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ADHESION OF ROAD BITUMEN EMULSIONS ON BOTH HYDROCHLORIC AND ORTHOPHOSPHORIC ACIDS FOR THE TECHNOLOGY OF SURFACE DRESSING

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This article presents the review of surface dressing wear courses, being the version of preservation and sealing of road structures' top layers. In the article, there is studied the adhesion of aggregates (from various granite quarries of Ukraine) with a bitumen binder, namely bitumen emulsion, for application in surface dressing for road pavements. In the article, there are used both the Ukrainian and European testing methods for the determination of adhesion in the bitumen-aggregate system. There was developed two bitumen emulsion formulations for the thin-layer motor-road pavements by the surface dressing depending on the type of acid used (either hydrochloric or orthophosphoric).

Key words: thin-layer road pavement; surface dressing; bitumen emulsion; hydrochloric acid; orthophosphoric acid; granite chips; limestone chips; emulsion-to-chips adhesion.

Introduction

In the modern conditions of motor-road operation the strategy of technical policy in the field of maintenance gradually changes towards the prevention of road pavement destruction formation. The transport-operation state of road pavement under the effect of repeated traffic loads and influence of natural factors is constantly changing. In the initial period of motor-road operation the changes happen slowly, while in later course the rate of worsening the transport-operation state starts growing. Taking into account that the substantial quantity of general-use roads need overhaul repairs, the extremely important is preservation of that roads, which have not yet exhausted their resource, as well as of those ones which are already repaired – in order to prolong their service time and between-repair intervals. It is due to motor-roads repairs and operational maintenance, that it may become possible to improve the transport-operation state of road pavements and prolong the service time and between-repair intervals. The efficiency of such works substantially depends upon the timely and periodical fulfillment of such preventive measures. One of such measures is paving of protective wearing courses. The protective wearing courses are intended for extension of between-repairs operation time for road pavements, ensuring the comfortable & safe traffic, improvement of roughness and evenness of road pavement, raising the adhesion index, as well as for pavement protection against the weather factors (temperature, humidity, precipitation, glazed and black frost). They combine the functions of protective courses, wearing courses and grip courses by means of special mix design (in the part of aggregate grading), special requirements towards the strength and wear-resistance of aggregates; type of binder and its properties; consumption norms; adding agents and choice of works technology.

In this article the attention is paid to so-called "thin-layer" pavements performed by surface dressing method. The application of surface dressings on motor-roads with average estimated forecast

traffic rate less than 1000 vehicle/day may be done in all the road-climatic areas of Ukraine – with mandatory substantiation of surface dressing types and road maintenance technologies depending upon the type and status of the existing pavement. The determinative factor for cohesion strength and durability of these wearing courses is adhesion between the surface dressing components, namely bitumen binder and aggregate. Therefore, the mix design for surface dressing by criterion of adhesion between the components is the actual problem, which can be resolved by both the efficient choice of bitumen binder & aggregate and determination of the necessary adhesion characteristics by correct and reliable investigation methods.

The analysis of information sources has shown that scientists in the present conditions pay attention to the detailed investigation of binder materials for surface dressing, as well as to adhesion with aggregate (E. Y. Hajj, 2011; Ozdemir, Ugurcan, 2018; Adams, 2019; M. Bueno, 2014; Oikonomou, 2007; Murat, 2005; Sidun, 2021; Pavlyuk, 2013).

The most wide-spread in the world method for determination of adhesion properties of the binder and filling aggregates for the pavements made by the surface dressing technology (Iu. Sidun, 2021) is Vialit Plate Shock Test (EN 12272-3:2004; Louw, 2004). Although there also exist other laboratory and field methods: Frosted Marble Cohesion Test (Howard Isaac L.,2013; Ozdemir, Ugurcan, 2016), Sweep Test of Bituminous Emulsion Surface Treatment Samples (Ozdemir, Ugurcan, 2016; ASTM D7000 – 19a), Pennsylvania Aggregate Retention Test (Kandhal, 1991), Australian Aggregate Pull-out Test (Queensland Department of Transport and Main Roads, 2012), British Pendulum Test (ASTM E303-93; EN 13036-4:2011), Pneumatic Adhesion Tension Test (Zhou, 2014). In general, the majority of scientists give special attention to technological aspects of performing the surface dressings (Ilchenko, 2011; Pavlyuk, 2010; Ilchenko, 2011; Junyan, 2013; Pavlyuk, 2011).

