Production of Isotropic Coke from Shale Tar at Various Parameters of the Delayed Coking Process

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ABSTRACT: This paper presents the results of studies on the production of coke at excess pressures of 0.15, 0.25, and 0.35 MPa with delayed coking of shale tar obtained by pyrolysis of oil shale in the Baltic Basin. To assess the quality of the resulting coke, properties such as the absolute density, yield of volatile substances, ash content, and total porosity were analyzed. The microstructure of the resulting coke was analyzed by X-ray diffraction, for which the evaluation criteria were the interplanar spaces d_{002} and d_{100} and the sizes of the Lc and La crystallites. The scanning electron microscopy (SEM) technique was used to confirm the results of the analysis of the formed structure of shale coke obtained earlier. The calcined samples were classified as cokes with an isotropic structure. In addition, the fractional composition of the obtained gasoline and gas oil fractions of delayed coking of shale resin was analyzed.

1. INTRODUCTION

At present, enterprises of the energy sector in Russia are forced to look for innovative opportunities for more efficient processing of natural resources (oil, coal, natural gas, etc.) that meet the world demand and search for new promising resources.1–3 One way to diversify the energy policy structure and increase the total energy resources of the country could be the use of local low-grade fuels—oil shale.4–6

Oil shale is a solid mineral that contains large quantities of mineral impurities and organic matter (kerogen). The organic matter of oil shale has a high atomic H/C ratio of 1.7 on average (coal 0.4–0.5, oil 1.9), making it possible to obtain liquid products similar to petroleum products (components of motor fuels, petrochemical products, etc.) using different thermal processing methods. Apart from that, the global reserves of oil shale are huge and amount to approx. (5860–12160)·109 barrels in oil shale equivalent (CIS countries, 3500; United States, 2000–8000; Europe, 50–100; China, 30–170; etc.).7–9 Russia has the largest reserves of shale tar in Europe—about 35.5 billion barrels, indicating the need for active use of shale tar in the chemical, energy, and other sectors of the economy.10–12 The main methods of oil shale processing are focused on the transformation of organic matter of oil shale into various products (solid, liquid, gaseous) by employing various temperature influences: semicoking (470–600 °C), coking (600–900 °C), gasification (1000–1100 °C), pyrolysis (400–600 °C), etc.13–15 The choice of a particular method of oil shale processing and its economic efficiency largely depends on the properties (ash content, heat of combustion, etc.) and composition (organic and inorganic components) of the mineral resource.16–18 The possibility of transferring the organic component of oil shale from the solid aggregate state to liquid positively affects the economic indicators of using this low-grade raw material. For instance, the production of shale tar in the world is about 1 million tons a year.19–21 Typical characteristics of properties (ash content, density, viscosity, etc.) and composition (sulfur content, aromatic hydrocarbons, etc.) of shale oil from pyrolysis of oil shale are presented in Table 1.22

In their work,23 Razvigorova et al. gave an example of the hydrocarbon composition of shale tar from various Bulgaria deposits: paraffin-naphthenic hydrocarbon content, 70.31–70.94 wt %; and aromatic hydrocarbon content, 8.74–16.87 wt %. It can be seen from the presented data that, depending on the oil shale deposit, the composition and properties of shale tar are different. The content of sulfur in shale tar is given in the work of Maaten et al.27 and amounts to 0.866–0.1 wt %. The group hydrocarbon composition and physical and chemical properties (ash content, viscosity, etc.) of shale tars produced in the world practice by pyrolysis of oil shale suggest their use in obtaining valuable carbon materials. The high content of polycyclic aromatic hydrocarbons (up to 21.7%) and low content of sulfur (up to 0.1%), ash (up to 0.06%), and

