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## STORAGE STABILITY OF CATIONIC BITUMEN EMULSIONS

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The compositions of cationic road bitumen emulsions based on special emulsifiers ensuring high storage stability were developed. The variability of storage stability results obtained by different test methods was analyzed, namely: sieving after 7 and 30 days (using 0.14 mm and 0.5 mm sieves) and the settling tendency test on day 7. The study showed that the emulsifier content, emulsifier type, and acid type have a significant influence on storage stability. Emulsions with higher emulsifier dosages exhibited improved stability according to all applied methods. Moreover, the use of hydrochloric acid proved more effective than orthophosphoric acid in maintaining storage stability. At the same time, the origin of bitumen (Ukrainian or Polish) had no significant effect on storage stability, either by sieving or settling tendency. Correlation analysis confirmed a clear relationship between the results of sieving methods and susceptibility to stratification.

**Keywords:** bitumen, emulsifier, cationic bitumen emulsion, characteristics of storage stability, storage stability by sieving, settling tendency.

### Introduction

Storage stability characterizes the ability of an emulsion to maintain homogeneity and resist phase separation over time. During storage, the bitumen droplets in the emulsion may undergo the following stages leading to unplanned breakdown:

- sedimentation (the process of bitumen droplets settling);
- flocculation (the process of droplets sticking together without complete coalescence);
- coalescence (the complete merging of bitumen droplets in the emulsion, leading to separation of the bitumen phase and breakdown of the emulsion).

Sedimentation of bitumen droplets in the emulsion occurs at the bottom of the container because the density of bitumen droplets is higher than that of water (in most cases), causing them to settle. As a result, the droplets move closer to one another, and their physical contact leads to flocculation. However, at the flocculation stage the emulsion can be restored to a homogeneous state by mixing at an appropriate temperature, thus preventing the onset of coalescence (Mallawarachchi, 2016; Cui, 2017; Porto, 2021; Dołżycki, 2019; Li, 2024; Carpani, 2025).

Overall, the storage stability of an emulsion depends on its composition, the frequency and duration of mixing, storage temperature, as well as the shape and material of the storage containers. Moreover, ensuring emulsion stability is important not only during storage but also during transportation, which likewise depends on time and temperature of transport, as well as the shape, material, and filling level of the transport containers (Schuster, 1996; Al-Sabagh, 2002; Liu, 2017; Ouyang, 2022; You, 2020).

All adverse effects that can cause premature breakdown of cationic bitumen emulsions during storage and transportation can be minimized by using highly stable emulsions (Salomon, 2006; Ouyang, 2018; Bidos, 2023; Sidun, 2023; Bidos, 2025).

Storage stability of emulsions is determined by the following indicators and corresponding test methods:

- storage stability by sieving – an indicator of emulsion homogeneity during storage, determined by the percentage residue retained after passing the emulsion through a specified sieve;

- settling tendency – an indicator characterizing the concentration of residual binder in the lower and upper layers of the emulsion, determined as the difference in water content between the upper and lower layers of a sample of given volume after conditioning for a certain time at ambient temperature;
- storage stability by electro-decanter method;
- particle size distribution of the emulsion – determination of changes in bitumen droplet size in the emulsion after a certain period of time, using laser diffraction or microscopic imaging;
- change in viscosity of the emulsion over time, determined using a Brookfield viscometer.

Storage stability is one of the requirements for the physico-technical properties of emulsions in accordance with the current Ukrainian standard DSTU B V.2.7-129:2013 and the European EN 13808, which is also in force in Ukraine. According to DSTU B V.2.7-129:2013, it is determined by the percentage of particles that do not pass through a 0.14 mm sieve (after 7 or 30 days of storage), while EN 13808 specifies that the test is carried out according to EN 1429 and is defined as the percentage of particles larger than 0.5 mm (after 7 days of storage).

These two indicators essentially correspond to homogeneity under DSTU B V.2.7-129:2013 and to residue after sieving under EN 13808, but both are measured after a certain storage period. It should be noted, that the residue after sieving in EN 13808 is determined on a 0.16 mm sieve, yet the standard does not require using this sieve when determining storage stability. On the contrary, DSTU B V.2.7-129:2013 regulates the determination of both homogeneity and storage stability on the same sieve of 0.14 mm. In general, these indicators are referred to as storage stability by sieving.

