UDC 662.74:552
CSCSTI 61.53.00

DOI: https://doi.org/10.54859/kjogi108656

Received: 13.06.2023.
Accepted: 15.09.2023.
Published: 30.12.2023.

Original article

Obtaining fuel products by combined hydrogenation of coal and shale

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ABSTRACT

Background: Coal and oil shale are one of the promising types of organic raw materials that can largely compensate and replace petroleum products and gas in the future. Unlike other types of solid fossil fuels oil shales contain significant amounts of hydrogen in organic matter. The possibility of obtaining liquid and gaseous hydrocarbons from a mixture of coal and oil shale similar in composition and properties to petroleum products and natural gas allows us to consider them as important strategic resources.

Aim: This article is devoted to the study of the process of obtaining fuel products of co-hydrogenation of coal and shale.

Materials and methods: Coal from the Taldykol deposit and slate from the Kiin deposit were taken as objects of research. The process of coal and shale liquefaction was carried out on a laboratory installation at a pressure of 5 MPa and a temperature of 425°C, a reaction time of 1 h. Gas chromatographic and elemental analyses were used.

Results: The research results showed that the optimal amount of shale added to coal is 15.0%. The implementation of the co-hydrogenation process under these conditions has increased the yield of liquid products by 10%, namely fractions with a boiling point of up to 200°C from 14.9% to 15.3%, fractions with a boiling point of 200–370°C from 22.1% to 26.4%, fractions with a boiling point above 370°C from 34.6% to 38.8%. Solid residue with a boiling point above 370°C tested as an organic binder for road construction.

Conclusion: The proposed process technology also makes it possible to obtain gasoline and diesel fractions, which after appropriate hydrotreating can be used as motor fuels. The addition of oil shale to coal allows the process to be carried out under optimal conditions with a high degree of conversion into liquid products without coke formation. The degree of transformation of the mixture of organic mass of shale and coal is much higher than just coal.

Keywords: *coal; shale; thermocatalytic processing; hydrogenation; product yield; gasoline fraction; diesel fraction.*

To cite this article:
Kairbekov ZK, Sarmurzina RG, Esenalieva MZ, Kairbekov AZ, Suimbaeva SM, Dzheldybaeva IM. Obtaining fuel products by combined hydrogenation of coal and shale. *Kazakhstan journal for oil & gas industry.* 2023;5(4):83–91. DOI: https://doi.org/10.54859/kjogi108656.

УДК 662.74:552
МРНТИ 61.53.00

DOI: https://doi.org/10.54859/kjogi108656

Получена: 13.06.2023.
Одобрена: 15.09.2023.
Опубликована: 30.12.2023

Оригинальное исследование

Получение топливных продуктов совместным гидрированием угля и сланца

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АННОТАЦИЯ

Обоснование. Угли и горючие сланцы являются одним из перспективных видов органического сырья, которые могут в значительной степени компенсировать, а в будущем и заменить нефтепродукты и газ. В отличие от других видов твердого горючего ископаемого, горючие сланцы содержат значительное количество водорода в органическом веществе. Возможность получения жидких и газообразных углеводородов из смеси угля и горючих сланцев, близких по составу и свойствам к нефтепродуктам и природному газу, позволяет рассматривать их как важные стратегические ресурсы.

Цель. Данная статья посвящена изучению процесса получения топливных продуктов совместным гидрированием угля и сланца.

Материалы и методы. В качестве объектов исследования были взяты уголь месторождения Талдыколь и сланец месторождения Киин. Процесс ожижения угля и сланца проводили на лабораторной установке под давлением 5 МПа и температуре 425°С, время реагирования 1 ч. Были использованы газохроматографический и элементный анализы.

Результаты. Результаты исследований показали, что оптимальное количество сланца, добавляемого к углю, составляет 15,0%. Осуществление процесса совместного гидрирования при данных условиях увеличил выход жидких продуктов на 10%, а именно фракций с температурой кипения до 200ºС с 14,9% до 15,3%, фракций с температурой кипения 200–370°С с 22,1% до 26,4%, фракций с температурой кипения выше 370°С с 34,6% до 38,8%. Твердый остаток с температурой кипения выше 370°С испытан в качестве органического вяжущего для дорожного строительства.

Заключение. Предлагаемая технология процесса также позволяет получать бензиновые и дизельные фракции, которые после соответствующей гидроочистки могут использоваться как моторные топлива. Добавка горючих сланцев к углю позволяет осуществлять процесс в оптимальных условиях с высокой степенью превращения в жидкие продукты без коксообразования. Степень превращения смеси органической массы сланца и угля гораздо выше, чем просто угля.

