



## UPGRADING OF HEAVY CRUDE OIL IN SUPERCRITICAL AQUEOUS FLUID IN THE PRESENCE OF ACTIVATED CHARCOAL

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### Abstract

The article presents the results of deep processing of heavy crude oil in supercritical aqueous fluid, which makes it possible to significantly reduce the content of sulfur and resinous asphaltene compounds in products, and to increase the yield of light fuel fractions. The possibility of reducing the temperature of upgrading of heavy crude oil due to the presence of active charcoal in the reaction medium is shown. The proposed technology provides environmentally safe and residue-free processing of heavy oil and further production of high-quality hydrocarbon raw materials enriched in low-boiling fractions.

### Keywords:

Upgrading;  
Heavy crude oil;  
Supercritical aqueous fluid;  
Activated charcoal.

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### 1. Introduction

Intensive consumption of conventional oil as the main hydrocarbon feedstock by our civilization has led to a significant reduction of their reserves on our planet. To date, an increase in the share of heavy oil (HO) is observed in the produced oil balance. The key factor complicating the transportation and further processing of heavy crude oil into light oil products is the high content of high-molecular and heteroatomic compounds, resins and asphaltenes, which tend to form coke at high temperatures and reduce the catalytic activity of catalysts. Huge reserves of heavy hydrocarbon resources in the world, as well as the desire to increase their processing depth at oil refineries, lead to the need to create new, more efficient technologies for their primary upgrading. In oil companies, heavy crude oil is upgraded into «synthetic» oil using expensive hydrogen and catalysts, and then converted oil is further refined into light oil products or into valuable petrochemical feedstock using standard technologies. The use of hydrogen and expensive catalysts for upgrading of heavy crude oil is not an efficient solution, since it leads to an increase in the cost of technology, as well as requires a complex catalyst regeneration stage. In this regard, re-search aimed at creating upgraded heavy crude oil, without hydrogen based technologies, focused on the destruction

of large molecules of hydrocarbons, resins and asphaltenes with the formation of low molecular weight compounds is of particular interest [1–6]. The first experimental studies on high-temperature transformations of petroleum hydrocarbons in the presence of water vapor and minerals with developed surface area were carried out in the late 1970s and early 1980s [7, 8]. Catalytic aquathermolysis at elevated temperatures is an attractive and environmentally friendly method of improving the composition and rheological properties of heavy crude oil [9,10]. The participation of catalysts based on transition metals is one of the most common solutions in improving the aquathermolysis process [4,11–13]. At the same time, the separation of these catalysts from the target product of aquathermolysis is difficult, which increases the cost, and negatively affects the subsequent stages of oil refining [14–16].

In recent years, significant progress has been made in the theoretical evaluation of the dissolution of petroleum hydrocarbons in water near its critical point (374 °C). Among the variety of processing methods, supercritical aqueous fluids are gaining popularity due to their ability to convert heavy oil components into valuable, clean hydrocarbon feedstock by suppressing coke formation and removing heteroatomic compounds [17]. In an effort to develop innovative technologies for the processing of heavy oil, corresponding to the principles of «green chemistry», research in the field of environmentally friendly supercritical aqueous fluids is gaining an increasing scale [18], specifically

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for upgrading and processing of heavy crude oil [19]. At supercritical temperatures, water becomes a solvent for gases formed during upgrading of heavy oil, including lower and higher hydrocarbons of liquid products [20]. The influence of the solubility of supercritical water on asphaltenes was investigated in [21,22]. An important aspect in the upgrading of heavy oil in super-critical aqueous fluid is the destruction of asphaltenes [23]. The formation of hydrogen and its role in the aquathermolysis of oil at supercritical water conditions was studied in the article [24]. Another work [25] highlights the results of heavy oil upgrading in supercritical water, in particular, the hydrogenation of unsaturated bonds of aliphatic hydrocarbons, being products of thermal decomposition of petroleum hydrocarbons.

The present paper proposes a solution to the problem of implication of expensive hydrogen and catalysts in the processing of heavy oil by using hydrothermal fluid in the supercritical state in the presence of activated charcoal capable of providing the reaction medium with a hydrogen proton. For the first time, the work revealed the distinctive features of changes in the composition and properties of liquid products of upgraded heavy oil in the medium of hydrothermal aqueous fluid at a temperature of 420 °C and a pressure of 24 MPa, as well as in the presence of activated charcoal at 375 °C and 23 MPa. It has been established that the formation of light fractions during conversion of heavy oil in the hydrothermal medium in the presence of activated charcoal occurs due to the destruction of sulfur-containing and resinous-asphaltene components.

## 2. Experimental

First object of study was heavy crude oil from the Ashalchinsky field, produced from terrigenous sediments, located on the western slope of the South Tatar arch of Volga-Ural oil and gas basin. Heavy oil is characterized by high values of density (0.9715 g/cm<sup>3</sup> at 20 °C) and viscosity (2771 MPa·s), high content of sulfur (2.8 wt.%), naphthenic and aromatic compounds (40.8 wt.%), polyaromatic compounds (13.7 wt.%), resins (37.7 wt.%) and asphaltenes (7.8 wt.%), insignificant content of solid paraffinic hydrocarbons (0.5 wt.%).

