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SYNTHESIS OF MODIFIERS FOR ASPHALT CONCRETE MIXTURES FROM PETROCHEMICAL WASTE

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Abstract. The relevance of this study is determined by the problem of solutions in the field of secondary processing of various waste products of petrochemical production for the purpose of their subsequent application for the synthesis of a new product. Recycled materials can be successfully used for the road construction industry as part of asphalt concrete coatings. This approach will significantly reduce the level of environmental pollution, and is also beneficial from an economic point of view. The aim of this work was to synthesize an organic amine-derived surfactant from petrochemical waste, which in future studies will be used as a modifying additive for asphalt concrete coatings with improved performance properties. Using the method of probabilistic-deterministic planning of the experiment, the modes of obtaining amine derivatives of various compositions by the reaction of amination of higher aldehydes with gaseous ammonia in the presence of lower aldehydes are optimized. The synthesis products are recommended for use as modifiers for the development of asphalt concrete mixtures with improved structural and mechanical characteristics. Due to the introduction of the synthesized AC-1 modifier at the level of 0.1-0.2%, the structural and mechanical characteristics of asphalt concrete mixtures are improved, which allows the use of this cost-effective modifier in the production of road surfaces with improved performance properties.

Keywords: amine-containing surfactants, petrochemical waste, synthesis of modifiers, asphalt-concrete mixtures, road coatings.

1. Introduction.

The most common type of improved road surfaces are asphalt concrete coatings made using petroleum road bitumen. An increase in the traffic intensity of cars

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leads to premature destruction of road surfaces, the service life of which is on average about 6 years.

One of the main causes of destruction of road surfaces is the unsatisfactory adhesion of bitumen to the surface of mineral materials, insufficient water and frost resistance of asphalt concrete. There are various ways to solve this problem, however, one of the most effective ways to ensure the required adhesion of bitumen to the surface of mineral materials is the use of surfactant additives in bitumen. Asphalt concrete mixtures with surfactant additives, adhesive bitumen additives and activators are used to increase the corrosion resistance of asphalt concrete pavements, increase the plasticity of asphalt concrete mixtures at all stages of the technological process, and increase the adhesion of the binder to mineral materials [1-2].

There is a large amount of waste from petrochemical industries that must be used as raw materials for the synthesis of various substances. This approach will significantly reduce the level of environmental pollution, as well as beneficial from an economic point of view [3-6]. The aim of this work was to synthesize an organic amine derivative of a surfactant, which in further studies will be used as a modifying additive for asphalt concrete pavements with improved operational properties.

Organic amine derivatives are amphiphilic compounds whose molecules consist of at least two parts, one of which is soluble in liquid (the lyophilic part), and the second part is insoluble (the lyophobic part). Such a structure determines their surface-active properties [7-10].

Currently, amine-containing surfactants are widely used in industrial applications as modifying additives for various functional purposes. The effectiveness of surfactants based on organic amine derivatives largely depends on the characteristics (structure, length) of the hydrocarbon radical in composition. Purposeful synthesis of primary and secondary amines is carried out mainly by reactions of direct amination of organic chlorine derivatives with ammonia or by reactions of concentration of higher aldehydes with lower amines. The disadvantages of these methods include multi-stage operations, which require the preliminary production of intermediate products and, as a result, the high cost of their production [11-12].

Taking into account the high cost of preparative reagents and the increased demand for amines, two tasks were envisaged in the development of the synthesis:

1. To carry out the synthesis of organic amine derivatives using waste and waste materials of petrochemical production, in which a mixture of aldehydes is concentrated.

2. To provide conditions for mass transfer of reagents and quantitative assimilation of ammonia.

2. Experimental part.

During the research we used:

1. Dimer fraction of distillation residues of the column distillation of butyraldehydes DF-270, formed in the technology of oxosynthesis of alcohols. According to its chemical composition, DF-270 is a mixture of reactive oxygen-containing aliphatic compounds of the limiting (2-methylpropanal) and unsaturated (methylpropenal) character. For the synthesis, we selected representative fractions of DF-270 with a weighted average molecular weight of 223 and a density of 0.925 g/cm^3. The molar ratio of higher to lower aldehydes varied in the sample in the range of 3.5-4.0 and the carbonyl and acid numbers at the level of 120-125 and 49-51 mg KOH/g, respectively. The total content of aldehydes in the samples was 55-60 %.

