Influence of parameters of delayed asphalt coking process on yield and quality of liquid and solid-phase products

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Paper studies the effect of excess pressure during delayed coking of asphalt, obtained by propane deasphaltization of tar, on yield and physical and chemical properties of hydrocarbon fuels' components and solid-phase product – petroleum coke. Asphalt was coked at a temperature of 500 °C and excess pressure of 0.15-0.35 MPa in a laboratory unit for delayed coking of periodic action. Physical and chemical properties of raw materials and components of light (gasoline), medium (light gasoil), and heavy (heavy gasoil) distillates obtained during experimental study were determined: density, viscosity, coking ability, sulfur content, iodine number, pour points, flash points, fluidity loss and fractional composition. Quantitative group hydrocarbon and microelement compositions and properties of obtained samples of petroleum coke (humidity, ash content, volatiles' yield, sulfur content, etc.) were also studied. Comparative assessment of their quality is given in accordance with requirements of GOST 22898-78 “Low-sulfur petroleum coke. Specifications”. In addition, patterns of changes in excess coking pressure on yield and quality indicators of distillate products and petroleum coke were revealed. With an increase in excess pressure of coking process from 0.15 to 0.35 MPa, content of paraffin-naphthenic hydrocarbons in light and heavy gasoils of delayed coking decreases. Common pattern in asphalt coking is an increase in yield of coke and hydrocarbon gas with an increase in excess pressure from 0.15 to 0.35 MPa.

Key words: delayed coking; asphalt; petroleum coke; gasoline; gasoils; fuels

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Introduction. Strategy for development of mineral resources base of the Russian Federation until 2035 (Decree of the Russian Federation Government dated December 22, 2018 N 2914-r) regarding the creation of new technologies for refining of oil, which belongs to second group of minerals significant for the country (according to quantity and quality of balance reserves of mineral raw materials in Russia), considers increasing the depth of oil refining and implementation of comprehensive modernization of oil refining industry. To do so, an increase in capacities for the processing of heavy petroleum raw materials will continue in Russia in the near future, the most marketable of which is delayed coking thermal process, which allows deepening of refining at the refinery to 98 % [7]. Total load of delayed coking plants for raw materials in Russia by 2020 will be about 13.6 million tons [9]. Using this process, it is possible not only to increase the volume of light (gasoline, kerosene, diesel) and dark (marine and boiler fuels) oil products, but also to expand the range of marketable products with carbon materials such as petroleum coke, pitch, coking additive and others [12]. Carbon materials – solid-phase products of delayed coking process – are widely used in metallurgy as raw materials for production of self-burning anodes and electrodes for electric steel smelting, as well as in the chemical industry as sorbents, and are used in other industries [4, 6].

Studying the influence of technological parameters of the delayed coking process (pressure, temperature, time, recirculation coefficient) depending on the quality of used raw materials
on its coking process will improve the yield and quality of the obtained liquid and solid products [1, 10].

I.R.Khairudinov et al. [5] investigated possibility of increasing the production of low-sulfur petroleum coke from tar, visbreaking residue, heavy gasoil of catalytic cracking and the extract of selective oil purification, obtained by processing a mixture of West Siberian oils at “Gazpromneft-ONPZ”, including effect of excess coking pressure (0.1 and 0.3 MPa) on the yield and quality of the liquid components of fuels and petroleum coke. Results of liquid and solid-phase coking products' yield obtained in a laboratory setup are shown in Table 1.

| Raw material components                  | Pressure, MPa | Products' yield, % mass | Time, h |
|------------------------------------------|---------------|-------------------------|---------|
|                                          | Coke | Distillate | Gas + losses |       |
| Tar                                      | 0.3  | 27.0       | 57.9         | 15.1   |
|                                          | 0.1  | 22.6       | 64.5         | 12.9   |
| Visbreaking residue                      | 0.3  | 28.2       | 58.2         | 13.1   |
|                                          | 0.1  | 24.1       | 65.5         | 10.4   |
| Heavy gasoil of catalytic cracking       | 0.3  | 26.1       | 59.3         | 14.6   |
|                                          | 0.1  | 21.8       | 68.9         | 9.3    |
| Extracts of selective oil purification   | 0.3  | 16.5       | 68.6         | 14.9   |
|                                          | 0.1  | 9.7        | 82.1         | 8.2    |

