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D.E. Aitbekova[1*], D.K. Makenov[1], E.I. Andreikov[2], A.G. Tsaur[3], Feng Yung Ma[4],
G.G. Baikenova[5, 6], A. Tusipkhan[1], A.A. Muratbekova[1], M.I. Baikenov[1]
[1]Karagandy University of the name of academician E.A. Buketov, Kazakhstan;
[2]Postovsky Institute of Organic Synthesis UB RAS, Ekaterinburg, Russia;
[3]Eastern Research Institute of Coal Chemistry, Ekaterinburg, Russia;
[4]Xinjiang University, Xinjiang Uyghur Autonomous Region, People's Republic of China;
[5]Karaganda Economic University of Kazpotrebsoyuz, Karaganda, Kazakhstan;
[6]South Ural State University, Chelyabinsk, Russia
*(*Corresponding author's e-mail: darzhan91@mail.ru)*

Hydrogen distribution in primary coke oven tar and its fractions

The aim of this work is to determine the hydrogen distribution in primary coke oven tar and its fractions. The hydrogen distribution in the primary coke oven tar of «ShubarkolKomir» JSC, its distillate fractions and distillation residue have been determined by the methods of elemental analysis, IR and PMR spectroscopy. The atomic ratio of C: H in the primary coke oven tar is 0.79. All fractions of the tar contain a large amount of alkyl-substituted aromatic compounds, phenols and other substances with alkyl groups. The initial tar characterized by a high content of hydrogen in the α- and β-positions to the aromatic ring, 29 % and 34 % respectively, which indicates a large number of alkyl substituents in the aromatic rings and near double bonds. The total amount of aliphatic and aromatic hydrogen in the tar is 79 % and 21 % respectively. Olefinic hydrogen is presented in the initial tar in an amount of 8 %. It is possible to make a choice of techniques for further processing (hydrogenation, coking, thermal cracking) to obtain products with high added value on the basis of determination of the elemental composition, quantitative distribution of hydrogen in the primary coke oven tar and its fractions by the using of above mentioned physical and chemical methods.

Keywords: primary coke oven tar, IR spectroscopy, PMR spectroscopy, elemental analysis, tar fractions, hydrogen distribution.

Introduction

Recently a decrease in reserves of low-viscosity, so-called «light» oils is observed. This requires the use of unconventional sources of hydrocarbon raw materials, new for oil refining, such as heavy oils and petroleum bitumens [1]. In addition, other types of alternative raw materials for oil refining and petrochemistry are coal products, which are not inferior in characteristics to their counterparts isolated from oil, and some have no analogues. This coal product is coke oven tar. Coke oven tars are obtained by the thermal processing of coal, which contain a significant amount of various organic compounds (hydrocarbons of all types and classes, heteroatomic, polyaromatic hydrocarbons, phenols and others). They can be used in oil refining, petrochemistry, ferrous and nonferrous metallurgy, nuclear industry. Coke-chemical tar is divided into two types: primary coke oven tar (PCOT) and high temperature coal tar [2–4]. It should be noted that high-temperature coke oven tar is used in industry, but primary coke oven tar doesn't find industrial application abroad and in our country, due to the high content of phenols (35–45 %) [5] and unstable low-boiling and resinous components [6]. The PCOT manufacturer in the Republic of Kazakhstan is «ShubarkolKomir» JSC [7]. All their products are sold in the near and far abroad as well as the entire produced volume of PCOT, which does not stimulate the development of innovative processes for obtaining multipurpose valuable low molecular weight hydrocarbons. Coke-chemical tar, consisting mainly of condensed aromatic hydrocarbons and other high-molecular compounds, refers to a difficult-to-process raw material. Knowledge of the primary coke oven tar chemical composition and its fractions allows to determine the choice of methods for further processing (hydrogenation, coking, thermal cracking) to obtain products with high added value.

The aim of this work is to determine the hydrogen distribution in the initial tar and its fractions by the methods of elemental analysis, IR and PMR spectroscopy. The quantitative distribution of hydrogen in the initial coke oven tar and its fractions of «ShubarkolKomir» JSC was established using physicochemical methods for the first time.

