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HYDROCHLORIC AND ORTHOPHOSPHORIC ACIDS USE IN THE QUICK-TRAFFIC SLURRY SURFACING MIX

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Abstract. This article is devoted to development and production (on a lab emulsion plant) of six compositions of slow-setting bitumen emulsions for a slurry-surfacing mix. An aggregate composition for Type 1 (0–5 mm) and Type 3 (0–15 mm) slurry-surfacing mixes was determined and selected. Results of the slurry surfacing mix composition selected by the criteria of a mixture decay, cohesion strength build-up rate and material loss by wet track abrasion testing – on the basis of bitumen emulsions produced from both distilled and oxidized bitumen, using various emulsifiers and both hydrochloric and orthophosphoric acids, are presented in the article. Advantages of using slurry-surfacing mixes based on emulsion produced with orthophosphoric acid and Redicote C-320E emulsifier were proved. Such a system either minimizes or eliminates in its composition the use of the mixture decomposition regulator and reduces the content of Portland cement. The application of such a system makes it possible to produce slurry surfacing mix on the basis of low acid oxidized bitumen made from light petroleum and rock material through the criterion of the total surface activity determined by methylene blue with high rates of mix curing, and consequently the transport rapid start on the arranged thin-layer coating. Besides, the wet track abrasion indices of this system are substantially lower than in traditional system on oxidized bitumen, and even lower in comparison with the system on distilled bitumen.

Keywords: slurry-surfacing mix, cohesive strength, bitumen emulsion, acid.

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1. Introduction

The slurry surfacing mix application is an effective way to maintain the operation characteristics of the new road surfaces and an economical way to renew the worn surface. Slurry surfacing mix forms a characteristic surface with better water resistance and adhesive qualities. It can be used on asphalt or concrete road surfaces [1-4].

Scientists [5-9] are unanimous that the slurry-surfacing mixes are definitely progressive, but there is a problem with the realization of this technology on domestic raw-material basis, because not all stone materials and especially bitumen are suitable for use in this technology. The optimal bitumen for slurry-surfacing mixes (by the criterion of the cohesion strength increase rate) is a high acid distilled bitumen obtained from heavy crude-oil, which provide rapid structure formation of the laid mixes. Such bitumen is expensive, which restricts their application. However, studies are underway to improve the bitumen properties through the modification [10-14].

Significant contribution into investigation of the problem of acid use for emulsions was made by James [15-17]. He asserts that alternative acids can provide the advantages in the operation indices at the stage of final emulsion application. Along with resulting pH changes, the reaction of acid with aggregate has a significant effect on the decay of cationic emulsions. Besides, the choice of acid influences the process, while the obtained products will potentially influence the adhesion and rheology of residues [18]. James and Ng [19, 20] analyzed the use of orthophosphoric acid for slurry seal and microsurfacing technologies and determined the peculiarities and advantages of this acid application for the mentioned technologies.

One of the most powerful investigators of slurry-technologies is Nouryon (AkzoNobel Asphalt Applications, Sweden), which has proposed (after a number of substantial investigations) the new system of rapid mix-set on arbitrary bitumen with orthophosphoric acid application. This system is called Redipave. In traditional slurry systems, hydrochloric acid is used to obtain the emulsions, while in the Redipave system – orthophosphoric acid together with patented emulsifiers. In Europe, the application of easy-to-use Redicote EM44 liquid emulsifier is recommended, while Redicote EM44-A and Redicote C-320 are used in

America. These emulsifiers allow both adopting emulsions for the chips with high reactivity and controlling mix-set time [19]. Correspondingly, the production of orthophosphoric acid emulsions in our country is not investigated and is not common. Although, as the world experience shows, using this acid in emulsions has a number of advantages.

The main advantages are: the possibility of using more common in Ukraine oxidized bitumen and non-optimum (high-reactive) aggregates by methylene-blue criterion, quick slurry-surfacing structure formation and the possibility of laying slurry-surfacing mix in cold weather and at night time; orthophosphoric acid does not require getting license for handling pre-cursors and is less corrosive unlike concentrated hydrochloric acid. In the works [21, 22] slurry-surfacing with application of orthophosphoric acid and Redicote EM44 emulsifier investigations are compared with traditional slurry-surfacing mixes, and efficiency of using system on orthophosphoric acid is proved.

The aim of this article is to determine the advantages of the slurry-surfacing mix with orthophosphoric acid and local mineral materials.

2. Experimental

The crude-oil road viscous oxidized bitumen of 70/100 grade (BND 70/100) from JSC Mozyr Refinery (Mozyr, Gomel Region, Belarus) and distilled bitumen Nybit E85 from Swedish company Nynas (Table 1), that correspond to the requirements [23], were used for the bitumen emulsion production.

