Quick-traffic slurry surfacing mix with orthophosphoric acid

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Abstract
Bitumen emulsions for slurry surfacing mix technology using oxidized bitumen and hydrochloric and orthophosphoric acids on laboratory DenimoTech bitumen-emulsion plant are made in the work. Methylene blue adsorption index of granite aggregate for use in slurry surfacing mix was investigated. Comparatively mix time and cohesion strength build-up of selected compositions slurry surfacing mix with hydrochloric and orthophosphoric acids depending on the variable content of bitumen emulsion. The advantage of using orthophosphoric acid in slurry surfacing mix according to the cohesion strength build-up criterion has been proved. The importance of correct distribution of bitumen drops in the emulsion was confirmed using a sedimentograph Mastersizer 2000. Two emulsions of the same component composition were compared, which differed in particle size. It has been established that it is not possible to design a slurry surfacing mix using the mix time criterion with the help of coarse bitumen emulsion.

1. Introduction

Today, the preservation of the existing road surface and the prevention of its destruction over time remains relevant. One of the progressive materials that solves this problem, both at the construction stage and at the stage of operation is a thin-layer pavement with slurry surfacing mix: slurry seal and micro surfacing (Bhargava et al., 2019; Demchuk et al., 2018; Gujar and Vakharia, 2019; Hou et al., 2018; Grilli et al., 2021; Sidun et al., 2019).

In addition, these coatings are economically feasible that will allow lean concept in the construction industries (Ulewicz and Ulewicz, 2020; Bobalo et al., 2021). The main function of the slurry surfacing mix is to seal the existing coating from damage and increase the roughness and coefficient of adhesion, which allows you to postpone the overhaul of the road.

2. Literature review

The classical formulation of bitumen cationic emulsion for slurry surfacing mix consists of oil road bitumen, emulsifier and water. It is known that the market of emulsifiers is rather broad, while the usage of different acids in this technology, as a rule, is restricted. The most popular for the use in emulsion still remains the hydrochloric acid (HCl). For its part, bitumen – as per technological method of production – can be used as oxidized or distilled (Zulu et al., 2018; Broughton et al., 2012; Labi et al., 2007; Yu et al., 2017; Watson and Jared, 1998; Smith and Beatty, 1999; Arbia et al., 2016; Robati et al., 2013; Giorgi et al., 2016; Mahmoud et al., 2015; Hwan et al., 2015). But for this technology, distillation bitumen from heavy high-resinous, low-paraffinic oil, usually from Venezuelan fields is optimal (Giorgi Claude et al., 2016; Pyshyev et al., 2015; Sidun et al., 2021). The topic of using oxidized bitumens in slurry surfacing technologies causes interest both among the producers and the scientists. Complexity and limitedness of usage for such bitumen consists in the problem of cohesion strength build-up in the mix laid. When using the optimal bitumens cohesion strength build-up falls within the 30 minutes limit. Systems with oxidized bitumen require up to 6-8 hours to reach the required indices of cohesion strength, while at low temperatures (+10°C) and high relative humidity the time for mix structure formation raises several times, and it does not correspond to the modern world-wide requirements for this technology. In addition, the particle size of the bitumen emulsion plays an important role in the formation of the slurry surfacing mix.

Bitumen and bitumen emulsions is not one and the only problematic section in slurry surfacing technology. As a mineral component of the road pavement the aggregate is used. In Slurry surfacing technology it is fine grade up to 15 mm. With
regard to such material very abruptly appears the problem of aggregate “reactivity”. Under the notion of aggregate “reactivity” we understand the presence of absorbing components (such as clay) in the pulp. It leads to rapid setting of bitumen emulsion, and – as a consequence – impossibility to receive the homogeneous mix, which is the essence of Slurry surfacing technology. The review of a number of published works on the topic of bitumen emulsions and Slurry surfacing has shown the possibility of using ortho-phosphoric acid (H$_3$PO$_4$) instead of hydrochloric one in this technology (James, 2005; James, 2006; Hogendoorn, 2016; Solodkyy et al., 2018; Sidun et al., 2020). In their turn, a lot of investigators encourage us to use the mixes with different chloric among them.