2. Residues from the production of butyl alcohols and 2 - ethylhexanol KOH - 92 (TU 38.302-75-03-92), which is a mixture of aldehydes (acetic – 5.6 %, oil – 4.4 %, crotonic – 1.1 %, 2-ethylbutenal – 12.8 %, hexanal – 2.4 %) and alcohols (2-ethylhexenol – 2.3 %, isohexanol – 1.6 %, 2-ethylhexanol – 4.0 %, butanol - the rest).

3. Crotonaldehyde with a basic substance content of 98.3%.

4. Gaseous ammonia from a cylinder (GOST 3760-79).

To intensify the mass transfer processes in the liquid and gas phases of the reaction space, we installed a sealed stainless-steel reactor (Kh18N10T) with a volume of 1.0 dm^3 (filling factor – 0.7). In the upper part, the reactor is equipped with an irrigation nozzle for continuous circulation and simultaneous stirring of the reaction mixture. To supply (under pressure) gaseous ammonia, a tubular dispersing device is located in the bottom of the reactor perimeter. Since the amination process is exothermic, the temperature in the reactor (25 °C) was maintained by circulating water through built-in heat exchangers. The synthesis was carried out at a fixed partial pressure of ammonia (0,025 MPa), which (as it was absorbed) was periodically fed to the reactor from a cylinder using a reducer.

To determine the optimal amination modes in the experiments, along with the duration factor (τ, 0 – 60 min), the circulation rate of the solution was varied (ω, 0.05 – 0.3 dm^3/min) and the quantitative consumption of crotonaldehyde in relation to higher aldehydes in the DF-270 composition were varied (G, 0.8–1.2 mole/mole). It was ensured by varying the volumes of crotonaldehyde in the reaction mixture.

The composition of KON-92 includes a mixture of higher aldehydes. Higher aldehydes do not directly react with ammonia, therefore, a condensation reaction is carried out with the products of ammonia amination of croton aldehyde according to the following scheme:

$$CH_3\text{-}CH{=}CH\text{-}COH + NH_3 \xrightarrow[-H_2O]{} CH_3\text{-}CH{=}CH\text{-}CH{=}NH_2,$$

$$CH_3\text{-}CH{=}CH\text{-}CH{=}NH_2 + R\text{-}CHOH \xrightarrow[-H_2O]{} R'\text{-}NH_2 + 3R'\text{-}NH\text{-}R'',$$

where R-CHOH are higher aldehydes,

R' – butyl, R" – 2-ethyl-2-hexenyl.

As a result of the synthesis, a mixture of substances is formed, but if the condition of a constant mass ratio "KOH-92 – crotonaldehyde – ammonia" is met, the target product is formed, which is a homogeneous light brown mass that dissolves in ethyl alcohol, white spirit, xylene, toluene, petroleum ether, kerosene and concentrated sulfuric and acetic acids.

The reactor was sealed, then ammonia was supplied from a cylinder, a circulation pump and a timer were turned on. The development of the aldehyde amination process was monitored by taking 5-7 ml samples at regular intervals (using a valve built into the circulation system). The degree of amination of higher aldehydes (α, %) and the composition of the products were determined from the results of chemical (GOST 25266-82) and spectral (Fourier spectrometer FSM 1202) analyzes.

3. Results and discussion

All experiments were performed in accordance with the plan-matrix of a 4-factor experiment at 3 levels of probabilistic-deterministic planning [13]. Experimental data on the degree of amination of higher aldehydes in the composition of DF-270 and KOH-92 are presented in table1.

