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STUDY OF THE USE OF PHENOLIC EXTRACT AS A PLASTICIZER IN THE PRODUCTION OF LINOLEUM

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ABSTRACT

The use of phenolic extracts from the purification of petroleum oils as secondary plasticizers in the production of composite materials, in particular, linoleum on a polyvinyl chloride (PVC) basis, was investigated. Partial replacement of expensive components - dioctyl phthalate and chlorinated paraffin - with a cheaper by-product of oil refining - phenolic extract from the purification of petroleum oils was studied. A technology for using phenolic extract in the production of PVC linoleum was developed. The results indicate higher physicochemical properties of linoleum obtained from the proposed polyvinyl chloride composition than those from a composition without phenolic extract. It has been established that satisfactory physical, chemical, mechanical and sanitary-hygienic properties of PVC linoleum are achieved by adding phenolic extract to the plasticizer in a ratio of dioctyl phthalate - chloroparaffin - phenolic extract equal to 3:1:1. The optimal ratio of the composition components was determined, wt.%: PVC - 51-55.5; plasticizer - 21.5-29; calcium stearate or urea - 0.6-0.8; titanium dioxide - 2.7-3.1; asbestos - 11.6-24. The proposed polyvinyl chloride linoleum has high strength, very high resistance to abrasion, is not subject to decay, has low thermal conductivity, and is very hygienic.

Keywords: Phenolic Extracts, Oil, Secondary Plasticizers, Linoleum, Polyvinyl Chloride.

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INTRODUCTION

Recently, along with the growth of production of film materials and linoleum, the requirements for their operational characteristics are also growing. Today, it is relevant to extend the service life of polymeric materials, reduce the cost of the target material by using cheap and affordable additives based on local raw materials.[1,2,3] The use of a mixture of plasticizers for polyvinyl chloride (PVC) is becoming increasingly important, which allows for the partial replacement of expensive plasticizers with cheaper ones and the wide variation of the properties of the plasticizer. The relevance of creating polyvinyl chloride linoleums that meet the increased requirements of international standards and the need to further improve their physical and mechanical properties using large-tonnage industrial waste,[4,5] in particular waste from oil refining industries,[6,7,8] served as the basis fo solving a number of scientific problems in research and finding ways to fill and plasticize polyvinyl chloride products.[9,10,11] The use of a mixture of plasticizers for polyvinyl chloride is becoming increasingly important, which makes it possible to partially replace expensive plasticizers with cheaper ones and vary the properties of the plastic over a wide range.[12,13]

Polyvinyl chloride linoleum is a roll material made of plastic mass-polyvinyl chloride. This type of linoleum is produced on a fabric base or baseless. The latter can be single-layer, double-layer, or multi-layer. In

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addition, they produce heat- and sound-insulating linoleum on a felt or porous base and on parchment.[14] The production of each type of linoleum has its own characteristics, technology, and equipment. Even the production of the same type of linoleum can have its own specifics. For example, the composite mass was applied to the fabric base in various ways, which in turn affects the quality of the linoleum produced. Polyvinyl chloride linoleum has great strength, very high resistance to abrasion, is not subject to decay, has low thermal conductivity, and is very hygienic.[15] The characteristics and physical and mechanical properties of polyvinyl chloride linoleums were given in Table-1.

Table–1: Characteristics of Polyvinyl Chloride Linoleums

Linoleum	Length of piece in m, min	Width of the canvas in mm	Web thickness in mm	Weight of 1 m^2 in kg	Weight loss due to abrasion on the MI-2 device in g/cm^2, max	Water absorption in 24 hours in %, max	Hardness according to the TShR-2 hardness tester in mm, max	Elasticity in %, min
On a fabric base	12	1400-1600	2.0-2.5±0.2	2.5-3.0	0.06	5	0.7	40
Baseless (single-layer and multi-layer)	12	1400-1600	1.5-2.5±0.1	2.8-4.5	0.05	4	0.3	40
On heat and sound insulating (felt or porous)	12	1400-1600	4.0-6.0±0.2	1.5-2.0	0.03	4	-	40