For the subsequent testing there was chosen the most reactive one in this technology. That is because (in case of correct mix design) the main purpose of this work is efficiency determination for using systems, produced from oxidized bitumen on ortho-phosphoric acid (in comparison with similar systems on hydrochloric acid), by the indices of cohesion strength build-up rate. Besides of that – determination of possibility of using reactive aggregates in such systems.

3. Experimental

Slurry surfacing testing was done according to the norms of International Slurry Surfacing Association (ISSA Technical Bulletin: A105, 2010, A 143, 2010, TB-113, 2017, TB-139, 2017). First there were produced two road bitumen emulsions on laboratory DenimoTech bitumen-emulsion plant SEP-0.3R by formulations No. 1 and No. 2 (Table 1), which differ only by type of the acid used.

### Table 1. Formulations of bitumen emulsions

| Emulsion components | Percentage of components in emulsion, % w/w. |
|---------------------|-----------------------------------------------|
|                     | No. 1  | No. 2  |                     |
| Bitumen             | 62     | 62     |                     |
| Redicote EM44 emulsifier | 1.1     | 1.1     |                     |
| Orthophosphoric acid | –      | –      | till pH 2.5 in water phase |
| Hydrochloric acid   | till pH 2.5 in water phase | –      |                     |
| Water               | Till 100 |        |                     |

For emulsion production there was used the oxidized bitumen produced by PJSC Ukrtatnafta (Ukraine), which corresponds to the brand 70/100 (EN 12591, 2009) and emulsifier Redicote EM44, produced by Nouryon (Sweden).

Different variants of Ukrainian granite aggregate were tested by criterion of absorption of methylene-blue (Table 2). For the subsequent testing there was chosen the most reactive among them - Ushytyskiy quarry’s aggregate.

### Table 2. Methylene blue adsorption index granite aggregate

| Quarry            | Methylene blue index, ml |
|-------------------|--------------------------|
| Vyrivskiy         | 10                       |
| Rokytynianski     | 17                       |
| Ushytyskiy        | 20                       |
| Vynogradivskiy    | 15                       |

For slurry surfacing mixes (type 1) production there were used Portland cement brands for strength, drinkable water, control additive (10% solution of emulsifier Redicote E-11, produced by Nouryon). Particle size distribution of bitumen emulsions was performed using a sedimentograph MasterSizer 2000 from Malvern Instruments (England).

### 4. Results and Discussion

Due to the use of the chosen aggregates and produced by formulations No. 1 and No. 2 bitumen emulsions there were selected the optimum slurry surfacing mix compositions, formed and tested the corresponding mix samples – by the index of cohesion strength build-up rate at the ambient temperatures of +25°C. The mix design was done based on setting-time parameter for the mix, while this parameter was determined as time period starting from the moment of blending of all the components of the mix and till the moment when the mix loses its mobility. To check the influence of the bitumen emulsion itself (that is, the type of acid) upon the mix there was decided to use the mixes with different emulsion content (12, 14 and 16 components – see Table 3). As far as the cement content in the systems on ortho-phosphoric acid is very important for stability, so in the system on hydrochloric acid it was also decided to use the constant quantity of cement, but a little bit changed – in order to reach the required setting time for the mix (> 180 sec). The water content was changed in inverse proportion to the content of emulsion – to achieve the necessary flowability of the mix.

### Table 3. Slurry surfacing mix designs on emulsion No. 1 and No. 2

| No. | Components content, g | Mix time, s |
|-----|-----------------------|-------------|
|     | aggregate              | cement      | water | control additive | emulsion |         |
| 1.1 | 100                   | 1.0         | 15.0  | 2.0             | 12       | 185     |
| 1.2 | 100                   | 1.0         | 13.0  | 2.0             | 14       | 181     |
| 1.3 | 100                   | 1.0         | 8.0   | 2.2             | 16       | 189     |