Experimental

Physicochemical characteristics of the tar are determined according to the indicators listed in [8]. In addition, the fractional composition has been determined by the method described in [9, 10].

Due to the close values of the density of tar and water, dehydration of tar by sedimentation is practically impossible, which complicates the process of tar fractionation. Toluene was added in an amount of 10 % of the coal tar in order to separate water from the tar before distillation. After distillation of the azeotropic mixture of toluene with water, fractionation was carried out, and the temperature was measured both in the vapor and in the liquid phase. Fractionation has been stopped when the temperature in the liquid reached 390–405 ºC, in order to avoid significant changes in the primary tar due to thermal reactions in the boiler. The fractions were selected as in the distillation of high-temperature coking tar. Fractions were selected in accordance with the range of their boiling points: up to 180 ºC; 180–210 ºC; 210–230 ºC; 230–270 ºC; above 270 ºC and up to the end of distillation. A comparative analysis with the high-temperature coking tar of «ArcelorMittal Temirtau» JSC was carried out.

Determination of the carbon, and nitrogen content in the solid residue of pyrolysis has been carried out by «CHN PE 2400-II» an automatic analyzer of «PerkinElmerInstruments». The sulfur content has been determined by the method of barium titration.

IR spectra of pyrolysis solid residues were obtained by FTIR diffuse reflection spectroscopy on a spectrometer with a Fourier transducer «Perkin-ElmerSpectrumBX-II». The samples for the spectral recording were prepared by applying a thin layer of a carefully ground sample on a special holder plate. IR transmission spectra of liquid products were obtained by taking of layer.

The PMR spectra of the fractions obtained by distillation of the semi-coking tar were recorded by a Broker DRX 400 spectrometer with TMS, as an internal standard in a $CDCl_3$ solution, at room temperature.

The hydrogen distribution in the samples was determined by the PMR spectra according to the following signal assignments for chemical shifts:

0–1.0 ppm — corresponds to the protons of methyl groups that are not bound with aromatic rings or are in the γ-position to them, further (H_γ);

1.0–2.0 ppm — corresponds to the protons of methyl, methylene and methine groups located in β-position with regard to the aromatic ring and double bond, as well as methylene groups in saturated structures (H_β);

2.0–4.4 ppm — corresponds to the protons in all alkyl groups in the α-positions of aromatic rings and double bonds (H_α);

4.5–6.7 ppm — corresponds to the protons of olefinic structures (H_{olef});

6.7–9.0 ppm — corresponds to the protons of aromatic groups (H_{ar}).

Results and Discussion

Tar main characteristics

Provided by manufacturer two samples of tar with 12.5 % and 2.2 % of water content were studied (Table 1).

The main characteristics of «ShubarkolKomir» JSC tar in comparison with the high-temperature coking tar are shown in Table 2.

In comparison with the high-temperature coking tar, the studied tar is characterized by: low density (1.045–1.062 g/cm^3); low content of insoluble substances in toluene and the absence of insoluble substances in quinoline; a reduced yield of distillation residue (pitch); a high content of phenols and a low content of naphthalene.

Table 1

Technical analysis of «ShubarkolKomir» JSC tar samples

Description	1st sample March 2019	2nd sample April 2019
Tar density at 20 ºC, g/cm^3	1.039	1.061
Tar density in terms of anhydrous at 20 ºC, g/cm^3	1.045	1.062
Water content, %	12.5	2.2
Mass fraction of substances insoluble in toluene, %	1.3	1.4
Mass fraction of substances insoluble in quinoline, %	–	–
Ash content, %	0.12	–

Table 2

Comparison of the characteristics of tar from «ShubarkolKomir» JSC and «ArcelorMittal Temirtau» JSC

