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INFLUENCE OF GROUP CHEMICAL COMPOSITION OF HIGHLY VISCOUS OIL RESIDUES ON QUALITY OF PETROLEUM ROAD BITUMENS



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The Kalamkas deposit's oil and its distillation products were studied, and research results on residues and road grade bitumen compositions obtained by oxidation of heavy oil residuest are presented. The atmospheric residue of Kalamkas oil (350 C°) was subjected to vacuum distillation with the selection of gas oil fractions boiling to 380, 400 and 420 C°. Samples of residues were subjected to oxidation in a laboratory installation, which is a batch cube at constant operating conditions.

The residues from different depths of gas oil fractions are characterized by different group chemical compositions. The group composition of the residues changes, the content of paraffin-naphthenic hydrocarbons decreases from 29.4 to 23.3% wt. and the proportion of heavy aromatic hydrocarbons increases from 26.8 to 27.8% wt. The content of asphaltenes (from 7.7 to 12.4% wt) and resins (from 17.4 to 22.5% wt) also increases. The ratio of asphaltenes and resins increases from 0.44 to 0.55. With an increase in the depth of selection of gas oil fractions to 420°C, the degree of aromaticity, the ratio of the amount of aromatic hydrocarbons to paraffin-naphthenic ones, increases to 1.79.

Road grade bitumens produced from the vacuum distillation residues of Kalamkas oil fuel oil using direct oxidation technology of raw materials have the best performance characteristics if they are obtained from residues boiling in the range of 380–400°C. Residues boiling above 380-400 C° can serve as raw materials for the production of oxidized road bitumen of BND70/100 grade according to ST RK 1373 - 2013.

KEY WORDS: *road bitumens; group chemical composition; tars; oxidation; fuel oil; quality; specifications.*

ТҰТҚЫРЛЫҒЫ ЖОҒАРЫ МҰНАЙ ҚАЛДЫҚТАРЫНЫҢ ТОПТЫҚ ХИМИЯЛЫҚ ҚҰРАМЫНЫҢ МҰНАЙ ЖОЛ БИТУМДАРЫНЫҢ САПАСЫНА ӘСЕРІ

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Мақалада Қаламқас мұнайы мен оның айдау өнімдеріне зерттеу жүргізіліп, Қаламқас кен орнынан алынған қалдықтардың және жол маркалы битумдардың құрамына жүргізілген зерттеу нәтижелері ұсынылған.

Қаламқас мұнайының атмосфералық қалдығы (350 С°) 380 С° , 400 С° және 420 С°-дейін қайнайтын газойль фракцияларын бөліп алумен вакуумдық айдауға ұшыратылды. Қалдық үлгілері тұрақты режимдік көрсеткіштерде зертханалық қондырғыда – мерзімді әрекетті кубта тотықтырылды.

Авторлар газойль фракцияларын бөліп алудың әр түрлі тереңдігіндегі қалдықтар әртүрлі топтық химиялық құрамдармен сипатталатындығын көрсеткен. Бұл кезде қалдықтардың топтық құрамы өзгереді – парафинді-нафтенді көмірсутектердің мөлшері масса бойынша 29,4-тен 23,3 мас.%-ға дейін төмендейді, ал ауыр ароматты көмірсутектердің үлесі 26,8-ден 27,8%-ға дейін артады. Асфальтендер (7,7-ден 12,4мас.%-ға дейін) және шайырлар (17,4-тен 22,5% мас. -ға дейін) мөлшері де артады. Асфальтендер мен шайырлардың (А/Ш) қатынасы 0,44-тен 0,55-ке дейін артады. Газойль фракцияларын іріктеу тереңдігінің 420°С-қа дейін ұлғаюымен ароматтылық дәрежесі—ароматты көмірсутектер мөлшерінің парафиндік-нафтенді көмірсутектерге қатынасы (АК/ПН) — 1,79-ға дейін артады.

