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USING BITUMEN EMULSIONS BASED ON OXIDATED, DISTILLATION AND MODIFIED OXIDATED BITUMENS FOR SLURRY SEAL PRODUCTION

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Abstract. Three commercial road emulsions on the basis of petroleum bitumens differing by their production method (oxidated, distillation and modified bitumens) and Redicote E-11 emulsifier were obtained. The obtained emulsions were used for the production of Slurry Seal and thin-layer road coatings on its basis. The coatings based on modified oxidated bitumen have more advantages compared with those based on oxidated bitumen. The expensive distillation bitumen may be exchanged for modified oxidative one to use it in a Slurry Seal technology. We propose to use the indene-coumarone resin for oxidated bitumen modification to improve its adhesive and cohesive properties.

Keywords: slurry seal, thin-layer coating, bitumen, bitumen emulsion, modifier, indene-coumarone resin.

1. Introduction

Every year the load on the road coatings increases due to the increase in number and tonnage of vehicles. The logical result is the increase in intensity and velocity of roadways destruction.

Production of thin-layer coatings (TLC) from Slurry Seal is one of the modern technologies allowing to solve the problem of protecting the roadway upper layers and fast recovery of their operational properties Slurry Seal is obtained *via* mixing macadam (with the particles size of less than 15 mm) or undersized crushed-stone, bitumen emulsion (BE), water, mineral filler, and regulator of emulsion lability at the ambient temperature not below 298 K. The main aim of the process of TLC production from Slurry Seal is extension of the service life of the existing coatings by cracks and small pot-holes pouring, considerable increase of roughness and adhesive properties, improvement of coating evenness, prevention of crack formation, protection from water saturation, and rebuilding of worn or preservation of the existing coating upper layer [1-3]. While using bitumen emulsions and Slurry Seal there are a lot of advantages compared with hot asphalt concrete technologies [4]:

- decrease of deleterious effect on the environment;
- lower power-consuming of the technological process due to the absence of drying, heating of stone materials and bitumen, maintaining of high temperature while stirring;
- combination of Slurry Seal preparing and laying in the same machine during its motion along the road;
- prolongation of the season for building due to the possible works at the temperatures of 278 K and higher;
- possibility to work with wet coatings.

To produce the high-quality Slurry Seal the expensive and deficit distillation bitumen is usually used. The mixtures produced according to Slurry Seal technology are obtained from oxidated petroleum bitumen with poor adhesive and cohesive properties. Inadequate adhesive properties lead to the destruction of TLC produced on its basis; low speed of required strength gain causes impossibility of fast recovery of vehicular traffic after the works.

One of the ways of improving the operational properties of the oxidated petroleum bitumen is its modification by adhesive, polymeric and cross-linking additives [5]. To produce bitumen modified by polymers (BMP) a series of modifiers is used, in particular Elvaloy

4170 (DuPont company), which is a copolymer of ethylene with butylacrylate and glycidylmethacrylate [6], thermo-elastoplasts of styrene-butadiene-styrene type Kraton D (Kraton Polimers company), synthetic latexes of Butonal NS type (BASF company) [7, 8], *etc*. However, in Ukraine the share of modified bitumen used in the road building is only 1–5 %. The reason is high cost of the modifiers.

The analysis of literature data [9] confirms that indene-coumarone resin (ICR) has high adhesive and emulsifying properties. This suggests that ICR can be used as a modifier for bitumen and bitumen emulsions. ICR is a copolymer of indene, coumarone, styrene and their derivatives; its cost is considerably lower than that of the above-mentioned modifiers [10]. Data represented in Ref. [11, 12] prove the possibility of ICR using as a modifier for bitumen modification with the aim of increasing its softening temperature and adhesion to mineral materials.

