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**IR ANALYSIS OF THE LIQUID PRODUCT OBTAINED
BY EXTRACTION PROCESSING COAL****S. D. Fazylov¹, Zh. B. Satpayeva¹, O. A. Nurkenov¹, A. B. Tateyeva²,
G. K. Karipova¹, D. S. Issabekova¹, Zh. S. Akhmetkarimova¹**¹Institute of Organic Synthesis and Coal Chemistry of the Republic of Kazakhstan,²Academician E. A. Buketov Karaganda State University, Karaganda, Kazakhstan

E-mail: iosu8990@mail.ru, satpaeva_zh@mail.ru, nurkenov_oral@mail.ru

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Abstract. The article describes the features of the composition, chemical and physico-chemical properties of some of the Karaganda coal basin and Maikuben coal as a raw material for deep technical-chemical processing. The extraction of coal of different brands benzene, benzene-alcohol solvent. Studies have shown that of all the samples studied carbon material, coal Maikuben and Kuu-Chek cuts very well undergo a process of extraction of bitumen materials with organic solvents. It is found that the extraction of coal studied most complete extraction of bitumen substances observed when using solvent system "alcohol-benzene". The analysis of the IR spectra obtained extracts of the studied coals. According to the spectral data bitumen of the alcohol-benzene bitumens enriched oxygen functional compounds, as evidenced by the presence of characteristic absorption bands of the carbon skeleton of aromatic and heterocyclic compounds in the respective spectral regions. Extraction with benzene leads to higher content of aromatic hydrocarbons obtained bitumens are mixtures of aliphatic and aromatic substances, said second overwhelmingly predominant. It is shown that in the long term, the correct choice of process conditions, they may be of interest as promising sources of bitumen products.

Coal is a valuable chemical-technological material with great chemical potential. Known processes for coal processing based on various destructive reactions that provide the product yield, but it loses much of valuable chemical compounds. Today many modern technologies for processing coal unprofitable and unable to compete with petrochemical manufacturers. Feature of the implementation of a new coal policy of Kazakhstan is the conversion of the coal industry of raw materials and energy in the deep processing of coal energy. Widespread coal in Kazakhstan, the high value of their organic part leads to conduct comprehensive scientific research to develop effective technologies for processing different coals. This policy is designed to ensure improvement of the competitiveness of coal production [1]. One of the most promising areas is the use of coal processing into liquid products, lignite waxes and sorption materials for different purposes [2-6].

One of the effective methods of processing coal is extraction with organic solvents. The extraction process is the dissolution of low molecular weight components (bitumen) located in the pores of the carbon material in the organic solvents and introducing in their place of the solvent molecules, i.e. the destruction of the supramolecular structure of coal [7, 8]. The quantity and composition of the solution passing into bitumen can vary within wide limits depending on the nature of the starting coal, the type of solvent and the conditions of preconditioning raw materials.

Important influence on the yield of bitumen is the nature of the solvent. To date, it investigated about 40 carbons extraction solvents. But summarizing the results obtained not only concluded that the yield of the extract in some cases increases with the boiling point of the solvent, probably due to thermal decomposition of coal bonds in the structure [7]. The authors of [9] give the following approximate ratios. If we take the activity of benzene extraction is unit, the petroleum ether - 0.42, gasoline - 0.83,

dichloroethane - 0.98, alcohol-benzene - 1.37. Rakovski [10] accentuate, depending on the ratio of the content in bitumen wax and resin amount is output in the bitumen extraction of peat from different solvents varies greatly. He points out that the poor solvent of bitumen is boiling hydrocarbons and fatty, such as petroleum ether. Bitumen better extracted aromatic hydrocarbons, such as benzene. Compared to gasoline and it is more soluble resin and wax. Ethyl alcohol is a good solvent of the resin bitumen and wax weaker. Therefore, the best bitumen solvents are mixed solvents such as alcohol-benzene[11, 12].

Bitumen fossil fuels can be divided into the wax and resin parts, which differ in their chemical composition and purpose of their use. Coal extracts are also of interest for structural studies.

Purpose is studying the extraction and the yield of liquid products from coal soluble by various organic solvents.

Experimental part

Research samples are subjected to some of the coal from the Karaganda coal basin and Maikuben: unenriched coal class mark coke oven section, "Kuu-Chekinsk"; unenriched coal gas fatty grade class section "Saryadyr"; grade coals coke fatty class mine "Kazakhstan" Shakhty cut and brown coals brand Maikuben coal basin.

In order to simplify the method in experiments with brown coal outputs extractable is measured not by a decrease in the organic mass of coal (WMD) of the sample, and by weight of substances that have fallen into the extracts. To the flask at reflux was placed 5 g of charcoal, pulverized and passed under a 0,2 mm sieve. Then add to 50 ml solvent (benzene, ethanol-benzene 1:1 2-propanol-benzene 1:1). The flask contents were refluxed for 4 hours on a magnetic stirrer at the boiling point of the solvent. Further, carbon residue from the extract filtered. The extract was subjected to evaporation and dried to constant weight and weighed. Thereby, the output value of the extract.

