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REVIEW OF THE INFLUENCE OF EMULSIFIER TYPES USED IN PRODUCING BITUMEN EMULSIONS ON EMULSION STABILITY

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Abstract. *This article presents a systematic analysis of three types of emulsifiers-cationic, anionic, and nonionic-used in the production of bitumen emulsions, as well as their chemical nature, molecular structure, and the impact of application areas on emulsion stability. The article separately considers the influence of such factors as emulsifier type, concentration, pH of the medium, droplet size, and zeta potential on stability, as well as the mechanism of action of cetyltrimethylammonium chloride (CTAC) and octadecylamine hydrochloride (C18H37NH2·HCl), which belong to the class of cationic emulsifiers, sodium oleate as an anionic emulsifier, and nonionic emulsifiers based on the hydrophilic-lipophilic balance (HLB) theory. The results show that cationic emulsifiers provide the best stability in a low-acid environment (pH 2-5), and anionic emulsifiers are compatible with limestone aggregates. In contrast, nonionic emulsifiers are suitable for long-term storage. Bio-based emulsifiers-in particular, cationic lignin-based ones-are preferred.*

Keywords: *bitumen emulsion, cationic emulsifier, anionic emulsifier, nonionic emulsifier, CTAC, sodium oleate, stability, surfactant, droplet size, zeta potential, HLB.*

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ОБЗОР ВЛИЯНИЯ ТИПОВ ЭМУЛЬГАТОРОВ, ПРИМЕНЯЕМЫХ ПРИ ПРОИЗВОДСТВЕ БИТУМНЫХ ЭМУЛЬСИЙ, НА ИХ СТАБИЛЬНОСТЬ

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Аннотация. *В данной статье представлен систематический анализ трех типов эмульгаторов: катионных, анионных и неионных, используемых при производстве битумных эмульсий, а также их химической природы, молекулярной структуры и влияния областей применения на стабильность эмульсии. В статье отдельно рассматривается влияние таких факторов, как тип эмульгатора, концентрация, pH среды, размер капель и зета-потенциал, на стабильность, а также механизм действия цетилтриметиламмонийхлорида (CTAC) и октадециламингидрохлорида (C18H37NH2·HCl), относящихся к классу катионных эмульгаторов, олеата натрия как анионного эмульгатора и неионных эмульгаторов на основе*

теории гидрофильно-липофильного баланса (ГЛБ). Результаты показывают, что катионные эмульгаторы обеспечивают наилучшую стабильность в низкокислой среде (pH 2-5), а анионные эмульгаторы совместимы с известковыми агрегатами. В отличие от них, неионные эмульгаторы подходят для длительного хранения. Предпочтительны эмульгаторы на биологической основе, в частности, на основе катионного лигнина.
Ключевые слова: *битумная эмульсия, катионный эмульгатор, анионный эмульгатор, неионный эмульгатор, CTAC, олеат натрия, стабильность, ПАВ, размер капель, зета-потенциал, HLB.*