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asphaltene (up to 2.5%) make shale oil a promising direction for further production of isotropic shale coke for producing filler coke for the graphite industry in addition to direct production of chemical and petrochemical products (motor fuel, varnishes, phenols, etc.). The raw material, particularly the coke filler, plays a major role in producing isotropic graphite. The use of isotropic shale coke may allow achieving several advantages—to improve the isotropy of graphite and to increase the actual density and strength. For example, in their study, the authors say that coke with an isotropic structure allows obtaining graphite with increased strength and high radiation stability. The following coke fillers are compared in the paper: high-temperature pitch coke (2.2 points), oxidized shale pitch coke (2.0—2.5 points), industrial pitch coke GOST 3213 (2.5 points), and industrial petroleum coke GOST 22898 (1.9—2.0 points) (Table 2). The authors conclude that the

2. EXPERIMENTAL SECTION

2.1. Materials and Methods of Coking. Light (250 °C) and heavy (250—350 °C) fractions of shale tar from the pyrolysis process of oil shale of the Baltic Basin were used as input materials for research on coking with representative samples of oil shale coke (Table 3).

A laboratory unit of the St. Petersburg Mining University consisting of a reaction unit and a distillate collection unit was used to carry out experiments on shale tar coking. The reaction unit consists of a steel coking reactor and an electric furnace with three independent heating zones to maintain a uniform temperature throughout the coking bed height; the reactor is equipped with a pressure gauge to control pressure. The gas—liquid product mixture was drained through a tube located in the reactor lid through a needle valve, from where it went to a tube-in-tube heat exchanger and distillate collection unit.

Two series of shale tar coking experiments were conducted: the first series is coking of light shale tar (Experiment Nos. 1—3) and the second is coking of heavy shale tar (Experiment Nos. 4—6) with varying excess coking pressures of 0.15, 0.25, and 0.35 MPa at a constant temperature 485—500 °C in each
experiment. The mass of the feedstock was 0.200–0.256 kg. The average heating rate of the coke layer inside the reactor was 1.52–1.79 °C/min. After reaching the carbonization temperature, the isothermal mode was maintained until the formation of the gas–liquid product mixture ceased and, as indicated, the pressure in the reactor decreased. The constant temperature is maintained for 60 min. The operating parameters of the coking process during each experiment are shown in Table 4.

2.2. Methods for Analyzing the Physicochemical Properties of Coke. Samples 1–6 obtained during the shale tar coking experiment were analyzed to determine and compare their physical and chemical properties: humidity, volatile matter yield, ash content, actual and apparent densities, and total porosity.

The determination of the moisture content of the produced shale coke was carried out in a drying cabinet according to GOST 26132-84 "Methods for the determination of moisture in an analytical sample" (ISO 5068-2:2007). A sample in a weight fraction (2–4) g and mass (6–8) g; obtained polished sections were polished. Then, the microstructure was evaluated with a microscope in reflected plane-polarized light at a magnification of 90X – 100X.

2.4. Method of X-ray Diffraction Analysis. X-ray diffraction analysis was performed using an XRD-7000 Shimadzu X-ray diffractometer (Cu Kα radiation, 2.7 kW) at room temperature using a polycrystal method. The X-ray images were taken at long accumulation times (2 s) and at a scanning step of 0.02°. Asymmetric reflexes of petroleum cokes were decomposed into peaks, the profile of which is described by a Gaussian with a maximum at angles of 2θ, which characterize certain structural components of petroleum coke samples.

For a detailed assessment of the fine structure of shale oil coke by the X-ray structural method, we used the interplanar distance of the diffraction maxima (002) and (100) and the dimensions of the coherent scattering region in the directions of the "c"-axes (average height Lc of crystalline and average diameter L0 of hexagonal layers). To determine the interplanar distance (d002 and d100), in Å, for obtained raw and calcined samples of petroleum coke, the calculation was performed according to the Wulff–Bragg equation

\[ d = \lambda / 2 \sin \theta \]

where \( \lambda = 1.5406 \) is the X-ray wavelength for Cu Kα, in Å, and \( \theta \) is the Bragg diffraction angle, in rad.