In addition to plasticizers, binders, thinners, fillers, dyes, and a fabric, felt, or porous base were used to produce polyvinyl chloride linoleum. Polyvinyl chloride was used as a binder. Polyvinyl chlorides are thermoplastic and are characterized by a linear structure of macromolecules. Polyvinyl chloride does not have noticeable toxicity, but when inhaled, it causes irritation of the respiratory tract. The maximum permissible concentration of polyvinyl chloride dust in work areas should not exceed 6 mg/m^3. Settled dust is fire-hazardous; its autoignition temperature is 1100°C. Polyvinyl chloride is resistant to acids and alkalis.[16] Polyvinyl chloride obtained by polymerization of vinyl chloride using the emulsion method was used for the production of linoleum. Good results were achieved by using different grades of polyvinyl chloride at the same time. A polyvinyl chloride composition for linoleum is known, including polyvinyl chloride, talc, asbestos, dioctyl phthalate, zinc white, resin, iron red lead, calcium stearate, and lead silicate.[17] The most well-known is the polyvinyl chloride composition for linoleum, including polyvinyl chloride, a plasticizer - a mixture of dioctyl phthalate and chlorinated paraffin in a ratio of 7:3, a heat-stable stabilizer - calcium stearate or urea, an inorganic filler - titanium dioxide, stearox, asbestos in the following ratio of components, wt%: polyvinyl chloride - 50.0; dioctyl phthalate - 14.0; chlorinated paraffin - 6.0; titanium dioxide - 2.0; stearox - 1.0; asbestos - 24.0; urea or calcium stearate - 2-3. However, the disadvantages of the known mixture are the high cost of linoleum and low resistance to the effects of fungal mold.[18] Depending on the production mode and the composition of the emulsion mixture, not all brands have the paste-forming properties necessary for the production of linoleum when mixed with plasticizers. Talc, barite (heavy spar), chalk, kaolin, and asbestos are most often used as fillers. When producing multi-layer linoleum, a particularly large amount of fillers is contained in the lower layers of linoleum that do not wear out. The filler must be finely dispersed and resistant to chemical reagents, not swell in water, not decompose at operating temperatures of production, be homogeneous, and not contain foreign inclusions.

Plasticizers are used to reduce intermolecular attractive forces and to make the material more flexible. The following requirements are imposed on plasticizers: minimal volatility, chemical stability, absence of odor, low melting point, low hygroscopicity, high flash point, resistance to light, compatibility with the plasticized polymer, and viscosity that ensures good processing of pigments into paste on paint grinding machines. The group of plasticizers is quite extensive. The most commonly used plasticizers are dibutyl phthalate, dioctyl phthalate. Although tricresyl phosphate is active as a plasticizer, it is toxic. The aim of the work is to study the use of phenolic extracts from the purification of petroleum oils as secondary plasticizers in the production of linoleum on a polyvinyl chloride basis.

EXPERIMENTAL

Recipe for Obtaining Linoleum

Phenolic extract from selective purification of petroleum oils is a by-product of oil refining and is a liquid from brown to dark brown in color. It consists of wt%: resins 31.75-43; aromatic hydrocarbons 34-50; paraffinic + naphthenic hydrocarbons 18.6-23.0. The composition was obtained as follows: a plasticizer was first prepared - a mixture of dioctyl phthalate (DOP), chloroparaffin (CP), and phenolic extract (PE) from the purification of petroleum oils.[19,20] Polyvinyl chloride was mixed with a plasticizer, titanium dioxide, asbestos, calcium stearate, or urea. The mixture was placed in a mixer and stirred until a homogeneous mass was obtained. Then it was fed into a plasticizing mixer, where, after being heated by friction to 130-150°C, it passed into a plastic state. From the plasticizing mixer, the composition was fed to a four-roll calender (roller temperature 110-125°C), where the linoleum sheet was formed.[21] For the production of linoleum, suspension polyvinyl chloride grade PVC-S-6358M was usually used. The grade was decoded as follows: the first four letters denote suspension polyvinyl chloride; the next two digits indicate the lower limit of the value of K, and the last letters, which are present in three special-purpose grades, denote: HS - heat-stabilized, H – hard, and S - soft.[22]

Production of Baseless Linoleum by the Roller-Calender Method

The roller-calender method is used in the production of both single-layer and multilayer baseless polyvinyl chloride linoleum. Extenders are used only in a mixture with primary plasticizers and in the preparation of polyvinyl chloride composite materials, in particular, linoleum. it is advisable to use mixtures of plasticizers.

Determination of Abrasion Resistance of Samples

Samples for testing were cut from the obtained fabric, and abrasion resistance was determined on a Zwick 6103 testing machine manufactured by Zwick Roell (Germany), including change in linear dimensions, absolute residual deformation during indentation, bond strength between layers, and specific volume resistance.