Extremely important for cold technologies is the composition of the original bituminous emulsion [25, 26]. Based on both recommendations from emulsion producers and practical experience of Ukrainian and European companies, a number of compositions was developed (with emulsions further produced), the most efficient (and high-quality) of which are presented in Table 2. Those compositions were also used for comparing the influence of orthophosphoric and hydrochloric acids upon the slurry-surfacing quality indices. According to the compositions, bitumen emulsions contained Swedish AkzoNobel emulsifiers Redicote 404, Redicote EM44 and Redicote C-320E, as well as co-emulsifier Redicote 540. Each of the compositions included SBR-polymer of Toptex B latex from Algol Chemicals (Finland).

The bitumen emulsions were produced on Danish laboratory batch bitumen-emulsion plant type DenimoTech SEP-0.3R

Established physico-technical indicators of the developed emulsion recipes confirmed that bitumen emulsions are cationic slow-setting emulsions (CSS) intended for use in slurry seal and microsurfacing technologies.

Table 1

Bitumen physico-mechanical parameters

Index	Actual value		Standard according to EN 12591-2009 [23]
	Nybit E85	Mozyr	
Penetration at 298 K ($m \cdot 10^{-4}$)	85	80	70–100
Softening temperature (ball & ring method), K	320	320	316–324
Fraass breaking point, K	262	252	≤ 263
Flash point, K	508	563	≥ 503
Solubility, %	99.1	99.4	≥ 99.0
Kinematic viscosity at 408 K, mm/s^2	508	513	≥ 503
Total acid number, mg KOH/g (according to ASTM D664 [24])	3.0	0.6	–

Table 2

Bitumen emulsion compositions for slurry-surfacing

Components, wt %	Emulsion compositions, No					
	1	2	3	4		
Bitumen	Nybit E85 – 62 %	Mozyr – 62 %				
Emulsifier	Redicote 404 – 1.1 %	Redicote 404 – 1.3 % Redicote 540 – 0.25 %	Redicote EM44 – 1.1 %	Redicote C-320E		
				1.2 %	1.6 %	1.8 %
Water phase pH (acid)	pH = 1.5 (HCl)	pH = 2.5 (HCl)	pH = 2.5 (H_3PO_4)			
Latex	Toptex B – 2 %					

The crushed stone and gravel bran of 0–5 mm and 0–15 mm fractions from LLC "Klesivsky quarry of non-metallic minerals TECHNOBUD Ltd. (Rivne region, Ukraine) were used for designing the grain composition of the mixture, corresponding to Type 1 and Type 3 slurry surfacing mix according to the standards of the International Slurry Surfacing Association (ISSA) [15-16].

It was noted that total surface activity determined by the methylene blue index (MB) is 15 ml, which exceeds the allowable norm (5–10 ml). However, previous studies [21, 22] have indicated that slurry-surfacing mixtures on orthophosphoric acid make it possible to use an aggregate with high MB values.

It was noted that aggregate gradation for the 0–5 mm falls within the limits of recommended limit-curves for slurry-surfacing mix Type 1 according to ISSA norms (Fig. 1), while for getting the closest size distribution curve to the requirements for slurry-surfacing mix Type 3 ISSA the combination of the screenings grade 0–5 mm and the chips grade 0–15 mm (in the amount of 75 % and 25 %, respectively) was prepared – with the resulting chips mixture grade 0–15 mm (Fig. 2).

The slurry-surfacing mixes were tested by the method to determine set and cure development of slurry surfacing systems by cohesion tester [29] and the method for wet track abrasion of slurry surfacing systems [30].

Cohesion strength build-up rate for the slurry-surfacing mix (traffic starts on the made surface) was determined by the corresponding mix-samples formation and testing them after certain time-period by cohesion tester. The slurry-surfacing mix samples were classified (on Types) by destruction character (Fig. 3):

1) N = Normal. Below 12 kg·cm, multiple radial cracks are apparent.

2) NS = Near Spin. Only one radial crack appears. The equivalent cohesion value is ~20 kg·cm. When reaching such type of destruction, the traffic start is possible with 40 km/h speed limit and prohibited turn.

3) S = Spin. No cracks appear but aggregate is dislodged directly beneath the foot and “roll” under the foot. The equivalent cohesion value is ~23 kg·cm.

4) SS = Solid Spin. There are no cracks or dislodged aggregate. The foot skids, or slides, over the surface. Some asphalt film may be removed. The equivalent cohesion value is ~26 kg·cm. With the achievement of this type of destruction, the traffic start is possible without speed limit or prohibitions.