The raw material for ICR production is one of by-products of coke production – indene-coumarone fraction (ICF) often called as "heavy benzene" [13]. ICF is a distillation product of gasoline fractions which are obtained, in their turn, *via* thermal destruction of coal organic matter during its coking. Nowadays indene-coumarone resins are not produced in Ukraine. The reason is that the main consumer (lacquer-paint industry) changed the resins for cheaper and more effective components. Therefore, application of ICR for bitumen modification not only improves the operational properties but allows to use the by-product (ICF) of coke production.

There are several industrial ICRs differing by softening temperature (T_s) [14]. It is logically to use resins with high T_s to decrease the modifier amount necessary to achieve the required softening temperature of BMP. On the other hand, it is unknown how the change of T_s will affect the adhesion properties of the modified bitumen.

Thus the aims of this work are:

- study of the effect of ICR softening temperature on the adhesive properties of the modified bitumen;
- obtaining of bitumen and bitumen emulsion modified by ICR;
- comparison of properties of bitumen emulsions based on modified bitumen with those based on non-modified oxidative and residual (distillation) bitumen;
- application of bitumen emulsions (including those modified by ICR) for Slurry Seal production.

2. Experimental

2.1. Initial Materials

The fraction 413–463 K was used as a raw material for ICR production. The fraction is obtained from wide ICF fraction withdrawn at JSC "Yasynovsky coke plant" (Donetsk, Ukraine). $TiCl_4$ was used as a catalyst. The characteristics of raw material are represented in Table 1.

To obtain BMP and BE we used: viscous oxidated road bitumen BND 60/90 withdrawn at JSC "Ukrtatnafta" (Kremenchuk, Ukraine); distillation bitumen Nynas 70-100 (Nynas company, Sweden); emulsifier Redicote E-11 (Akzo Nobel, Sweden). The official distributor of the latter two components is "Prologue TD" Ltd company (Lviv, Ukraine). The characteristics of bitumens are represented in Tables 2 and 3.

To obtain Slurry Seal we used: fraction 0–10 mm of granite scree (JSC "Tomashgorsky scree plant, Tomashgorod, Ukraine); portlandcement PC II/A-Sh-400 (JSC "Mykolaivcement", Mykolaiv, Ukraine); regulating additive – 10% aqueous solution of Redicote E-11.

Table 1

Characteristics of indene-coumarone fraction boiled within 413-463 K

Temperature, K	Yield of the fraction 413–463 K, wt %	Bromine number, g Br_2/100 g product	Content of unsaturated compounds, wt %[1]
412	0.0	74.12	63.47
413	10.0		
413	20.0		
414	30.0		
415	40.0		
419	50.0		
423	60.0		
428	70.0		
433	80.0		
441	90.0		
453	95.0		
478	99.5		

[1]Average molecular mass of unsaturated compounds present in the fraction 413–463 K is equal to 138

Table 2

Characteristics of road bitumen BND 60/90

Index	Actual value	Standard according to DSTU 4044-2001 [15]
Penetration at 298 K ($m \cdot 10^{-4}$)	72	61–90
Softening temperature (ball & ring method), K	322	320–326
Ductility, м$\cdot 10^{-2}$ (cm) at 273K at 298 K	 3 84	 ≥ 3 ≥ 55
Change of properties after heating:		
Change of weight after heating, %	0.12	≤ 0.8
Residual penetration, %	72	≥ 60
Change of softening temperature, K	4	≤ 6
Fraaß breaking point, K	255	≤ 255
Flash point determined in open firepot, K	557	≥ 503
Adhesion to glass, %	32	Not standardized
Low-temperature adhesion, %	28	Not standardized
Mass part of paraffins, %	8.0	Not standardized
Solubility in organic solvents, %	99.5	≥ 99.00
Penetration index	-0.6	-2.0 ± 1.0