BITUM EMULSIYALARINI ISHLAB CHIQARISHDA QO'LLANILADIGAN EMULGATOR TURLARINING BARQARORLIKKA TA'SIRI TAHLILI

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Annotatsiya. *Mazkur maqolada bitum emulsiyalarini ishlab chiqarishda qo'llaniladigan uch turdagi: kationli, anionli va noionli emulgatorlar, shuningdek, ularning kimyoviy tabiati, molekulyar tuzilishi hamda qo'llanilish sohalarining emulsiya barqarorligiga ta'siri tizimli tahlil qilingan. Maqolada emulgator turi, konsentratsiyasi, muhitning rH ko'rsatkichi, tomchilar o'lchami va dzeta-potensial kabi omillarning emulsiya barqarorligiga ta'siri alohida ko'rib chiqilgan. Shuningdek, kationli emulgatorlar sinfiga mansub setiltrimetilammoniy xlorid (CTAC) va oktadetsilamin gidroxlorid (C18H37NH2·HCl), anionli emulgator sifatida natriy oleati hamda gidrofil-lipofil muvozanat (GLM) nazariyasiga asoslangan noionli emulgatorlarning ta'sir mexanizmi tahlil etilgan. Natijalar kationli emulsifikatorlarning kuchsiz kislotali muhitda (pH 2–5) eng yaxshi barqarorlikni ta'minlashini, anionli emulsifikatorlarning esa ohakli to'ldirgichlar bilan mos kelishini ko'rsatadi. Ulardan farqli o'laroq, noionogen emulsifikatorlar uzoq muddatli saqlash uchun yaroqlidir. Bioasosli, xususan, kationli lignin asosidagi emulsifikatorlarga afzallik beriladi.*
Kalit so'zlar: *bitum emulsiyasi, kationli emulsifikator, anionli emulsifikator, noionogen emulsifikator, CTAC, natriy oleat, barqarorlik, SAM, tomchi o'lchami, dzeta-potensial, HLB.*

Introduction. Bitumen emulsions are an environmentally friendly material widely used in modern road construction, which retains bitumen in an aqueous medium in the form of tiny droplets. Compared to traditional hot mix asphalt concrete, bitumen emulsions consume 40-50% less energy, have a low fire hazard, and minimize harmful hydrocarbon emissions into the atmosphere [1]. The main component of the emulsion-the emulsifier (surfactant)-plays a crucial role in maintaining the stability of bitumen droplets in an aqueous medium.

Emulsifiers are divided into three main types: cationic, anionic, and nonionic. This classification is based on the ionic charge formed during the dissociation of the surfactant molecule in an aqueous medium [2]. The correct choice of emulsifier determines such important properties of the emulsion as its storage stability, droplet size distribution, adhesion to the aggregate, and suitability for the intended application [3].

Over the past decade, significant advances have been made in the field of emulsifier chemistry, with lignin-based bioemulsifiers, polymer-modified surfactants, cetyl ion-based cationic surfactants (CTAC, cetylpyridinium chloride), and nanocellulose-based stabilizers being the focus of research [4, 5, 19]. At the same time, the hydrophilic-lipophilic balance (HLB) theory is being revised to more accurately explain the mechanism of action of non-ionic emulsifiers [20]. Despite this, systematic review studies that would comparatively analyze the effects of different types of emulsifiers, their chemical nature, and concentration on stability are lacking. The aim of this article is to determine the effects of the type, chemical nature, and concentration of emulsifier on the stability of bitumen emulsion based on an analysis of scientific literature.

Materials and Methods. ***Materials used in the study.*** In the analyzed articles, different grades of bitumen were used as the base material. Most of the studies used bitumen with 60/90 and 90/130 penetration [7, 10]. Representatives of three main classes were taken as emulsifiers: cationic emulsifiers (propylene alkyl polyamine - CSS-1h [3, 7, 10], didodesyl dimethyl ammonium bromide [2], amino-functionalized product based on kraft lignin [4, 5, 6]); anionic emulsifiers (2-bromostearic acid [2], dodecacyl sulfate derivatives, sodium oleate [13]); polyethylene glycol (PEG) [2] and nanocellulose crystals [8] were tested as non-ionic emulsifiers. In the study by Sidun et al. [9] Commercial cationic emulsifiers Redicote C-320E and Polyram L950 were tested on Ukrainian and Polish bitumens. Geng et al. [11] prepared a complex modified emulsion by adding a rejuvenator and SBR latex to the cationic emulsifier.