Infrared spectra sample coal and bituminous substances removed the instrument FT-IR spectrometer NIKOLET AVATAR-320 Fourier transformer in tablets with KBr in the range of 4000-450 cm^{-1} .

Results and discussion

It is known that coal different in different field's humidity, quantity and composition of minerals, and other characteristics. These technical analyses of coal samples under study are shown in Table 1. Technical analysis of coals on indicators made in accordance with GOST 11022-95 and GOST 27314-91.

Table 1 – Technical characteristics of samples investigated coals wt. %

| Coal | A^d | W_t | V^{daf} | Calorific value Q^f_{is} , kJ/kg | S (total) |
|-----------------------------------|-------|-------|-----------|---------------------------------------|--------------|
| Brand Coke cut "Kuu-Chekinsk" | 37,53 | 2,57 | 26,33 | 19854 (4742) | 0,38 |
| Brand gas fat cut "Saryadyr" | 35,47 | 5,11 | 34,01 | 18924 (4520) | 0,59 |
| Brand coke fatty cut "Kazakhstan" | 26,31 | 5,56 | 29,9 | 8314 (5562) | 0,68 |
| Brandbrown cut "Maikuben" | 16,8 | 7,3 | 48 | 7160 (5240) | 0,82 |

As seen from Table 1, the studied coals differ significantly in their characteristics. Sample Kuu-Chekinsk grade coal Coke class has high ash content (A_d) 37,53% and humidity (W_a) 2,57%. Coal gas fat class brand Saryadyr cut different volatile components (V^{daf}) 34,01%. Samples "Saryadyr" and "Kazakhstan" coal are close in their characteristics: moisture 5,11 and 5,56%, respectively. Maikuben coal differs it's sharply lower values of ash content (A_d) 16,8%, reflecting the benefit of higher organic mass and the prospects for bituminous materials.

The use of mechanical processing of coal is one of the stages of their preparation for processing. A mechanical activation substance occurs during intense dispersion of the processed material. There occurs

it dispersion and accumulation of activation energy. Mechanical activation can be considered as coal grinding, which increases the specific surface area by reducing the geometrical dimensions of the particles and opening previously inaccessible pore [13, 14]. It should also be borne in mind that under intense mechanical action on the coals along with dispersing their activation occurs, accompanied by significant structural changes WMD. Therefore, we carried out the preliminary mechanical activation of coal in the plate mill ML-1 to the size of 0,2-0,1 mm.

Extraction was performed bitumen such as benzene solvents, a mixture of ethanol and benzene (1:1) with 2-propanol and benzene (1:1). Originally the effect of organic solvents on the yield of resinous substances studied on the example of raw coal of mark "Coke" cut class "Kuu-Chekinsk." It was found that the best bituminous substances extracted coal, aromatic hydrocarbons (e.g., benzene, toluene), and the use of low-boiling hydrocarbons and fatty (petroleum ether, etc.) are incomplete extraction of coal tar substances.

Out extract bituminous substances increases with the boiling point of the extracting, possibly due to thermally cleavage of the carbon in the structure [15-20]. Benzene, compared with gasoline, and better dissolves the resin and wax. Ethyl alcohol is a good solvent of the resin bitumen, wax and bad. Good extraction properties are toluene and xylene, though they are high-boiling solvents (b.p.>1000C). Therefore, the most the best solvents for the extraction of bitumen were mixed solvents, such as benzene, ethanol, benzene-2-propanol (among the studied solvents). The most optimal ratio of these solvents is: 2:1 and 1:1. However, according to the results on the degree of extractability bituminous substances also affect the origin of the carbon material. For example, the solvent system "isopropyl alcohol, benzene" poorly extracted (for only 0,4%) of bituminous coal tar Maikuben incision and from coals Kuu- Chekinsk section extraction was maximal (2,4%). In this connection, in the study of bituminous materials bitumen is also necessary to examine the content of waxes, resins, acid number, the number of etheric, saponification number, iodine number, and the melting point. Sometimes it is necessary to additionally install an ash content, the carbon content and hydrogen content of waxes and oils in the composition derived wax materials i.e. depending on the type of the solvent varies not only the output of bitumen, but also for their properties.

