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OVER-STABILIZED BITUMEN EMULSIONS MADE FROM EMULSIFIERS FOR SLOW-SETTING EMULSIONS

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Formulations of road over-stabilized cationic bitumen emulsions with orthophosphoric, sulfuric and hydrochloric acids and cationic emulsifiers for slowly disintegrating emulsions have been designed. The physical and mechanical properties of the manufactured over-stabilized bitumen emulsions and their breaking behavior were determined. It has been established that over-stabilised bitumen emulsions demonstrate good storage stability for 180 days. The obtained results of breaking behavior bitumen emulsions indicate that emulsions based on hydrochloric and sulfuric acid have a shorter fines mixing time and greater mixing stability with cement than emulsions based on orthophosphoric acid. For over-stabilized bitumen emulsions on orthophosphoric acid, the method according to DSTU EN 12848:2020 is not effective, due to the high reactivity of emulsions on orthophosphoric acid during interaction with portland cement.

Keywords: cationic bitumen emulsions, hydrochloric acid, orthophosphoric acid, sulfuric acid, breaking behaviour, mixing stability with cement.

Introduction

Cationic bitumen emulsion is a popular material in road construction (Sidun, 2019; Sidun, 2019; Dołżycki, 2019; Pérez, 2013; Liu, 2020). Breaking behavior and storage stability are among the most important physical and technical properties of these emulsions. According to DSTU B V.2.7-129:2013, according to the breaking value, emulsions are divided into the following types: rapid-setting, medium-setting and slow-setting. Emulsions for the breakdown of which more mineral material is needed or the end of their breakdown occurs later than in slow-setting can be attributed to over-stabilized (super-stable) (Sidun, 2023). Over-stabilised bitumen emulsion can be used in the following road technologies: installation of slurry surfacing road pavements, installation of base layers using "cold recycling" technology, soil stabilization and others. The use of over-stabilized bitumen emulsion when setting up slurry surfacing road pavements prevents premature disintegration of the mixture directly in the machine. After all, in regions with high air temperature (and as a result, the basis for applying slurry surfacing, the issue of premature breakdown of the mixture is critical (Sidun, 2021; Sidun, 2021; Sidun, 2023, Wang, 2013; Grilli, 2012). During the application of over-stabilized bitumen emulsion in "cold recycling" technology makes it possible to postpone the time of emulsion breakdown, which allows for more thorough mixing of all the components of the mixture during the execution of work with the help of recyclers and to increase the transportation time of the mixture, if it is made in a factory (Grilli, 2013; Grilli, 2018; Grilli, 2016; Pasetto, 2020; Ouyang, 2018; Romeo, 2018; Godenzoni, 2018; Lesueur, 2004; Salomon, 2006). In addition, over-stabilised bitumen emulsions are also characterized by better storage stability, which allows to increase the time for their use.

The purpose of the article was to prove the possibility of obtaining over-stabilized bitumen emulsions using conventional emulsifiers for slow-setting bitumen emulsion.

Materials and Methods

Emulsions were made on oxidized bitumen brand 70/100, produced by PJSC "Ukrtatnafta", and emulsifiers of the company Nouryon (Sweden), and corresponding acids. Orthophosphoric, sulfuric and hydrochloric acids were used with Redicote E-11 emulsifier, and only orthophosphoric acid was used with Redicote C-320E. The compositions of the emulsions are given in Table 1. Bitumen emulsions were produced in laboratory conditions using a bitumen-emulsion installation of the colloid mill type.

Table 1

Compositions of emulsions

№ bitumen emulsion	Bitumen content, % by mass	Emulsifier Redicote		Type of acid	pH in the aqueous phase
		brands	Content, % by mass		
1(HCl;1,1 % E-11)	60	E-11	1,1	HCI	2,5
2(H_2SO_4;1,1 % E-11)		E-11		H_2SO_4	
3(H_3PO_4;1,1 % C-320E)		C-320E		H_3PO_4	
4(HCl;1,1 % E-11)	61	E-11		HCI	
5(H_3PO_4;1,1 % C-320E)		C-320E		H_3PO_4	
6(H_3PO_4;1,2 % C-320E)	62	C-320E	1,2	H_3PO_4	2,3
7(H_3PO_4;1,6 % C-320E)		C-320E	1,6	H_3PO_4	
8(H_3PO_4;1,8 % C-320E)		C-320E	1,8	H_3PO_4	

The physical and mechanical properties of emulsions were studied in accordance with DSTU B V.2.7-129:2013, including the breaking value. Also, breaking behavior of bitumen emulsions was determined according to the requirements of DSTU EN 13808:2020, time (state) to disintegration according to DSTU EN 13075-2:2020, stability during mixing with Portland cement according to DSTU EN 12848:2020

Results and discussion

For all eight bitumen emulsions, physical and mechanical properties were determined according to DSTU B V.2.7-129:2013, as well as additionally storage stability after 180 days (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 DSTU EN 13075-2:2020 and mixing stability with cement according to DSTU EN 12848:2020 are given in Table 3.