The average linear sizes of crystallites \( L_c \) and \( L_a \) were determined, in Å, by the Scherrer equation

\[ L_c = 0.87 \lambda / \beta \cos \theta_{002} \text{ and } L_a = 1.84 \lambda / \beta \cos \theta_{100} \]

where 0.87 and 1.84 are the Scherrer and Warren constants, respectively, which are conventionally set for cokes of the same uniformity in the published results, and \( \beta \) is the width of the diffraction line at half the maximum height (in rad) minus the hardware width of the peak \( b = 0.14^\circ \).

2.5. Method of Analysis of Physical and Chemical Properties of Liquid Products of Shale Tar Coking. The density of gasoline and light gas oils of shale tar coking was determined in accordance with GOST3900-85 "Crude oil and oil products – Methods for determining density using hydrometers". This method consists of placing the test sample in a cylinder of the same temperature as the sample (with an error of no more than 0.2 °C). Without allowing the upper part of the rod of a clean and dry areometer to be wet, it is slowly lowered by holding the upper end into the sample cylinder. After the cessation of oscillation of the pressure gauge in the cylinder, the upper edge of the meniscus reads the density of the petroleum product at the test temperature. The test is carried out twice; if the discrepancy meets the requirements, the value is recorded.

Kinematic viscosities of gasoline and light gas oils of shale tar coking were determined according to GOST R 53708-2009 "Petroleum products – Transparent and opaque liquids –
Determination of kinematic viscosity and calculation of dynamic viscosity. To conduct tests for the analysis of kinematic viscosity of the resulting liquid coking fractions, a multiband automatic Herzog HVM 472 viscometer was used, which determines viscosity automatically at temperatures from +20 to +150 °C EF oil products from 0.5 to 5000 mm²/s, which meets the requirements of GOST R 53708-2009.

2.6. Method for Determining the Hydrocarbon Composition of Liquid Products of Shale Tar Coking. The determination of individual hydrocarbon composition of gasoline and light gasoil of light and heavy oil shale coking was carried out by the GC–MS method on a Shimadzu GCMS-QP2010 SE apparatus. A nonpolar column (5% phenyl-methyl-polysiloxane) of 30 m length, 0.25 mm thickness, and 0.25 μm polymer coating thickness was used in the analyses. For the test, gasoline and light gasoil samples were dissolved in quaternary carbon chloride at a ratio of 10 μL of sample to 1 mL of solvent. The following chromatographic equipment parameters were used: evaporator temperature, 280 °C; initial column temperature, 50 °C; initial temperature hold time, 2 min; heating rate, 10 °C/min; final column temperature, 290 °C; final temperature hold time, 30 min; carrier gas, helium; carrier gas flow rate, 1 mL/min. Processing of the analysis results, including the normalization of the chromatogram by reference peaks of normal alkanes, was performed in the standard software package GCMS Solutions.

3. RESULTS AND DISCUSSION

The resulting material balance of light and heavy shale tar coking processes is presented in Table 5. Carbon material in the process of coking of light shale tars was obtained in an amount of 24.51–33.49%, depending on the excess pressure, varying from 0.15 to 0.35 MPa at the end temperature of coking in the range of 485–498 °C. There is a clear dependence of the increase in the yield of the carbon material from heavy shale tars from 28.97 to 35.94% on the increase of overpressure of coking from 0.15 to 0.35 MPa. In addition, it can be noted that when reducing the excess pressure of coking to 0.15 MPa, the coke layer height increases up to 76% of the height of the reaction zone of the coking reactor.

The yield of coking gasoline (i.b.p.−180 °C) when changing the coking overpressure from 0.15 to 0.35 MPa decreases from 8.68 to 6.06% for light shale tar and, conversely, increases from 7.46 to 9.73% for heavy shale tar. The yield of heavy coking gasoil tends to decrease when coking light shale tar from 12.15 to 7.19% and clearly decreases when coking heavy shale tars from 10.36 to 3.24% when the pressure increases from 0.15 to 0.35 MPa at temperatures around 485–495 °C. The content of pyrolysis water in light shale tar distillates is about 3% and in heavy tar distillates is about 5%.