3. Results and Discussion

The compositions of the slurry surfacing mix were selected on 100 g of stone material, depending on the recipe of bitumen emulsion (Table 1) and the type of stone material (Figs. 1, 2), including Portland cement and 20 % blast furnace slag, water and decomposition regulator (10 % aqueous solution of Redicote E-11 emulsifier).

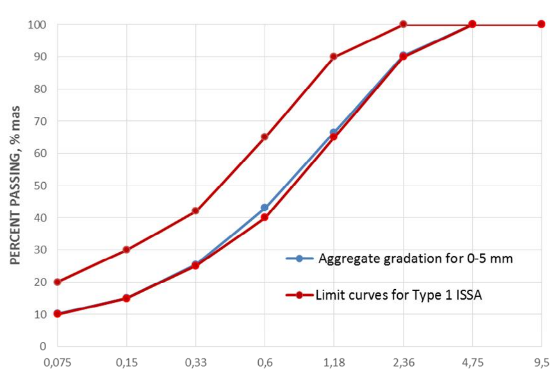


Fig. 1. Aggregate gradation for the screenings grade 0–5 mm according to Type 1 ISSA

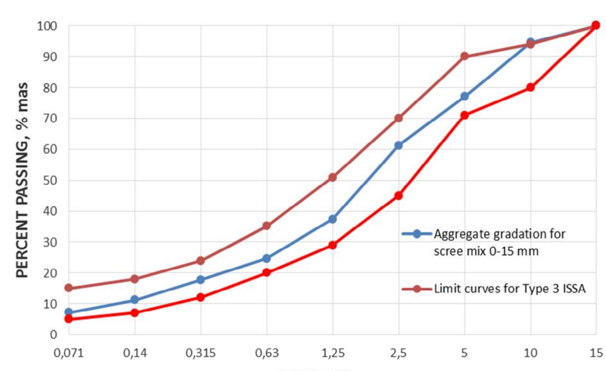
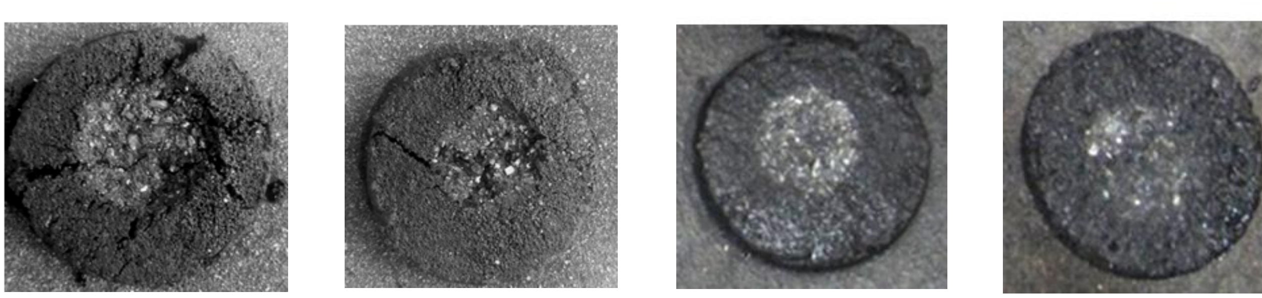


Fig. 2. Aggregate gradation for the selected chips mix grade 0-15 mm according to Type 3 ISSA



1) N 12–13 kg·cm 2) NS 20–21 kg·cm 3) S 23 kg·cm 4) SS 26 kg·cm

Fig. 3. Types of the slurry-surfacing mix samples destruction

The compositions of the slurry surfacing mix were selected according to the breakage criterion (*P*) and a new indicator – start of cohesion strength build-up (*SCS*) [31]. As a start of cohesion strength there is considered time interval from the breakage of the mixture to the moment of its transfer into a quasi-solid state. The mixture turns into a quasi-solid state after loss of mobility and the possibility of further mixing, when it begins to lump. In laboratory conditions the start time for cohesion strength build-up is proposed to be when it is possible to form and hold a lump from the formed mixture for several seconds on a spatula. In the absence of unfavorable weather or mechanical factors throughout the slurry surfacing mix formation period, the sooner the cohesive strength build-up begins, the sooner the hard cover will attain the required cohesive strength to open traffic. In laboratory conditions, it is established that the beginning of cohesive strength build-up must occur no later than 30 s after the mixture breakage. If it occurs later, the produced slurry-surfacing mix will have a low rate of cohesive strength gain [31]. The optimal compositions of the slurry surfacing mix by the mentioned criteria are given in Table 3. Table 2 shows that slurry-surfacing systems on orthophosphoric acid and Redicote C-320E with dosage rates of 1.6 and 1.8 wt % do not require mix-set control dope, while the cement dosage is 2 times less than in systems on hydrochloric acid.