No.	Indicator	Tar types		Determination method
		«ShubarkolKomir» JSC tar	«ArcelorMittal Temirtau» JSC high-temperature coke oven tar	
1	Density at 20ºC, g/cm^3	1.062	1.190	State Standard [11, 12]
2	Fractional composition, mass fraction (from anhydrous tar), %			Specification [9], [10]
	till 180 ºC	5.1	0.5	
	180–210 ºC	4.4	2.5	
	210–230 ºC	4.8	10	
	230–270 ºC	15.8	8	
	up to 270 ºC by vapor till 390 ºC in a still	19.7	19	
	up to 270 ºC by vapor till 405ºC in a still	26.1		
	Distillation residue (pitch) yield, %			
	at 390 ºC in a still	49.8		
	at 405 ºC in a still	40.0	60	
3	Mass fraction of substances insoluble in toluene (α-fraction), %	1.4	6–10	State Standard [13], specification [8]
4	Mass fraction of substances insoluble in quinoline (α1-fraction), %	–	4–6	State Standard [14], specification [8]
5	Ash content, %	0.12	<0.3	Specification [8]
6	Phenol content, %	20.6	1–2	Calculated by the content of phenols in distillate fractions up to 390 °C in a still
7	Naphthalene content, %	1.6	7–12	According to GLC/MS data of distillate fractions

Elemental analysis of fractions

Data on the density of fractions and the elemental composition of fractions and pitch are shown in Table 3.

From the data in Table 3 it follows:

- the carbon content reaches 80.6–82.5 % in the fractions, and 83–84 % in pitch;
- the hydrogen content is 9.1–9.8 % in the fractions and it decreases to 7.4–7.8 % in the pitches;
- the atomic ratio of C: H is at the level of 0.71–0.79 in the fractions and it increases to 0.90–0.94 in pitch;
- fractions and pitch contain more than 9–10 % of the total oxygen and sulfur, while the sulfur content is less than 0.3 %, the oxygen content declines considerably in the distillation residues.

Table 3

Density of fractions and results of elemental analysis

Name product	Density, g/cm^3	Content, % mass.				C/H atom
		C	H	N	S+O	
Dehydrated tar	1.039	81.45	8.57	0.69	9.29	0.79
180–210 °C fraction	0.954	80.60	9.11	1.07	10.29	0.74
210–230 °C fraction	0.965	80.64	9.41	1.06	8.89	0.71
230–270 °C fraction	0.970	80.89	9.23	0.93	8.95	0.73
up to 270 °C fraction by vapor till 405 °C in a still	 0.993	82.46 83.39	8.73 9.78	0.91 1.29	7.98	0.79
up to 390 °C distillation residue in a still	–	83.11	7.40	0.87	8.62	0.94
up to 405 °C distillation residue in a still	–	84.10	7.78	0.68	7.44	0.90

Description of IR spectra

Figure 1 shows the IR spectrum of the initial tar.