Table 2 – Dependence of bituminous resin on the nature of the solvent

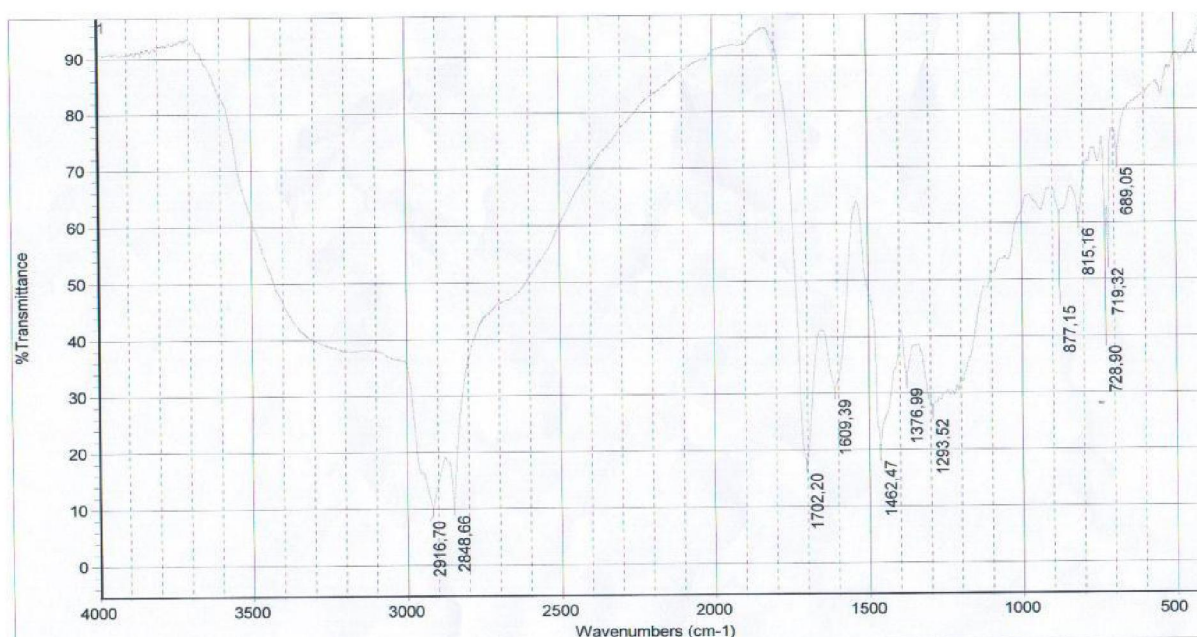
| Solvent | Coal brand coke cut "Kuu-Chekinsk" | Coal brand gas fat cut "Saryadyr" | Coal brand coke fatty cut "Kazakhstan" | Coal brand brown cut "Maikuben" |
|-------------------------|------------------------------------|-----------------------------------|--|---------------------------------|
| | Out extract, % | | | |
| Benzene | 1,0 | 0,2 | 0,2 | 1,0 |
| Ethanol-benzene 1: 1 | 0,4 | 0,2 | 0,2 | 2,4 |
| 2-propanol-benzene 1: 1 | 2,4 | 0,2 | 0,2 | 0,4 |

As can be seen from Table 2, the output of bitumen varies not only depending on the type of solvent and the nature of the coal. Resin components of bitumen produced from coal samples Maikuben concentrated mainly in alcohol-benzene extract.

The obtained IR spectrum of the benzene-alcohol extract is shown in Fig. The IR spectra of the benzene extracts bitumen's presence of strong absorption bands in the region 1290-1609 cm^{-1} indicate the nature of the aromatic components of the extract. According to the spectral data bitumen's enriched oxygen functional compounds, as evidenced by the presence of absorption bands of the carbon skeleton of aromatic and heterocyclic compounds in the 1340-1500 cm^{-1} . Which exhibits strong absorption bands associated with non-planar deformation vibrations of C-H deformation vibrations and cycle. Medium-intensive absorption band at about 1609 cm^{-1} may belong to both the aromatic structure and double C=C bond. Having the average intensity of the band at 1462 cm^{-1} can also confirm the presence of the benzene ring. The broad band at 3500-3600 cm^{-1} indicates the valence vibrations of the hydroxyl group.

The spectra are shown bifurcated bitumen coals band at 2916-2848 cm^{-1} , indicating the stretching vibrations of the aliphatic group (CH_2) and (CH_3). There is a common absorption band at 1702 cm^{-1} corresponding to oscillations group (C=O) in the ester group $\text{CH}_2\text{C}(\text{O})\text{OR}$. The presence of poorly soluble bands from 1050 to 1300 cm^{-1} shows different CO bond.

Alcohol-benzene extracts of other coal mines produce similar IR spectra.



IR ethanol-benzene extract Maikuben brown coal of class

By analyzing the spectral picture as a whole, it must be concluded that the spectrum is the range of bitumen mixture of aliphatic and aromatic substances, and the second is dominated overwhelmingly. IR spectra insoluble solid residues Maikuben coal (and others as well), after extraction, are given less information.