With an increase in excess pressure from 0.1 to 0.3 MPa, the coke yield increases by 4.1-4.4 % for tar, visbreaking residue and heavy gasoil of catalytic cracking and for extracts of selective oil purification by 6.8 %. Yield of distillates' amount, on the contrary, decreases: by 6.6 and 6.8 % for tar and visbreaking residue, by 9.6 and 13.5 % for heavy gasoil of catalytic cracking and extracts of selective oil purification. Accordingly, the gas yield increases (taking into account losses) by 2.2-6.7 %.

M.O.Andropov et al. [2] studied process of thermolysis for averaged M-40 fuel oil of West Siberian oil at a temperature of 420 and 450 °C, a pressure of 0.01 and 0.20 MPa and a thermolysis time of 3.5 to 14.0 h. Material balance of process regarding thermolytic decomposition of fuel oil was obtained under various conditions (Table 2).

| Products' yield during thermolysis of raw materials with obtaining of coke [2] |
|------------------------------------------|------------------|------------------|---------|
| Pressure, MPa | Temperature, °C | Coke | Distillate | Gas | Time, h |
| 0.01 | 420 | 8.28 | 76.67 | 15.05 | 12.0 |
|       | 450 | 7.40 | 77.18 | 15.42 | 10.0 |
| 0.20 | 420 | 10.06 | 74.71 | 15.23 | 14.0 |
|       | 450 | 9.56 | 73.71 | 16.73 | 13.5 |

Depending on duration of the process, temperature and pressure, a highly viscous liquid residue, pitch or petroleum coke can be extracted. To obtain coke at temperatures of 420 and 450 °C, as final solid-phase product, duration of thermolysis should be 10-12 hours at a pressure of 0.01 MPa and 13.5-14.0 hours at 0.20 MPa. An increase in the thermolysis pressure from 0.01 to 0.20 MPa at an equal process temperature leads to an increase in the yield of coke by 2.16 and 1.78 %, a decrease in the amount of distillates of 1.96 and 3.47 %, and also to an increase in the amount...
the resulting gases by 0.18 and 1.31 %, respectively, at 420 and 450 °C. An increase in temperature from 420 to 450 °C at constant pressure leads to an increase in the total yield of distillates and hydrocarbon gas and to a decrease in the yield of coke.

Work [3] presents studies on influence of oil residues types obtained at the Atyrau oil refinery: fuel oil, semi-tar and tar during coking, in a laboratory reactor on yield (Table 3) of distillate products and crude coke under excess pressure 0.20 MPa, coking time 2.0-2.5 hours, counted from the moment vapors appear in the receiver until the end of the experiment, carried out at a temperature of 460-470 °C, followed by a rise in temperature to 550-600 °C for coke drying and aging within 30 minutes. As a result of oil residues' coking, following products were obtained: crude coke 9.7-17.9 %, sum of coking distillates 72.9-80.0 %, and hydrocarbon gas about 7.9-8.9 %.

The purpose of this work is to establish the influence of excess pressure of the asphalt coking process of propane tar deasphaltization from a mixture of West Siberian oils on the yield and quality indicators of obtained distillate products and petroleum coke.

### Table 3

| Products of coking | Raw materials of coking |
|--------------------|------------------------|
|                    | Fuel oil | Semi-tar | Tar  |
| Wet gas            | 8.9      | 8.7      | 7.9  |
| Gasoline           | 15.4     | 14.7     | 13.1 |
| Light gasoil       | 39.6     | 37.5     | 38.6 |
| Heavy gasoil       | 25.0     | 23.4     | 21.2 |
| Crude coke         | 9.7      | 14.2     | 17.9 |
| Losses             | 1.7      | 1.5      | 1.3  |

**Methodology.** For experiment, asphalt obtained by process of propane tar deasphaltization from a mixture of West Siberian oils was used. Three parts of experiment were carried out on the coking of asphalt at a constant process pressure: first at 0.15 MPa, second at 0.25 MPa, third at 0.35 MPa. Temperature of coking process' end in all parts of experiment was 500 °C. Representative samples of petroleum coke and sum of distillates were obtained in each part of experiment; the latter were subsequently distilled at atmospheric pressure to gasoline fraction (IBP – 180 °C), light (180-340 °C) and heavy (340 °C – EBP) gasoil.