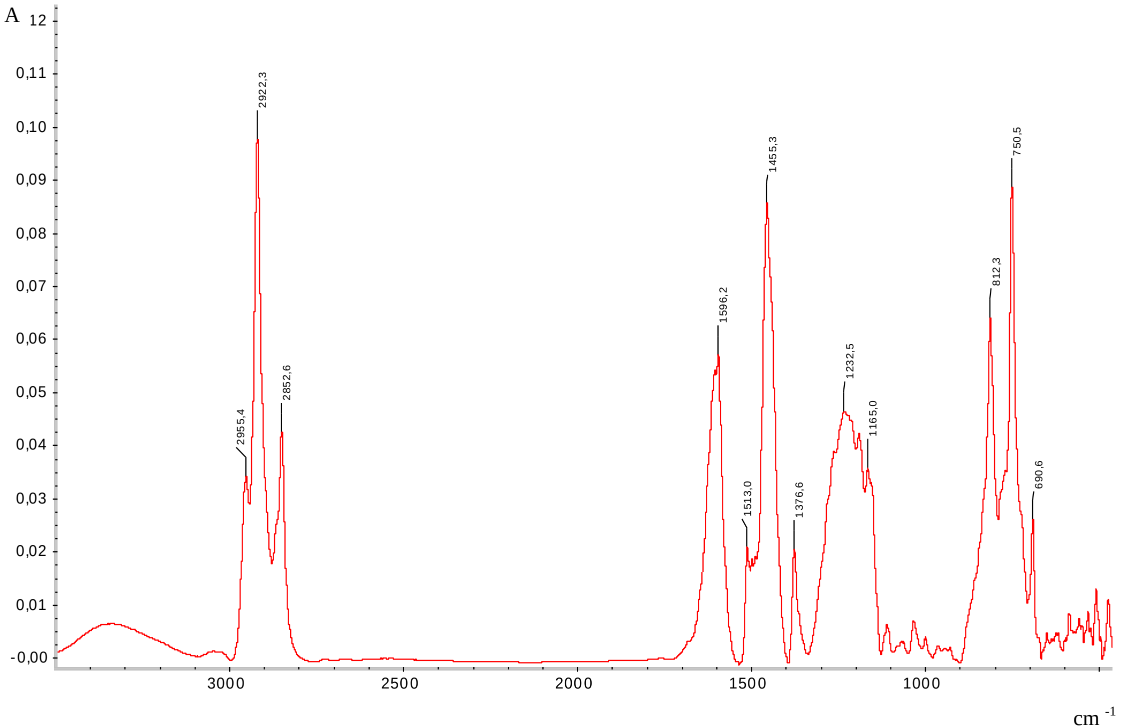

Figure 1. IR spectrum of dehydrated tar

The general view of the spectra for fractions and residues of tar distillations is similar, which indicates the presence of identical organic functional groups in all samples.

The most important information is the absorption in the region of 3.040–2.800 cm^{-1}. Strong absorption below 3.000 cm^{-1} (C-H bonds of methyl and methylene groups) indicates a significant amount of alkyl groups. In addition, the vibrations of $-CH_3$ and $-CH_2$ groups correspond to absorption bands at about 1,450 and 1,375 cm^{-1}. At the same time, the absorption of aromatic hydrogen at 3.020 cm^{-1} is weak.

The second feature is a wide band with a maximum at 3.380–3.370 cm^{-1} which is related to phenolic hydroxyls.

The bands in the region of 900–700 cm^{-1} correspond to the vibrations of the aromatic ring c-h bonds. The most pronounced bands are at 815 cm^{-1} (1,4-substituted aromatic ring) and a peak at about 750 cm^{-1} (1,2-substituted aromatic ring). Strong absorption around 1,600 cm^{-1} belongs to the aromatic ring. The bands in the range of 1,280–1,050 cm^{-1} refer to the Ar-O group in phenols.

The following small changes in the IR spectra for the coal tar and pitch fractions can be noted: the fractions (up to 230 °C and 230–270 °C) are characterized by stronger absorption at 1,514 cm^{-1} (vibration of the benzene ring) and in the region of 900–700 cm^{-1}, which indicates a higher content of compounds with one benzene ring.

Description of PMR spectra

Figures 2, *a-c* show the PMR spectra of the initial tar, distillate fraction 230–270 ºC and distillation residue. Quantitative data on the distribution of hydrogen types according to the PMR data in tar, distillate fractions and distillation residues are given in Table 4.

