixture of unreacted fatty acids, a quantity of styrenated acids and a considerable amount of polystyrene. The product is formed by slow addition of a mixture of styrene with the catalyst to fatty acids. It becomes transparent and homogeneous. The presence of free polystyrene is not determined, but it was established that with increasing amounts of the loaded amount of styrene reacting with fatty acids increases. At the same time the molar ratio of styrene to fatty acid is 4 : 1, taken in formulation (see Table 1), is considered to be optimal.

The formulation of obtaining alkyd resins, modified by styrene for synthesis according to method 1 is also known (Table 2) [11].

A mixture comprising dehydrated castor oil acids, styrene,  $\alpha$ -methyl-styrene and benzoyl per-

oxide is put into a flask according to the recipe, and then it is heated to 150–170°C for 3 hours. Then glycerine is added to the derived copolymer and the mixture is heated to 200°C. This temperature has been maintained for 21 hours and then charged with phthalic anhydride. The resulting mixture is heated to 245°C and kept at this temperature for 4 hours.

Table 2
Formulation of alkyd-styrene resin for synthesis
according to method 1

Components	Component content, wt %
acids of dehydrated castor oil	34.8
Styrene	24.4
α-Methylstyrene	10.4
Glycerine	13.1
Phthalic Anhydride	17.3
Total	100.0

Note. Initiator – 2% benzoyl peroxide (from styrene content).

Deriving alkyd styrene resin according to method 2 (Fig. 4) is under consideration.

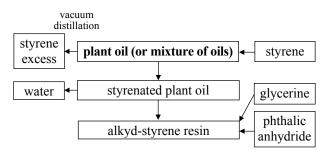


Fig. 4. The scheme for producing alkyd-styrene resin according to method 2

It is known [9] that in 1946 a mechanism of copolymerization of oils with styrene was proposed which is different for oils with isolated and conjugated double bonds. In the first case, the oil serves as a chain transfer agent, playing the same role as the solvent. In the presence of oils with isolated double bonds chain stopping can occur due to unstable hydrogen of the methylene group located in the  $\alpha$ -position to the double bond.

Drinberg A. Ya., Fundyler B. M. and Lifits L. A. have also investigated the copolymerization of styrene with vegetable oils. They found that the copolymerisation reaction runs only with tung oil containing a conjugated bond system. Linseed oil is possible to introduce into the copolymerization reaction, preoxidized it by atmospheric oxygen. Crude sunflower oil as well as oxidized do not enter in the copolymerization reaction with styrene. Copolymerization reaction takes place only when oxidized mixture of sunflower and tung oils are used together and this is sufficient to introduce 5% of tung oil. It is also established that the rate of conversion into a three-dimensional polymer increases with increasing styrene content in the co-polymer [12].

Polystyrenes have relatively low melting, but at normal temperature they are incompatible with the oil. Homogeneous product is formed when heated of oil styrene monomer with reflux condenser and peroxidate catalyst as a result of copolymerization, which proceeds readily with oils having conjugated bonds, and only to a small extent with oils having nonconjugated bonds [5].

When a high content of conjugated double bonds in oils, their copolymerization with styrene can easily cause gelation as a result of the formation of spatial polymers due to crosslinking radicals of fatty acids [9].

Styrene and oil can be copolymerized either in solution or in block. Copolymerization in solution makes it possible to control the process well, but it is slow and, of course, if you need a product without solvent, it must then be removed. Usual formulation comprises 25 parts of oil, 25 parts of styrene and 50 parts of solvent. The mixture is heated until the desired degree of conversion. The residual monomer styrene can be distilled to remove, and it is accompanied by removal of most solvent, which requires subsequent addition of fresh solvent.

Block copolymerization proceeds much faster than in solution, but in this case styrene polymerrizes itself readily as well as it copolymerizes. When copolymerized by this method, the products are incompatible and turbid due to the incompatibility of polystyrene with oil. Therefore, to obtain bright and homogeneous products it is recommended to use a mixture of styrene and  $\alpha$ -methylstyrene [4].

Table 3 shows the possible formulations of styrenated oils and describes the technological copolymerization process in the block.

The table shows that almost all the copolymerization products dry quickly, except made by a mixture of soybean and tung oils. The films produced from a mixture of 50% linseed and 50% dehydrated castor possess the best drying ability. However, the least time-consuming process is the copolymerization of dehydrated castor oil with a mixture of styrene and  $\alpha$ -methylstyrene.

The formulation [6] of obtaining alkyd-styrene resins with oxidized soybean oil according to method 2 is known (Table 4).

Alkyd-styrene resins obtained by this formulation have an acid number of not more than 16 mg KOH/g. The films of these alkyds dry at  $(20 \pm 2)^{\circ}$ C to degree 1 for 22 minutes, and up to degree 3 for not more than 8 hours. They have good elasticity, and resistance to water, alkalis, and solvents.