Table 3

Characteristics of road bitumen Nynas 70/100

Index	Actual value	Standard according to EN 12591-2009 [16]
Penetration at 298 K ($m \cdot 10^{-4}$)	85	61–90
Softening temperature (ball & ring method), K	321	320–326
Change of properties after heating:		
Change of weight after heating, %	0.8	≤ 0.8
Residual penetration, %	46	≥ 46
Change of softening temperature, K	4	≤ 6
Fraaß breaking point, K	262	253–263
Flash point determined in open firepot, K	505	≥ 503
Solubility in organic solvents, %	99.4	≥ 99.00
Density at 298 K, g/cm^3	1.06	1.0–1.07
Kinematic viscosity at 408 K, mm^2/s	231	≥ 230

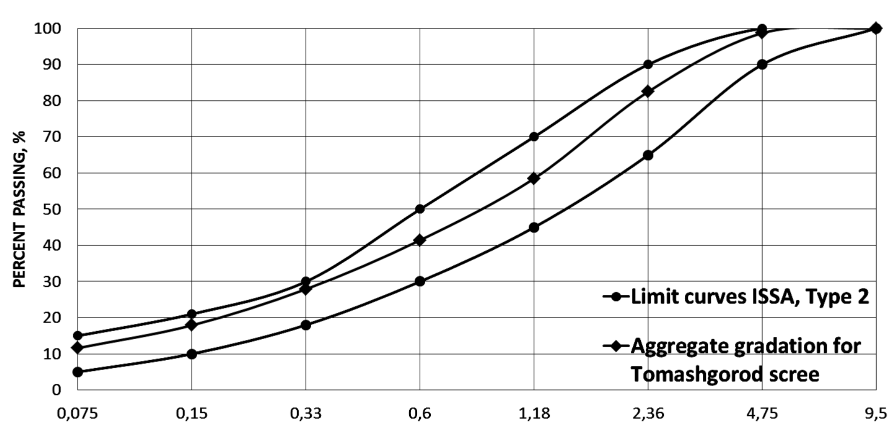

Fig. 1. Grain-size composition of Tomashgorod scree according to ISSA

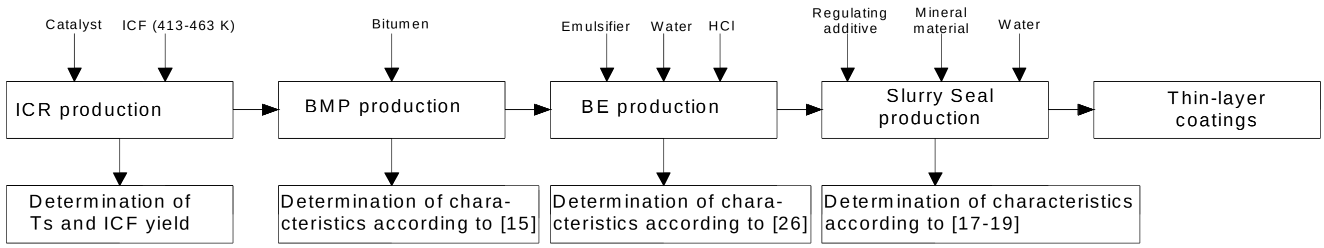

Fig. 2. Scheme of experimental investigations

According to its grain-size composition (Fig. 1) the granite scree refers to the type 2 (International Slurry Surfacing Association, ISSA) [17] and may be used for Slurry Seal production. Slurry Seal based on this filler is recommended to be used for the urban and local roads, as well as for aerodrome landing strips.

2.2. Experimental Procedure

The obtaining of modifier (ICR), bitumen modification by it, preparation of emulsions and slurry seal, as well as their use for TLC production were conducted according to the Scheme given in Fig. 2.

2.2.1. ICR production

ICF is pretreated before the cooligomerization: it is dried and pyridine bases are removed using 72% sulphuric acid. Such pretreatment allows to decrease the catalyst amount and increase ICR yield and softening temperature. The prepared raw material is loaded into a reactor, then the process conditions (time, temperature, catalyst amount) are set and oligomerization is carried out under constant stirring. The obtained product is washed by water till the reaction becomes neutral. The unreacted raw material is purified from ICR using vacuum distillation.