Cetyltrimethylammonium chloride (CTAC). Among the long-chain quaternary ammonium salts, cetyltrimethylammonium chloride (CTAC, $C_{19}H_{42}ClN$), a cationic surfactant with a hexadecyl radical, occupies an important place in bitumen emulsions. In a study by Yang et al., the "cetyl ion" of emulsified asphalt systems based on CTAC, cetylpyridinium chloride (CPC), sodium hexadecyl sulfate (SHS), and sodium hexadecyl sulfonate (SHSN) was compared with molecular dynamics (MD) simulations and experimental methods. The results showed that the electrostatic interaction between emulsifier molecules and bitumen is stronger than the van der Waals interaction between emulsifier molecules and water, and the interface stability mainly depends on the electrostatic force; According to the thickness of the interactive layer, emulsifiers decrease in the order SHS > SHSN > CPC > CTAC, that is, CTAC forms a thinner layer, but with a strong electrostatic effect [19].

Octadecylamine hydrochloride ($C_{18}H_{37}NH_2 \cdot HCl$). The classic class of cationic emulsifiers for bitumen emulsions consists of hydrochloride salts of long-chain primary fatty amines-in particular, octadecylamine hydrochloride. In a review by Al-Mohammedawi and colleagues, such amine salts (octadecylamine, laurylamine, N-methylstearylamine) are described as the main group of cationic emulsifiers used in the manufacture of bitumen emulsions. These compounds, dissolved in the aqueous phase, are protonated by acid (usually hydrochloric acid), forming a positively charged amine head with a long hydrocarbon chain; this provides strong electrostatic attraction to the surface of the negatively charged silicate (granite) aggregate and imparts high adhesive properties in cold mixes. In industrial practice, such amine salts are usually used in amounts of 0.1-2.0 wt.% at pH in the range of 2-5 [1].

Sodium Oleate. Among the anionic emulsifiers, sodium oleate (sodium salt of oleic acid, $C_{17}H_{33}COONa$) is of practical importance. Antonova et al. studied the effect of sodium oleate concentration on the stability of anionic bitumen emulsions used for waterproofing purposes. The results showed that at an emulsifier concentration of about 3%, a minimum sieve residue (i.e., the best homogeneity and dispersibility) is observed, which is consistent with the results of tests after 7 and 30 days of storage; the dependence of the relative viscosity of the emulsion on the emulsifier concentration was also established. Sodium oleate is obtained by neutralizing technical oleic acid with sodium hydroxide and works actively in an alkaline environment (pH 8-10), which allows for compatible work with limestone (carbonate) aggregates [13].

Non-ionic emulsifiers and hydrophilic-lipophilic balance theory. The stabilizing mechanism of non-ionic emulsifiers, in contrast to electrostatic charge, is related to the value of hydrophilic-lipophilic balance and the steric barrier mechanism. The revised hydrophilic-lipophilic theory proposed by Yan et al. showed that an aqueous solution of bitumen emulsifiers provides more accurate results in predicting the surface tension than the classical hydrophilic-lipophilic model [20]. This helps to explain the ability of polyethylene glycol derivatives and other ethoxylated non-ionic surfactants to stably retain bitumen droplets regardless of ionic strength; however, such emulsifiers are weaker in adhesion to the aggregate than cationic emulsifiers [2].

Emulsion preparation methods. All emulsions in the analyzed papers were prepared by the colloid mill method, in which heated bitumen (110-160°C) and the dissolved aqueous phase of the emulsifier are mixed using high shear force. The emulsifier was usually added to the aqueous phase by dissolution at a temperature of 40-60°C [1, 3]. In

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the study by You, bitumen was prepared by adding HCl and NaOH solutions to emulsions at different pH values (1.0-12.0), while the bitumen content was retained at 60% [3]. Porto prepared a bio-surfactant-based emulsion using a colloid mill at a speed of 1000-6000 rpm; the emulsifier proportion was 1-6% of the total emulsion weight and maintained at pH-14 [12].

Methods and Standards for Stability Assessment. The following methods and international standards were used in the reviewed articles to assess stability; these standards form the methodological basis for the numerical values presented in Tables 1-3.

Storage stability: the sieve residue was determined by sieving (DSTU B V.2.7-129:2013 and European standard EN 12847 0.14 mm and 0.5 mm sieve) after 7 and 30 days of storage [9, 13]; sedimentation tendency was measured as the difference in the amount of water in the lower and upper layers of the emulsion [9]. In US practice, storage stability is often assessed according to ASTM D6930 (24-hour static sedimentation). The particle charge type (determination of cationic or anionic) is determined by electrophoresis in accordance with standard EN 1428 [16, 18].