The investigation results on the determination of slurry-surfacing mix cohesion strength build-up rate are presented in Table 4.

Analyzing Table 4, we can see that the highest cohesion strength build-up rates for slurry-surfacing mix on the emulsions used are provided by the compositions on the distilled bitumen Nybit E85, as well as on oxidized bitumen BND 60/90 produced by JSC Mozyr Refinery with Redicote C-320E emulsifier at the dosage rate of 1.6 and 1.8 wt % of emulsion depending on the mixture type. Therefore, the use of orthophosphoric acid and the Redicote C-320E emulsifier makes it possible to use the emulsified form oxidized bitumen in slurry surfacing mix technology on an equal basis with distilled bitumen due to the mix curing time. In general, it can be observed that Type 3 slurry-surfacing mix (0–15 mm), regardless of the bitumen emulsion formulation, is characterized by more intensive mix-curing rates than Type 1 slurry-surfacing mix (0–5 mm). For further testing, the slurry-surfacing mix No. 3 was rejected, as far as the rates of cohesion strength build-up for the mix 0–5 mm were unsatisfactory in contradistinction to the investigations results [21, 22]. Thus, the formulation of bitumen emulsion No. 3 is not universal and depends on the aggregate used and on its reactivity.

For determination of wet track abrasion of slurry surfacing systems the mix type 0–5 mm was chosen. This testing simulates deterioration (in wet conditions) of thin-layer coating made of slurry-surfacing mix, namely its mass loss due to traffic. Besides, it allows evaluating the bitumen-binder-to-aggregate adhesion and the minimum required amount of bitumen in the mixture. Analyzing the tests results (Table 5), we observe the lowest aggregate loss at the wet track abrasion (same as with test on cohesion strength build-up rate) in slurry-surfacing mix on the basis of distilled bitumen Nybit E85 and oxidized bitumen BND 60/90 produced by JSC Mozyr Refinery with Redicote and Redicote C-320E emulsifier in the amount of 1.8 wt %. In general, the mixtures with bitumen emulsion dosage of 14 parts are more economical, especially since the increase in the flow rate of the emulsion to 16 parts is not followed by the impressive decrease of material loss at wet track abrasion.

Also, the samples of slurry surfacing mix using orthophosphoric acid have a more saturated dark color and characteristic stickiness.

4. Conclusions

Based on technical comparison of the mixtures designed by three criteria: mix-set, cohesion strength build-up rate and material loss at wet track abrasion, the efficient bitumen emulsion compositions (for slurry-surfacing mix) with high-reactive aggregate were created: composition No. 1 (Nybit E85 – 62 %, Redicote 404 –

Table 3

Composition of slurry-surfacing mixes

Mix No.			Components, g				Breakage, m:s	SCS, m:s	Mix type, mm
			Cement	Water	Control dope	Emulsion			
1. Nybit E85 62 %; Redicote 404 – 1.1 %; pH = 1.5 (HCl)		1.1	0.7	12.0	2.0	14.0	2:20	2:15	0–5
		1.2	1.0	10.0	1.15	14.0	2:20	2:30	0–15
2. Mozyr – 62 %; Redicote 404 – 1.3 %; Redicote 540 – 0.25 %; pH = 2.5 (HCl)		2.1	1.0	13.0	1.3	14.0	2:10	2:20	0–5
		2.2	1.0	10.0	0.7	14.0	2:10	2:25	0–15
		2.3	1.0	11.0	1.0	16.0	2:10	2:20	0–5
		2.4	1.0	11.0	0.5	16.0	2:20	2:30	0–15
3. Mozyr – 62 %; Redicote EM44 – 1.0 %; pH = 2.5 (H_3PO_4)		3.1	0.5	11.0	3.0	14.0	2:10	2:40	0–5
		3.2	0.5	10.0	1.4	14.0	2:30	2:55	0–15
4. Mozyr – 62 %; Redicote C-320E pH = 2.5 (H_3PO_4)	1.2 %	4.1	0.5	12.0	2.25	14.0	2:40	3:05	0–15
	1.2 %	4.2	0.5	10.0	0.5	14.0	2:30	2:55	0–15
	1.6 %	4.3	0.5	8.0	–	14.0	2:35	3:00	0–15
	1.8 %	4.4	0.5	14.0	–	14.0	3:19	3:33	0–5