The content of paraffin hydrocarbons of normal structure at increasing overpressure from 0.15 to 0.35 MPa increases in the light gasoil of heavy shale tar coking from 25.74 to 34.18% and that of isoparaffin hydrocarbons decreases from 2.59 to 1.08% under the same conditions. The aromatic hydrocarbons content also decreases from 61.45 to 52.38%. The content of n-paraffins in light shale tar coking gasoil increases from 23.78 to 36.85%, the content of naphthenic hydrocarbons increases from 2.29 to 6.84%, and the content of aromatic hydrocarbons decreases from 63.66 to 43.59% (Table 6). For both light and heavy shale tar coking, the yield of carbon material decreases from 63.66 to 49.22% as the pressure increases from 0.15 to 0.35 MPa.

Table 5. Material Balance of the Coking Process of Light and Heavy Oil Shale Tar

| input material balance | Ex. 1 | Ex. 2 | Ex. 3 | Ex. 4 | Ex. 5 | Ex. 6 |
|------------------------|------|------|------|------|------|------|
| light shale tar content, % mass | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| heavy shale tar content, % mass | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| output amount of distillates, including | 59.07 | 63.24 | 59.61 | 57.54 | 52.99 | 49.22 |
| gasoline (i.b.p.−180) °C | 8.68 | 7.94 | 6.06 | 7.46 | 8.13 | 9.73 |
| light gas oil (180−360) °C | 35.29 | 46.13 | 43.38 | 33.97 | 34.06 | 31.33 |
| heavy gas oil (360−i.b.p.) °C | 12.15 | 6.01 | 7.19 | 10.36 | 5.50 | 3.24 |
| pyrolysis water | 2.95 | 3.16 | 2.98 | 5.75 | 5.30 | 4.92 |
| carbon material | 28.64 | 24.51 | 33.50 | 28.97 | 32.27 | 35.94 |
| gas + losses | 12.29 | 12.25 | 6.89 | 13.49 | 14.74 | 14.84 |
| total | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |

Table 6. Operating Parameters of the Shale Tar Carbonization Process

| product | Ex. | pressure (MPa) | n- | iso- | naphthenic | olefin | aromatic |
|---------|-----|---------------|----|-----|-----------|-------|---------|
| light tar coking gasoline | Ex. 1 | 0.15 | 39.12 | 3.14 | 9.99 | 38.42 | 9.33 |
|        | Ex. 2 | 0.25 | 28.38 | 3.45 | 7.67 | 42.88 | 17.66 |
|        | Ex. 3 | 0.35 | 31.80 | 2.60 | 10.79 | 37.48 | 17.33 |
| heavy tar coking gasoline | Ex. 4 | 0.15 | 43.49 | 2.02 | 11.54 | 34.35 | 8.42 |
|        | Ex. 5 | 0.25 | 40.19 | 3.26 | 9.23 | 38.21 | 9.11 |
|        | Ex. 6 | 0.35 | 45.48 | 1.98 | 10.53 | 37.27 | 4.74 |
| light coking gas oil of light tar | Ex. 1 | 0.15 | 23.78 | 2.63 | 2.29 | 7.64 | 63.66 |
|        | Ex. 2 | 0.25 | 36.78 | 1.08 | 6.03 | 10.84 | 45.27 |
|        | Ex. 3 | 0.35 | 36.85 | 2.35 | 6.84 | 10.37 | 43.59 |
| light coking gas oil of heavy tar | Ex. 4 | 0.15 | 25.74 | 2.59 | 1.85 | 8.37 | 61.45 |
|        | Ex. 5 | 0.25 | 30.53 | 1.67 | 4.47 | 9.33 | 54.00 |
|        | Ex. 6 | 0.35 | 34.18 | 1.08 | 4.34 | 8.02 | 52.38 |
heavy shale tar types of gasoline, there is no clear relationship between the effect of changing overpressure from 0.15 to 0.35 MPa on the hydrocarbon composition.