2.2.2. BMP production

BMP is prepared as follows: necessary amount of bitumen is heated to the given temperature, then modifier is added and stirring is turned on (*Re* = 1200). The modification time is 1 h, the modification temperatures are given in [11]. The amount of polymer is determined by the experiments based on the need to obtain BMP with T_s = 325–327 K.

2.2.3. BE production

Bitumen emulsions are prepared as follows:

– "aqueous phase" preparation;

– "bitumen phase" preparation;

– two phases mixing by means of laboratory bitumen-emulsion plant SEP-0.3R (Denimotex company, Denmark).

Aqueous and bitumen phases are loaded into the special vessels of the plant, then they are heated till given temperature (aqueous phase – 343 K, bitumen phase – 413 K) and bitumen emulsion is produced by mixing in colloid mill.

To calculate the components amount in the emulsion we used computer program appended to the plant.

2.2.4. TLC production from Slurry Seal

Slurry Seal is prepared and tested according to ISSA requirements [17, 18] and EN standard [19] at 298 K and air relative humidity of 77 %. Slurry Seal composition is designed to achieve necessary time of its break (time from all component mixing till the moment the mixture loses its mobility and possibility of further mixing). This criterion determines the time during which the mixture should be prepared and layered. The general procedure for Slurry Seal break is as follows:

– mixing of mineral components, water, regulating additive and bitumen emulsion in the enamel ware;

– manual mixing of the obtained mixture by means of spatula in the tilted enamel vessel to evaluate the mixture mobility during all experiment time;

– fixing of mixture break.

The content of all components, when mixture break continues not less than 180 s is considered to be the optimum one.

TLC model is prepared by filling forming rings with the mixture. The ring diameter is 60 mm, height is 10 mm and it is situated on the plate made of ruberoid. When the mixture loses mobility and fluidity, the samples are delivered from the rings and tested according to [18, 19] after definite periods of time. Each sample is classified according to the destruction character and corresponding torque value (M_0). Ultimate shear strength is calculated according to formula (1):

$$t = \frac{16 \cdot M_0}{p \cdot d^3} = 0.21 \cdot M_0 \quad (1)$$

where t – ultimate shear strength, MPa; M_0 – torque, H·m; d – diameter of rubber point equal to $29\cdot10^{-3}$ m.

According to the standards [17, 18] such characteristics of Slurry Seal speed of cohesive strength gain as destruction character, torque values and ultimate shear strength must comply with the values represented in the Table 4.

2.3. Analysis of Raw Material and Products

The raw material, resin, bitumen and bitumen emulsions were analyzed according to the standard methods [20-23].

The essence of the method described in [24] is to determine bitumen adhesive properties while adhesion to glass after several freezing-unfreezing cycles. The freezing is carried out at 258 K, unfreezing – at 298 K for 12 h. Adhesion to glass is determined after samples are taken off the refrigerator. The investigation scheme is as follows:

– preparation of necessary numbers of samples;

– determination of adhesive properties of the initial samples;

– determination of adhesive properties after one freezing-unfreezing cycle;

– determination of adhesive properties after four freezing-unfreezing cycles;

– determination of adhesive properties after four freezing-unfreezing cycles followed by maintaining at 258 K for 4 days;

The value of low-temperature adhesion is calculated according to the formula (2):

$$A_{-15} = \frac{1}{2}\left(\frac{A_1 + A_2 + A_3}{3} + A_4\right) \qquad (2)$$

where A_{-15} – low-temperature adhesion (adhesion to glass at 258 K), %; A_1 – initial value of adhesion to glass, %; A_2 – adhesion to glass after one cycle, %; A_3 – adhesion to glass after four cycles, %; A_4 – adhesion to glass after four cycles and four days of maintaining, %.

The filler for Slurry Seal (granite scree) is analyzed according to ISSA standards [17].

3. Results and Discussion

3.1. BE Production

As it was mentioned above, the softening temperature of bitumen modifiers may affect bitumen adhesive properties. According to the procedures described in Subsection 2.2.1 and Ref. [12, 25] we obtained ICR with different softening temperatures (Table 5).