The purpose of this article is tudying bitumen emulsions on hydrochloric and orthophosphoric acids for performing the protective wearing courses by surface dressing technology at high and low (operation) temperatures.

Materials and Methods

The testing of bitumen emulsions formulations was conducted according to State Standard of Ukraine DSTU B V.2.7-129:2013.There were tested the following bitumen emulsions properties:

- Appearance.
- Particle charge test.
- Residue on sieving (sieve No. 014).
- Binder content.
- Viscosity – Efflux time, 4 mm – 25 °C.
- Breaking Behaviour.

Testing of adhesion for extracted-from-emulsion binder with chips surface was done by two methods: high-temperatures methods and Vialit Plate Shock Test at operation temperatures.

Determination of adhesion for extracted-from-emulsion binder with chips surface was done by two methods which differed by the testing temperature. By the method No. 1 the testing started from the choice of two chips, which were washed and cleared from dust & dirt and placed in desiccator for reaching the constant weight.

Each chip was winded by rope and immersed for 1–2 seconds into the vessel with distilled water. After the removal of chip from water it was allowed to flow-off during (15±5) seconds, while thereafter it was immersed into bitumen emulsion and hanged up on clamp stand.

The emulsion-treated chips were retained during one day at (20±5) °C and thereafter tested. To provide for that the 3-liter flask was filled for 2/3 of its volume and heated till (95±1) °C. The emulsion-treated chips were immersed into the preheated water in such way as not to touch the walls and bottom, while they were retained at 95 °C during 30 minutes. Binder rising to the water surface during boiling was removed by filter paper.

The adhesion quality was estimated visually (in points, with step 0.5 point) by characteristic of binder film on a chip surface after boiling (DSTU 8787: 2018) (Table 1).

Table 1

Evaluation of quality for adhesion of bituminous binder with chips

Degree of retaining the bituminous binder film on the chips surface	Evaluation of adhesion quality, points
Bituminous binder film remained on the chips surface; it is admissible to have not more than 2 places non-covered by bituminous binder, the diameter of each of which not exceeding 1.0 mm	5.0
Bituminous binder film partly separated from angles or ribs and/or remained on more than 90 % of chips surface	4.5
From 75 % to 90 % of chips surface remained coated by the bituminous binder film	4.0
From 60 % to 75 % of chips surface remained coated by the bituminous binder film	3.5
From 40 % to 60 % of chips surface remained coated by the bituminous binder film	3.0
From 20 % to 40 % of chips surface remained coated by the bituminous binder film	2.5
Bituminous binder film remained on less than 20% of the chips surface	2.0
Note: During the adhesion quality evaluation each range of chips surface covered by binder contains the value of bottom limit and does not contain the value of top limit	

Method No 2 was similar to method No. 1. Still, the testing conditions as to temperature were harder: chips were boiling in water-with-glycerin in ratio 5 to 1 at (100 ±1) °C.

Testing according to Vialit Plate Shock Test consists in determination of binder ability to retain the aggregate on metal plate upon shock influence. To provide for the studies we used the pre-screened granite chips grade 5–10 mm and according to "DSTU EN 12272-3:2020" there were chosen (for testing) 100 chips of the said grade for each plate. There was determined the binder quantity required to be spread on the plate. The plate dimensions constitute 200×200 mm (0.04 m^2). For the chips grade 5–10 mm the bitumen consumption norm constitutes 1.00 kg/m^2. Thus, the weight of bitumen to be spread on the plate constitutes: 1.0×0.04=0.04 kg = 40 g. Thereafter we determine the quantity of 65 % bitumen emulsion to be spread on the plate (40×1000/65) = 61.54 g.