Шикізатты тікелей тотықтыру технологиясын қолдана отырып, Қаламқас мазутының вакуумды айдау қалдықтарынан алынған жол маркалы битумдар, егер олар 380–400 С° аралығында қайнайтын қалдықтардан алынса, ең жақсы өнімділік сипаттамаларына ие. 380-400 С° жоғары қайнайтын қалдықтар ҚР СТ 1373 - 2013 бойынша тотыққан жол битумының БНД 70/100 маркасын алу үшін шикізат ретінде қызмет ете алады.

ТҮЙІНДІ СӨЗДЕР: *жол битумдары; топтық химиялық құрам; гудрондар; тотығу; мазут; сапа; техникалық сипаттамалар.*

ВЛИЯНИЕ ГРУППОВОГО ХИМИЧЕСКОГО СОСТАВА ОСТАТКОВ ВЫСОКОВЯЗКОЙ НЕФТИ НА КАЧЕСТВО НЕФТЯНЫХ ДОРОЖНЫХ БИТУМОВ

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Проведено исследование Каламкасской нефти и продуктов ее перегонки, приведены результаты исследований состава остатков и битумов дорожных марок, полученных окислением тяжелых остатков нефти месторождения Каламкас.

Атмосферный остаток Каламкасской нефти (350 С°) подвергали вакуумной перегонке с отбором газойлевых фракций, выкипающих до 380 С°, 400 С° и 420 С°. Образцы остатков подвергали окислению на лабораторной установке - кубе периодического действия при постоянных режимных показателях.

Авторами показано, что остатки различной глубины отбора газойлевых фракций характеризуются разным групповым химическим составом. При этом изменяется групповой состав остатков — снижается содержание парафинонафтеновых углеводородов с 29,4 до 23,3% мас. и повышается доля тяжелых ароматических углеводородов с 26,8 до 27,8% мас. Повышается также содержание асфальтенов (с 7,7 до 12,4% мас.) и смол (с 17,4 до 22,5% мас). Соотношение асфальтенов и смол (А/С) при этом увеличивается с 0,44 до 0,55. При увеличении глубины отбора газойлевых фракций до 420°С степень ароматичности — отношение количества ароматических углеводородов к парафинонафтеновым (АУ/ПН), возрастает до 1,79.

Битумы дорожных марок, вырабатываемые из остатков вакуумной перегонки мазута Каламкасской нефти по технологии прямого окисления сырья, имеют наилучшие эксплуатационные характеристики, если они получены из остатков, выкипающих в диапазоне 380 – 400 С°. Остатки, выкипающие выше 380 – 400 С°, могут служить сырьем для получения окисленных дорожных битумов марки БНД 70/100 по СТ РК 1373 – 2013.

***КЛЮЧЕВЫЕ СЛОВА:** дорожные битумы; групповой химический состав; гудроны; окисление; мазут; качество; технические характеристики.*

Introduction. The properties of petroleum bitumen mainly depend on its group chemical composition, which in turn is determined by the nature of the petroleum feedstock and the bitumen production technology [1]. It is known that commercial bitumen with the same physical and chemical properties, softening point, extensibility and other indicators can have different performance properties.

In this aspect, the issue of involving special types of raw materials in bitumen production is relevant. The most suitable for bitumen production are highly aromatized, highly resinous oils of an aromatic, naphthenic-aromatic base. There is positive world experience in obtaining high-quality bitumen from Venezuelan, heavy Arab, and Mexican oils. Favorable raw materials for the production of bitumen are heavy, highly resinous oils from Western Kazakhstan [2-4].

Residues of highly resinous aromatic oils are the best type of raw material for the production of oxidized bitumen. At the same time, sulfur, which is part of the heavy residues from the distillation of resinous sulfurous oils, contributes to the production of high-quality bitumens [5, 6].

The relationships between asphaltenes, resins and oils, as well as their various ratios on bitumen quality indicators, have been studied [7]. It is suggested that resins and asphaltenes are characterized by the manifestation of "collective" properties due to the similarity of their nature and structure. The boundary between asphaltenes and resins is very arbitrary, so it is more expedient to consider their joint influence on the characteristics of bitumen.

