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OVER-STABILISED BITUMEN EMULSION FOR SLURRY SURFASING BY MIX TIME CRITERION

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Slurry Surfacing was investigated using over-stabilised emulsions and highly reactive stone material with a methylene blue index of 18 at high temperatures. Unmodified emulsions were produced on four different emulsifiers at the same dosage –1.2 % by mass of the bitumen emulsion itself. Slurry Surfacing was designed on the basis of the investigated emulsions, screening of the 0–4 mm fraction, water, Portland cement and mix time regulator. Setting the mix time for each of the Slurry Surfacing based on six emulsions made it possible to state that there is a relationship between the characteristic of the breaking behavior of the bitumen emulsion mixing stability with cement and mix time. It was established that at a temperature of 30 °C, the use of an over-stabilised emulsion with a special emulsifier at its dosage of 1.2 % allows the production of mixtures with highly reactive screenings without including a mix time regulator in the mixture.

Keywords: over-stabilised bitumen emulsion, Slurry Surfacing, breaking behaviour, mixing stability with cement, mix time, cohesive strength.

Introduction

Slurry Surfacing (SS) is a bitumen-emulsion technology used to install thin-layer coatings for wearing courses of highways, city streets, airfields, and parking lots. The SS technology dates back to the end of the 19th century and has gradually gained widespread use due to its obvious advantages (Grilli, 2019; Johnson, 2007; Broughton, 2012; Shropshire, 2011; Hajj, 2011; Peshkin, 2004; Hajj, 2013; Wang, 2013; Hu, 2022; Bhargava, 2019). Like all technologies using bitumen emulsions, SS has limitations regarding low application temperatures. The installation of wearing courses with SS is allowed at air temperatures not lower than 5 °C and in the absence of precipitation. As a rule, wearing courses with SS are arranged in late spring, summer and early autumn, when the ambient temperature is close to or above 20 °C. But when the ambient temperature reaches about 30 °C, this rapidly affects the mix time of SS. It becomes shorter, which can lead to premature breaking of the mixture in the slurry spreader, which will lead to a stoppage of work and the need to clean the machine's bunkers from the mixture that is no longer cast. Therefore, at high temperatures, it is recommended to increase the emulsifier content in the bitumen emulsion or increase the content of the mix time regulator (additive) in the form of an aqueous solution of a special emulsifier (surfactants such as fatty polyamines, amidoamines, imidazolines and quaternary ammonium salts) in the mixture. At the stage of designing SS in laboratory conditions, as a rule, the emulsifier content is close to 1.0 wt. % bitumen emulsion, and the amount of mix time regulator depends on the “reactivity” of the stone material according to the methylene blue (MB) indicator, the composition of the bitumen emulsion and its acidic pH, the amount of water and Portland cement in the mixture and the temperature and humidity of the environment. (Jamion, 2014; Sun, 2014; Ye, 2017; Lerose, 2019; Ceratti, 2011; Kucharek, 2010; Sidun, 2023).

In turn, increasing the mix time regulator can negatively affect the consistency and speed of gaining cohesive strength, and as a result, the opening of traffic even at high temperatures can be long. In addition, the use of highly reactive stone materials also forces an increase in the amount of additive in the mixture. In order to minimize the amount of additive, the content of the classical emulsifier in the emulsion can be increased, but in practice this method is rarely used. Another promising option for this is the use of over-stabilised bitumen emulsions at high temperatures of the installation and use of highly reactive stone materials in the composition of SS (Usman, 2019; Sidun, 2019). However, it remains not fully studied how over-stabilised emulsions will interact with Portland cement during mixing of the mixture and what their effect will be on mix time and cohesion of mix.

Therefore, the aim of the work is to investigate SS compositions using over-stabilised emulsions and highly reactive stone material at high temperatures.

Materials and methods

To produce bitumen emulsions, traditional emulsifiers for slow-setting emulsions were used: Redicote E-11, Redicote C-320E, Polyram L950, and the emulsifier for super-stable Redicote E 4875 NPF. The bitumen for the emulsions was 70/100 PKN Orlen, and two acids were also used: hydrochloric and orthophosphoric (Table 1).

Table 1

Compositions of emulsions

No. bitumen emulsion	Bitumen content, % by mass	Emulsifier Redicote		Type of acid	pH in the aqueous phase
		brands	Content, % by mass		
1	60	Redicote E-11	1.2	HCl	2.5
2		Redicote E4875 NPF			
3		Polyram L950			
4		Redicote C-320E			
5		Polyram L950		H ₃ PO ₄	
6		Redicote C-320E			

For the preparation and manufacture of SS samples, in addition to BE, a screening with an MB index of 18 ml (determined according to SOU 42.1-37641918-119:2014), Portland cement, drinking water and a disintegration regulator (10 % solution of the emulsifier Redicote E-11) were also used. The screening was deliberately chosen to be “reactive” with a high MB index, because according to SOU 42.1-37641918-119:2014, the MB should not exceed 10 ml.