Determination of mechanical adhesion: this is binding chips together in dry state along with their natural dust or fines, which form an inhibitor film on a chips surface. To provide for mechanical adhesion the bitumen emulsion is heated to 70 °C (this is the temperature of bitumen emulsion application on the road), while thereafter it is evenly applied upon the preheated (during 5 minutes to the temperature of 50 °C) steel plates. Afterwards the un-cleaned chips are placed evenly on a plate surface, along with sinking them slightly into emulsion. The samples are not compacted. The samples formed are placed for 24 hours into a desiccator at 30 °C with forced ventilation.

After 24 hours the samples are removed from desiccator and placed for 20 minutes (±2 min) into a climatic chamber at (5±1) °C. Thereafter the samples are brought to the testing temperature and the testing starts. The steel plate is put upside down (with chips downwards), and the metallic ball weighing 500 g is dropped upon it three times within 10 seconds.

Determination of brittle temperature – that is the lowest temperature for conducting testing, at which the number of all the fixed & retained on plate surface chips constitutes not less than 90 %.

The preparation works are conducted the same way as for the test on mechanical adhesion. The testing is started at the temperature of 5 °C by placing the samples into a climatic chamber and cooling them down to (5±1) °C, while thereafter they are tested on Vialit device by dropping (three times during 10 seconds) the 500 g ball upon the upside-down-turned plate.

Grip by weight (R) both for the mechanical adhesion and the brittle temperature was determined by the formula (1)

$$R = ((B–E)/B)\times 100\ \%, \quad (1)$$

where B – chips weight before testing, g; E – weight of unfixed chips after testing, g.

The weight of unfixed chips after testing was determined as a sum of weights for unfixed chips, whose surface is not covered by binder after rinsing by solvent for removing binder.

The quantitative counting of chips was done as follows:

– a' – quantity of unfixed chips, whose surface is not covered by binder;

– b' – quantity of unfixed chips, whose surface is partly covered by binder;

– c' – quantity of fixed chips.

According to quantity of chips applied and the used grades by chip-size the arithmetical sum is checked: $a'+b'+c' = 100$. The calculation is made for average values on parallel determination as to each chips quantity, namely: a, b and c.

In accordance with this, the indices of grip or adhesion are expressed in general quantity of units (or in percent) for all the fixed and unfixed chips, whose surface is partly covered by binder after shock-method testing.

Grip index and/or adhesion index constitutes:

– $b + c$ – when using 100 chips for each sample;

where b and c – average values of parallel determination of chips quantity.

For testing there were used the following formulations of cationic bitumen emulsion, which differed just by the type of acid (Table 2).

Table 2

Cationic bitumen emulsion formulations

Components	Formulation No, % by weight	
Bitumen 70/100 (Motor Oil-Greece) – 65 %	1	2
Redicote EM-44 emulsifier	0,25 %	0,25 %
Water-phase pH (acid)	pH = 2.5 (HCL)	pH = 2.5 (H_3PO_4)

For bitumen emulsions production there was used grade 70/100 bitumen of the Greek company Motor Oil, as well as rapid-setting-emulsions emulsifier Redicote EM-44 of the Netherlands company Nouryon. Physico-technical parameters of these emulsions were determined (Table 3).

In course of breaking index determination upon adding the quartz sand the orthophosphoric-acid-based emulsion was not breaking completely and remaining for the long time in thick liquid state. Besides, H_3PO_4-based bitumen emulsion was characterized by breaking index exceeding the norm even for slow-setting emulsions (170–230). Such special features of emulsion breaking are caused by different chemical nature of interaction of orthophosphoric & hydrochloric acid with quartz sand used for testing. Thus, the emulsion on hydrochloric acid is a rapid-setting one, while the emulsion on orthophosphoric acid is an over-stable one. Besides of that, by the index of miscibility with dense-grade and open-grade mixes the emulsion on H_3PO_4 also shall be referred to a slow-setting one.

