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ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
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Қ. И. Сәтпаев атындағы Қазақ ұлттық техникалық зерттеу университеті

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ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
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Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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SMOKELESS FUEL PRODUCTION – SEMI-COKE FROM COAL

Abstract. In the article, the coal pyrolysis of the Saryadyr field (layer "Pyatimetrovyi") was carried out in the temperature range 250-550 ^{o}C at a heating rate of 10-15 ^{o}C/min and with holding time at 550 ^{o}C for 1, 2 and 3 hours. As a result of pyrolysis, semi-coke was produced (as a smokeless fuel), as well as combustible gas and liquid products (resin). The technical and elemental compositions of the obtained pyrolysis products were studied using thermogravimetric and chromatographic analysis, energy dispersive X-ray spectroscopy. The combustible gas with the highest calorific value was obtained at a temperature of 350 °C. The dependence of the influence of the holding time in the pyrolysis process (at 550 °C) on the yield and elemental composition of the products.

Key words: coal, Saryadyr, pyrolysis, semi-coke, combustible gas, resin.

Introduction. During the direct combustion of coals in the ordinary form in furnace with layer combustion, significant fuel losses occur due to underburning and entrainment. The efficiency of use of coal in these furnaces (containing up to 50% of fines) is not more than 50-60%, and when burning coal and coal fines (class 0-6 mm) thermal efficiency decreases to 30-40% [1].

The modern ecological situation in the world tightens the requirements to the quality of the burned municipal and domestic fuel. At the same time, fuel burned in the municipal sector should have low sulfur content, the reduced smoke and the necessary granulometric composition [2].

Thus, the organization of production of smokeless fuel - semi-coke for domestic consumers has a great economic importance, both in terms of economic efficiency, and rational use of natural resources and ecological safety of the environment [3-6]. At the same time, non-caking and weakly caking coals should be used in order to obtain the semi-coke.

The main methods of thermochemical processing of solid fuels are gasification, high-temperature coking of coals and thermal treatment of fuels at temperatures of 500-600 °C (semi-coking) [4]. At the same time, for the last process, the main for the last several decades is semi-coking in furnaces with internal heating by a gas coolant. All other methods have found industrial application only in individual cases or are promising in connection with new developments.

Analysis of the world practice of producing smokeless fuels [5, 7-14] shows that the process of pyrolysis with the simultaneous production of still gas and liquid products is particularly relevant, and especially in connection with high ecological requirements, is of definite scientific and practical interest.

The process of semi-coking allows the production of a medium-temperature char, which can then be burned without the emission of any harmful emissions (carbon dioxide CO_2, hydrogen sulfide H_2S, nitrogen oxides NOx, etc.) [15, 16]. In addition, the products of the semi-coking of solid fuels are also combustible gas (due to the predominant content of CO, H_2, CH_4) and liquid products (resin) [17]. Liquid pyrolysis products can be used as heating oil. Combustible gas (after purification) and semi-coke can also

be used as an energy fuel for its combustion (in cogeneration plants, boiler houses, domestic furnaces) to generate thermal energy, as well as in GPI and gas turbine stations for generating electricity. Refined flammable gas (with a certain ratio of H_2/CO) can be a raw material for the production of motor fuels (gasoline, diesel fuel) and other valuable chemical products (methanol, ethanol, etc. more than 300 species). The acquisition of these products makes it possible to increase the efficiency of the use of primary energy of solid fuels in comparison with the process of direct combustion.