For all eight bitumen emulsions, physical and mechanical properties were determined according to DSTU B V.2.7-129:2013 (Table 2). The results of determining the characteristics of the breakdown of bituminous emulsions, namely: breaking behavior (index) according to DSTU B V.2.7-129:2013, fines mixing time according to EN 13075-2 and mixing stability with cement according to EN 12848 are given in Table 2. SS were investigated in accordance with ISSA A105, A143 and SOU 42.1-37641918-119:2014 standards by indicators: mixture breakdown, cohesive strength.

Results and discussion

In Table 2, the emulsion based on the specialized Redicote E4875 NPF is the most resistant to breaking in terms of its breaking behavior characteristics, which confirms previous studies (Sidun, 2023, 2023; Bidos, 2023). The fastest breaking is characterized by a special emulsion for fast-hardening SS based on Redicote C-320E 1.2 H₃PO₄, which is also consistent with studies (Sidun, 2019, 2021, 2023). It is also indicative that according to EN 13808, the mixing stability with cement according to the operational class 10 is ≤ 2 . This class can be attributed to the most resistant emulsions in terms of mixing stability with cement (Bidos, 2023, 2024). According to Table 2, the requirements for class 10 of EN 13808 are met by

bitumen emulsion No. 3–5. Mixing stability with cement is an indicative parameter for those technologies in which the emulsion is used with Portland cement, and SS is one of them. Analysis of Table 2 shows that the bitumen emulsion on the Redicote C320E emulsifier disintegrates most quickly at the breaking value and during mixing stability with cement.

Table 2

Physical and mechanical properties of bitumen emulsions

No. bitumen emulsion	Particle charge test, pH	Residue on sieving (sieve No. 014), %;	Binder content, %	Viscosity – Efflux time, 4 mm – 25 °C	Stability during storage: residue on sieving (sieve No. 014), %, not more than:		Breaking behavior		
					– after 7 days	– after 30 days	Breaking value (index), %	Fines mixing time	Mixing stability with cement
1 (C320E-1.2 % HCl)	5.30	0.01	59.28	8.6	0.08	0.04	243	>300	64.3
2 (L950 -1.2 % HCl)	3.70	0.08	59.49	10.0	0.08	0.15	306		51.3
3 (E4875 NPF-1.2 % HCl)	2.80	0.03	59.37	7.6	0.05	0.07	330		0.1
4 (E11-1.2 % HCl)	3.01	0.02	59.19	8.8	0.05	0.08	263		1.6
5 (L950 H ₃ PO ₄ -1.2 %)	2.19	0.07	60.39	10.8	0.04	0.06	314		2.1
6 (C-320E 1.2 H ₃ PO ₄)	2.33	0.02	59.92	7.6	0.07	0.09	242	291	81.3

To determine the breaking characteristic for SS, the mix time indicator is used, which should be at least 120 s or 180 s, depending on the type of SS. In our case, for SS based on screenings and unmodified bitumen emulsions, it should be at least 180 s. The mix time for each of the emulsions was determined based on the basic composition (Table 3), in which all components except the mix time regulator were constant. The mix time was determined at a temperature of 20 °C.

Table 3

Basic composition of SS

Content of components, parts				
Dropped out (MB 18)	Portland cement	Water	Emulsion	Disintegration regulator
100.0	1.0	10.0	14.0	variable

In Table 4, the amount of mix time regulator required for mix time of at least 180 s was selected. Only SS on bitumen emulsion 3. E4875 NPF-1.2 % HCl does not require a mix time regulator at all to achieve a value of 180 s or more. However, it is indicative that this emulsion leads to a mix time of up to 240 s, that is, it is the most stable among the studied ones according to the breaking criterion. If we draw an analogy between Table 3 and Table 4, then there is a convergence between mixing stability with cement and mix time SS. That is, those emulsions that quickly breaking when mixed with cement (mixing stability with cement is significantly more than 2) do not form SS according to the mix time criterion without a mix time regulator.

Table 4

**The amount of disintegration regulator required
for a mix time of at least 180 s at a temperature of 20 °C**

No. bitumen emulsion	Disintegration regulator, parts	Mix time, c
1. C320E-1.2 % HCl	–	immediately
	3.2	185
2. L950 -1.2 % HCl	–	immediately
	2.2	188
3. E4875 NPF-1.2 % HCl	–	249
4. E11-1.2 % HCl	–	32
	1.0	184
5. L950 1.2 % H ₃ PO ₄	–	immediately
	1.2	186
6. C-320E 1.2 H ₃ PO ₄	–	immediately
	1.5	184