Table 1 – Modes and indicators of amination of higher aldehydes in the composition of DF-270 and KOH-92

Experimental conditions			Amination degree,%	
G, mole/mole	ω, dm^3/min	τ, min	DF -270	KOH-92
1.2	0.05	10	60.1	52.3
1.2	0.2	20	94.3	81.8
1.2	0.3	30	98.7	90.2
1.0	0.05	30	80.2	71.2
1.0	0.2	10	75.4	64.7
1.0	0.3	20	96.8	80.2
0.8	0.05	20	55.1	48.4
0.8	0.2	30	67.3	60.7
0.8	0.3	10	49.9	40.3

After sampling the experimental array, partial dependences were obtained (Fig.), reflecting the influence of ω, τ and G on the degree of amination of higher aldehydes in the composition of KOH-92 and DF-270. The analysis showed that the processes of amination develop most intensively in the presence of DF-270; for the quantitative amination of aldehydes in the composition at G=1.0 mole/mole and ω=0.2 dm^3/min, it took at least 30 minutes.

The duration of the amination of aldehydes in KOH-92 under the same conditions is 1.7–1.8 times longer and amounted to approximately 50 minutes. An increase in the circulation rate of the reaction mixture above 0.2 dm^3/min is impractical, since the increase in the degree of conversion of aldehydes turned out to be insignificant. It is expedient to set the consumption of crotonaldehyde for the quantitative amination of higher aldehydes in the composition of KOH-92 and DF-270 at a level of 1.0 – 1.1 mole.

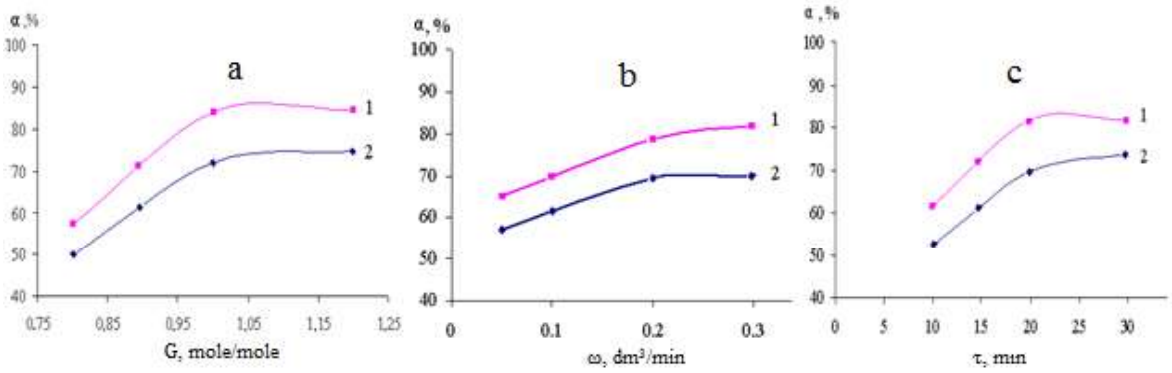

Figure. Influence of crotonaldehyde consumption (a), solution circulation rate (b) and synthesis duration (c) on the degree of amination of higher aldehydes in DF-270 (1) and KOH-92 (2)

To assess the resulting contribution of all three factors to the degree of amination of DF-270 and KOH-92, generalized equations were obtained, 1 and 2, respectively:

$$\alpha 1 = (-0.058 \cdot G2 + 0.13 \cdot G - 0.055) \cdot (69.8 \cdot \omega + 62.4) \cdot (33.5 \cdot \tau\ 0.28) \quad (1)$$

$$\alpha 2 = (-0.057 \cdot G2 + 0.13 \cdot G - 0.055) \cdot (\ 54.4\omega + 55.5) \cdot (\ 25.1 \cdot \tau\ 0.33) \quad (2)$$

By differentiating the multi-factor dependencies 1 and 2, equations for calculating the rate of amination of aldehydes are obtained. The average amination rates of aldehydes in the composition of DF-270 and KOH-92 at the specified modes G=1.0 mole/mole and ω = 0.2 dm^3/min were 3.1 and 2.0 %/min.