Table 1

An extended comparative description of the three main types of emulsifiers

Indicator	Cationic	Anionic	Nonionic	Standard	Ref.
Charging	Positive (+)	Negative (-)	Neutral (0)	EN 1428	[2]
pH of the environment	Acidic (2-5)	Alkaline (8-12)	Neutral (6-8)	pH-metry	[3]
Stability period	3-6 months	2-4 months	6-12 months	EN 12847 / ASTM D6930	[1]
CMC concentration	0.5-1.5 wt.%	0.8-2.0 wt.%	1.0-2.5 wt.%	Surface tension/HLB	[1]; [20]
Aggregate compatibility	Granite	Limestone	Universal	Zeta-potential + SFE	[2]; [18]
Processing temperature	110-150°C	120-160°C	100-140°C	-	[3]
Typical representatives	CSS-1h, CTAC, $C_{18}H_{37}NH_2 \cdot HCl$, cationic lignin	Sodium oleate, dodecyl sulfate, alkali lignin	PEG, nanocellulose, ethoxylated alcohols	-	[21]; [13]
Scope	Cold mix, tack coat, chip seal	Waterproofing, limestone aggregate	Special industrial coatings	-	[4]

Droplet size and distribution: D50 and D90 values were determined by laser diffractometry [4, 7, 8, 19]; electron microscopy (EM) was used in some studies to confirm droplet morphology. Zeta potential: the electrostatic charge on the droplet surface was measured by electrophoretic light [1, 4, 14, 16, 18] using the Smoluchowski equation; according to the literature, a zeta potential with an absolute value above 30 mV is an indication of a stable colloidal system [14].

Results. ***General characteristics of the selected sources.*** The physicochemical and technological characteristics of the three main types of emulsifiers, as well as the standards used to measure them, are summarized in Table 1.

Emulsifiers and stability. Cationic emulsifiers are the most common type in bitumen emulsions, imparting a positive charge to bitumen droplets. The effect of pH on stability and rheology was studied by modifying a cationic, slowly breaking emulsion with HCl and NaOH solutions. This study demonstrated a significant improvement in the storage stability of the emulsion at pH 2.5-4.0 [3].

Among recent studies, the molecular dynamics analysis by Yan et al. is particularly significant: they compared "cetyl ionic" emulsions based on CTAC, cetylpyridinium chloride (CPC), hexadecyl sulfate (SHS), and hexadecyl sulfonate (SHSN), confirming the primary role of electrostatic action in interface stability [19]. This result is also true for classical amine salts such as octadecylamine hydrochloride: the long hydrocarbon chain penetrates the bitumen phase and the positively charged amine head forms an electrostatic barrier at the water-bitumen interface [1].

Anionic emulsifiers impart a negative charge to bitumen droplets. Razali compared the properties of emulsified modified bitumen prepared using three types of emulsifiers: 2-bromostearic acid (anionic), didodesyl dimethyl ammonium bromide (cationic), and polyethylene glycol (non-ionic). The anionic emulsifier demonstrated superior stability, releasing only 1.67% water, while the cationic emulsifier provided the fastest drying time (8 hours) [2].

Studies using sodium oleate showed that the stability of anionic bitumen emulsions for waterproofing purposes was greatest at the optimum emulsifier concentration [13]; this result confirms the general pattern that stability is maximized at emulsifier concentrations near the CMC.

Non-ionic emulsifiers stabilize emulsions through a steric barrier mechanism rather than electrostatic stabilization. This mechanism offers advantages during long-term storage due to its low sensitivity to ionic strength [2].