EN 13808 also contains a requirement for the settling tendency of emulsions, which is tested according to EN 12847 (the U.S. analogue is ASTM D6930-19). Storage stability by sieving and settling tendency are similar emulsion properties, however since an emulsion may undergo phase separation without significant changes in the particle size distribution, meaning these parameters may not correlate.

Storage stability by electro-decantation, according to the French standard NF T66-022, evaluates the tendency of an emulsion to separate (decant) under the action of an electric field. This test provides an indirect and rapid assessment of emulsion stability, since it does not require keeping the emulsion for a long storage period.

The determination of emulsion storage stability by particle size distribution and by viscosity change over time is quite popular worldwide (Wang, 2012; Ivanov, 1997; Campanelli, 1989; Tadros, 2004), but not in Ukraine due to the lack of necessary equipment.

Thus, storage stability characteristics of bitumen emulsions are defined as the properties of a bitumen emulsion determined by specific test methods, which establish the possible storage period of the emulsion before its breakdown without contact with mineral surfaces.

According to DSTU B V.2.7-129:2013, storage stability is determined on the 7th and 30th day of storage by the homogeneity method, i. e., with certain modifications and clarifications. Specifically, emulsions were stored in 2-liter plastic bottles at a temperature of  $20 \pm 2$  °C. The emulsions were mixed once a week by inverting the bottles upside down during storage.

### **Materials and Methods**

The following bitumens were used to produce bituminous emulsions:

- oxidized road petroleum bitumen grade BND 70/100, manufacturer PJSC “Ukratnafta” (Ukraine), hereinafter referred to as Ukratnafta;
- oxidized road petroleum bitumen grade 70/100, manufacturer PKN Orlen (Poland), hereinafter referred to as Orlen.

The results of the study of the physico-mechanical properties of the bitumens used are presented in Table 1.

Both bitumens belong to grades BND 70/100 and 70/100 according to DSTU 4044 and DSTU EN 12591:2017, respectively. The bitumen produced by PKN Orlen (Poland) shows slightly higher penetration and a softening point that is 1 °C higher compared to the bitumen produced by PJSC “Ukratnafta” (Ukraine). In addition, the Fraas brittleness temperature of the Polish bitumen is 1 °C lower. The adhesion to glass of this bitumen is also better than that of the Ukrainian bitumen. The ductility of both bitumens is greater than 100 cm.

Table 1

**Physico-mechanical properties of the bitumens**

| No. | Indicator                                              | Origin and grade requirements according to |       |                                          |                                                  |
|-----|--------------------------------------------------------|--------------------------------------------|-------|------------------------------------------|--------------------------------------------------|
|     |                                                        | Ukratnafta                                 | Orlen | Requirements                             |                                                  |
|     |                                                        |                                            |       | BND 70/100,<br>according to<br>DSTU 4044 | 70/100,<br>according to<br>DSTU EN<br>12591:2017 |
| 1   | Needle penetration at 25 °C, 0.1 mm                    | 83                                         | 89    | 71÷100                                   | 70÷100                                           |
| 2   | Softening point, °C                                    | 47.4                                       | 46.4  | 45÷51                                    | 43÷51                                            |
| 3   | Fraas brittleness temperature, °C                      | −13                                        | −14   | Not higher than                          |                                                  |
|     |                                                        |                                            |       | −13                                      | −10                                              |
| 4   | Ductility at 25 °C, cm                                 | >100                                       | >100  | Not less than 60                         | –                                                |
| 5   | Adhesion to glass surface, %                           | 20.4                                       | 24.1  | Not less than 18                         | –                                                |
| 6   | Flash point in open crucible, °C                       | 288                                        | 291   | Not less than                            |                                                  |
|     |                                                        |                                            |       | 230                                      | 230                                              |
| 7   | Solubility in organic solvent, %                       | 99.94                                      | 99.91 | Not less than                            |                                                  |
|     |                                                        |                                            |       | 99.00                                    | 99.00                                            |
| 8   | Change in properties after heating:                    |                                            |       |                                          |                                                  |
| 8.1 | Mass change after heating, %                           | 0.13                                       | 0.11  | Not more than                            |                                                  |
|     |                                                        |                                            |       | 0.90                                     | –                                                |
| 8.2 | Residual penetration, %                                | 59.1                                       | 78.8  | Not less than                            |                                                  |
|     |                                                        |                                            |       | 59                                       | –                                                |
| 8.3 | Change in softening point, °C                          | 5.7                                        | 4.8   | Not more than                            |                                                  |
|     |                                                        |                                            |       | 6                                        | –                                                |
| 9   | Penetration index                                      | −0.72                                      | −0.73 | −2.0÷1.0                                 | −1.5÷0.7                                         |
| 10  | Ductility at 0 °C, cm                                  | 4.8                                        | 7.2   | Not less than 3.8                        | –                                                |
| 11  | Temperature at which penetration equals 800·0.1 mm, °C | 48.7                                       | 48.7  | 45÷51                                    | –                                                |
| 12  | Penetration index at temperature T800                  | −0.31                                      | −0.05 | −2.0÷1.0                                 | –                                                |
| 13  | Dynamic viscosity at 135 °C, Pa·s                      | 0.305                                      | 0.359 | –                                        | –                                                |