	Composition of the mixture of oils, wt %			
Components	100% dehydrated castor	90% linseed 10% tung	80% soybean 20% tung	50% linseed and 50% dehydrated castor
Formulations of styrenated oils				
Oil	45	45	45	50
Mixture of styrene and $\alpha$ -methylstyrene	55	55	55	50
Reaction time, h				
Addition of styrene	6	6	6	6
Temperature rise to 250°C	5	6	4	5
Exposure at 250°C	6	6	6	7
Heating 250–285°C	0.5	_	_	-
Heating 250–300°C	-	0.5	0.5	1
Exposure at 285°C	1.5	_	_	-
Exposure at 300°C	-	2.5	3.5	3
Overall process	19	21	20	22
Rates of obtained styrenated oils				
Amount of unreacted styrene, %	1.2	1.5	2.0	1.2
Drying time at temperature $(20 \pm 2)^{\circ}$ C, h, not				
more:				
a) to degree 1	0.75	1	2.5	0.5
b) to degree 3	6.0	7.0	24	5

Features of copolymerization process in the block according to method 2

Notes. In all cases before styrene adding oil was heated to 160°C. Initiator – 3% benzoyl peroxide (from styrene content). Solidification was carried out with a mixture of desiccants: 0.5% Pb and 0.02% Co based on the weight of metal to oil. The dilutant was a mixture of 70% mineral spirits and 30% solvent resulted in obtaining of 60% solution.

Another known method of obtaining styrenated alkyds (method 3) consists in styrenating of monoglycerides and their subsequent esterification (Fig. 5).

Table 4

Components	Component content, wt %
Oxidized soya bean oil	37.7
Styrene	19.1
α-Methylstyrene	8.2
Glycerine	10.9
Phthalic anhydride	24.1
Total	100.0

Formulation of alkyd-styrene resin for the synthesis according to method 2

*Notes.* Initiator -3% benzoyl peroxide (from styrene content). The reaction of transesterification was carried out with 0.01% (based on the oil content) of calcium oxide.

Table 5 shows the formulation of alkyd-styrene resins with a mixture of linseed and dehydrated castor oils to obtain alkyd-styrene resin [6].

According to the formulation given in Table 6, the process of preparation of styrenated alkyd consists of several stages:

1) alcoholysis of oils: linseed and dehydrated castor oil, glycerol and calcium oxide is heated to 230°C and kept at that temperature for about 1 hour to obtain a product which is soluble in methanol, 1 : 3;

2) styrenazing of monoglycerides: the half part of the styrene and the initiator solution are added to monoglycerides and the mixture is heated to  $160^{\circ}$ C, then a reflux condenser on and the residue of styrene is added for 3 hours. Then in the next 4–5 hours the temperature is raised to  $210^{\circ}$ C;

3) esterification for obtaining of the styrenated alkyd: phthalic anhydride is added to styrenated monoglycerides and xylene is added for azeotropic solution. The mixture of these substances is heated at 215–230°C for about 3 hours to achieve the required viscosity and acid number, after which the resin is dissolved and filtered.

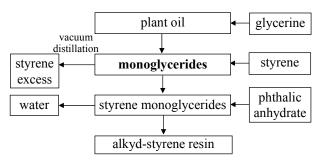


Fig. 5. The scheme for obtaining of alkyd-styrene resins according to method 3

Such a styrene-alkyd resin has an acid number of not more than 7 mg KOH/g. Films of alkyds dry at  $(20 \pm 2)^{\circ}$ C to 1 degree for 10 minutes.

Table 3

Components	Component content, wt %
Linseed oil	19,0
Dehydrated castor oil	19.0
Glycerine	8.7
Styrene	35.0
Phthalic anhydride	18.3
Total	100.0

Table 5 Formulation of alkyd-styrene resin for the synthesis according to method 3

*Notes.* The reaction of transesterification was carried out with 0.06% (based on the oil content) of calcium oxide. 4.62% (by weight basis) of xylene was added for azeotropic distillation of water of reaction. Copolymerization reaction initiator – 0.85% (the content of the styrene) of cumene hydroperoxide (73% solution in xylene).

The most common method of obtaining industrial alkyd-styrene resins is method 4 (Fig. 6).

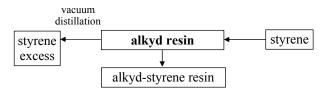


Fig. 6. The scheme for obtaining alkyd-styrene resin according to method 4

Low-viscosity alkyd is used for copolymerizable with styrene. Homogeneous resins with good properties are obtained when applying medium fatty alkyd resins. Fatty acids with conjugated double bonds, are used, unsaturated dibasic acids or a mixture of saturated dibasic acids with a small amount of maleic anhydride are used in the synthesis of alkyd.