Conclusion. Thus, studies have shown that of all the samples studied carbon material, Maikuben and Kuu-Chekinsk coals cuts very well undergo a process of extraction of bituminous materials with organic solvents. To increase the yield of extractable coal material in terms of the experiments, it is necessary to examine the influence of various physical and chemical (e.g., wave) and chemical methods of influencing the structural degradation of the carbon material.

In the future, if properly selected, the process, they may be of interest as a promising source of bituminous products. Further studies Maikuben and Kuu-Chekinsk coals are of interest in obtaining practically important materials.

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ИК-АНАЛИЗ ЖИДКИХ ПРОДУКТОВ, ПОЛУЧЕННЫХ ПУТЕМ ЭКСТРАКЦИОННОЙ ПЕРЕРАБОТКИ УГЛЕЙ

С. Д. Фазылов¹, Ж. Б. Сатпаева¹, О. А. Нуркенов¹, А. Б. Татеева²,
Г. Ж. Карипова¹, Д. С. Исабекова¹, Ж. С. Ахметкаримова¹

¹Институт органического синтеза и углехимии Республики Казахстан,

²Карагандинский государственный университет им. Е. А. Букетова, Казахстан

Ключевые слова: уголь, битум, экстракция, свойства, воск.

Аннотация. В статье рассмотрены особенности состава, химических и физико-химических свойств некоторых углей Карагандинского и Майкубенского угольных бассейнов как сырья для глубокой химической переработки. Изучена экстракция углей разных марок бензольным, бензольно-спиртовыми растворителями. Проведенные исследования показали, что из всех изученных образцов угольного материала угли Майкубинского и Куу-Чекинского разрезов достаточно хорошо подвергаются процессу экстракции битуминозных веществ органическими растворителями. Установлено, что при экстракции изучаемых углей наиболее полное извлечение битуминозных веществ наблюдается при использовании системы растворителей «спирт-бензол». Проведен анализ ИК-спектров полученных экстрактивных веществ из изучаемых углей. Согласно спектральным данным спирто-бензольные битумоиды обогащены кислородсодержащими функциональными соединениями, что подтверждается наличием характеристических полос поглощения углеродного скелета для ароматических и гетероциклических соединений в соответствующих областях спектров. Экстракция бензолом приводит к повышению содержания ароматических углеводородов, полученные битумоиды представляют собой смеси алифатических и ароматических веществ, причем вторые преобладают в подавляющей степени. Показано, что в перспективе, при правильном выборе режима технологического процесса, они могут представлять интерес как источники перспективных битуминозных продуктов.

КӨМІРДІ ЭКСТРАКЦИЯЛЫҚ ҚАЙТАӨНДЕУ ЖОЛЫМЕН АЛЫНҒАН СҰЙЫҚ ӨНІМДЕРІНІҢ ИҚ-АНАЛИЗІ

С. Д. Фазылов¹, Ж. Б. Сәтбаева¹, О. А. Нұркенов¹, А. Б. Тәтеева²,
Г. Ж. Кәріпова¹, Д. С. Исабекова¹, Ж. С. Ахметкәрімова¹

¹Қазақстан Республикасының Органикалық синтез және көмірхимия институты,

²Е. А. Бөкетов атындағы Қарағанды мемлекеттік университеті

Тірек сөздер: көмір, битум, экстракция, қасиеттер, балауыз.

Аннотация. Мақалада терең техномиялық қайтаөндеу үшін шикізат болып табылатын Қарағанды мен Майкубен көмір бассейндерінің құрамының және физико-химиялық қасиеттерінің ерекшеліктері қарастырылған. Маркалары әр түрлі көмірдің бензолмен, спиртті-бензол еріткіштерімен экстракциясы зерттелген. Жүргізілген зерттеулер бойынша, барлық зерттелген көмір үлгілерінен Майкубен мен Кушоқы көмірлері, битуминозды заттарды органикалық еріткіштермен экстракциялау үрдісіне жақсы талдауға алынады. Зерттелген көмірлердің экстракциясы кезінде, битуминозды заттардың толық бөлініп алуы, «спирт-бензол» еріткіштер қоспасын қолданғанда анықталды. Зерттелінген көмірлерден алынған экстрактивті заттардың ИҚ-анализі жүргізілді. Спектралды мәліметтер бойынша спиртті-бензолды битумоидтар оттегі-құрамды функционалды қосылыстармен бай, бұл көмір қаңқасының ароматтық және гетероциклдік қосылыстар үшін, өзіне тән жұтылу аймағымен түсіндіріледі. Бензолмен экстракциялау ароматтық көмірсутектердің құрамының көбеюіне әкеледі. Алынған битумоидтар алифаттық және ароматтық заттардың қоспасынан құралған, соның ішінде екіншісі басымырақ. Технологиялық үрдістің дұрыс тәртібін қолданған жағдайда, болашақта битуминозды заттарды алуда шикізат көзі ретінде қызығушылықты тудырады.

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