Table 2

**The properties of the cationic emulsifiers**

| Indicator                                | Redicote                         |         |         | Polyram L950 |
|------------------------------------------|----------------------------------|---------|---------|--------------|
|                                          | E-4875 NPF                       | E-11    | C-320E  |              |
| Flash point, °C                          | 59 (ISO 2719)<br>>100 (ISO 2592) | 18      | >100    | >100)        |
| Pour point, °C                           | <-5                              | 20      | 0       | <-30         |
| Density, lb/gal                          | 9.09                             | 7.51    | 7.35    | 8.35         |
| Dosage for slow-breaking emulsion, wt. % | 0.8–2.0                          | 0.6–1.5 | 0.8–1.5 | 0.5–1.5      |

In addition to bitumen, the production of the bituminous emulsions included the following components: emulsifiers (Tables 2, 3), acids, and water (Table 4).

Cationic emulsifiers from Nouryon (Redicote E-4875 NPF, Redicote E-11, Redicote C-320E) and Arkema (Polyram L950) were selected for the study. Standard properties of the cationic emulsifiers, as

specified in the product specifications, are presented in Table 2, while the properties determined according to DSTU 9187:2022 are presented in Table 3.

Table 3

### Specified indicators of cationic emulsifiers

| Indicator              | Redicote E-4875 NPF   | Redicote E-11 | Redicote C-320E | Polyram L950 |
|------------------------|-----------------------|---------------|-----------------|--------------|
| Appearance             | Dark brown liquid     | Yellow liquid |                 |              |
|                        | No foreign inclusions |               |                 |              |
| Density, g/cm³         | 1.07                  | 0.90          | 0.88            | 0.95         |
| Kinematic viscosity, s | 119.6                 | 5.4           | 5.2             | 46.0         |

In this study, the storage stability was evaluated using the storage stability by sieving method according to the national standard DSTU B V.2.7-129:2013 with a 0.14 mm sieve (after 7 and 30 days of storage) and according to the current European standard in Ukraine, DSTU EN 1429, with a 0.5 mm sieve (after 7 and 30 days of storage). The settling tendency was also determined in accordance with DSTU EN 12847.

### Results and discussion

For comparison, the storage stability of the emulsions was studied using 18 formulations based on Orlen bitumen and 18 corresponding formulations based on Ukratnafta bitumen. In these formulations, the emulsifier content and type of acid were varied (Table 4).