For experiment on coking of heavy petroleum raw materials (asphalt), a laboratory unit was used consisting of a reaction unit and a distillate collection unit. Reaction unit includes a steel coking reactor and an electric furnace with three independent heating zones to maintain a uniform temperature along the height of the coking layer; the reactor is equipped with a pressure gauge. The gas-liquid product mixture was discharged through a tube located in the reactor lid through a needle valve, from where it entered the pipe-in-pipe heat exchanger and the distillate collection flask, and hydrocarbon gas to the exhaust system. Installation configuration and the coking process are described in more detail in [8]. The feed load did not exceed 2/3 of the reactor volume.

Raw material of coking process – asphalt, was analyzed by following quality indicators: density, coking ability, Brookfield viscosity, content of ash, sulfur, trace elements, flash points and yield points, fractional composition and group hydrocarbon composition.

For light distillate products – gasoline fractions, the following were determined: density, iodine number, content of sulfur, hydrogen sulfide, fractional composition, kinematic viscosity, group hydrocarbon composition.

For middle distillate fractions – light coking gasoils: density, sulfur content, iodine number, hydrocarbon group composition, fraction composition, kinematic viscosity, pour point and flash point were determined.
Following physical and chemical properties were determined for heavy distillates (gasoils) of coking: density, coking ability, sulfur content, kinematic viscosity, flash points and fluidity loss, group hydrocarbon composition, fractional composition.

Analysis of group hydrocarbon composition of asphalt, as well as obtained medium and heavy distillates, was carried out on a “Gradient-M” chromatograph (JSC SUE “INHP RB”, Russia) with a thermal conductivity detector using liquid-adsorption chromatography on glass columns 30 cm high with diameter of 1.2-1.4 mm. Modified silica gel ASK was used as an adsorbent, and a complex mixture of solvents with a gradient-displacement feed mode was used as eluates. Studied objects are divided into following seven groups: paraffin-naphthenes, aromatics (light, medium and heavy), resins (I and II) and asphaltenes.

Physical and chemical properties of crude petroleum coke samples obtained during each of the three parts of experiment were investigated. Moisture, ash content, volatiles yield, porosity, actual and apparent density (without preliminary calcination), sulfur content and trace elements were determined.

Sulfur and trace elements in the obtained samples of crude petroleum coke were determined using X-ray fluorescence spectrometry without preliminary ashing of samples using the addition method on a sequential wave dispersive X-ray fluorescence spectrometer XRF-1800 Shimadzu (Center for Collective Use of Saint-Petersburg Mining University). Device is equipped with an x-ray tube with a copper anode of 2.7 kW power. Detailed method for determining sulfur and trace elements is shown in [11].

**Discussion.** Quality of coking raw materials was evaluated by indicators that directly or indirectly characterize its ability to be converted to petroleum coke during high-temperature thermolysis.

Asphalt quality indicators are as follows:

| Physical and chemical properties          | Values |
|-------------------------------------------|--------|
| Density at 20 °C, kg/m³                   | 1024.0 |
| Coking ability, %                         | 15.32  |
| Sulfur content, %                         | 1.60   |
| Brookfield viscosity at 80 °C, s          | 391    |
| Ash content, %                            | 0.18   |
| Flash point, °C                           | > 344  |
| Temperature of fluidity loss, °C          | 60     |

**Fractional composition, % volume:**

| Initial boiling point, °C                 | 457    |
| 5 % boils out at temperature             | 512    |
| 10 % boils out at temperature            | 532    |
| End boiling point, °C                     | 543    |
| Yield, % volume                          | 14     |

Indirectly assessing the tendency to coke formation of oil products is possible by values of density and viscosity indicators – the higher they are, the higher this indicator is. However, both its and physical and chemical indicators of coking raw materials' quality are determined by group hydrocarbon composition (Table 4). Density and viscosity of petroleum products increase in following hydrocarbon groups: paraffin-naphthenic, aromatics (light, medium, heavy), resins (I and II), asphaltenes.