Having analyzed the physical and mechanical parameters of the produced emulsions No. 1-No. 3 with the same binder content of 60 % and the emulsifier content in the aqueous phase of 1.1 %, it can be stated that the bitumen emulsion on sulfuric acid has a higher pH and viscosity - efflux time. If we compare bitumen emulsions with the same emulsifier content of 1.1 %, but different binder content of 60 % and 61 % (№1-№5), then emulsions with a higher binder content are characterized by a higher viscosity - efflux time and a higher pH value, although pH in the aqueous phase in them is the same. Comparing emulsions with the same binder content of 62 % and pH in the aqueous phase of 2.3 (No. 6-No. 7), but with different content of emulsifier in the aqueous phase (from 1.2 to 1.8 % by mass), we establish that the pH of emulsions is almost the same, and the viscosity - efflux time becomes higher with an increase in the emulsifier content.

According to Table 2, residue on sieving of manufactured bitumen emulsions is almost the same. In general, there is a dependence between residue on sieving and storage stability for 30 days and 180 days, emulsions that showed better storage stability in the early stages after production, kept this trend during long-term storage.

Analyzing Table 3 and Fig. 1 bitumen emulsion on orthophosphoric acid mixes longer with mineral aggregate, respectively, they have a higher breaking value according to DSTU B V.2.7-129:2013 and fines

mixing time according to DSTU EN 13075-2:2020, but show worse results for determining mixing stability with cement according to DSTU EN 12848:2020.

Table 2

Physical and mechanical properties of bitumen emulsions according to DSTU B V.2.7-129:2013

№ 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:		
					- after 7 days	- after 30 days	- after 180 days
1(HCl;1,1 % E-11)	2,89	0,01	59,82	6,2	0,06	0,12	0,24
2(H2SO4;1,1 % E-11)	3,29	0,01	60,08	8,8	0,04	0,11	0,23
3(H3PO4;1,1 % C-320E)	2,97	0,02	59,73	6,9	0,10	0,22	0,30
4(HCl;1,1 % E-11)	2,95	0,01	60,99	6,4	0,08	0,14	0,26
5(H3PO4;1,1 % C-320E)	3,02	0,02	60,87	7,0	0,11	0,26	0,40
6(H3PO4;1,2 % C-320E)	3,10	0,02	61,85	7,6	0,13	0,28	0,42
7(H3PO4;1,6 % C-320E)	3,06	0,01	62,31	7,8	0,15	0,29	0,43
8(H3PO4;1,8 % C-320E)	3,08	0,01	62,18	8,0	0,12	0,28	0,41

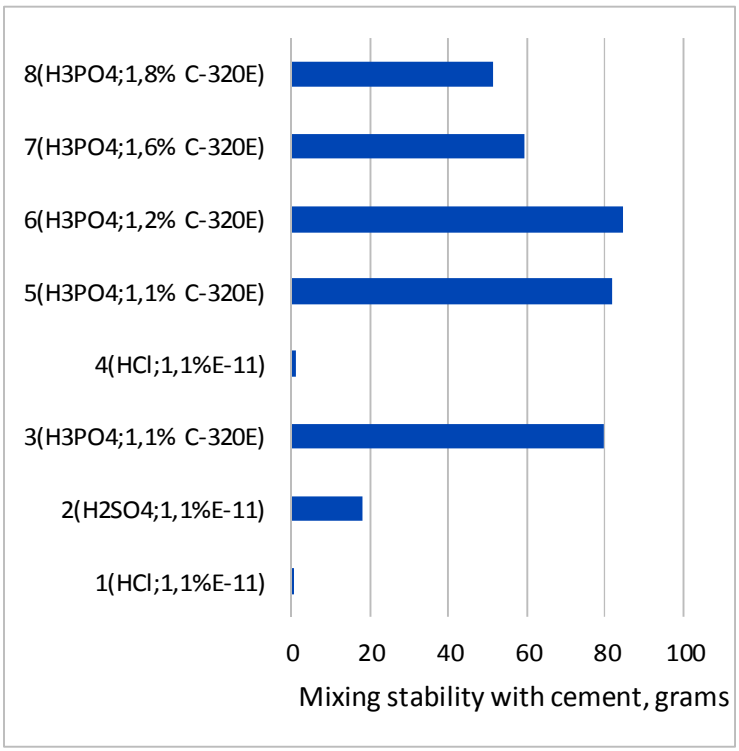
It can be assumed that increasing the amount of emulsifier in the emulsion increases the breaking value. At the same time, emulsions with various acids and emulsifiers at a content of 1.1 % demonstrate high breaking value and storage stability.