Table 4

### Compositions of emulsions

| No. | Bitumen             |                    | Emulsifier         |                    | type of acid                   | pH in the aqueous phase |
|-----|---------------------|--------------------|--------------------|--------------------|--------------------------------|-------------------------|
|     | manufacturer        | content, % by mass | brands             | content, % by mass |                                |                         |
| 1   | Orlen / Ukrtatnafta | 60                 | Redicote E-11      | 0.9                | HCl                            | 2.5                     |
| 2   |                     |                    |                    | 1.2                |                                |                         |
| 3   |                     |                    |                    | 1.5                |                                |                         |
| 4   |                     |                    | Redicote E4875 NPF | 0.9                |                                |                         |
| 5   |                     |                    |                    | 1.2                |                                |                         |
| 6   |                     |                    |                    | 1.5                |                                |                         |
| 7   |                     |                    | Polyram L950       | 0.9                |                                |                         |
| 8   |                     |                    |                    | 1.2                |                                |                         |
| 9   |                     |                    |                    | 1.5                |                                |                         |
| 10  |                     |                    | Redicote C-320E    | 0.9                |                                |                         |
| 11  |                     |                    |                    | 1.2                |                                |                         |
| 12  |                     |                    |                    | 1.5                |                                |                         |
| 13  |                     |                    | Polyram L950       | 0.9                | H <sub>3</sub> PO <sub>4</sub> |                         |
| 14  |                     |                    |                    | 1.2                |                                |                         |
| 15  |                     |                    |                    | 1.5                |                                |                         |
| 16  |                     |                    | Redicote C-320E    | 0.9                |                                |                         |
| 17  |                     |                    |                    | 1.2                |                                |                         |
| 18  |                     |                    |                    | 1.5                |                                |                         |

All emulsions prepared according to the formulations presented in Table 4 did not break after production and were suitable for the determination of storage stability characteristics (Table 5).

By analysing the results in Table 5, it can be stated that the emulsifier content has the most significant effect on storage stability. Emulsions with a higher emulsifier content exhibit better storage stability by sieving and settling tendency. Among the studied emulsifiers, Redicote E-4875 NPF provides the best storage stability by sieving on 0.14 mm and 0.5 mm sieves after 7 and 30 days. In addition, the settling tendency after 7 days is also the lowest. It is known that this emulsifier is used for so-called over-stabilized bitumen emulsions (Salomon, 2006; Ouyang, 2018; Bidos, 2023; Sidun, 2023; Bidos, 2025).

Table 5

**Storage stability characteristics**

| No. | Storage stability by sieving, % of bitumen emulsion on bitumen Orlen/Ukrtatnafta |           |           |           | Settling tendency<br>7 days, % |
|-----|----------------------------------------------------------------------------------|-----------|-----------|-----------|--------------------------------|
|     | 7 days                                                                           |           | 30 days   |           |                                |
|     | 0.14 mm                                                                          | 0.5 mm    | 0.14 mm   | 0.5 mm    |                                |
| 1   | 0.09/0.10                                                                        | 0.02/0.02 | 0.06/0.09 | 0.02/0.03 | 2.06/2.46                      |
| 2   | 0.08/0.08                                                                        | 0.01/0.01 | 0.04/0.06 | 0.01/0.02 | 1.83/1.93                      |
| 3   | 0.05/0.04                                                                        | 0.01/0.01 | 0.21/0.06 | 0.01/0.02 | 1.58/1.78                      |
| 4   | 0.01/0.03                                                                        | 0,11/0.08 | 0.02/0.08 | 0.05/0.03 | 1.63/1.83                      |
| 5   | 0.08/0.06                                                                        | 0.01/0.02 | 0.15/0.06 | 0.04/0.03 | 1.35/1.55                      |
| 6   | 0.04/0.05                                                                        | 0.01/0.01 | 0.09/0.05 | 0.02/0.02 | 1.11/1.21                      |
| 7   | 0.05/0.09                                                                        | 0.01/0.02 | 0.08/0.10 | 0.05/0.03 | 2.33/2.63                      |
| 8   | 0.05/0.08                                                                        | 0.01/0.02 | 0.07/0.08 | 0.03/0.02 | 2.01/2.31                      |
| 9   | 0.04/0.06                                                                        | 0.01/0.01 | 0.05/0.06 | 0.02/0.02 | 1.73/1.97                      |
| 10  | 0.08/0.10                                                                        | 0.01/0.03 | 0.10/0.10 | 0.04/0.03 | 1.98/2.08                      |
| 11  | 0.05/0.08                                                                        | 0.01/0.02 | 0.08/0.07 | 0.02/0.02 | 1.79/1.89                      |
| 12  | 0.05/0.05                                                                        | 0.01/0.01 | 0.05/0.04 | 0.01/0.01 | 1.58/1.68                      |
| 13  | 0.070.09                                                                         | 0.03/0.03 | 0.08/0.09 | 0.03/0.03 | 2.26/2.56                      |
| 14  | 0.04/0.06                                                                        | 0.02/0.02 | 0.06/0.07 | 0.01/0.02 | 1.96/2.16                      |
| 15  | 0.03/0.02                                                                        | 0.02/0.01 | 0.05/0.05 | 0.01/0.01 | 1.64/1.84                      |
| 16  | 0.09/0.09                                                                        | 0.03/0.03 | 0,11/0,12 | 0.03/0.03 | 2.02/2.32                      |
| 17  | 0.07/0.06                                                                        | 0.02/0.01 | 0.09/0.08 | 0.01/0.02 | 1.88/2.18                      |
| 18  | 0.06/0.04                                                                        | 0.02/0.01 | 0.08/0.06 | 0.01/0.01 | 1.64/1.84                      |