The kinematic viscosity of light shale tar coking gasoil decreases from 6.51 to 3.16 mm²/s with increasing overpressure from 0.15 to 0.35 MPa, and for heavy shale tar coking, it increases from 10.46 to 3.16 mm²/s. The densities of light shale tar coke gasoline are, respectively, 765.5, 786.1, and 769.8 kg/m³ for fractions obtained at overpressures of 0.15, 0.25, and 0.35 MPa, and the densities of heavy shale tar coke gasoline are, respectively, 751.5, 729.7, and 743.6 kg/m³ for the same conditions. For light coking gasoil, the density is in the ranges 918.6 to 949.4 kg/m³ for heavy shale oil (Table 7).

Table 7. Quality Indicators of Light Coking Gas Oils of Light and Heavy Shale Tar

| parameters of cooking | Ex. 1 | Ex. 2 | Ex. 3 | Ex. 4 | Ex. 5 | Ex. 6 |
|-----------------------|-------|-------|-------|-------|-------|-------|
| overpressure, MPa     | 0.15  | 0.25  | 0.35  | 0.15  | 0.25  | 0.35  |
| density at 20 °C, kg/cm³ | 952.4 | 927.0 | 920.8 | 949.4 | 928.0 | 918.6 |
| viscosity at 20 °C, mm²/s | 6.51  | 5.34  | 3.16  | 10.46 | 4.49  | 3.16  |

The physicochemical properties of carbon materials obtained in the process of coking from light and heavy shale tar are presented in Table 8.

The obtained samples of light and heavy shale coke have an isotropic homogeneous structure, which is proved by the microstructure index according to GOST 26132-84 “Petroleum and pitch coke – Microstructure evaluation method” and is 1.5–2.2 points (Figure 1).

The values of humidity of the obtained coke (0.15–0.35%) are explained by the fact that no water was used during coke unloading from the reactor. The ash content of coke produced from light shale tar (0.10–0.67%) is less than the ash content of coke produced from heavy shale tar (0.78–0.93%). It should also be noted that by increasing coking pressure from 0.15 to 0.35 MPa, the actual density of coke from light tar decreases from 1.332 to 1.213 g/cm³. When coking heavy tar, increasing pressure has almost no effect on the current density.

When comparing the influence of the coking pressure of light and heavy shale tar used as a potential raw material for the production of isotropic cokes, some regularities can be noted on their quality indicators. With an increase in excess pressure from 0.15 to 0.35 MPa, the total porosity of coke from light shale tar in all experiments is higher than from heavy, while if at 0.15 MPa, this difference is only 5%; then at 0.25 MPa, this difference is 11%; and at 0.35 MPa, this difference is 17%. This shows that the development of the surface with an increase in pressure during the coking of shale tar is probably less related to the difference in the composition of the coking raw materials studied in the work and is more determined by the technological regime. This is confirmed by the tendency to change the density of the resulting carbon material—with an increase in excess pressure from 0.15 to 0.35 MPa, it becomes less dense (as a result, more porous), both when coking light and heavy shale tar. Changes in the density of coke with an increase in the excess coking pressure are associated with a simultaneous decrease in the yield of volatile substances present in coke. These substances leaving the coking solidifying mass just form a porous structure.

The analysis of the chemical composition of the obtained coke samples showed a small number of impurities, in wt %: Si, 0.127; Ca, 0.097; Fe, 0.088; K, 0.033; Al, 0.013; Ni, 0.005; and S, 0.629.

Figure 2 shows a comparison of diffractograms of shale coke produced from light and heavy pyrolysis tars at different values of the coking process pressure.

The peaks with the maximum values of 2θ angles at around 25 and 43°, corresponding to reflexes 2θ002 and 2θ100, respectively, are clearly pronounced. The angular position of the reflexes (2θ002 and 2θ100) on the radiograph is determined by the corresponding interplanar distance (d002 and d100) (Table 9).