Kraft lignin chemically modified by the Mannich reaction and bioethanol lignin were used as cationic emulsifiers in bitumen emulsions. Cationic kraft lignin effectively stabilized a 60% bitumen-containing emulsion at a concentration of 0.25-0.75%, at a pH of 13, while maintaining a droplet size of approximately 4.5 μm [4]. Furthermore, Losa tested nanocellulose crystals as an emulsifier, proving them to be a viable alternative to commercial emulsifiers [8].

The results showed that the type of emulsifier significantly affects the storage stability and degree of dispersion of the bitumen.

Comparative analysis of emulsifier concentration and stability parameters.

Table 2

Bitumen emulsion stability parameters for different emulsifier types and concentrations

Emulsifier Type	Concentration (wt%)	pH	Droplet size (μm)	Storage stability (day)	Zeta potential (mV)
Cationic (amen)	1.0	3.5	5.2	>90	+35 ± 3
Cationic (amen)	1.5	3.5	3.8	>120	+48 ± 4
Anionic (fatty acid)	1.0	9.5	6.8	60-75	−32 ± 4
Anionic (fatty acid)	1.5	9.5	5.1	75-90	−45 ± 5
Non-ionic (PEG)	2.0	7.0	7.4	>180	≈0
Cationic lignin	0.5	13.0	4.5	>150	+28 ± 5

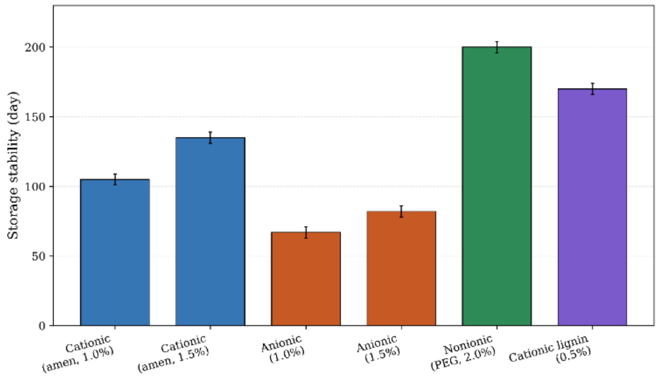

Fig.1. Shelf life of bitumen emulsion depending on the type and concentration of emulsifier.

As can be seen from Table 2, increasing the cationic emulsifier concentration from 1.0 to 1.5 wt.% leads to an increase in the zeta potential from +35 to +48 mV and an increase in shelf life from 90 to 120 days. The nonionic emulsifier provides stability for over 180 days, but the adhesive forces are low due to the zeta potential being close to zero. The cationic lignin emulsifier provided stability for over 150 days at a relatively low concentration of 0.5 wt.% and a high pH of 13 with a positive zeta potential of 28 mV. The following diagrams (Fig. 1 and Fig. 2) visually illustrate this relationship.

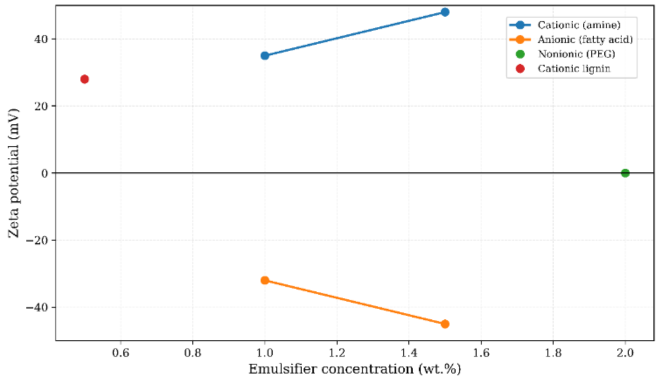

Fig.2. Effect of emulsifier type and concentration on zeta potential.

Advantages and Applications of Emulsifiers. The technological advantages, disadvantages, and practical applications of the three types of emulsifiers are summarized in Table 3. This classification is used to select the most appropriate emulsifier type for specific construction conditions (unit type, climate, repair timeframe) at the design stage.