Sanitary and Hygienic Assessment of Linoleum Samples

The sanitary and hygienic assessment of linoleum samples was carried out using the gas-liquid chromatography method on the LHM-7A chromatograph with a flame ionization detector. The sample to be tested was preliminarily placed in a 2-liter metal cylinder. To determine the resistance of PVC linoleum plasticized with PE to the effects of fungal mold, a two-week fungal culture grown on a Czapek-Dox nutrient medium at a temperature of 28±2°C was used. The test samples were placed in sterile Petri dishes with Van Interson medium, then inoculated with a spore suspension. After sowing, they were placed in a thermostat with a temperature of 27°C, relative humidity of 60%, and pH of the medium of 5-8. Linoleum samples without PE were affected by fungi within 60 days, especially in slightly acidic environments. With the addition of the PE plasticizer, the induction period increased to 20 days.[23]

RESULTS AND DISCUSSION

Physicochemical Properties of Linoleum

Partial replacement of expensive components - dioctyl phthalate and chlorinated paraffin - with a cheaper one - a by-product of oil refining (phenolic extract from the purification of petroleum oils) was studied.

The compositions developed during the experiment and the physicochemical properties of the resulting linoleum are given in Table-2. The data presented in Table-2 indicate high physical and chemical properties of linoleum obtained from the proposed polyvinyl chloride composition than from a composition without phenolic extract. As a result of the experiment, an increase in the resistance of linoleum to the effects of fungal mold was established: the one obtained from the composition without PE was affected after 60 days; the one obtained from the composition using PE was affected after 80 days.

Table-2: Physicochemical Properties of Linoleum

Composition and properties of the composition	Examples				
	1	2	3	4	Known composition
Polyvinyl chloride	52.85	51.0	55.5	55.5	50.0
Plasticizer					
Dioctyl phthalate	13.5	12.9	17.4	17.4	14.0
Chloroparaffin	4.5	4.3	5.8	5.8	6.0
Phenolic extract from cleaning petroleum oils	4.5	4.3	5.8	5.8	-
Titanium dioxide	3.1	2.9	2.7	3.1	2.0
Calcium stearate	0.75	-	0.6	-	3.0
Urea	-	0.76	-	0.8	-
Asbestos	20.8	20.6	12.2	11.6	24.0
Stearox	-	-	-	-	1.0
Abrasion, μm	36.5	37.0	26.5	36.0	37.0
Deformability under indentation, mm	0.24	0.28	0.32	0.34	0.4
Absolute deformation, mm	0.08	0.10	0.15	0.18	0.20
Change in linear dimensions, %	0.6	0.65	0.8	0.75	1.0

Investigation Compatibility of the Phenolic Extract with Industrial Plasticizers

Polyvinyl chloride composition for linoleum includes polyvinyl chloride, plasticizer - a mixture of dioctyl phthalate (DOP) and chloroparaffin (CP), calcium stearate or urea, inorganic filler - titanium dioxide and asbestos. The plasticizer additionally contains a by-product of oil refining, phenolic extract (PE) from the selective purification of petroleum oils in a ratio of DOP: HP: PE equal to 3:1:1. The proposed composition for linoleum has the following ratio of components, wt%: polyvinyl chloride - 51-55.5; plasticizer - 21.5-29.0; calcium stearate or urea - 0.6-0.8; titanium dioxide - 2.7-3.1; asbestos - 11.6-24. Table-3 shows the requirements for PVC-S-6358M grade polyvinyl chloride and the standards for three grades. In the production of linoleum, plasticizers were used to reduce intermolecular attractive forces and to make the material more flexible.

Table-3: Technical Standards for PVC-S-6358M Grade Polyvinyl Chloride

Indicators	Standards for varieties		
	Higher	1st	2nd
The value of K	63-65	63-65	63-65
Bulk density, g/cm^3, min	1.4	1.38	1.34
Moisture and volatile matter content, %, max	0.3	0.4	0.5
Ash content, %, max	0.02	0.02	0.02
Bulk density, g/cm^3	0.45-0.55	0.45-0.55	0.45-0.60
Duration of absorption plasticizer, min, max	10	30	40
Dispersion:			
Residue on sieve with mesh No. 025, %, max	1.0	1.0	1.0
Residue on sieve with mesh No. 0063, %, max	80	50	-

The following requirements are imposed on plasticizers: chemical stability, minimal volatility, no odor, low flash point, resistance to light, compatibility with the polymer, and viscosity that ensures good processing of pasty mixtures on rollers of various purposes.[24] The further objective of the study was to investigate the compatibility of the phenolic extract with industrial plasticizers. As a result of the study, the optimal composition of a non-stratifying at room temperature mixture of extracts with a secondary plasticizer chloroparaffin, as well as with a primary plasticizer dioctyl phthalate was determined: PE:DOP: CP=(15-25):(55-65):(15-25). Experiments were conducted to obtain linoleum samples in laboratory conditions with a PE content in a mixture of DOP and CP plasticizers from 0 to 40% by weight.