The obtained results allow to assert that ICR addition to BND 60/90 road bitumen increases its softening temperature and significantly improves its adhesive properties (Fig. 3, Table 2). The increase in the softening temperature of the initial ICR leads to the improvement of BMP adhesive properties, including those at low temperatures. To prepare BMP with good adhesive properties (around 100%) it is necessary to use ICR with $T_s \geq 403$–413 K.

Table 4

Requirements for the speed of cohesive strength gain of slurry seal

Time to achieve necessary cohesive strength, h	Strength characteristics			Stages of Slurry Seal formation
	Destruction character	Torque (M_0), H·m	Ultimate shear strength (t), MPa, not less than	
At the latest 0.5	N – Normal	1.2–1.3	0.25	Setting
At the latest 1	NS – Normal Spin	2.0–2.1	0.42	Self-consolidation (opening of traffic with a speed limit of 40 km/h)
Not standardized	S – Spin	2.3	0.48	Consolidation
Not standardized	SS – Solid Spin	2.6	0.54	Cured (opening of traffic without speed limit)

Table 5

Conditions for ICR obtaining and its softening temperatures

Conditions for ICF cooligomerization			ICR softening temperature according to B&R method, K
Catalyst content, wt %	Temperature, K	Time, min.	
1	333	40	370
3	313	80	393
3	293	5	401
3	293	80	408
3	293	20	413

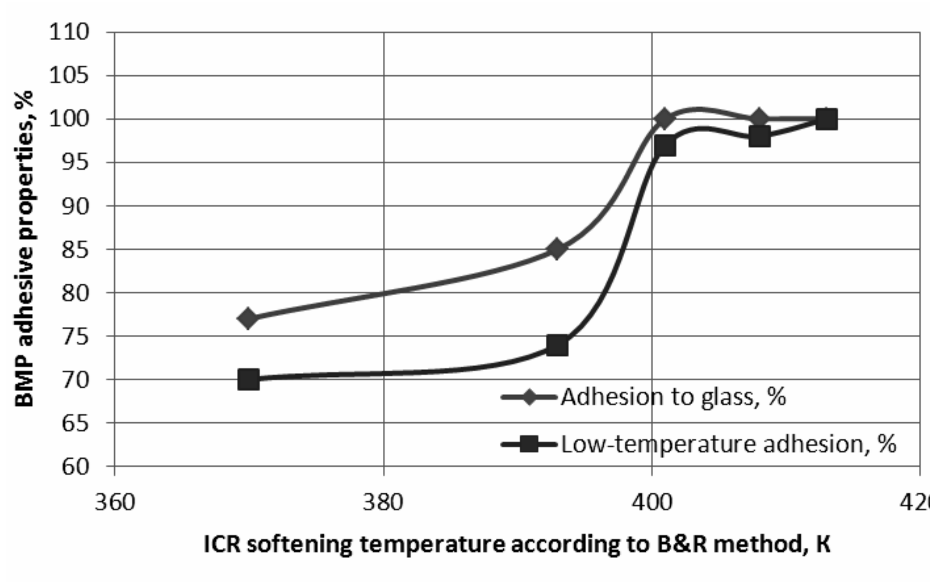

Fig. 3. BMP adhesive properties *vs*. ICR softening temperature

Table 6

Compositions of bitumen emulsions

Emulsion components	Content of components in the emulsion, wt %		
	BE 1	BE 2	BE 3
Bitumen	62	62	62
Redicote E-11 emulsifier	1.1	1.1	1.1
Hydrochloric acid	till pH = 2.5 in aqueous phase	till pH = 2.5 in aqueous phase	till pH = 2.5 in aqueous phase
Water	till 100	till 100	till 100