Table 3

Advantages, Disadvantages, and Applications of Emulsifiers

Emulsifier Type	Advantages	Disadvantages	Main areas of application	Ref.
Cationic	Strong adhesion to silicate aggregates; fast drying (approximately 8 hours)	low pH; some amine salts irritate skin and eyes; relatively high price	Cold mixes, tack coat, chip seal (closed coating), PMBE (polymer-modified emulsion)	[1]; [2]
Anionic	Relatively inexpensive; well compatible with carbonate aggregates; low toxicity	Stability period is shorter (2-4 months); when mixed with cement, it may delay hydration.	Waterproofing coatings, road construction in limestone regions, CA (cement-bitumen) mortar	[13]; [21]; [22]
Non-ionic	Longest shelf life stability (>180 days); low sensitivity to ionic strength and electrolytes.	Weak adhesion to the aggregate (zeta potential ≈ 0); high cost.	Special industrial coatings, long-term storage emulsions, mixed (complex) formulas	[2]; [20]

Discussion. The reviewed sources confirm a general trend: the type, chemical nature, and concentration of the emulsifier are the most important factors affecting the stability of bitumen emulsions. Cationic emulsifiers remain the most common type for road construction, as their positively charged droplets are electrostatically attracted to the negative surface charge of most silicate aggregates [2, 3].

Sidun's study showed that the type of acid used in the preparation of cationic emulsions-HCl and H_3PO_4-has a significant impact on storage stability. Emulsions prepared with HCl showed superior results in screening stability and sedimentation tendency compared to H_3PO_4 [9]. This result further emphasizes the importance of acid selection for cationic emulsifiers.

The electrochemical approach based on zeta potential and surface free energy has become the leading method for assessing emulsion stability in recent years. While Pinto and Buss demonstrated that zeta potential is a reliable quantitative measure of asphalt emulsion stability [14], Cuaran-Cuaran extended this approach to assessing electrochemical interactions with aggregate [18]. Meanwhile, Yang confirmed the electrostatic superiority of cationic surfactants such as CTAC in interface stability at the microscopic level using molecular dynamics simulations [19], allowing us to link macroscopic zeta potential measurements to the molecular mechanism.

Conclusion. This systematic review led to the following key findings across emulsifier types.

Cationic emulsifiers. Cationic emulsifiers such as CSS-1h, CTAC, and octadecylamine hydrochloride provide the highest stability and adhesion properties for materials used in acidic environments with a pH of 2-5 and in combination with silicate aggregates. Their superiority is explained by the mechanism of electrostatic attraction, and stability is further enhanced at concentrations above the SMC of 1.0-1.5 wt%.

Anionic emulsifiers. Anionic emulsifiers based on sodium oleate and other fatty acid salts are preferred for limestone-based aggregates and waterproofing applications. An optimal concentration (~3% for sodium oleate) significantly improves stability; however, they require careful hydration when mixed with cement.

Non-ionic emulsifiers. Non-ionic emulsifiers, such as PEG derivatives, provide the longest shelf stability, i.e., over 180 days, due to steric stabilization, but have weaker adhesion to aggregates than cationic emulsifiers and are used in specialized industrial construction.

Bioemulsifiers. Lignin- and nanocellulose-based bioemulsifiers have the potential to become a worthy alternative to traditional chemical emulsifiers: cationic kraft lignin exhibits effective stabilizing properties at levels of 0.25-0.75%.

Furthermore, the production method for polymer-modified bitumen emulsions, along with the type of emulsifier, significantly impacts shelf stability and mechanical properties. Cationic emulsions prepared with HCl acid have superior shelf stability to H3PO4-based emulsions, and the choice of acid type is also an important parameter.

Assessing the future industrial performance of bioemulsifiers. Of current importance are studies aimed at more deeply understanding the molecular mechanisms of cationic surfactants such as CTAC and octadecylamine hydrochloride, analyzing their long-term stability under various climatic conditions, and optimizing emulsifier formulations using HLB theory and numerical modeling methods.

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