Result of excess pressure influence during coking on the yield of obtained products is presented in Table 5. In the process of asphalt coking at a temperature of 500 °C, hydrocarbon gas, petroleum coke and sum of distillates were obtained, which was subsequently distilled at atmospheric pressure to gasoline fraction (IBP – 180 °C), light (180-340 °C) and heavy (340 °C – EBP) gasoil of coking.
Common pattern in the asphalt coking is an increase in the yield of coke and hydrocarbon gas with an increase in excess pressure from 0.15 to 0.35 MPa. With increasing pressure, evaporation degree of reactive molecules decreases, since self-evaporation slows down, their time being in liquid phase increases, which leads to a decrease in the yield of liquid products and an increase in the yield of coke. This pattern is caused by cracking reactions of resulting liquid distillates to hydrocarbon gases and thermopolycondensation reactions of polycyclic aromatic hydrocarbons to coke. In this case, accordingly, with an increase in coking pressure, yield of distillates amount decreases, namely, heavy gasoil.

Analysis of individual distillate fractions' yield showed that when coking pressure changes from 0.15 to 0.35 MPa, gasoline yield increases by 5.09 % (from original), light gasoil – by 5.78 %, and heavy gasoil decreases by 31.99 % (of its initial content at a coking pressure of 0.15 MPa).

Analysis of physical and chemical properties of gasoline, asphalt coking (table 6) showed that with an increase in excess pressure from 0.15 to 0.35 MPa, content of olefinic hydrocarbons decreases from 19.4 to 18.5 %, which confirms the value of the iodine number, characterizing the content of unsaturated bonds, which also decreases from 64.7 to 57.8 g I₂/100 g.

If analyzing results of quality indicators for light gasoils of tar coking and asphalt (table 6) obtained at different pressures, one can note some regularities: as pressure increases from 0.15 to 0.35 MPa, low-temperature properties of light gasoils of tar coking are improved: pour point decreases by 5 °C (to –29 °C). Sulfur content with increasing coking pressure slightly decreases for light coking gasoil. Values of viscosity and density pass through an extremum with a minimum at a pressure of 0.25 MPa. Iodine number passes through an extremum with a maximum at an excess pressure of asphalt coking process of 0.25 MPa.

With an increase in excess pressure of coking process from 0.15 to 0.35 MPa, content of paraffin-naphthenic hydrocarbons in light gasoil decreases from 60.5 to 57.6 % (see Table 4).
At the same time, quality indicators of light gasoil vary insignificantly, values of quality parameters for heavy gasoil of asphalt coking noticeably change (Table 6) in terms of group hydrocarbon composition and coking ability. All this is due to enhanced destruction of gasoil components in raw materials during a longer stay in coking zone (due to increased pressure), which is manifested in a sharp decrease in the yield of heavy gasoil (see Table 5) and an increase in content of aromatics in this fraction (see table 4).

As in the case of light gasoils, with an increase in excess coking pressure from 0.15 to 0.35 MPa, content of paraffin-naphthenic hydrocarbons decreases. Resin content practically does not change during coking (8.2-8.5 %) with a change in studied pressure range.

Results of a study considering physical and chemical properties of obtained samples of petroleum coke from asphalt at various process excess pressures are given in Table 7.