Table 7

Physico-technical indices of bitumen emulsions

Index		Requirements according to [26]		BE		
		ECS-60	ECSM-60	1	2	3
Appearance		Homogeneous dark-brown liquid		Fits the requirements		
Hydrogen ions concentration, pH		1.5-6.5		3.1	2.6	4.1
Homogeneity (sieve No.014 residue), %, at most		0.25	0.3	0.06	0.09	0.07
Content of residual binding agent		58–62		61	61	61
Assumed viscosity at 293 K (apparatus with hole diameter of 4 mm), s		5–25		10	9	8.4
Stability during storage: sieve No.014 residue, %, at most - after 7 days - after 30 days		 0.3 0.4	 0.4 0.5	 0.12 0.21	 0.13 0.23	 0.11 0.19
Adhesion of residual binding agent to macadam, points, no less		5.0		5.0	5.0	5.0
Miscibility with mixtures of grained composition	porous	Yes		Yes	Yes	Yes
	dense	Yes		Yes	Yes	Yes

To find the conditions for ICF cooligomerization, under which ICR maximum yield would be achieved ($T_s \geq 403$ K) we developed experimental statistical mathematical model (ESM) of this process [25]. On the basis of regression equations, which describe this model, by means of uniform search technique we found the optimum conditions for ICR obtaining (temperature 310 K, time 40 min., catalyst concentration 3.3 wt %). The resin yield is 33.3 wt % relative to the raw material and its T_s is 408 K.

The obtained ICR and BND 60/90 bitumen were used to prepare BMP (bitumen content is 93.3 %, ICR content – 6.7 wt %). The BMP characteristics are: softening temperature according to B&R method is 325 K, adhesion to glass – 100 %, low-temperature adhesion – 97 %. According to the mentioned indexes the modified bitumen corresponds to BMP 60/90-52.

The oxidated initial and modified bitumens as well as distillation bitumen were used to produce bitumen emulsions:

– BE 1 – on the basis of BND 60/90 bitumen;

– BE 2 – on the basis of BMP 60/90-52 modified bitumen;

– BE 3 – on the basis of Nynas 70/100 bitumen.

BE compositions are represented in Table 6 and their physico-technical indices – in Table 7.

On the basis of represented results we can assert that obtained BE fit the normative documents. Thus, BE 1 and BE 3 correspond to ECS-60 type (cationic slow-breaking emulsion) and BE 2 – to ECSM-60 type (cationic slow-breaking modified emulsion).

3.2. Determination of Slurry Seal Optimum Composition and Speed of Cohesive Strength Gain

The obtained bitumen emulsions were used for the production of Slurry Seal together with other components. The optimum compositions of Slurry Seal according to the break criterion are represented in Table 8.

On the basis of obtained slurry seals we formed the samples of TLC models according to the procedure described in Subsection 2.2.4 and determined the speeds of cohesive strength gain after definite periods of time (Table 9).

Table 8

Optimum compositions of Slurry Seal according to the break criterion

Slurry Seal, No.	Components content, g					Break.time, s
	Granite scree	Portlandcement	Water	Regulating additive	Bitumen emulsion	
BE 1						
1	100	1.25	10	2.00	14	206
BE 2						
2	100	1.25	10	2.25	14	180
BE 3						
3	100	1.00	10	0.50	14	190

Table 9

Speeds of cohesive strength gain for the samples of Slurry Seal

Slurry Seal, No.	Experiment time, hr	Destruction character	M_0, H·m	t, MPa	Mixture class
1	0.5	Normal (N)	1.2	0.252	Quick-setting, slow self-consolidation
	1.0	Normal (N)	1.2	0.252	
	3.0	Normal (N)	1.3	0.273	
	4.5	Normal Spin (NS)	2.0	0.42	
	6.0	Spin (S)	2.3	0.483	
	7.0	Solid Spin (SS)	2.6	0.546	
2	0.5	Normal (N)	1.2	0.252	Quick-setting, quick self-consolidation
	1.0	Normal Spin (NS)	2.1	0.441	
	2.0	Spin (S)	2.3	0.483	
	3.0	Solid Spin (SS)	2.6	0.546	
3	0.25	Normal Spin (NS)	2.1	0.441	Super quick-setting, super quick self-consolidation
	0.5	Spin (S)	2.4	0.504	
	0.75	Solid Spin (SS)	2.7	0.567	