According to the conducted studies, the type of bitumen used for emulsion preparation has little effect on storage stability characteristics, whereas the type of acid used has a noticeable impact. Emulsions prepared with HCl show better storage stability by sieving and settling tendency compared to those with  $H_3PO_4$ , as can be seen for the emulsifiers Redicote C-320E and Polyram L950.

A correlation is also observed between the results of storage stability by sieving after 7 and 30 days using a 0.14 mm sieve and the settling tendency after 7 days. Regarding storage stability by sieving on a 0.5 mm sieve, all obtained results are no more than 0.1 %, indicating that no large bitumen droplets form in the emulsions during storage for up to 30 days.

**Conclusions**

A series of cationic bitumen emulsions were developed and tested using various emulsifiers and acids, allowing a comprehensive assessment of the influence of composition on storage stability. It was demonstrated that increasing the emulsifier dosage positively affects the stability of emulsions according to all test methods. The emulsifier Redicote E-4875 NPF exhibited the best performance, confirming its effectiveness for producing over-stabilized emulsions. The use of HCl acid provides better stability than  $H_3PO_4$ , regardless of the emulsifier type. The origin of the bitumen ("Ukrtatnafta" or PKN "Orlen") does not have a significant effect on storage stability.

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### **СТІЙКІСТЬ ЗА ЗБЕРІГАННЯ КАТІОННИХ БІТУМНИХ ЕМУЛЬСІЙ**

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У статті викладено результати комплексних досліджень стабільності під час зберігання катіонних бітумних емульсій, що є важливим параметром їх ефективності у дорожньому будівництві. Проаналізовано вплив типу та дозування емульгаторів, а також виду кислоти на показники збереження однорідності та схильності до розшарування катіонних бітумних емульсій. Для виготовлення емульсій використано бітуми марок 70/100 виробництва ПАТ “Укртатнафта” (Україна) та PKN “Orlen” (Польща), що дало змогу порівняти властивості емульсій залежно від походження в’язучого. В роботі застосовано емульгатори компаній “Nouryon” (Redicote E-4875 NPF, Redicote E-11, Redicote C-320E) та “Arkema” (Polygam L950), а також різні кислоти (соляну та ортофосфорну). Експериментальна програма охоплювала оцінювання стабільності за методиками ДСТУ Б В.2.7-129:2013 – стабільність за зберігання за допомогою ситового аналізу (сито 0,14 мм); ДСТУ EN 1429 – стабільність за зберігання за допомогою ситового аналізу (сито 0,5 мм), ДСТУ EN 12847 – визначення схильності до розшарування, а також аналіз впливу дозування емульгатора на якість отриманих емульсій. Результати показали, що вирішально впливають на стабільність саме вміст і тип емульгатора: підвищення концентрації емульгатора покращує показники як за ситовими методами, так і за схильності до розшарування. Найкращі результати продемонстрував емульгатор Redicote E-4875 NPF, відомий застосуванням у надстійких катіонних бітумних емульсіях. Встановлено, що використання соляної кислоти забезпечує кращі характеристики зберігання порівняно із ортофосфорною, незалежно від виду емульгатора. Водночас походження бітуму (українського чи польського виробництва) на стійкість за зберігання емульсій істотно не впливало. Кореляційний аналіз підтвердив зв’язок між результатами ситових методів і схильністю до розшарування.

**Ключові слова:** бітум, емульгатор, катіонна бітумна емульсія, характеристики стійкості за зберігання, стійкість під час зберігання за ситовим методом, стабільність до розшарування.