Balance experiments were carried out in optimized modes and pilot batches of products were accumulated. The degree of conversion of aldehydes in the composition of DF-270 and KOH-92, in 35 and 55 minutes was at least 98.5 %. The amination product of KOH-92 (hereinafter AC-1) is a homogeneous light brown mass that dissolves in ethyl alcohol, white spirit, xylene, toluene, petroleum ether, kerosene and concentrated sulfuric and acetic acids. The amination product DF-270 (hereinafter AC-2) is a homogeneous dark brown mass with a boiling point over 200 °C. Chemical analysis showed that the total content of primary and secondary amines in the composition of AC-2 is 1.8-2.0 times higher than in AC-1.

One of the possible applications of organic amine derivatives is the modification of asphalt concrete mixtures. According to the results of preliminary tests, it was found that the introduction of AC-1 into the composition of asphalt concrete compositions significantly improves the quality characteristics of road surfaces - durability, adhesion, water resistance, compressive strength, swelling, heat resistance, etc. The above indicators are provided when using a different-grade mineral filler (crushed stone, sand). The following components were used for the preparation of the asphalt-concrete mixture: bitumen of the BND 90/130 brand, mineral filler consisting of granite-based crushed stone (10-20 mm fraction), surfactant AC-1.

The process of preparing the asphalt mixture includes heating the bitumen to 130-160 °C. A paste-like product AC-1 is introduced into the bitumen melt and mechanical mixing is carried out for 15-25 minutes. The resulting mixture is processed mineral filler to complete impregnation. Table 2 shows the compositions of the studied asphalt-concrete mixtures.

Table 2 – Compositions of asphalt concrete mixtures, wt.%

Component	Sample					
	1	2	3	4	5	6
Bitumen	6.5	5.0	5.6	6.0	6,5	5.6
AC-1	0.25	0.10	0.15	0.05	0.20	0.17
Mineral filler	93.25	94.9	94.25	93.95	93.6	94.23

The test results of these compositions are shown in table 3.

Table 3 – Structural and mechanical properties of asphalt concrete mixtures

Indicator	1	2	3	4	5	6
Water saturation	2.3	2.4	2.4	2.7	2.3	2.8
The coefficient of water resistance	1.8	1.5	1.6	1.8	1.8	1.2
Swelling	0.08	0.08	0.07	0.09	0.07	0.09
Compressive strength, kgf/cm	34	31	31	26	34	25
R50	29	26	27	22	29	22

As evidenced by the data obtained, a decrease in the consumption of AC-1 below 0.1 % leads to a deterioration in the indicators of the structural and mechanical properties of asphalt concrete mixtures (sample 4, table 3). An increase in the AC-1 consumption over 0.2 % does not impair the structural and mechanical properties of the mixtures, however, it is undesirable due to the overrun of the modifier.

4. Conclusion

Thus, according to the results of the studies performed, we can conclude:

1. Used waste and waste materials of petrochemical production allow to synthesize a modifier for asphalt concrete mixtures.

2. The process of ammonia amination of higher aldehydes in the composition of the distillation residues KOH-92 and DF-270 is quantitatively realized in the presence of a lower aldehyde - crotonic.

3. The amination process is intensified in the modes of forced circulation of the reaction mixture. At a given partial pressure of ammonia of 0.025 MPa and a circulation rate of the reaction mixture of 0.2 dm^3/min, no less than a 2-fold increase in the rate of the process and, at the same time, quantitative assimilation of ammonia were provided. The consumption of the latter does not exceed 1,1 mole per 1 mole of crotonaldehyde.

4. Due to the addition of the synthesized modifier AC-1 at the level of 0.1-0.2 %, the structural and mechanical characteristics of asphalt concrete mixtures are improved, which makes it possible to use this economically beneficial modifier in the production of road surfaces with improved operational properties.

Conflict of Interest: The authors declare that they have no competing interests.