The experimental results (Table 9, Fig. 4) show that the quickest gain in the mixture cohesion is observed during application of Nynas 70/100 bitumen (Slurry Seal No.3). In 15 min after TLC laying it is possible to open traffic with speed limit of 40 km/h and after 45 min – without speed limit. The speed of cohesion strength gain of Slurry Seal based on oxidated BND 60/90 (Slurry Seal No.1) is unsatisfactory. The modification of the oxidated bitumen by ICR improves not only their adhesive properties but the speed of cohesive strength gain as well. Slurry Seal No.2 on the basis of BMP 60/90-52 exhibits high speeds of TLC structure formation. While using such coatings the traffic with speed limit of 40 km/h is possible after 1 h after coating laying and after 3 h the traffic has no speed limits.

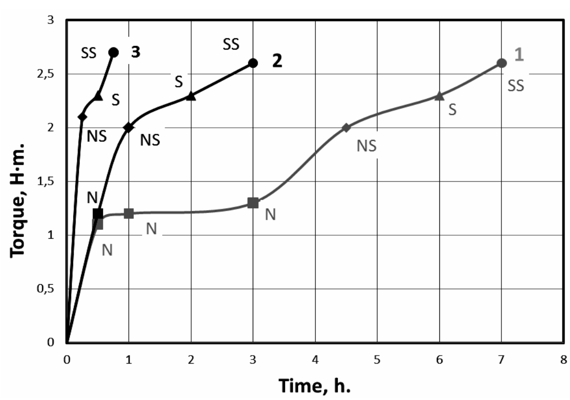

Fig. 4. Speed of cohesive strength gain for TLC based on Slurry Seals No. 1-3

4. Conclusions

1. To improve the adhesive properties of petroleum road bitumen considerably, including low-temperature adhesion, it is necessary to use indene-coumarine resin in the amount of 6–8 wt % with the softening temperature of 403–413 K.

2. The commercial emulsion of ECSM-60 type and Slurry Seal with the break time of 180 s are obtained on the basis of the oxidated bitumen and Redicote E-11 emulsifier. Both products meet the standards.

3. Slurry seals based on the petroleum bitumen modified by ICR have higher speeds of cohesive strength gain compared those obtained on the basis of the oxidated bitumen.

4. Modified bitumen may be used instead of deficient distillation bitumen for the production of thin-layer coatings from slurry seal.

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ЗАСТОСУВАННЯ БІТУМНИХ ЕМУЛЬСІЙ НА ОСНОВІ ОКИСНЕНИХ, ДИСТИЛЯЦІЙНИХ І МОДИФІКОВАНИХ ОКИСНЕНИХ НАФТОВИХ БІТУМІВ ДЛЯ ВИРОБНИЦТВА SLURRY SEAL

Анотація. *З нафтових бітумів, що відрізнялися способом виробництва (окисненого, залишкового, модифікованого окисненого), і емульгатора «Redicote E-11» одержано три товарних дорожніх емульсії, які надалі використано для приготування Slurry Seal та тонкошарових дорожніх покриттів (ТП) на їх основі. Показано переваги тонкошарових дорожніх покриттів, у виробництві яких використовувався модифікований окиснений нафтовий бітум, над тонкошаровими покриттями на основі окисненого бітуму та встановлено можливість заміни дороговартісного дистиляційного бітуму окисненим модифікованим бітумом для застосування в технології Slurry Seal. Запропоновано для модифікації окисненого бітуму використовувати інден-кумаронову смолу, що дає можливість суттєво покращити його адгезійні та когезійні властивості.*

Ключові слова: *лита емульсійно-мінеральна суміш, тонкошарові покриття, бітум, бітумні емульсії, модифікатори, інден-кумаронова смола.*