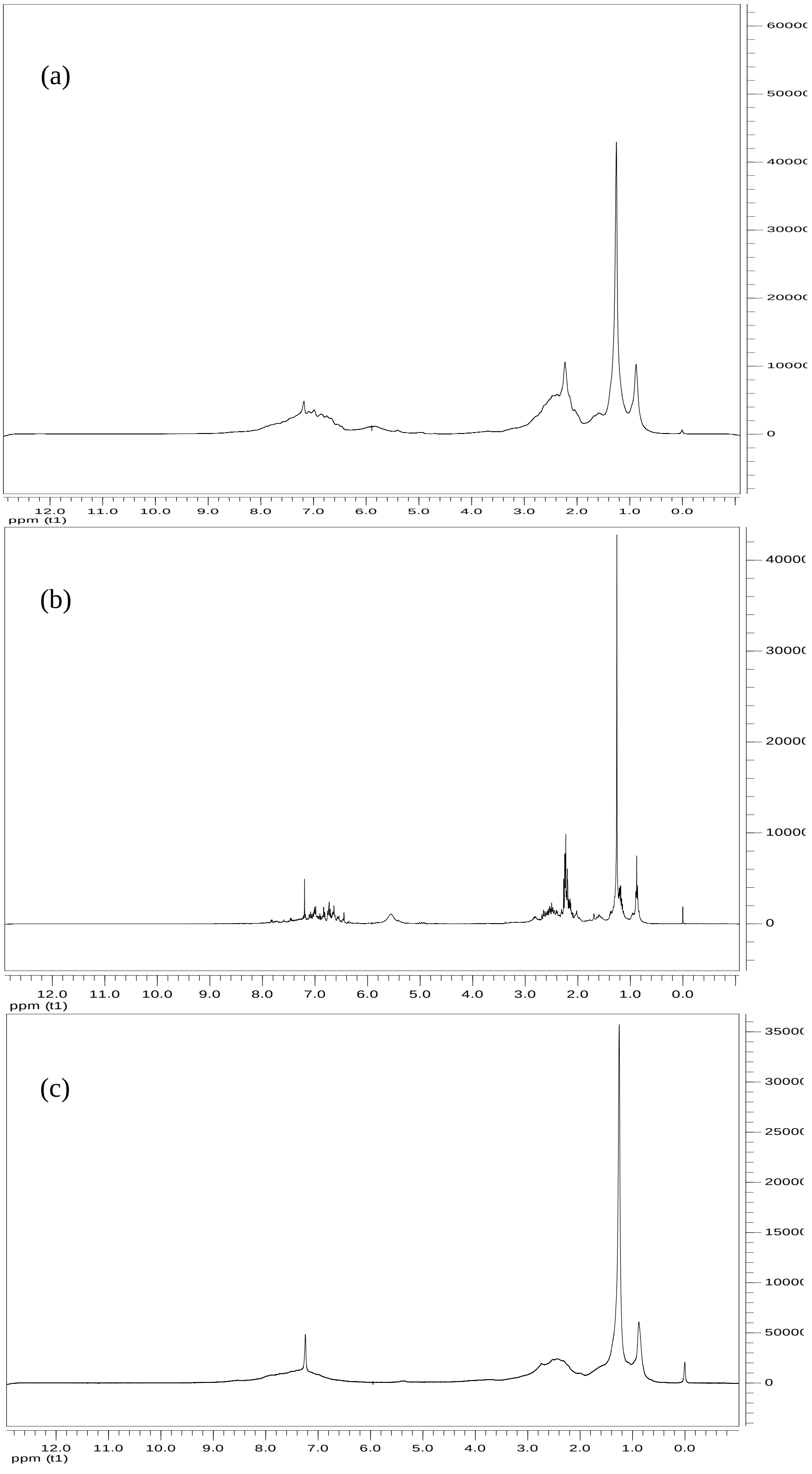

Figure 2. PMR spectra of dehydrated tar (*a*), 230–270 ºC distillate fraction (*b*) and up to 405 ºC distillation residue

Table 4

Distribution of hydrogen types in tar, distillate fractions and distillation residues

Sample	Proton distribution, %				
	H_{γ}	H_{β}	H_{α}	H_{olef}	H_{ar}
Tar	8.42	33.84	29.38	7.74	20.62
180–210 ºC fraction	8.57	28.79	28.36	10.03	24.25
210–230 ºC fraction	8.95	32.59	29.36	10.29	18.81
230–270 ºC fraction	8.55	32.65	31.28	10.51	17.01
up to 270 ºC by vapor and till 405 °C in a still	8.46	39.93	28.54	7.70	15.40
up to 390 ºC distillation residue in a still	11.66	40.56	26.57	–	21.21
up to 405 ºC distillation residue in a still	9.79	43.29	25.17	–	21.75

All fractions and distillation residues are characterized by high content of hydrogen at the α- and β-positions to the aromatic ring. The number of alkyl groups with more than three carbon atoms in the chain is small, as $H_{\gamma}<H_{\alpha}$.