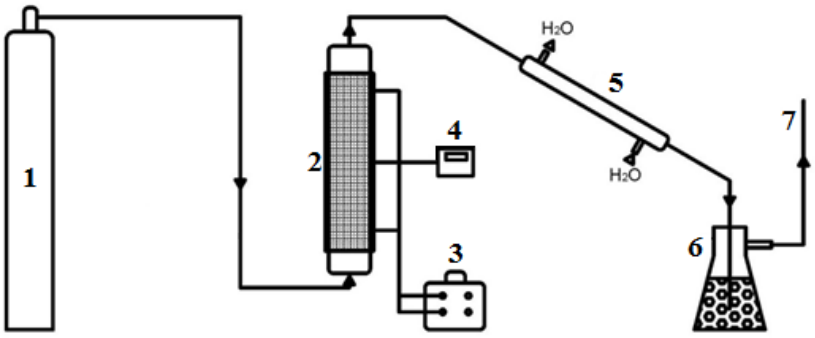
The purpose of this paper is to investigate the pyrolysis process to produce semi-coke, flammable gas, and liquid products (resins).

Research methodology. The coal of the Saryadyr field ("Pyatimetrovyi" layer) located in the Akmola region (Erementau district) was chosen as the object of the study.

The humidity, ash content, and volatility of the Saryadyr coal and the resulting semi-coke were obtained from the Thermoster Eltra thermogravimetric analyzer (according to ASTM D7582-12 Standard Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis).

Elemental analysis of carbon samples was carried out on the elemental analyzer Euro EA-3000, and also by energy dispersive X-ray spectroscopy using a SEM device (Quanta 3D 200i) with an attachment for energy dispersive analysis from EDAX at a resolution of 3.0 nm at 30 kV (high vacuum mode); <12 nm at 3 kV (low vacuum mode); with an accelerating voltage from 200 V to 30 kV and with an increase from x50 to x100000. Elemental analysis is determined from Be to U. Samples were attached to a copper holder using conductive adhesive paper. Previously, a thin conducting layer of carbon was applied to the surface of the samples in a special vacuum installation for better passage of the charges. The energy of the exciting electron beam during the analysis was 15 keV, the operating distance was 15 mm.

Pyrolysis of coal was carried out on a laboratory installation, shown in figure. Preliminary, the coal was fractioned to fractions of 20-40 mm and loaded into a cylindrical quartz reactor (3).



Schematic diagram of a laboratory pyrolysis installation:
1 - gas cylinder (argon); 2 - reactor; 3 - LATR; 4 - thermal sensor; 5 - direct refrigerator;
6 - a flask for cleaning gas from tar; 7 - gas outlet

The reactor is wrapped in a nichrome spiral and insulated with asbestos to heat the furnace. The temperature in the reactor was set by means of a heating element (LATR) (4) and monitored according to the indications of a digital thermal sensor "Aries TRM1" (5) equipped with a thermocouple of the chromel-alumel type introduced into a special pocket of the reactor. The temperature in the reactor was maintained with an accuracy of ± 0.2 °C.

The technological process was carried out in an inert argon medium in the temperature range 250-550 ^{o}C at a heating rate of 10-15 ^{o}C/min and held at 550 ^{o}C in three stages: for 1, 2 and 3 hours. Argon was supplied from a cylinder (1) to a reactor at a predetermined flow rate of 20 ml/min, which was set using a flowmeter. After the reactor, the gas was sent to the cooler (6), from where part of the condensed gas was drained into the flask (7) to purify it of resinous substances. Unreacted gases were sent to vent (8).

The elemental composition of the gas (produced during pyrolysis) and liquid products was determined on a chromatograph of the Chromos GC-1000.

The calorific value (kcal/m^3) of the pyrolysis gas, consisting mainly of the combustible components CO, H2, CH4, was determined by the formula:

$$Q_r = 0{,}01(n_{CO}\,Q_{CO} + n_{H2}\,Q_{H2} + n_{CH4}\,Q_{CH4} + n_{H2S}\,Q_{H2S}),$$

where n_{CO}, n_{H2}, n_{CH4}, n_{H2S} – components concentration CO, H_2, CH_4, H_2S (v. %) in gas; Q_{CO}, Q_{H2}, Q_{CH4}, Q_{H2S} – the calorific values of the components respectively CO, H_2, CH_4, H_2S (numerical values were used from [18]).

Results and discussion. The results of the analysis of technical and chemical compositions of Saryadyr coal are given in tables 1, 2.