For further studies at a temperature of 30 °C (Table 5), bitumen emulsion No. 3. E4875 NPF-1.2 % HCl was used, as the most stable according to the mix time criterion, and No. 4. E11-1.2 % HCl, which requires the smallest amount of mix time regulator compared to others to achieve a mix time of at least 180 s. The temperature of all components, as well as the temperature of the room in which the test was conducted, was 30 °C. Analysis of Table 5 shows that the use of a super-stable emulsion with a special emulsifier Redicote E4875 NPF at its dosage of 1.2 % allows the production of SS with highly reactive screening (MB 18) and a temperature of 30 °C without including a mix time regulator in the mixture. In contrast, 0.4 of the mix time regulator must be included in the composition of the SS on an emulsion with Redicote E11-1.2 % by weight. Regarding the cohesive strength of SS, both compositions showed the same time for opening the movement of vehicles, which was determined by the nature of the fracture of SS samples:

– Near Spin fracture in the form of a single radial crack. It characterizes the self-compacting stage of the mixture, it is possible to open the movement with a speed limit of up to 40 km/h and a prohibition of turning. The equivalent cohesion value is ~ 26 kg·cm.

– Solid Spin is characterized by the absence of fractures, but displacement or removal of binder particles is possible. It characterizes the hardening stage of the mixture, it is possible to open the movement without prohibitions. The equivalent cohesion value is ~ 26 kg·cm.

Table 5

Mix time and cohesive strength of SS at 30 °C

No. bitumen emulsion	Disintegration regulator, parts	Mix time, c	Test time, hours: minutes	Nature of destruction
3. E4875 NPF-1.2 % HCl	–	185	2:00	Normal Spin (Solid Spin)
4. E11-1.2 % HCl	1.7	183	(3:00)	

In terms of cost-effectiveness, the SS composition with the special emulsifier Redicote E4875 NPF is 42 % cheaper than the composition with Redicote E11, due to the lower cost of Redicote E4875 NPF than with Redicote E11 and the absence of the need to include a disintegration regulator in the SS.

Conclusions

The relationship between the characteristic of the breaking behavior of bitumen emulsion mixing stability with cement and mix time of unmodified Slurry Surfacing based on screenings has been

established. Emulsions that are characterized by a higher mixing stability with cement require a larger amount of mix time regulator based on the mix time criterion (not less than 180 s).

It has been determined that over-stabilised cationic bitumen emulsions for Slurry Surfacing technology, namely the composition with Redicote E 4875 NPF, should be used when using highly reactive screenings and ambient temperatures of 30 °C and above. Such Slurry Surfacing does not require a mix time regulator of the mixture, which can lead to technological and economic advantages.

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НАДСТІЙКІ БІТУМНІ ЕМУЛЬСІЇ ДЛЯ ЛИТИХ ЕМУЛЬСІЙНО-МІНЕРАЛЬНИХ СУМІШЕЙ ЗА КРИТЕРІЕМ РОЗПАДУ СУМІШІ

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Досліджено литі емульсійно-мінеральні суміші із використанням надстійких емульсій та високо-реактивного кам'яного матеріалу із показником метилену синього 18 за високих температур. Немодифіковані емульсії виготовляли на шести різних емульгаторах за однакового їх дозування –1,2 % масових від маси самої бітумної емульсії. Чотири бітумні емульсії були виготовлені за допомогою хлоридної кислоти, а дві за допомогою ортофосфорної. Встановлено їхні основні фізико-механічні властивості, особливу увагу приділено характеристикам розпаду. Литі емульсійно-мінеральні суміші проектували на основі досліджених емульсій, відсіву фракції 0–4 мм, води, портландцементу та регулятора розпаду. Встановлення часу змішування для кожної із литих емульсійно-мінеральних сумішей на основі шести емульсій дало підставу стверджувати, що є взаємозв'язок між такою характеристикою розпаду бітумної емульсії, як стійкість під час змішування із портландцементом, та часом змішування. Емульсії, які характеризуються стійкістю під час змішування з портландцементом значно більше ніж 2, не здатні за критерієм часу змішування (не менше ніж 180 с) утворювати суміші без регулятора розпаду. Дослідивши литі емульсійно-мінеральні суміші за критерієм часу змішування за температури 20 °С, ми вилучили чотири емульсії для подальшого дослідження за температури 30 °С. Було вибрано дві емульсії, одна з яких є надстійкішою за критерієм часу змішування, а друга потребує порівняно з іншими найменшу кількість регулятора розпаду для досягнення часу змішування не менше ніж 180 с. Встановлено що за температури 30 °С використання надстійкої емульсії зі спеціальним емульгатором за його дозування 1,2 % дає змогу виготовляти суміші із високореактивним відсівом без введення регулятора розпаду в суміш.

Ключові слова: надстабільна бітумна емульсія, литі емульсійно-мінеральні суміші, характеристики розпаду, стабільність змішування із цементом, час змішування, когезійна міцність.

Подяка. Робота виконана у межах реалізації проєкту “Інноваційні комплексні підходи для відновлення транспортних споруд” від Національного фонду досліджень України (грант № 2023.05/0026).