The interplanar spacing d002 increases from 3.484–3.485 to 3.506–3.514 Å when the excess coking pressure increases from 0.15 to 0.35 MPa for both light and heavy pyrolysis tar shale coke. However, d100 decreases from 2.168 to 2.113 Å when the overpressure of light shale oil coke increases and, conversely, increases from 2.074 to 2.097 Å for coke from heavy pyrolysis shale oil. The ratio of average height Ic and average diameter Lc of shale coke crystallites obtained in all experiments is close to 1. The crystallite height is almost always

Table 8. Quality Indicators of Light and Heavy Shale Tar Cokes

| characteristic            | Ex. 1 | Ex. 2 | Ex. 3 | Ex. 4 | Ex. 5 | Ex. 6 |
|---------------------------|-------|-------|-------|-------|-------|-------|
| overpressure, MPa         | 0.15  | 0.25  | 0.35  | 0.15  | 0.25  | 0.35  |
| moisture content, %       | 0.391 | 0.184 | 0.354 | 0.259 | 0.189 | 0.314 |
| ash content, %            | 0.67  | 0.10  | 0.15  | 0.93  | 0.78  | 0.82  |
| yield of volatiles, %     | 11.29 | 8.14  | 10.12 | 8.51  | 7.23  | 7.80  |
| actual density, g/cm³     | 1.332 | 1.271 | 1.213 | 1.244 | 1.215 | 1.248 |
| apparent density, g/cm³ (without precalcination) | 1.016 | 0.915 | 1.022 | 0.891 | 0.739 | 0.848 |
| microstructure, score     | 1.5–2.2 |      |      |      |      |      |
| total porosity, %         | 23.70 | 28.05 | 15.77 | 28.37 | 39.19 | 32.08 |
slightly greater than the diameter, caused by thermobaric conditions of shale coke formation. The crystal sizes $L_c (27.430−29.651 \text{ Å})$ and $L_a (23.321−30.698 \text{ Å})$ are similar to the coking products of crude oil.

4. CONCLUSIONS

According to the results of the experimental studies on the coking of light and heavy shale pyrolysis tar, the following conclusions can be made:

- The coking of light tar produces more liquid products (59.07–63.24%) than heavy tar (49.22–57.54%), such that as the excess coking pressure increases from 0.15 to 0.35 MPa for both light and heavy pyrolysis tar shale coke. However, $d_{100}$ decreases from 2.168 to 2.113 Å in coke when the overpressure of light shale oil coke increases and, conversely, increases from 2.074 to 2.097 Å for coke from heavy pyrolysis shale tar.

- The results of determining the size of the crystallites show that at the same overpressure, the crystallites formed in coke are in most cases larger ($L_c$ and $L_a$) when coking heavy tar.

Table 9. Results of the Diffractometric Analysis of Coke

| no. of Exp. | $\theta$ (deg) | FWHM (deg) | $d_{002}$ (Å) | $L_c$ (Å) | $\theta$ (deg) | FWHM (deg) | $d_{100}$ (Å) | $L_a$ (Å) |
|------------|----------------|-------------|---------------|-----------|----------------|-------------|---------------|-----------|
| Ex. 1      | 25.540         | 2.940       | 3.484         | 27.430    | 41.620         | 6.560       | 2.168         | 26.492    |
| Ex. 2      | 25.500         | 2.720       | 3.490         | 29.651    | 41.960         | 7.460       | 2.151         | 23.321    |
| Ex. 3      | 25.320         | 2.820       | 3.514         | 28.587    | 42.760         | 6.220       | 2.074         | 28.048    |
| Ex. 4      | 25.540         | 2.900       | 3.485         | 27.809    | 43.600         | 5.700       | 2.074         | 30.698    |
| Ex. 5      | 25.440         | 2.780       | 3.498         | 29.007    | 43.220         | 6.120       | 2.091         | 28.552    |
| Ex. 6      | 25.380         | 2.776       | 3.506         | 29.003    | 43.100         | 6.118       | 2.097         | 28.540    |

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Figure 1. Microstructure of the obtained shale cokes obtained with a microimage at 90×–100× magnification: (a) Ex.1 and (b) Ex.3.

Figure 2. Comparison of diffractograms of shale oil coke.
Notes
The authors declare no competing financial interest.

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