Table 1 − Chemical composition of Saryadyr coal («Pyatimetrovyi» layer)

Indicator, wt.%	Content
humidity on the working mass W^r, %	2.56
ash content A^r, %	19.71
volatile V^{daf}, %	50.54
sulfur on the working mass S^r, %	0.59
carbon content C^{daf}, %	82.98
hydrogen content H^{daf}, %	5.59
oxygen content O^{daf}, %	9.76
nitrogen content N^d, %	1.08
Combustion heat of fuel Q^r_i, kcal/kg	5215
Chemical composition of mineral part, wt.%	
SiO_2	59.6
Al_2O_3	14.5
Fe_2O_3	8.2
CaO	1.9
MgO	1.3
K_2O+Na_2O	11.7
SO_3	2.8

The results of the energy-dispersive X-ray spectroscopy for Saryadyr coal samples are given in table 2.

Table 2 − Chemical composition of Saryadyr coal («Pyatimetrovyi» layer)

Element	Initial coal		Coal, 1 hour - holding time		Coal, 2 hours - holding time		Coal, 3 hours - holding time	
	wt.%	at. %	wt.%	at. %	wt.%	at. %	wt. %	at. %
C	77.07	84.05	87.80	91.80	92.69	95.55	89.88	93.07
O	14.84	12.15	8.09	6.35	3.71	2.87	7.32	5.69
Al	2.30	1.12	1.09	0.51	0.42	0.19	0.64	0.29
Si	5.48	2.56	2.93	1.31	3.02	1.33	2.01	0.89
S	0.30	0.12	0.09	0.04	0.15	0.06	0.16	0.06

Chromatographic analysis of the composition of pyrolysis gas from Saryadyr coal, held at 550°C for 1, 2 and 3 hours, showed that the most caloric gas is observed at temperatures of 350 and 450°C. Their calorific values are ≈3500-3900 kcal/m^3 and ≈4100-4400 kcal/m^3, respectively, due to mainly, except for CO (≈2-8%) and H2 (≈9-27%), high methane content (up to 43%). There are H_2S (≈1-3%), ethane (≈3-6%), propane + propylene (1-2%) in small quantities.

With a further increase in temperature to 550 ^{0}C, the calorific value of the gas is significantly reduced to 3200, 3400, 2700 kcal/m^3, respectively, with a holding time of 1, 2, 3 hours.

Tables 3–5 show the material balances of pyrolysis products of Saryadyr coal (per 400 grams of coal).

The data from table 6 show that the appreciable influence of the holding time of coal during pyrolysis affects the calorific value of the produced semi-coke. At the shortest time (t = 1 hour), the calorific value has the highest value and is 6271 kcal/kg, which is significantly higher than the similar parameter of the initial coal (5215 kcal/kg). With further increase in the exposure time, this parameter decreases.

Table 3 − Material balance of pyrolysis products of Saryadyr («Pyatimetrovyi» layer) (T = 550 ^{0}C, t = 1 hour)

Pyrolysis products of coal (400 g)	Content		
	g	m^3	%
Solid residue (semo-coke)	334.7	–	83.67
Gas	18.5	16.1	4.62
Liquid products (resin)	6.5	–	1.62
Water	30.7	–	7.67
Balance discrepancy	9.6	–	2.40
Total	400.0	–	100.00

Table 4 − Material balance of pyrolysis products of Saryadyr («Pyatimetrovyi» layer) (T = 550 ^{0}C, t = 2 hour)

Pyrolysis products of coal (400 g)	Content		
	g	m^3	%
Solid residue (semo-coke)	287.4	–	71.85
Gas	44.2	–	11.05
Liquid products (resin)	35.0	–	8.75
Water	25.0	–	6.25
Balance discrepancy	8.4	–	2.10
Total	400.0	–	100.00

Table 5 − Material balance of pyrolysis products of Saryadyr («Pyatimetrovyi» layer) (T = 550 ^{0}C, t = 3 hour)

Initial products	Content		
	g	m^3	%
Solid residue (semo-coke)	263.5	–	65.87
Gas	62.4	54.3	15.60
Liquid products (resin)	10.2	–	2.55
Water	53.7	–	13.42
Balance discrepancy	10.2	–	2.55
Total	400.0	–	100.00

Table 6 presents data on the technical and elemental analysis of semi-coke from Saryadyr coal.