The total amount of aliphatic hydrogen is several times higher than that of aromatic hydrogen. The 180–210 ºC fraction contains the largest amount of aromatic hydrogen.

Olefinic hydrogen (unsaturated compounds) is present in the initial primary coke oven tar and distillate fractions, its relative amount decreases for the heaviest fraction, and olefinic hydrogen is not found in the distillation residues.

Conclusions

Thus the results of IR spectral studies show that all fractions of the coke oven tar and the pitch obtained from it contain a large amount of alkyl-substituted aromatic compounds, phenols and other substances with alkyl groups. Aromatic compounds generally contain substituents at the ortho- and para- positions. The IR spectrum of the pitch does not fundamentally differ from the IR spectra of the fractions, i.e. it also contains hydrogen mainly in alkyl groups and contains phenolic groups.

According to the results of the elemental analysis the atomic ratio of C: H in the primary coke oven tar is 0.79.

Primary coke oven tar has a high content of hydrogen in the α- and β-positions to the aromatic ring, 29 % and 34 % respectively, which indicates a large number of alkyl substituents in the aromatic rings and near double bonds. The total amount of aliphatic hydrogen is 79 %. It is several times more than the amount of aromatic hydrogen, which is 21 %. The high content of alkyl substituents in aromatic rings allows using 180–230 °C distillate fractions of the tar to increase the octane and cetane number in the production of motor fuel.

Fractions with a boiling point above 270 ºC can be used to obtain boiler fuel, carbon black, coke and needle coke.

The products manufactured by oil refinery or coal chemical enterprises can be diversified through knowledge of the hydrogen distribution and the use of the compounding method.

Thus the results obtained in this study can be the basis for choosing the most preferable direction for further processing of raw materials.

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Д.Е. Айтбекова, Д.К. Макенов, Е.И. Андрейков, А.Г. Цаур, Фен Юн Ма,
Г.Г. Байкенова, А. Түсіпхан, А.А. Муратбекова, М.И. Байкенов

Біріншілік таскөмір шайыры мен оның фракцияларында сутегінің таралуы

Элементтік талдау, ИҚ- және ПМР-спектроскопия әдістерімен «Шұбаркөл Көмір» АҚ бастапқы таскөмір шайырында, оның дистилляттық фракцияларында және дистилляция қалдығында сутектің таралуы анықталды. Біріншілік көмір шайырындағы C : H атомдық қатынасы 0.79 құрайды. Шайырдың барлық фракцияларында алкилорынбасылған ароматты қосылыстар, фенолдар және алкил топтары бар басқа қосылыстар көп. Бастапқы шайыр ароматты сақинаға α- және β-жағдайларда орналасқан сутектің жоғары құрамымен сипатталады, сәйкесінше 29 % және 34 %, бұл ароматты сақиналарда және қос байланыстардың жанында алкилді орынбасушылардың көп мөлшерін көрсетеді. Біріншілік таскөмір шайырындағы алифатты және ароматты сутектің жалпы мөлшері сәйкесінше 79 % және 21 % құрайды. Біріншілік шайырда олефинді сутегі 8 % құрайды. Жоғарыда аталған физика-химиялық әдістердің көмегімен біріншілік таскөмір шайырындағы және оның фракцияларындағы сутектің элементтік құрамын, сандық таралуын анықтау жоғары құнды өнімдерді алу үшін одан әрі қайта өңдеу (гидрогенизация, кокстеу, термиялық крекинг) әдістерін таңдауды айқындауға мүмкіндік береді.

Кілт сөздер: біріншілік таскөмір шайыры, ИҚ-спектроскопия, ПМР-спектроскопия, элементтік талдау, шайыр фракциялары, сутегінің таралуы.