In the production of bitumen by oxidation of petroleum feedstock, the group hydrocarbon composition of the feedstock determines both the quality of the resulting bitumen and the intensity of its oxidation.

It has been shown [8] that regulation of the physicochemical properties of oxidized bitumen is possible by determining the hydrocarbon group composition of tars and selecting the optimal ratio of hydrocarbon groups in the feedstock. Stabilization of raw materials of optimal composition, as well as the balance of the ratio of hydrocarbon groups in this raw material, make it possible to change the quality of road bitumen in accordance with the regulatory requirements of modern standards.

Thus, the development of a rational technology for the production of high-quality road bitumen from specially selected raw materials, in particular, heavy resinous oils, having a high range of plasticity and resistance to thermal-oxidative aging is an urgent task in oil refining.

Materials and methods. In the experimental part, a method was used for conducting research on Kalamkas oil and its distillation products, as well as the results of studying the composition of residues and bitumen of road grades obtained using traditional technology for producing bitumen by high-temperature oxidation of residues of different depths of gas oil fractions.

To find ways to rationally use oil from the Kalamkas field on the Buzachi Peninsula, the main physical and chemical characteristics were determined and the possibility of producing bitumen from it was assessed.

Kalamkas oil has a density of 912 kg/m^3, sulfurous (1.62% wt.), paraffinic (paraffin content 3.15% with a melting point of 57 C^o), highly resinous (1.4% asphaltenes, 17.42% silica gel resins, coking properties - 4.7%), low-hardening (–27°C).

According to the A + C indicator - 2.5P [9], Kalamkas oil is completely suitable for the production of high-quality bitumens of a wide range.

Straight-run fuel oil from Kalamkas oil was subjected to vacuum distillation using an automatic installation with computer control Dist D-1160 CC to carry out distillation analyzes of petroleum products in accordance with the ASTM D 1160 standard. Samples of tar boiling above 380 C^o, 400 C^o and 420 C^o were obtained. Samples of tar were subjected to oxidation in a laboratory installation, which is a batch cube at constant operating parameters: process temperature 250 ^{o}C; air consumption for oxidation is 1.5 liters per 1 kg of raw materials per minute. Experimental samples of bitumen were tested for compliance with the requirements of the current regulatory and technical documentation and, based on the test results obtained, a sample with optimal technical characteristics was selected.

Residues and bitumens were analyzed by determining the group chemical composition and generally accepted standard quality assessment methods used in oil refining; the analysis results are presented in *Tables 1* and *2*.

Results and discussion. As the selection deepens, the content of paraffin-naphthenic, light and medium (mono- and bicyclic) aromatic hydrocarbons in the oil residue decreases. Due to this, the proportion of heavy polycyclic aromatic compounds increases.

Thus, the heavier the residue, the higher its content of resins and asphaltenes. As the depth of selection of vacuum distillates increases, the density and relative viscosity of tar increase.

It is known that the higher the ratio of asphaltenes to resins in oil and the lower the content of solid paraffins, the better the quality of the resulting bitumen and the simpler the technology for their production [10,11].

From the data in *Table 1* it follows that the residues of different depths of selection of distillate fractions are characterized by different group chemical compositions. At the same time, the group composition of the residues changes: the content of paraffin-naphthenic

Table 1 – **Characteristics of residues of different sampling depths obtained from Kalamkas oil**