Table 6 – Results of technical and elemental analysis of semi-coke
from Saryadyr coal, obtained at a temperature of 550 °C and a holding time of 1-3 hours

Parameter name and unit of measurement	Coal, 1 hour - holding time	Coal, 2 hour - holding time	Coal, 3 hour - holding time
Humidity analytical, W^a, %	1.36	1.64	2.24
Ash content, A^d, %	23.72	26.13	27.92
S^d_t, %	0.32	0.27	0.28
Q_s^d, kcal/kg	6271	5987	5689
C^{daf}, %	67.97	68.17	67.80
H^{daf}, %	3.07	1.78	0.68
N^d, %	1.34	1.20	1.23
O^{daf}, %	3.58	2.45	2.09

Conclusions. The analysis of the results showed that, depending on the type of the main product to be obtained, one should select a particular regime with a certain holding time of the coal (1-3 hours) at a given temperature. At holding time in the process of pyrolysis of coal at a temperature of 550 ^{0}C for 1, 2 and 3 hours, the main products are respectively the smokeless fuel (semicoke) in an amount of 83.67 (wt.%) with the highest calorific value, resin 8.75 %) and a combustible gas of 15.60 (wt %).

Thus, for the most rational use of solid fuels (in contrast to their direct combustion), their complex processing is necessary to produce various hydrocarbon products with high added value, since their total cost can significantly exceed the cost of the raw materials.

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ТАС КӨМІРДЕН ЖАРТЫЛАЙ КОКС –
ТҮТІНСІЗ ОТЫН АЛУ

Аннотация. Мақалада "Сарыадыр" («Пятиметровый» пласты) кенішінің көмірінің пиролизі келтірілген, 250-550 °С температура аралығында, қыздыру жылдамдығы 10-15 °С / мин құрады және 550 ° С-да 1, 2 және 3-сағат ұсталды. Пиролиз нәтижесінде жартылай кокс (түтінсіз отын), жанғыш газ және сұйық өнімдер (шайыр) алынды. Термогравиметрлік және хроматографиялық талдау әдістерімен, энергодисперсиялық рентгенді спектроскопия арқылы пиролиз нәтижесінде алынған өнімнің техникалық және элементтік құрамы анықталды. Жылуы айтарлықтай жоғары газ 350 °С температурада түзілді. Пиролиз үрдісі кезінде (550 °С) ұсталу уақытының өнімнің элементтік құрамы мен шығымына тәуелділігі анықталды.

Түйін сөздер: көмір, Сарыадыр, пиролиз, жартылай кокс, жанғыш газ, шайыр.

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ПОЛУЧЕНИЕ БЕЗДЫМНОГО ТОПЛИВА –
ПОЛУКОКСА ИЗ КАМЕННОГО УГЛЯ

Аннотация. В статье проведен пиролиз угля месторождения «Сарыадыр» (пласт «Пятиметровый») в интервале температур 250-550 ^{o}C при скорости нагрева 10-15 ^{o}C/мин и с выдержкой при 550 ^{o}C в течение 1, 2 и 3 часа. В результате пиролиза получены полукокс (в качестве бездымного топлива), а также горючий газ и жидкие продукты (смола). Методами термогравиметрического и хроматографического анализов, энергодисперсионной рентгеновской спектроскопии изучены технический и элементный составы полученных продуктов пиролиза. Горючий газ с наибольшей теплотворной способностью получен при температуре 350 ^{o}C. Установлена зависимость влияния времени выдержки в процессе пиролиза (при 550 ^{o}C) на выход и элементный состав продуктов.

Ключевые слова: уголь, Сарыадыр, пиролиз, полукокс, горючий газ, смола.

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