Table 3

Physical-technical indices of bitumen emulsions

Name of index	Bitumen emulsion formulations No.		Requirements of DSTU B V.2.7-129:2013 for Cationic rapid-setting emulsion-65
	1 – HCL	2 – H_3PO_4	
1. Appearance	Homogeneous dark-brown liquid		Homogeneous dark-brown liquid
2. Particle charge test, pH	4.20	2.96	1.5–6.5
3. Residue on sieving (sieve No. 014), %	0.01	0.01	Not more than 0.25
4. Binder content, %	64.34	64.15	63–67
5. Viscosity – Efflux time, 4 mm – 25 °C	9.4	9.0	From 15 to 70
6. Miscibility with chips-grading mixes, yes/no: – open-grade; – dense-grade	 no no	 yes yes	 no no
7. Breaking Behaviour (index), %	84.02	245.11	50–130

Determination of adhesion for the (extracted from emulsion) binder with chips surface was done by two methods differing by chips boiling temperature (Table 4). For the testing there were used granite chips from Mokrianskiy and Novograd-Volynskiy limestone chips.

Table 4

Emulsion-to-chips adhesion from three different quarries by methods No. 1 and No. 2

No and formulation of bitumen emulsion	Emulsion-to-chips adhesion from quarry in points		
	Mokrianskiy (granite)	Novograd-Volynskiy (granite)	Monastyrskiy (limestone)
	Method No. 1 (T = 95±1 °C)		
1. Bitumen: 65 % Redicote EM-44 pH = 2.5 (HCL)	3.5	4.0	3.5
2. Bitumen: 65 % Redicote EM-44 pH = 2.5 (H_3PO_4)	4	4.0	4.5
	Method No. 2 (T = 95±1 °C)		
1. Bitumen: 65 % Redicote EM-44 pH = 2.5 (HCL)	2.5	4.0	3.0
2. Bitumen: 65 % Redicote EM-44 pH = 2.5 (H_3PO_4)	3.5	4.0	3.5

Results and discussion

By analyzing Table 4 we determine that (regardless from testing method at high temperatures) the best adhesion indices are inherent to bitumen emulsion on orthophosphoric acid (besides of testing on Novograd-Volynskiy granite chips). When using limestone chips there were observed the following specific features during boiling: the distilled water acquiring dark-brown coloring, with crumbling of rock-particles from limestone chips.

To provide for testing on binder-to-filling-aggregate adhesion by shock method with application of Vialit plate at operation temperatures (determination of mechanical adhesion and brittle temperature) there were used the pre-screened grade of granite chips 5–10 mm according to "DSTU EN 12272-3:2020". Actually, there were chosen 100 chips of each grade for each plate. For the testing there were chosen Novograd-Volynskiy granite chips among the rest, as far as their adhesion indices (Table 4) are the same by two proposed methods at high temperatures – for emulsion on both the hydrochloric and orthophosphoric acids.

Determination of adhesion of extracted-from-emulsion binder with chips-surface by two methods (Table 4) has shown that usage of orthophosphoric acid for the production of bitumen emulsion is optimum by criterion of adhesion at high temperatures. Still, the testing by Vialit method has shown that by criterion of adhesion at low operation temperatures the advantage shall be given to the usage of bitumen emulsion with hydrochloric acid.

The first samples with emulsions on orthophosphoric and hydrochloric acids for determination of mechanical adhesion were tested at the temperature of 14 °C, while the subsequent ones at the temperature of 10 °C. Besides, there was determined the brittle temperature by Vialit method, and it constituted 5 °C (Table 5).