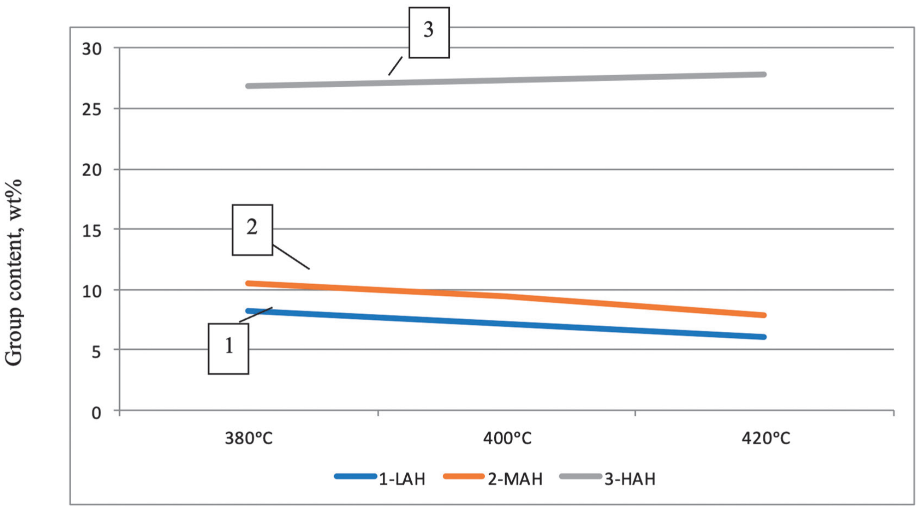
Indicators	Measurement	Indicator values for residues boiling above °C		
		380	400	420
Relative density at 20 C°	kg/m³	964,0	969,2	975,8
Total sulfur content	% wt.	1,82	1,97	2,06
Coking according to Conradson	%	8,5	10,3	11,4
Flash point	°C	214	219	228
Conditional viscosity at 80 °C	sec.	8,6	16,8	20,2
Group chemical composition: hydrocarbon content - paraffin-naphthenic - aromatic, incl. - light - medium - heavy - resins - asphaltenes	% wt.	29,4 45,5 8,2 10,5 26,8 17,4 7,7	26,7 43,9 7,2 9,4 27,3 19,6 9,8	23,3 41,8 6,1 7,9 27,8 22,5 12,4
Ratio of A/R		0,44	0,50	0,55
Ratio of AH/PN		1,55	1,64	1,79
Reaching oil	% wt.	56,1	53,4	51,6

hydrocarbons decreases from 29.4 to 23.3% wt. and the proportion of heavy aromatic hydrocarbons increases from 26.8 to 27.8% wt. The content of asphaltenes (from 7.7 to 12.4% wt) and resins (from 17.4 to 22.5% wt) also increases. The ratio of asphaltenes and resins (A/R) increases from 0.44 to 0.55. With an increase in the depth of selection of distillate fractions to 420 °C, the degree of aromaticity i.e. the ratio of the amount of aromatic to paraffin-naphthenic hydrocarbons (AH/PN) increases to 1.79.

Figure 1 shows the data on the changes in the content of individual groups of aromatic hydrocarbons depending on the depth of residue selection. In the figure and below, PNH are paraffin-naphthenic hydrocarbons, LAH are light aromatic hydrocarbons, MAH are medium aromatic hydrocarbons and HAH are heavy aromatic hydrocarbons. As the selection deepens in the residues, the content of paraffin-naphthenic, light (LAH) and medium (mono- and bicyclic) aromatic compounds (MAH), which are removed from the vacuum column with side streams i.e. light and heavy vacuum gas oils. Due to this, the proportion of heavy polycyclic aromatic compounds (HAH) increases.

The high oil content in the raw material provides bitumen with good frost resistance [11]. Thus, the sample obtained by oxidation of the residue above 380 °C has the highest low-temperature properties (brittleness temperature is minus 26°C). At the same time, the sample has low thermal-oxidative stability, as evidenced by tests after heating in a thin film at a temperature of 163°C in the presence of atmospheric oxygen according to the EN 12607-1 (RTFOT) method.

The heavier the residue, the higher its content of resins and asphaltenes. With increasing sampling depth, density, viscosity, sulfur content and the value of the coking indicator also increase.



Indicator values for residues boiling above
LAH – light aromatic hydrocarbons; MAH – medium aromatic hydrocarbons; HAH – heavy aromatic hydrocarbons

Figure 1 – **The nature of changes in the content of individual groups of aromatic hydrocarbons in the distillation residues of Kalamkas oil at different sampling depths**

The results of the study of the physico-chemical characteristics of tars (the residues of vacuum distillation of Kalamkas oil fuel oil with different depths of oil fractions selection) are presented in *Table 2*. To compare and contrast the obtained qualitative characteristics of experimental bitumens, the same table presents the requirements of ST RK 1373 - 2013.