The dependence of the physical and mechanical properties of linoleum, the processability of the mass, and the presence of sweating of oily substances on the surface of the samples on the content of phenolic extract was determined. With an increase in phenolic extract over 35%, the processability of the mass worsens, its processing becomes difficult, over 30% - the physical and mechanical properties of linoleum decrease, over

23% - sweating of oily substances appears on the surface. The optimal content of PE in the composition was 20%.
Using existing technology and PE, linoleum of the VK and MP types (linoleum grades) was manufactured according to the following recipe (kg) (Fig.-1 and 2).

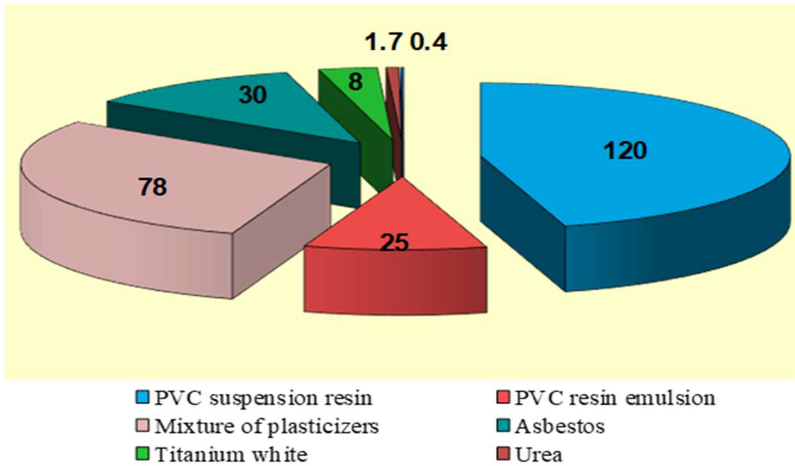

Fig.-1: Recipe for obtaining linoleum of the VK-S brand (kg)

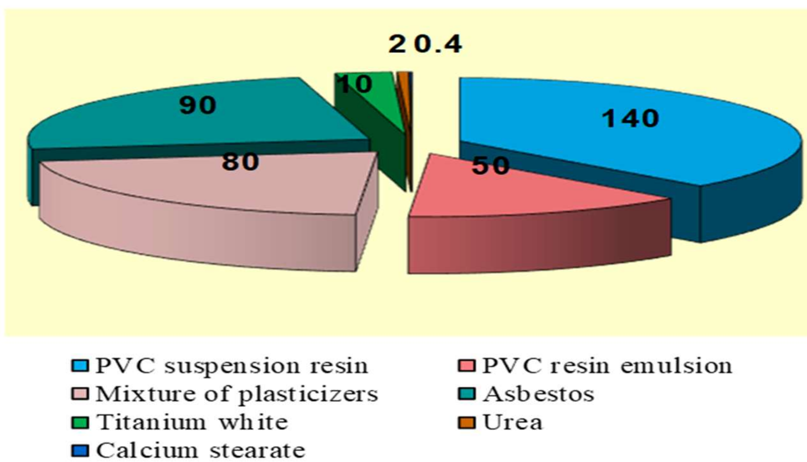

Fig.-2: Recipe for Obtaining Linoleum of the MP Brand (kg)

It was found that the processability of the mass during processing on a roller-calender line is satisfactory. In terms of the physical and mechanical properties of the front surface, linoleum is not inferior to serial products (Table-4).

Table-4: Physical and Mechanical Properties of Linoleum Plasticized with PE

Indicator	VK-S		MP	
	BS EN ISO 10874:2012	Actual indicators	BS EN ISO 10874:2009	Actual indicators
Width, mm	1500±10	1505	1500±10	1500
Abrasion, µm	90	38.5	60	24.5
Change in linear dimensions, %, max	-	-	±1.0	- 0.6 / + 0.1
Informativity in indentation, mm, max Absolute absolute - residual	 1.6 -	 1.24 -	 0.4 0.2	 0.24 0.08
Bond strength between layers, kgf/cm^2, min	0.3	0.3	0.8	1.44