Ключевые слова: *уголь, сланец, термокаталитическая переработка, гидрогенизация, выход продуктов, бензиновая фракция, дизельная фракция.*

Как цитировать:
Каирбеков Ж.К., Сармурзина Р.Г., Есеналиева М.З., Каирбеков А.Ж., Суймбаева С.М., Джелдыбаева И.М. Получение топливных продуктов совместным гидрированием угля и сланца // *Вестник нефтегазовой отрасли Казахстана.* 2023. Том 5, № 4. С. 83–91. DOI: https://doi.org/10.54859/kjogi108656.

ӘОЖ 662.74:552
ҒТАХР 61.53.00

DOI: https://doi.org/10.54859/kjogi108656

Қабылданды: 13.06.2023.
Мақұлданды: 15.09.2023.
Жарияланды: 30.12.2023

Түпнұсқа зерттеу

Көмір мен тақтатасты бірлесіп гидрлеу арқылы отын өнімдерін алу

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АННОТАЦИЯ

Негіздеу. Көмірлер мен жанғыш тақтатастар органикалық шикізаттың перспективті түрлерінің бірі болып табылады, олар болашақта негізінен мұнай өнімдері мен газ тапшылығын айтарлықтай дәрежеде өтей алады, тіпті оларды алмастыра да алады. Қатты жанғыш қазбалардың басқа түрлерімен салыстырғанда, жанғыш тақтатастардың органикалық құрамында сутектің мөлшері едәуір. Көмір мен жанғыш тақтатастардың қоспасынан мұнай өнімдері мен табиғи газдардың құрамы мен қасиеттеріне жақын сұйық және газтәрізді көмірсутектерді алу мүмкіндігі оларды маңызды стратегиялық ресурстар ретінде қарастыруға мүмкіндік береді.

Мақсаты. Бұл мақала көмір мен тақтатасты бірлесіп гидрлеу арқылы отын өнімдерін алу процесін зерттеуге арналған.

Материалдар мен әдістер. Зерттеу объектілері ретінде Талдыкөл кен орны көмірі мен Киин кен орны тақтатасы алынды. Көмір мен тақтатастарды сұйылту процесі 5 МПа қысымда және 425°С температурада зертханалық қондырғыда жүргізілді, реакциялану уақыты 1 сағ. Газды хроматографиялық және элементтік талдау әдістері қолданылды.

Нәтижелері. Зерттеу нәтижелері көмірге қосылатын тақтатастың оңтайлы мөлшері 15,0% екенін көрсетті. Берілген шарттарда бірлескен гидрлеу процесін жүзеге асыру сұйық өнімдердің шығымын 10% -ға арттырды, атап айтқанда, қайн.темп. 200ºС дейінгі фракцияны 14,9%-дан 15,3%-ға дейін, қайн.темп. 200–370°С болатын фракцияны 22,1%-ден 26,4%-ға дейін, қайн. темп. 370°С жоғары фракцияны 34,6-дан 38,8%-ға дейін жоғарлатты. Қайн.темп. 370°С жоғары болатын қатты қалдық жол құрылысы үшін органикалық байланыстырғыш ретінде сыналған.

Корытынды. Процестің ұсынылған технологиясы тиісті гидротазалаудан кейін мотор отыны ретінде пайдалануға болатын бензин мен дизель фракцияларын алуға мүмкіндік береді. Көмірге жанғыш тақтатастарды қосу процесті кокссіз сұйық өнімдерге айналудың жоғары деңгейімен оңтайлы жағдайда жүзеге асыруға мүмкіндік береді. Тақтатас пен көмірдің органикалық заттар қоспасының айналу дәрежесі көмірге қарағанда әлдеқайда жоғары.

Негізгі сөздер: *көмір, тақтатас, термокаталитикалық өңдеу, гидрогенизация, өнім шығымы, бензин фракциясы, дизель фракциясы.*

Дәйексөз келтіру үшін:
Қайырбеков Ж.Қ., Сармурзина Р.Ғ., Есеналиева М.З., Қайырбеков А.Ж., Суймбаева С.М., Джелдыбаева И.М. Көмір мен тақтатасты бірлесіп гидрлеу арқылы отын өнімдерін алу // *Қазақстанның мұнай-газ саласының хабаршысы.* 2023. 5 том, № 4, 83–91 б. DOI: https://doi.org/10.54859/kjogi108656.