Mix-design based on emulsion No. 1

| No. | Components content, g | Mix time, s |
|-----|-----------------------|-------------|
|     | aggregate              | cement      | water | control additive | emulsion |         |
| 2.1 | 100                   | 0.8         | 14.0  | 2.5             | 12       | 182     |
| 2.2 | 100                   | 0.8         | 12.0  | 2.5             | 14       | 182     |
| 2.3 | 100                   | 0.8         | 10.0  | 2.3             | 16       | 184     |

The optimum formulations presented in Table 3 were used to form the mix samples in the rings of 60 mm in diameter and
6 mm high – to determine cohesion strength build-up rate by the means of modified cohesion tester. The objective of experiment was determination of time-period required for slurry surfacing sample to achieve the necessary strength to start traffic with speed limit up to 40 km/hour (type of destruction NS at the corresponding torque) and without speed limit (type of destruction SS at the corresponding torque) (Table 4).

Table 4. Indices of cohesion strength build-up rate

| Mix-design | Time, min | 60 | 120 | 180 | 240 | 300 | 360 | 420 |
|------------|-----------|----|-----|-----|-----|-----|-----|-----|
| Destruction|           | N  | N   | N   | S   | S   | S   |     |
| Torque, kg cm |       | 12 | 12  | 12  | 20  | 23  | 23  | 23  |

| Mix-design | Time, min | 60 | 150 | 200 | 210 | 270 | 300 | 360 |
|------------|-----------|----|-----|-----|-----|-----|-----|-----|
| Destruction|           | N  | N   | N   | NS  | S   | S   | S   |
| Torque, kg cm |       | 12 | 12  | 12  | 20  | 23  | 23  | 23  |

| Mix-design | Time, min | 30 | 60  | 90  | 150 | -   | -   | -   |
|------------|-----------|----|-----|-----|-----|-----|-----|-----|
| Destruction|           | N  | N   | NS  | S   | S   | S   |     |
| Torque, kg cm |       | 12 | 12  | 12  | 20  | 26  | -   | -   |

| Mix-design | Time, min | 30 | 60  | 90  | 120 | 180 | 240 | 300 |
|------------|-----------|----|-----|-----|-----|-----|-----|-----|
| Destruction|           | N  | N   | NS  | S   | S   | S   | S   |
| Torque, kg cm |       | 12 | 12  | 12  | 12  | 12  | 12  |     |

| Mix-design | Time, min | 30 | 60  | 90  | 120 | 150 | -   | -   |
|------------|-----------|----|-----|-----|-----|-----|-----|-----|
| Destruction|           | N  | N   | NS  | S   | SS  | S   |     |
| Torque, kg cm |       | 12 | 12  | 12  | 20  | 23  | 26  | -   |

| Mix-design | Time, min | 30 | 60  | 90  | 90  | 105 | -   | -   |
|------------|-----------|----|-----|-----|-----|-----|-----|-----|
| Destruction|           | NS | NS  | NS  | S   | SS  | S   |     |
| Torque, kg cm |       | 20 | 20  | 20  | 23  | 26  | -   | -   |

Because of the high water content mix designs 1.1, 2.1 harden slowly, respectively, these compositions are not acceptable. All other slurry surfacing formulations were selected for comparison (Fig. 1).

As one can see on the figure 1, at +25°C the mix on orthophosphoric acid (with emulsion content 16 components) already after 30 min allows start of traffic with speed limit up to 40 km/hour, while for hydrochloric acid system it will take 3 times more. Possibilities of starting traffic without limitations also are on the part of orthophosphoric-acid-based mixes. With more detailed examination of cohesion strength build-up rate for the mix on hydrochloric acid for composition No. 1.3 (emulsion content 16) and the mix on orthophosphoric acid (emulsion content 14) it proves to be practically identical. Still, for the mix on hydrochloric acid with emulsion content 14 the indices of “starting traffic without limitations” are not reached even after 6 hours. Achieving fast cohesion strength build-up mix on orthophosphoric acid can be explained by the interaction of cement with orthophosphoric acid. Acid and cement form calcium ions, but the level of soluble calcium ions in the orthophosphoric acid system is lower. Calcium ions reduce the adsorption of emulsifiers by minerals, measured by the methylene blue indicator, so minerals become more reactive in phosphoric acid systems. In addition, the charge of the cement itself becomes positive in the hydrochloric acid system and negative in the orthophosphoric acid system. Therefore, the cement itself can directly react with the positively charged emulsion droplets..