Sanitary and Chemical Studies of Linoleum

Sanitary and chemical studies were conducted under simulated conditions on the possibility of migration of hydrogen chloride, DOP, unsaturated and aromatic hydrocarbons during operation of prepared linoleums at temperatures of 20 and 40°C. It was established that the migration of volatile components into the air from linoleum plasticized with PE is significantly less than from the standard. Its back side and side cuts were sealed with silicate glue. Samples of the gases released were taken at a volume saturation of 0.4 m3/m^3 with material and an air exchange of 0.5 rev/h at 20°C. Concentration of the released volatile products was carried out by freezing in hollow loops at -78°C. The first analysis of the samples was carried out after two weeks of weathering: a large amount of volatile substances pentene-2 and hexene-1 was found. Then the linoleum samples were weathered for another two weeks. The results of the analysis were presented in Table-5. Based on the calculation of average values of concentrations of identified substances (Table-5), according to permissible levels (PL) and maximum permissible concentrations (MPC), the indicators of «total toxicity» were determined. The studied samples of PVC linoleum, containing up to 30% of the mass of PE (by the sum of plasticizers), meet the requirements of the international quality and safety standard in terms of sanitary and chemical properties and can be used in housing construction.[25]

Table-5: Concentration of Substances Released from Linoleum

Identified substance	Concentration, mg/m^3				MAC*, mg/m^3
	Standard with FE-1	Baseless with a printed area	Standard with FE-1	Baseless with a printed area	
Pentene-2	0.320	1.200	0.140	0.200	3
Benzene	0.008	0.018	0.997	0.015	0.08
Toluene	0.015	0.042	0.020	0.030	0.60
Ethylbenzene	0.010	0.030	0.010	0.015	0.02
Cyclohexanone	0.008	0.120	-	-	0.04
Kumol	0.012	0.024	0.010	0.020	0.01
Mesitylene	-	-	0.012	0.010	-

Note-* The permissible level for ethylene is used

Furthermore, taking into account the specific conditions of use of the coatings, the resistance of PVC linoleum plasticized with PE to the effects of fungal mold was determined. The data presented indicate the possibility of using linoleum with a new plasticizer as a material that is biologically stable in various environments. For a more reliable hygienic assessment of the material, a toxicological experiment was conducted under conditions simulating the use of the coating in practice: 60 white rats weighing 130-160 g were subjected to chronic inhalation baiting for 4 months. The animals were divided into two groups, 20 individuals in each. The control group was placed in a chamber into which clean air was supplied. During the experiment, various integral and specific tests were used to characterize the negative impact of chemical ingredients migrating into the air:

- Systematic observation of the general condition and behavior of animals (appearance, motor activity, appetite, etc.)
- Determination of animal weight in dynamics
- Study of the nervous system function by determining the chronaxie of antagonist muscles using the Pure Enrichment BD15525 TENS electronic pulse stimulator
- Study of the morphological composition and peripheral blood
- Determination of cholinesterase in the blood using the Pokrovsky method
- Weight coefficient of internal organs
- Vitamin C content in the blood

A toxicological experiment confirmed that replacing DOP and its plasticizing mixture with HP with PE up to 30% does not have a toxic effect on the body of experimental animals with chronic round-the-clock refueling.

CONCLUSION

Thus, technological solutions have been developed for the use of PE in the production of PVC linoleum as a secondary plasticizer. Satisfactory physical, chemical, mechanical and sanitary-hygienic properties of

PVC linoleum are achieved by adding PE to the plasticizer in a ratio of dioctyl phthalate - chloroparaffin - phenolic extract equal to 3:1:1. The optimal ratio of the composition components has been determined, wt%: PVC - 51-55.5; plasticizer - 21.5-29; calcium stearate or urea - 0.6-0.8; titanium dioxide - 2.7-3.1; asbestos - 11.6-24. The results of laboratory studies and experiments allow us to conclude that the FE product - an extract of selective purification of petroleum oils of fractions II and III can be used as a plasticizer for carbon-chain polymers with side active functional groups. In the process of processing under production conditions, FE showed excellent processability with the PVC composition. Its application will allow, without changing the basic technological regulations, to obtain single-layer polyvinyl chloride linoleum, double-layer polyvinyl chloride roll floor covering with a printed pattern, and polyvinyl chloride linoleum on a heat-insulating base with savings on the scarce and expensive primary plasticizer dioctyl phthalate and chlorinated paraffin. The conducted studies show the possibility of using phenolic extracts in the manufacture of rubber compounds, PVC film materials, artificial leather, and cable plasticizer.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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