Table 5

Mechanical adhesion and brittleness indices by Vialit method with Novograd-Volynskiy chips

Bitumen emulsion formulation	Adhesion indices by Vialit method				
	Test No. and temperature	*a*	*b*	*c*	*R, %*
Mechanical adhesion					
1. Bitumen: 65 % Redicote EM-44 pH = 2.5 (HCL)	I – 14 °C	0	5	95	94.18
	II – 10 °C	0	38	62	52.72
2. Bitumen: 65 % Redicote EM-44 pH = 2.5 (H_3PO_4)	I – 14 °C	0	18	82	72.72
	II – 10 °C	0	70	30	18.82
Brittle temperature					
1. Bitumen: 65 % Redicote EM-44 pH = 2.5 (HCL)	I – 5 °C	0	57	43	39.88
	II – 5 °C	0	50	50	48.76
2. Bitumen: 65 % Redicote EM-44 pH = 2.5 (H_3PO_4)	I – 5 °C	0	53	47	43.51
	II – 5 °C	0	73	27	24.91

Conclusions

1. There were chosen two testing methods: adhesion with chips by methods of boiling in distilled water and in water with glycerin and adhesion of binder with filling aggregate by shock method with application of Vialit plate, due to which there was determined the efficiency of developed bitumen emulsion formulations by criterion of high and operational (low) temperatures.

2. There were studied the physical-technical indices of bitumen emulsions for thin-layer (slurry) motor-road pavements by surface dressing technology for the formulations which differed by the type of the acid used (hydrochloric or orthophosphoric).

3. It was proved that the special influence upon binder-to-granite-chips adhesion is made due to change of testing temperature modes. At the temperature of 95 °C and 100 °C the adhesion of emulsion on orthophosphoric acid is better than that one on hydrochloric acid, while at the temperatures of 5 °C, 10 °C and 14 °C there is observed the better adhesion of emulsion with hydrochloric acid. The thin-layer (slurry) pavement performed by method of surface dressing works at operational temperatures. Therefore, the

Vialit method is the preferential one for determination of adhesion by this technology, while the optimum formulation for cationic bitumen emulsion is that one with application of hydrochloric acid.

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ЗЧЕПЛЮВАНІСТЬ ДОРОЖНІХ БІТУМНИХ ЕМУЛЬСІЙ НА СОЛЯНІЙ ТА ОРТОФОСФОРНІЙ КИСЛОТАХ ДЛЯ ТЕХНОЛОГІЇ ПОВЕРХНЕВОЇ ОБРОБКИ

Розглянуто шари зносу за технологією поверхневої обробки, що є варіантом збереження та герметизації верхніх шарів дорожніх конструкцій. Однією із ключових характеристик якості цих шарів є зчеплюваність (адгезія) між бітумним та мінеральним матеріалом. В статті досліджено зчеплюваність кам’яних матеріалів різних гранітних кар’єрів України із бітумним в’яжучим, а саме бітумною емульсією, для застосування в технології поверхневої обробки дорожніх покриттів. В роботі використано українські та європейські методи досліджень визначення зчеплюваності в системі бітум – кам’яний матеріал. За українським методом визначено зчеплюваність залишкового в’яжучого, виділеного з емульсії після її розпаду, із поверхнею щебеню після кип’ятіння у дистильованій воді та в розчині гліцерину і дистильованої води. Як європейський метод для досліджень використано ударний метод із застосуванням плити Віаліт. Розроблено два склади бітумних емульсій для тонкошарових покриттів автомобільних доріг за технологією поверхневої обробки залежно від типу використаної кислоти (соляна чи ортофосфорна). Результати випробувань за вибраними методами схожі, що дало змогу вибрати оптимальний кам’яний матеріал серед досліджених і відповідно оптимальний склад бітумної емульсії. Доведено, що на зчеплюваність в’яжучого із гранітним щебенем особливо впливає зміна температурних режимів ведення випробувань. За температур 95 °С та 100 °С зчеплюваність емульсії на ортофосфорній кислоті вища, ніж у соляної, а за 5 °С, 10 °С, 14 °С краща зчеплюваність у емульсії із соляною кислотою.

Ключові слова: тонкошарове дорожнє покриття; поверхнева обробка; бітумна емульсія; соляна кислота; ортофосфорна кислота; гранітний щебінь; вапняковий щебінь; адгезія емульсії до щебеню.