To establish the importance of the size of the distributed drops of bitumen in the emulsion, the particle size distribution of emulsion No. 2 (Fig. 2a) was established and an emulsion of similar composition with No. 2, but with non-standard size of bitumen drops in the emulsion (Fig. 2b) was made.

Fig. 2. Particle size distribution of emulsions: a) - with standard particle size, b) - with non-standard.

Indexes of particle size distribution are given in Table 5.
Table 5. Indexes of particle size distribution

| Indexes          | a)   | b)   | Recommendations Nosuryay |
|------------------|------|------|-------------------------|
| D 10, µm         | 1.95 | 9.821| 1 - 2                   |
| D 50, µm         | 3.624| 30.140| 3 - 8                  |
| D 90, µm         | 6.994| 81.247| 10 - 20                |
| Specific surface, m²/g | 1.85 | 0.328| -                       |
| D[3.2], µm       | 3.252| 18.312| -                      |
| D[4.3], µm       | 4.224| 38.943| -                      |

Slurry surfacing mix (Table 6) was made on the basis of bitumen emulsions No. 2a) and No. 2b). Mix time emulsion No. 2b) is unstable, because without control additive the decomposition of the mixture occurs instantly, and with the inclusion of a minimum amount of control additive the decomposition of the mixture occurs extremely late, which will lead to late hardening of the coating.

Table 6. Slurry surfacing mix designs on emulsion No. 2a) and b)

| No. 1 | Components content, g | Mix time, s |
|-------|-----------------------|-------------|
| 1.    | aggregate: 100, cement: 8.0, water: 12.0, control additive: 2.5, emulsion: 14.0 | 187         |
| 1b    | Mix-design based on emulsion No. 2b)                                                                 |
|       | 100, 8.0, 12.0, - 0.2, 14.0 | 04 358      |
| 2b    | Mix-design based on emulsion No. 2b)                                                                 |

Therefore, smaller and monodisperse particles help to optimize the properties of the emulsion, such as viscosity, disintegration rate and adhesion, the best results are given by a narrow range of distribution of small particles 1-5 µm.

4. Summary and conclusion

The possibility was proven for the use (in slurry surfacing technologies) of aggregates with high index of methylene blue (i.e. high reactivity) when applying emulsions on oxidized bitumens with orthophosphoric acid.

The novelty of the obtained results is due to the fact that was determined slurry surfacing mixes on orthophosphoric acid need less quantity of the control additive to reach the required setting time for the mix.

There was proven the advantage of using emulsions on orthophosphoric acid comparatively to emulsions on hydrochloric acid – in slurry surfacing mixes on oxidized bitumens: with the equal content of bitumen emulsion the usage of orthophosphoric acid increases the cohesion strength build-up rate. It provides for starting traffic without limitations at lower emulsion content in the mix (if to compare with hydrochloric acid), while it also allows saving bitumen emulsion (bitumen).

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与正磷酸的快速流动泥浆堆焊混合料

关键词
泥浆铺面混合物
沥青乳液
正磷酸
盐酸
氧化沥青

摘要
在实验室 DenimoTech 沥青乳液设备上，使用氧化沥青和盐酸和正磷酸为泥浆表层混合技术制造沥青乳液。研究了用于泥浆堆焊混合料的花岗岩骨料的甲基蓝吸附指数。根据沥青乳液的可变含量，比较混合时间和所选组合物的泥浆表面混合物与盐酸和正磷酸的混合时间和内聚强度增加。根据内聚强度建立标准，在浆料堆焊混合料中使用正磷酸的优势已被证明。使用沉积仪 Mastersizer 2000 确认了乳液中沥青滴正确分布的重要性。比较了两种具有相同组分组成但粒径不同的乳液。已经确定不可能在粗沥青乳液的帮助下使用混合时间标准来设计泥浆表层混合物。