### Table 6

| Physical and chemical properties | Gasoline | Light gasoil | Heavy gasoil |
|----------------------------------|----------|--------------|--------------|
| Process pressure, MPa           | 0.15     | 0.25         | 0.35         |
| Density at 20°C, kg/m³          | 768.1    | 755.7        | 756.9        |
| Conradson Coking, % mass        | –        | –            | –            |
| Content of total sulfur, %      | 0.41     | 0.41         | 0.41         |
| Content of hydrogen sulfide, %  | –        | 0.0020       | –            |
| Iodine number, g I₂/100 g      | 64.7     | 65.0         | 57.8         |
| Viscosity at 20 °C, mm²/s      | 0.88     | 0.88         | 0.91         |
| Viscosity at 100 °C, mm²/s     | –        | –            | –            |
| Pour point, °C                  | –        | –            | –            |
| Temperature of fluidity loss, °C| –        | –            | –            |
| Flash point, °C                 | –        | –            | –            |
| Fractional composition, % volume: |       |              |              |
| Initial boiling point, °C       | 69       | 71           | 70           |
| 5 %                              | 88       | 92           | 91           |
| 10 %                             | 105      | 109          | 107          |
| 50 %                             | 137      | 132          | 132          |
| 90 %                             | 181      | 182          | 185          |
| 95 %                             | 195      | 191          | 193          |
| End boiling point, °C           | 208      | 207          | 210          |

### Table 7

| Physical and chemical properties | Pressure, MPa (exc.) |
|----------------------------------|----------------------|
|                                  | 0.15  | 0.25  | 0.35  |
| Humidity, %                     | 0.90  | 0.95  | 1.0   |
| Volatiles' yield, %             | 4.8   | 4.4   | 4.1   |
| Ash content, %                  | 0.44  | 0.43  | 0.45  |
| Actual density, g/cm³           | 1.61  | 1.59  | 1.73  |
| Apparent density, g/cm³         | 0.75  | 0.84  | 0.83  |
| Total porosity, %               | 53.0  | 47.0  | 52.0  |
| Microstructure, points          | 2.5   | 2.6   | 2.6   |
| Vanadium content, ppm           | 210   | 190   | 190   |
| Sulfur content, %               | 1.80  | 1.76  | 1.72  |

Moisture content in petroleum coke (Table 7) obtained from asphalt at a pressure of 0.15; 0.25 and 0.35 MPa, is 0.90; 0.95 and 1.0 %, respectively. Volatiles' yield for petroleum coke is in the
range of 4.1-4.8 % mass. Ash content is not more than 0.45 %. Vanadium content from 190 to 210 ppm shows that this coke can be used as raw material for its extraction. A separate work [14] is devoted to this process. Obtained values of humidity, volatiles' yield and ash content meet the requirements of GOST 22898-78 “Low-sulfur petroleum cokes. Specifications”: humidity ≤ 3.0 %, volatiles' yield ≤ 6-8 % and ash content ≤ 0.30-0.80 %.

Conclusion. Obtained data show that with directed coking process of heavy petroleum raw materials (using asphalt as an example), composition and quality of light, medium and heavy distillate products can be controlled in pressure range from 0.15 to 0.35 MPa.

With intended purpose of coking process for production of medium and heavy distillates, for example, to use them as components for marine fuels, it is rational to conduct process at a lower pressure (in studied interval it is 0.15 MPa). At this pressure, yield of heavy asphalt coking gasoil was 21.87 %. However, tendency to foaming significantly limits possibility of reducing the pressure in industrial process of delayed coking, therefore, in industrial implementation, it is necessary to control degree of coke chamber's filling in order to avoid transfer of foaming mass to fractionation column.

Analysis results of yield and quality of petroleum cokes from asphalt of a mixture of West Siberian sulphurous oils processed at Russian refineries confirmed the possibility of improving these parameters by increasing the pressure in delayed coking plants to 0.35 MPa. High sulfur content (1.72-1.80 %) does not allow classification of obtained coke samples in accordance with GOST 22898-78 “Low-sulfur petroleum cokes. Specifications”. There is a high probability of non-compliance with the standard for low-sulfur electrode cokes even after calcining at 1100 °C, at which sulfur content decreases up to 50 % of its content in raw coke. However, sulfur coke from asphalt can be used, for example, as fuel in production of Portland cement, since permissible content of sulfur oxide (IV) in it should be from 1 to 4 %.

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