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Түйіндеме

МҰНАЙ-ХИМИЯЛЫҚ ҚАЛДЫҚТАРДАН АСФАЛЬТ-БЕТОНДЫҚ ҚАТЫСТЫРМАЛАРДЫ МОДИФИКАТОРЛАР СИНТЕЗІ

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Бұл зерттеудің өзектілігі - мұнай-химия өндірісінің әртүрлі қалдықтарын кейіннен жаңа өнімді синтездеу үшін қолдану мақсатында қайта өңдеу саласындағы мәселе шешімдер анықталады. Қайта өңделген материалдарды асфальтбетон жабындарының құрамында жол құрылысы индустриясы үшін сәтті пайдалануға болады. Бұл тәсіл қоршаған ортаның ластану деңгейін төмендетуге мүмкіндік береді, сонымен қатар экономикалық тұрғыдан тиімді. Осы жұмыстың мақсаты - мұнай-химия қалдықтарынан беттік белсенді заттың органикалық амин туындысын синтездеу болып табылды, оны әрі қарай зерттеу барысында өнімділік қасиеттері жақсартылған асфальтбетон жабындарына өзгертетін жетілдірілген қоспа ретінде қолданылады. Тәжірибені ықтималдық-детерминдік жоспарлау әдісін қолдана отырып, төменгі альдегидтердің қатысуымен жоғары альдегидтердің газ тәрізді аммиакпен аминдеу реакциясы арқылы әр түрлі құрамдағы амин туындыларын алу режимдері оңтайландырылған. Синтез өнімі жақсартылған құрылымдық-механикалық сипаттамалары бар асфальт-бетон қоспаларын өңдеуші ретінде

қолдануға ұсынылады. 0,1-0,2% деңгейінде синтезделген АС-1 модификаторының енгізілуімен асфальтбетон қоспаларының құрылымдық-механикалық сипаттамалары жақсаруына байланысты осы экономикалық жағынан тиімді модификаторды пайдалану қасиеттерін жетілдірілген жол төсемдері өндірісінде пайдалануға мүмкіндік туғызады.

Түйінді сөздер: құрамында амин бар беттік белсенді заттар, мұнай-химия қалдықтары, модификаторлар синтезі, асфальтбетон қоспалары, жол төсемдері.

Резюме

СИНТЕЗ МОДИФИКАТОРОВ ДЛЯ АСФАЛЬТОБЕТОННЫХ СМЕСЕЙ ИЗ ОТХОДОВ НЕФТЕХИМИИ

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Актуальность настоящего исследования определяется проблемой решений в области вторичной переработки различных отходов нефтехимического производства с целью их последующего применения для синтеза нового продукта. Переработанные материалы могут успешно использоваться для дорожно-строительной отрасли в составе асфальтобетонных покрытий. Данный подход позволит существенно снизить уровень загрязнения окружающей среды, а также выгоден с экономической точки зрения. Целью настоящей работы являлся синтез органического аминопроизводного поверхностно-активного вещества из отходов нефтехимии, которое в дальнейших исследованиях будет применено в качестве модифицирующей добавки для асфальтобетонных покрытий с улучшенными эксплуатационными свойствами. С применением метода вероятностно-детерминированного планирования эксперимента оптимизированы режимы получения аминопроизводных различного состава по реакции аминирования высших альдегидов газообразным аммиаком в присутствии низших альдегидов. Продукты синтеза рекомендованы к использованию в качестве модификаторов для разработки составов асфальтобетонных смесей с улучшенными структурно-механическими характеристиками. За счет введения синтезированного модификатора АС-1 на уровне 0,1-0,2 % улучшаются структурно-механические характеристики асфальтобетонных смесей, что позволяет применять данный экономически выгодный модификатор при производстве дорожных покрытий с улучшенными эксплуатационными свойствами.

Ключевые слова: аминосодержащие поверхностно-активные вещества, отходы нефтехимии, синтез модификаторов, асфальтобетонные смеси, дорожные покрытия.