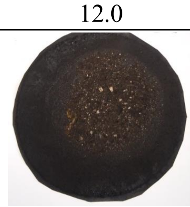
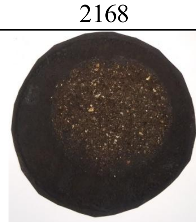
Table 4

Slurry-surfacing mix cohesion strength build-up rate

Mix No. according to Table 3	Mix type	Emulsion dosage	Time, h:min			
			Destruction character			
1.1	0–5	14	0:15	0:25	0:30	0:45
			N	S	S	SS
1.2	0–15	14	0:15	0:20	0:25	0:30
			S	S	S	SS
2.1	0–5	14	2:00	3:00	4:00	–
			NS	S	SS	–
2.2	0–15	14	1:00	1:30	2:00	3:30
			NS	NS	S	SS
2.3	0–5	16	1:00	2:15	3:00	4:00
			N	S	S	S
2.4	0–15	16	1:00	2:00	3:00	4:00
			N	NS	S	S
3.1	0–5	14	2:00	4:00	4:30	5:00
			N	N	NS	S
3.2	0–15	14	1:15	1:30	2:00	2:30
			N	NS	NS	S
4.1	0–5	14	0:30	1:00	2:30	4:00
			N	N	N	NS
4.2	0–15	14	1:00	1:15	1:30	–
			S	S	SS	–
4.3	0–15	14	0:30	0:45	–	–
			S	SS	–	–
4.4	0–5	14	0:20	0:30	0:45	–
			S	S	SS	–

Table 5

Slurry-surfacing mix loss at wet track abrasion

Mix No.	Emulsion dosage, g per 100 g of aggregate / Material loss in mass at wet track abrasion, g/m^2		
	12.0	14.0	16.0
1			
	2168	36	16
2			
	2395	1066	46
4.4			
	204	23	20

1.1 %, pH = 1.5 (HCl)) and No. 2 (BND 70/100 (Mozyr) – 62 %, Redicote C-320E – 1.8 %, pH = 2.5 (H_3PO_4)).

The possibility and efficiency of slurry-surfacing systems application based on oxidized bitumen on orthophosphoric acid and Redicote C-320E emulsifier, with reaching the efficient indices of cohesion strength build-up rate and wet track abrasion for the coating – in comparison with more wide-spread and known systems on hydrochloric acid was proven. Besides, this system is less dependent upon the aggregate reactivity than hydrochloric acid-based systems, while it provides for usage of actually all aggregates which correspond to the grading requirements for the mix Types.

The investigation analysis shows that the usage of orthophosphoric based systems is technically efficient. Therefore, the large-scale implementation of such technologies into production is recommended.

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ВИКОРИСТАННЯ ХЛОРНОЇ ТА ОРТОФОСФАТНОЇ КИСЛОТИ ДЛЯ ШВИДКОТВЕРДНУЧИХ ЛИТИХ ЕМУЛЬСІЙНО-МІНЕРАЛЬНИХ СУМІШЕЙ

Анотація. *Розроблено та виготовлено шість складів повільнорозкладних бітумних емульсій для литих емульсійно-мінеральних сумішей (ЛЕМС). Визначено та підібрано зернові склади кам'яного матеріалу для ЛЕМС типу 1 (0-5 мм) та типу 3 (0-15 мм). Проведено підбір складу ЛЕМС за критеріями розкладу суміші, швидкості набору когезійної міцності та втрати матеріалу за вологого абразивного зносу на основі бітумних емульсій з використанням дистиляційних та окиснених бітумів, різного роду емульгаторів та хлорної та ортофосфатної кислот. Доведено переваги використання ЛЕМС з ортофосфатною кислотою та емульгатором Redicote C-320E. Показано, що така система приводить до мінімуму або виключає використання в її складі регулятора розкладу суміші і зменшує вміст портландцементу. Використання такої системи дає можливість виготовляти ЛЕМС на основі низькокислотного окисненого бітуму, виготовленого з легкої нафти та реактивного кам'яного матеріалу за критерієм загальної поверхневої активності, визначеної за показником метилену синього, з високими темпами твердіння суміші, а відповідно, і швидким пуском транспорту по влаштованому тонкошаровому покритті. Встановлено, що показники вологого абразивного зносу покриття цієї системи є значно нижчими показників традиційної системи на основі окисненого бітуму та нижчими навіть в порівняні з системою на основі дистиляційного бітуму.*

Ключові слова: *лита емульсійно-мінеральна суміш, когезійна міцність, бітумна емульсія, кислота.*