Table 2 – **Main technical characteristics of oxidized residues obtained during vacuum distillation of Kalamkas oil fuel oil**

Indicators. Measurement.	ST RK 1373 - 2013		Actual values of indicators for residues boiling above. °C:		
	BND 70/100	BND 100/130	380	400	420
Needle penetration depth, 0.1mm at 25 °C at 0 °C	71-100 not lower than 22	101-130 not lower than 30	84 42	75 33	63 22
Softening temperature, °C, not lower	45	43	50,7	51,4	51,2
Ductility, cm, at 25 °C at 0 °C	not less than 75 3,8	not less than 90 4,0	more than 100 4,7	more than 100 3,8	more than 100 3,5
Brittleness temperature, °C	not above -20	not above -22	-24,8	-22,3	-18,7
Penetration index	from - 1,0 to + 1,0		-0,3*	-0,3*	-0,3*
After heating at 163°C for 5 hours					
Weight loss. % wt.	no more than 0,6	no more than 0,8	0,38	0,24	0,09
Change in softening temperature, °C	no more than 7	no more than 8	4,0	4,1	4,5
Penetration at 25 °C, 0.1 mm % of original value	not less than 60	not less than 50	70,0	72,7	74,3

** The penetration index is determined according to ST RK 1373 - 2013. clause 8.2.2: Note.*

A negative value of the penetration index indicates that the resulting bitumen as a colloidal system has a “sol” structure and is a stabilized suspension of asphaltenes in a dispersion medium highly structured with resins.

During the oxidation of oil residues, many sequential-parallel reactions simultaneously occur: condensation reactions, in which the size of the molecules increases; reactions in which the sizes of molecules change little or do not change; destruction reactions in which the size of molecules decreases; concentration with distillation of light oil fractions [12].

The group chemical compositions of road bitumen of the same degree of oxidation, obtained by oxidation of Kalamkas oil residues boiling above 380, 400 and 420 °C, are presented in *Table 3*.

Table 3 – **Group chemical compositions of road bitumens obtained by oxidation of Kalamkas oil residues**

Residue	Content of individual hydrocarbon groups, % wt.						Ratio	
	PNH	LAH	MAH	HAH	Total resins (R)	Asphaltenes (A)	A/R	AH/PN
above 380 °C	24,7	7,6	9,4	24,4	20,2	13,7	0,68	1,68
above 400 °C	22,5	7,0	8,8	24,5	22, 8	14,4	0,63	1,79
above 420 °C	19,6	6,0	7,6	25,8	25, 7	15,3	0,60	2,01

From the presented experimental material it follows that oxidized bitumen contains a slightly smaller amount of paraffin-naphthenic hydrocarbons in comparison with the original residues. Since compounds of this type are oxidized extremely slightly under the given conditions, it can be assumed that the main part of them, the most volatile, is removed from the reaction mass in the form of vapors along with oxidation gases without any special destructive transformations.

The content of aromatic compounds in bitumen is also less than in the residue from which it is obtained. However, in this case, the reason for the decrease in the content of aromatic compounds is that a certain part of them enters into chemical reactions and is spent on transformations into heavier compounds i.e. polycyclic aromatic compounds with a higher molecular weight, viscosity and a higher boiling point. These compounds include resins. In turn, the reactive resins are compacted into asphaltenes.

The process of “compaction” of residues is caused by an increasing loss of hydrogen during oxidation, which, in combination with cyclization reactions, leads to the formation of high molecular weight products characterized by a high degree of aromaticity - asphaltenes and resins.

As a result of comparing the requirements of the current standards for road bitumen and the actual quality indicators of experimental laboratory samples of bitumen produced from residues of different sampling depths obtained during the distillation of Kalamkas oil, the following conclusions can be drawn:

- Oxidized bitumen obtained from vacuum residues of Kalamkas oil, boiling above 380 and 400 °C correspond to petroleum road bitumen grade BND 70/100 according to ST RK 1373 - 2013; the residue that boils above 420 °C does not meet the requirements for petroleum road bitumen of the grade in terms of penetration at 25 °C.
- With increasing depth of selection of distillate fractions in the residue, the plasticity of bitumen obtained from it decreases;
- The most plastic bitumen with the best low-temperature characteristics is obtained by oxidation of the lightest residue, boiling above 380°C.