Introduction

Recently, researchers have been paying great attention to the study of the processes of joint thermocatalytic processing of coal and shale. This is most relevant for the deposits of the Republic of Kazakhstan, where both coal and shale are present at the same time. Numerous studies have shown that the organic and mineral parts of oil shale have an activating effect on the thermocatalytic transformation of coals, heavy residual petroleum products, liquid high-boiling waste and some oil and chemical industries [1–2].

A number of processes of thermochemical processing of coal and oil shale have been developed in NJSC al-Farabi Kazakh National university, the Faculty of chemistry and chemical technology, the Department of physical chemistry, catalysis and petrochemistry, as well as the Research Institute of New chemical technologies and materials [3–14], which are based on the results of our research on the complex chemical and technological processing of hard and brown coals of Kazakhstan, conducted in 1990–2020 [15–19]. As the research results have shown, the catalytic properties of oil shales allow for the hydrogenolysis of the organic mass of coals and heavy petroleum raw materials with a high degree of their transformation into liquid distillate products without intensive coke and pellet formation under optimal conditions [3–6, 9, 20].

The mineral part of the shale, containing aluminosilicates, iron oxides and other catalytically active forms of metals, in turn activates the cracking reactions [21]. The presence of such a complex of properties in oil shales causes a change not only in the reactivity of the components of hydrocarbon raw materials, but also in the sizes of supramolecular structures that are an integral part of such a dispersed system, and the preparation of the raw mixture, during its heating and hydrogenolysis [22–24].

It is proved [21, 25, 26] that an important indicator of the possibility of obtaining synthetic liquid products from solid hydrocarbon raw materials is the ratio C : H. In coal it is 0.6–0.8%, while in shale oil it reaches 1.5%, which is similar to the indicators for natural oil. In the process of coal liquefaction, the presence of a hydrogen donor is necessary, for which heavy oil fractions can be used. On this basis, work has begun on the study of joint processing of coal and shale oil, during which it is possible to use them as a "supplier" of hydrogen for coal liquefaction in parallel with the liquefaction of shale oil. This technology will make it possible to obtain a wider range of liquid products with a high content of "light" fractions.

Materials and methods

In this paper, the process of joint processing of brown coal of Taldykol deposit and oil shale of Kiin deposit is studied.

The Taldykol deposit is located in Bayanaul district of Pavlodar region, 65 km south-east of Ekibastuz and 160 km south-west of the regional center of Pavlodar. Taldykol coal has the following physical and chemical characteristics: W^a = 12.0% (moisture analytical), A^{daf} = 7.4% (dry ash), V^{daf} = 41.2% (volatile substances), C^{daf} = 74.5% (carbon of the dry ashless state), H^{daf} = 5.43% (dry state hydrogen), S^{daf} = 0.53% (total dry state sulfur), O + N = 19.54% (amount of carbon and nitrogen), H/C = 0.87% (the ratio of hydrogen to carbon). The insignificant content of ash (7.4%) and a fairly high content of hydrogen (5.43%) make it possible to consider the coal of the Taldykol deposit as a favorable raw material for hydrogenation processing into liquid fuel [27].

The Kiin deposit is located on the territory of the Stepnoy district of the Aktobe region in the upper reaches of the Kiya River, the left tributary of the Ural River. It was opened in 1940 by A.L. Yanshin. Kiin shale has the following physicochemical characteristics: A^{daf} = 72% (dry ash), V^{daf} = 19.3% (volatile substances), C^{daf} = 74% (carbon of the dry ashless state), H^{daf} = 7.6% (dry state hydrogen), S^{daf} = 1.04% (total dry state sulfur), H/C = 0.103% (the ratio of hydrogen to carbon), calorific value per dry substance 265–1537 kcal/kg, heat of combustion of combustible mass 7314–7744 kcal/kg, resin yield per organic mass 21–27%. Yield of semi-coking products in the retort: resin – 4.1–6.5%, semi-coke – 86–93%, pyrogenic water – 2.0–3.7% and gas + losses 1.0–4.3% [27].

The process of liquefaction of coal and shale was carried out at a laboratory installation under a pressure of 5.0 MPa and a temperature of 425°C. To intensify the coal liquefaction process, a catalytic system consisting of fine solid particles of polymetallic ore enrichment sludge was introduced. Destructive processes are additionally implemented on the surface of these particles. Under the conditions of experiments in the process of catalytic processing of a mixture of coal and shale, coke-shaped products on the walls of the installation and in the volume of the reaction mixture were not formed. Liquid products obtained in the process were subjected to distillation with fraction selection with boiling point up to 200°C, fractions with boiling point up to 200–370°C. The residue from the boiling point above 370°C contained in its composition the insoluble organic matter of shale and coal and their mineral part [3–4].