Д.Е. Айтбекова, Д.К. Макенов, Е.И. Андрейков, А.Г. Цаур, Фен Юн Ма,
Г.Г. Байкенова, А. Түсипхан, А.А. Муратбекова, М.И. Байкенов

Распределение водорода в первичной каменноугольной смоле и ее фракциях

Методами элементного анализа, ИК- и ПМР-спектроскопии установлено распределение водорода в первичной каменноугольной смоле АО «Шубарколь Комир», ее дистиллятных фракциях и неперегоняемом остатке дистилляции. Атомное соотношение C : H в первичной каменноугольной смоле составляет 0,79. Все фракции смолы содержат большое количество алкилзамещенных ароматических соединений, фенолов и других соединений с алкильными группировками. Исходная смола характеризуется высоким содержанием водорода, находящегося в α- и β-положениях к ароматическому кольцу, 29 и 34 % соответственно, что свидетельствует о большом количестве алкильных заместителей в ароматических кольцах и рядом с двойными связями. Общее количество алифатического и ароматического водорода в смоле составляет 79 и 21 % соответственно. В исходной смоле олефиновый водород присутствует в количестве 8 %. Установление элементного состава, количественного распределения водорода в первичной каменноугольной смоле и ее фракциях с помощью названных выше физико-химических методов позволяет определиться в выборе методов дальнейшей переработки (гидрогенизация, коксование, термический крекинг) для получения продуктов, имеющих высокую добавленную стоимость.

Ключевые слова: первичная каменноугольная смола, ИК-спектроскопия, ПМР-спектроскопия, элементный анализ, фракции смолы, распределение водорода.

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Information about authors

Aitbekova, Darzhan Ergalievna (*corresponding author*) — doctoral student, Karagandy University of the name of academician E.A. Buketov, Universitetskaya street, 28, 100028, Karaganda, Kazakhstan; e-mail: darzhan91@mail.ru; https://orcid.org/0000-0002-6839-9711

Makenov, Daulet Kairzhanovich — doctoral student, Karagandy University of the name of academician E.A. Buketov, Universitetskaya street, 28, 100028, Karaganda, Kazakhstan; e-mail: mckenov.daulet@gmail.com; https://orcid.org/0000-0002-1011-7662

Andreikov, Eugeny Iosifovich — doctor of chemical sciences, Postovsky Institute of organic synthesis UB RAS, Sofia Kovalevskaya, 22, 620108, Ekaterinburg, Russia; e-mail: cc@ios.uran.ru; https://orcid.org/0000-0002-9285-9036

Tsaur, Anatoly Grigorevich — senior researcher, JSC «Eastern research Institute of coal chemistry», 8-Marta, 14, 620990, Ekaterinburg, Russia; e-mail: tsaur@rosfirm.ru

Ma, Feng Yung — PhD doctor, professor, Xinjiang University, Urumqi, People's Republic of China, ma_fy@126.com

Baikenova, Gulzhan Causilevna — doctor of chemical sciences, professor, Karaganda Economic University of Kazpotrebsoyuz, Karaganda, Kazakhstan; South Ural State University, Chelyabinsk, Russia; e-mail: murzabek_b@mail.ru; https://orcid.org/0000-0002-2816-3341

Tusipkhan, Almas — PhD doctor, senior lecturer, Karagandy University of the name of academician E.A. Buketov, Universitetskaya street, 28, 100028, Karaganda, Kazakhstan; e-mail: almas_kz_22@mail.ru; https://orcid.org/0000-0002-6452-4925

Muratbekova, Aigul Akizhanovna — candidate of chemistry sciences, docent, Karagandy University of the name of academician E.A. Buketov, Universitetskaya street, 28, 100028, Karaganda, Kazakhstan; e-mail: aigulmuratbekova@mail.ru; https://orcid.org/0000-0002-2156-9306

Baikenov, Murzabek Ispolovich — doctor of chemical sciences, professor, Karagandy University of the name of academician E.A. Buketov, Universitetskaya street, 28, 100028, Karaganda, Kazakhstan; e-mail: murzabek_b@mail.ru; https://orcid.org/0000-0002-8703-0397