The patterns found are in good agreement with the results of studies of the group chemical composition of bitumen and the residues from which they were obtained.

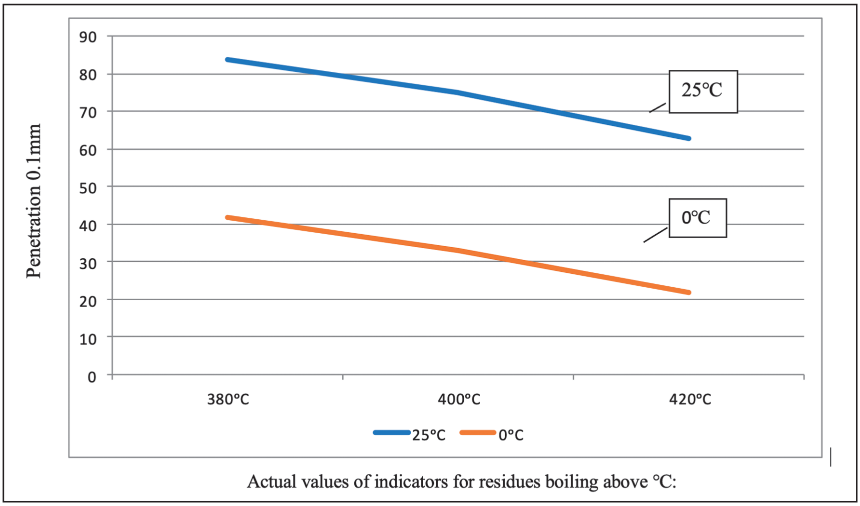


Figure 2 – **Change in penetration of road bitumen obtained from Kalamkas oil residues of different sampling depths**

The decrease in the plasticity of bitumen as the depth of selection of gas oil fractions during oil distillation increases, the boiling point of the residue, is explained by a decrease in the content of both bitumen and the residue of paraffin-naphthenic and aromatic hydrocarbons, which have a plasticizing effect on bitumen. It is for this reason that bitumen from the residue that boils above 380 °C is the most plastic.

The good low-temperature characteristics of bitumens obtained from residues of low sampling depths are explained by the increased content of paraffin-naphthenic compounds in the composition of these bitumens.

From *Table 3* it follows that the highest content of paraffin-naphthenic and aromatic hydrocarbons falls precisely on the portion of the residue that boils above 380 °C. Consequently, the distillation residue of Kalamkas oil of precisely this depth of selection has the most balanced ratio of the main groups of hydrocarbons in its composition.

Due to this, the quality of bitumen obtained by direct oxidation of such residue is the highest in comparison with bitumen obtained from heavier residues.

Conclusions. The heavy, highly resinous oil of the Kalamkas field is most suitable for the production of oxidized and residual bitumen from its residues. Based on experimental data, we can conclude that road grade bitumens produced from the residues of vacuum distillation of Kalamkas oil fuel oil using direct oxidation technology of raw materials have the best performance characteristics, provided they are obtained from residues boiling in the range of 380–400 °C.

Residues boiling above 380 and 400 °C can serve as raw materials for the production of oxidized road bitumens of the BND 70/100 grade.

Bitumen with a structure close to a sol, grades BND, have very high rates of deformability, heat resistance, crack resistance and aging resistance. It is advisable to use such bitumen in asphalt concrete in the IV and V road climatic zones of Kazakhstan.

Thus, the possibility of obtaining high-quality oxidized road bitumen from the residues of vacuum distillation of heavy oil from the Kalamkas field has been experimentally confirmed. The use of bitumen of this quality level makes it possible to achieve high performance characteristics of asphalt concrete. It will help to increase the maintenance-free service life of road surfaces not only in normal conditions, but also in conditions of intense traffic loads on the roadway.

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