Results

Based on the data of Table 1, it follows that the optimal amount of shale added to coal is 15.0%. When using Kiin shale in the accepted conditions of thermocatalytic processing (test 1), a high yield of the gasoline fraction with boiling point up to 200°C – 15.3% is obtained based

 DOI: 10.54859/kjogi108656

Table 1. Results of thermal catalytic processing of coal with different shale content (425°C, 5.0 MPa, reaction time 1.0 h, intensively shaken reactor)

Product Yield	No. of tests			
	1	2	3	4
Taken, wt.%:				
Coal	85.0	90.0	92.0	94.8
Shale, including:	15.0	10.0	8.0	5.2
Obtained based on coal, wt.%:				
Gas	11.2	14.1	11.0	11.2
Water	5.3	4.7	4.6	4.9
Fraction with boiling point up to 200°C	15.3	12.5	12.1	11.9
Fraction with boiling point up to 200–370°C	26.4	25.9	24.7	21.3
Total yield of light distillates	41.7	38.4	36.8	33.2
Balance with boiling point above 370°C	38.8	39.3	42.5	43.5
Coke content on the mineral part of shale, wt.%	3.0	3.5	5.1	7.2

on coal and diesel fraction with boiling point up to 200–370°C – 26.4%. With a decrease in Kiin shale additives to 10.0%, the total yield of gasoline and diesel fraction decreases from 41.7% to 38.4%. A further decrease in the amount of shale added to 8.0% and 5.2% leads to a significant decrease in the yield of the fractions of motor fuels to 36.8% and 33.2%, respectively, the yield of the heavy residue and coke increases with boiling point above 370°C.

An increase in the content of shale in the coal shale mixture above 15.0% is impractical, since this will lead to a complication of the process technology, an increase in erosion of the equipment by the mineral part of the shale, a delamination of the reaction mixture into liquid and solid phases and a complication of the hardware design of the unit for isolating solid components from liquid products of thermocatalytic processing.

The results of the joint thermal catalytic processing of brown coal of the Taldykol deposit and shale of the Kiin deposit are summarized in Table 2.

As shown in Table 2, the addition of shale to coal under optimal process conditions makes it possible to carry out the process of co-hydrogenation of the organic mass of coal with a high degree of conversion into liquid products without coke formation. In comparison with the process of hydrogenation of coal alone, the degree of transformation of the process of co-hydrogenation of coal and shale is high, since as a result, there is an increase in the yield of liquid products, namely fractions with boiling point up to 200°C from 14.9% to 15.3%, fractions with boiling point 200–370°C from 22.1% to 26.4%, fraction with boiling point above 370°C from 34.6% to 38.8%. The solid residue was

Table 2. Thermocatalytic processing of Taldykol coal and Kiin shale mixture

Process indicators	Coal	Coal +Oil shale
Process Conditions		
Coal: Paste Former (shale + coal): pasting agent	1 : 1.3	(0.6 + 0.4) : 1.3
Shale Organic Mass: Coal Organic Mass		1 : 0.9
Temperature, °C	420	420
Pressure, MPa	5.0	5.0
Duration, min	15	30
Product Yield, %		
Fraction with boiling point up to 200°C	14.9	15.3
Fraction with boiling point up to 200–370°C	22.1	26.4
Fraction with boiling point above 370°C	34.6	38.8
Solid products, gas + water	26.8	18.0
Losses	1.6	1.5

Table 3. Characteristics of distillate products of thermal catalytic processing of coal in a mixture with shale

Indicator	Fractions with boiling point		
	up to 200°C	200–370°C	above 370°C
Density at 20°C, g/cm³	0.7547	0.8891	0.9394
Contents, Vol. %:			
Phenols	2.7	1.8	–
Nitrogenous bases	1.5	4.5	–
Group hydrocarbon composition, wt. %			
paraffinic + naphthenic	73.5	48.3	23.1
aromatic	26.5	51.7	57.1
silica gel resins	–	–	16.5
asphaltenes	–	–	3.3
Iodine number, g I2/100g of product	25.6	34.2	12.3
Elemental composition, wt. %:			
C	85.71	86.30	86.65
H	13.93	12.40	11.24
S	0.27	1.11	1.83
N	0.09	0.19	0.28
Content, g/t			
V	–	–	6
Ni	–	–	19

used as an organic binder for road construction. The bitumen obtained on the basis of products of thermocatalytic processing of a mixture of shale and coal meets the requirements of GOST 22245-76 for petroleum bitumen [17].