Maleic anhydride is involved in the reaction of a polyesterification and serves the source of both double bonds necessary for copolymerization with styrene. The maleic anhydride content in alkyd should be precisely calculated. Optimal conditions for the copolymerization are provided for the introduction of maleic anhydride in the amount that one double bond of maleic anhydride is on 3 molecules of phthalic anhydride. In this case the final product is transparent and has a relatively stable viscosity [13].

Further copolymerization process is carried out at about temperature from 140 to 170°C in xylene with initiator. The main initiator is di-tret-butyl peroxide, the amount of which depends on the temperature and varies from 1 to 4% from weight of monomer. Pre-oxidation and polymerization of oils (linseed, sunflower) increases the yield of the copolymer. The copolymerization involves method, in which styrene is gradually introduced into the mixture for several hours together with an initiator. After addition of monomers and initiators the reaction temperature is maintained; the degree of conversion is determined by measuring the mass of the non-volatile part. If necessary, for the completeness of the reaction additionally inject a small amount of initiator. The quantitative ratio of the alkyd resin and the copolymer is generally from 60 : 40 to 85 : 15. The reaction is usually carried out until it terminates at 95–97%. The residue of unreacted styrene is removed under suction at the end of the process.

To improve the odor and light-fastness it is desirable to remove small amounts of residual monomer styrene. For this,  $CO_2$  is blown through the reaction product before it is left to cool. The content of volatile substances in the drying oils negligible, and thus, the degree of conversion can be estimated by collecting matter removed by blowing [4].

The formulation of a styrene-alkyd resin for synthesis based on glyptal resin is known by me-thod 4 (Table 6) [14].

Table 6

Formulation of alkyd-styrene resin for the synthesis according to method 3

Components	Component content, wt %
Glyptal resin (50%-concentration)	50.0
Styrene	25.0
Xylene	25.0
Total	100.0

*Note.* Initiator -2% tertiary butyl peroxide (from styrene content).

Xylene solution of glyptal resin (50%), styrene and xylene are placed into flask. The resulting mixture is stirred for 30 min and is heated. When temperature runs up to temperature  $140^{\circ}$ C, 50% solution of t-butyl peroxide or other peroxide in xylene is given into the reaction mixture in four equal portions at intervals of 1.5 h. Aggregate exposure is carried out at 140°C for 20–25 h to obtain a dry residue 49.5–50.0%. The viscosity of the reaction solution should be 45–50 seconds. Then the solution is cooled to room temperature and filtered.

Alkyd-styrene resin obtained by this formulation has an acid value of not more than 7 mg KOH/g, the drying time up to 3 degrees at  $(20 \pm 2)^{\circ}$ C – less than 8 hours.

Depending on the styrene content styrenealkyd resins can be divided into 3 groups [8]:

1) resins containing 30% of styrene and more; they dry faster, diluted with white spirit and are suitable after dilution for brushing. On the basis of these resins solid, water-resistant, fast drying coating are obtained (drying from dust for 20 minutes, the real drying 1.5 h). Lacquers and enamels, based on these resins have a high concentration of filmforming substance and they may be applied for painting the chassis of motor vehicles, heavy machinery and equipment, cables, etc. and decks;

2) resins containing 15–25% of styrene are used and in the primers and in baking enamels. Particularly their use is appropriate with melamine-formaldehyde resins with which they conjunct well. The obtained coating of baking possess have high hardness, luster, resistance to water, alkalis and detergents, as well as weather resistance. They can be used for dyeing of washing machines, etc.;

3) resins containing 10% of styrene are well diluted with white spirit, and can be brushed. These resins can be used for colouring of the inside of a building.

Alkyd-styrene resins are available as solutions in white spirits or xylene. For quick hardening films of air drying xylene solution is more profitable, but for grinding pigments and improving coatings with high gloss one should be applied the solution in slowly evaporating white spirit [5].

Film formation occurs primarily through physical drying (evaporation of solvent), as well as oxidative polymerization due to the remaining double bonds of the fatty acid residues of oils. Air curing is carried out with siccatives [15].

It is known [5] that usual combination of lead and cobalt driers are added to the coatings of airdrying based on styrenated alkyds. The amount of the metal input depends on the type of pigment, as well as in conventional oil and alkyd lacquers. In styrenated alkyds, drying at 120°C or below is put a small amount of cobalt, and drying at a temperature above 120°C driers are not usually put.

**Conclusion.** This paper deals with possible methods of obtaining, formulations, as well as the possibility of applying alkyd-styrene resins in paint and varnish production.

On the basis of this work one can distinguish 4 main methods of the synthesis of alkyd-styrene resins which have their own characteristics, advantages and disadvantages. But the most common method is copolymerization of alkyd with styrene. Synthesis of alkyd-styrene resins by this method can be carried out on the production lines of JSC "Lakokraska" Lida.

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