Table 3

Emulsions breaking behaviour

№ bitumen emulsion	Breaking value (index) за DSTU B V.2.7-129:2013, %	Fines mixing time according to DSTU EN 13075-2:2020, sec	Mixing stability with cement according to DSTU EN 12848:2020, grams
1(HCl;1,1 %E-11)	198	199	0,8
2(H2SO4;1,1 %E-11)	253	>300	18,00
3(H3PO4;1,1 % C-320E)	247	270	79,8
4(HCl;1,1 %E-11)	196	205	1,1
5(H3PO4;1,1 % C-320E)	245	278	82,0
6(H3PO4;1,2 % C-320E)	257	>300	84,5
7(H3PO4;1,6 % C-320E)	301	>300	59,3
8(H3PO4;1,8 % C-320E)	320	>300	51,2

The obtained results of breaking behavior of bitumen emulsions indicate that emulsions on hydrochloric and sulfuric acid have a shorter fines mixing time and greater mixing stability with cement than emulsions on orthophosphoric acid. For bitumen emulsions on orthophosphoric acid, it is observed that with an emulsifier dosage of 1.2 % and above, the fines mixing time is more than 300s, but stability with cement increases with an emulsifier dosage of 1.6 % and above. Bitumen emulsions with orthophosphoric acid interact with cement in a different way than bitumen emulsions with hydrochloric and sulfuric acid, which is confirmed by the method according to DSTU EN 12848:2020. Therefore, for slow-setting and over-stabilized bitumen emulsions containing orthophosphoric acid, the method according to DSTU EN 12848:2020 is not effective, due to the high reactivity of emulsions on orthophosphoric acid during interaction with portland cement.



Bitumen emulsions mixing stability with Portland cement

For all investigated emulsions, there is a correlation between breaking behavior (index) according to DSTU B V.2.7-129:2013 and fines mixing time according to DSTU EN 13075-2:2020.

Conclusions

The compositions of bitumen emulsions with hydrochloric, sulfuric, orthophosphoric acids and emulsifiers have been selected. The physical and mechanical properties of the produced emulsions and breaking behavior were determined.

It has been established that over-stabilised bitumen emulsions demonstrate good storage stability for 180 days. There is a linear relationship between storage stability for all investigated emulsions for 7, 30, 180 days.

It can also be argued that the definition of breaking behavior depends on the components from which the emulsion is made, since the mixing stability with cement method is not effective for bitumen emulsions made on orthophosphoric acid. While the breaking value method according to DSTU B V.2.7-129:2013 and fines mixing time method according to DSTU EN 13075-2:2020 can be used for emulsions on orthophosphoric, hydrochloric and sulfuric acids.

The possibility of obtaining over-stabilized bitumen emulsions from the usual emulsifiers, for slow-setting emulsions, has been proven, since according to the method of DSTU B V.2.7-129:2013 their breaking value is more than 230; according to the method according to DSTU EN 13075-2:2020 emulsions with fines mixing time more than 180 for No. 1, No. 3, No. 4, No. 5 of the studied bitumen emulsions and more than 300 for No. 2, No. 6, No. 7, No. 8 of the studied bitumen emulsions . Also, bitumen emulsions No. 1, No. 4 containing hydrochloric acid according to the DSTU EN 12848:2020 method demonstrate good mixing stability with cement (less than 2 g).

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НАДСТАБІЛЬНІ БІТУМНІ ЕМУЛЬСІЇ ВИГОТОВЛЕНІ ЗА ДОПОМОГОЮ ЕМУЛЬГАТОРІВ ДЛЯ ПОВІЛЬНОРОЗПАДНИХ ЕМУЛЬСІЙ

Запроектовано склади дорожніх надстійких катіонних бітумних емульсій із ортофосфорною, сірчаною та соляною кислотами та катіонактивними емульгаторами для повільнорозпадних емульсій. Визначено фізико-механічні властивості виготовлених надстабільних бітумних емульсій та їх характеристики розпаду. Було встановлено вплив типу кислоти та вмісту емульгатора на фізико-механічні показники бітумних емульсій та характеристики розпаду за трьома методами: індекс розпаду, час (стан) до розпаду, стійкість під час змішування з портландцементом. Проаналізувавши фізико-механічні показники виготовлених емульсій із одинаковим вмістом бітуму 60 % та вмістом емульгатора у водній фазі 1,1 % можна стверджувати, що бітумна емульсія на сірчаній кислоті має більший pH та в'язкість. Якщо ж порівнювати бітумні емульсії з одинаковим вмістом емульгатора 1,1 %, але різним вмістом бітуму 60 % та 61 %, то емульсії із більшим вмістом бітуму характеризуються більшим pH та в'язкість, хоча pH в водній фазі є одинаковим. Порівнюючи емульсії із одинаковим вмістом бітуму 62 % та pH в водній фазі 2,3, але різним вмістом емульгатора у водній фазі (від 1,2 до 1,8), pH є майже однаковим, а в'язкість збільшується із збільшення вмісту емульгатора.

Встановлено, що надстабільні бітумні емульсії демонструють хорошу стійкість при зберіганні протягом 180 діб. Отримані результати характеристик розпаду бітумних емульсій свідчать про те, що емульсії на соляній та сірчаній кислоті мають менший час до розпаду та більшу стійкість під час змішування з портландцементом ніж емульсії на ортофосфорній кислоті. Для надстійких бітумнинх емульсій на ортофосфорній кислоті метод за ДСТУ EN 12848:2020 не підходить, через високу реактивність емульсій на ортофосфорній кислоті під час взаємодії із портландцементом.

Keywords: бітумні катіонні емульсії, соляна кислота, ортофосфорна кислота, сірчана кислота, характеристика розпаду, стійкість під час змішування з портландцементом.