The fuel products obtained were investigated using gas chromatographic and elemental analysis. The results are shown in table 3.

According to the results of gas chromatographic analysis, it can be seen that the content of paraffin-naphthenic hydrocarbons in the fractions with a boiling point up to 200°C is high (73.5 wt.%) compared to fractions of boiling point 200–370°C (48.3 wt.%) and with a boiling point above 370°C (23.1 wt.%). And the content of aromatic hydrocarbons has an inverse correlation, that is in fractions with a boiling point above 370°C – 57.1 wt.%, in fractions with a boiling point 200–370°C – 51.7 wt.%, in fractions with a boiling point up to 200°C – 26.5 wt.%. And also in fractions with a boiling point above 370°C resins were found (16.5 wt.%) and asphaltenes (3.3 wt.%).

The content of nitrogenous bases in light distillates (1.5–4.5 vol.%), phenols (2.7–1.8 vol.%), high sulfur content (0.27–1.83 wt.%), as well as the presence of vanadium and nickel indicate that the distillates obtained do not meet the requirements for the quality of motor fuels and need additional processing, such as hydrotreating, cracking, reforming, etc.

Conclusions

Thus, the results of studies of the joint hydrogenation of coal and shale showed that the implementation of the process in the presence of shale of the Kiin deposit increased the total yield of liquid products. The optimal amount of shale added to coal is 15.0%. In this case, there is an increase in the total yield of liquid products by 10%. The solid residue with a boiling point above 370°C has been tested as an organic binder for road construction. The proposed process technology also makes it possible to obtain gasoline and diesel fractions, which after appropriate hydrotreating can be used as motor fuels. Gasoline fraction obtained has a high content of paraffin-naphthenic hydrocarbons (73.5 wt.%) and a moderate amount of aromatic hydrocarbons (26.5 wt.%), as well as unsaturated compounds. The addition of oil shale to coal allows the process to be carried out under optimal conditions with a high degree of conversion into liquid products without coke formation. The degree of transformation of the mixture of organic mass of shale and coal is much higher than just coal.

ДОПОЛНИТЕЛЬНО

Источник финансирования. Работа выполнена в рамках проекта грантового финансирования AP14869180 «Разработка эффективных технологии совместной гидрогенизационной переработки углей и горючих сланцев РК для получения компонентов моторных топлив и химических веществ».

Конфликт интересов. Авторы декларируют отсутствие явных и потенциальных конфликтов интересов, связанных с публикацией настоящей статьи.

Вклад авторов. Все авторы подтверждают соответствие своего авторства международным критериям ICMJE (все авторы внесли существенный вклад в разработку концепции, проведение исследования и подготовку статьи, прочли и одобрили финальную версию перед публикацией). Наибольший вклад распределён следующий образом: Каирбеков Ж.К. – интерпретация данных исследования, проверка результатов, написание и редактирование рукописи, Сармурзина Р.Г. – концепция исследования, Есеналиева М.З. – интерпретация данных, контроль за выполнением работы, Каирбеков А.Ж. – проведение исследования, Суймбаева С.М. – проведение экспериментов, Джелдыбаева И.М. – сбор, анализ, интерпретация данных, написание и редактирование рукописи.

ADDITIONAL INFORMATION

Funding source. The work was carried out within the framework of the grant financing project AP14869180 «Development of effective technologies with joint hydrogenation processing of coal and oil shale of the Republic of Kazakhstan for the production of motor fuel components and chemicals».

Competing interests. The authors declare that they have no competing interests.

Authors' contribution. All authors made a substantial contribution to the conception of the work, acquisition, analysis, interpretation of data for the work, drafting and revising the work, final approval of the version to be published and agree to be accountable for all aspects of the work. The largest contribution is distributed as follows: Zhaksyntay K. Kairbekov – interpretation of research data, verification of results, writing and editing of the manuscript; Raushan G. Sarmurzina – research concept; Manshuk Z. Esenalieva – interpretation of data, control over the performance of work; Altay Zh. Kairbekov – conducting research; Saltanat M. Suimbaeva – conducting experiments; Indira M. Dzheldybaeva – data collection, analysis, interpretation, writing and editing of the manuscript.

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