Second object of study was liquid products of heavy crude oil upgraded in hydrothermal medium, third – oil upgraded in the presence of activated charcoal of the BAU-A grade (GOST 6217-74. Wood crushed activated carbon. Specifications), consisting of up to two-thirds of amorphous carbon and graphite crystallites formed by packs of hexagonal rings. An increase in the adsorption properties of charcoal was achieved by its activation, due to its preliminary impregnation with calcium carbonate, and subsequent heat treatment of the coal at 800 °C. Activated charcoal is a fairly reactive compound capable of oxidation by water vapor [26–28]. Activated charcoal was used with particle size of 294-638 nm, specific surface area – more than 1000 m<sup>2</sup>/g and a pore diameter – more than 50 nm.

Experiments on upgrading of heavy crude oil were carried out in a closed heated tubular batch reactor (autoclave) with a volume of 230 cm<sup>3</sup>, equipped with thermocouples, manometers, fittings for feedstock input and products withdrawal, at temperatures and pressures close to the critical point of water (375 °C, 22.1 MPa). Before the experiment, the initial reaction mixture was prepared, which presented oil-water emulsion. In the first experiment (1), the ratio of heavy crude to water in the initial mixture was 2:1. When the re-actor reached a temperature of 420 °C, the pressure was 24 MPa. In order to reduce the upgrading temperature of heavy crude oil, due to the adsorption of high-molecular compounds on a developed specific surface, activated charcoal was introduced into the initial mixture in the second experiment (2) in an amount of 4 wt.% to oil. Within heavy crude oil to water ratio of 2:1 of the second experiment, it resulted in the following experimental conditions: pressure – 3 MPa, upgrading temperature – 375 °C. In summary, the temperature and pressure in the first experiment were 420 °C and 24 MPa, in the second experiment – 375 °C and 23 MPa, respectively. The prepared initial mixture was loaded into the autoclave through the upper fitting, hermetically sealed, and the temperature was increased to a predetermined value, while the pressure in the closed system increased simultaneously with an increase in temperature. The duration of the experiment at a given temperature was 40 minutes. At the end of the experiment, the heating was turned off, and as the reactor reached room temperature, the conversion products were removed. The resulting liquid product was first subjected to settling in a separating funnel to remove the contained water, then analyzed. The content of the low-boiling fraction (i.b.p.-200 °C) in the initial oil and liquid products of the experiments was determined by distillation.

Determination of the component composition of the stripped oil (i.b.p.-200 °C fraction preliminarily distilled off) of the initial and converted oil was carried out by precipitating asphaltenes from them with a 40-fold volume of 40-70 °C petroleum ether and, then separating the deasphalted oil into oils and resins by column liquid-adsorption chromatography on GASK silica gel. For the analysis of resinous-asphaltene compounds, ASK silica gel (GOST 3956-76. Silica gel for industrial use. Specifications) of 0.25-0.5 mm fraction was used, which was preliminarily dried at 150°C for 8 hours. As a hydrocarbon (HC) desorbent, a mixture of petroleum ether (85 vol.%) and carbon tetrachloride (15 vol.%) was used. Resinous compounds were isolated with an alcohol-benzene mixture: benzene and isopropyl alcohol in a ratio of 1:1 by volume.

The content of sulfur and nitrogen in the initial heavy oil and conversion products was determined by burning 0.1 g weighed portions on a CHN-3 analyzer in a glass tube filled with copper at 1000 °C.

Elemental analysis of charcoal before and after the experiment was carried out on a RigakuZSXPrimus II X-ray fluorescence spectrometer.

IR spectra was recorded by Vector-22 spectrometer (Bruker) in 2000-400  $\text{cm}^{-1}$  range with a resolution of 4  $\text{cm}^{-1}$ . To study the structural group composition, spectral coefficients were used, defined as the ratio of the optical density values at the peaks of the corresponding absorption bands:  $C_1 = D1600/D720$  (aromaticity);  $C_2 = D1710/D1465$  (oxidation);  $C_3 = D1380/D1465$  (branching);  $C_4 = (D720 + D1380) / D1600$  (wax content);  $C_5 = D1030/D1465$  (sulfur content).

The molecular weight distribution of n-alkanes in the saturated part (HC) of the oil was determined by gas chromatography/mass spectrometry (GC/MS) on a PerkinElmer-Claruss 680 gas chromatograph with an SQ8 MS mass spectrometric detector, capillary column, in the temperature programming mode from 70 to 270  $^{\circ}\text{C}$ .

High molecular weight components – heavy oil asphaltenes and activated charcoal before and after experiments were investigated on a powder X-ray diffractometer from BrukerAxs with a Bragg-Brentano shooting geometry in the  $2\theta$  angle range from 2 to 70 $^{\circ}$ . Screening step – 0.04 $^{\circ}$ , exposure time – 2 s/step. During the shooting, a standard disk polymethyl methacrylate cell containing the sample was rotated in its own plane at 30 rpm speed. The phase composition of the samples was determined using the DIFFRAC.EVA software module and diffraction database.

The data on the micrograph of the surface of the initial activated charcoal and solid products of the experiments were obtained on an electron microscope «SEM TM-1000», Hitachi with microanalysis system.

The study performed using the equipment of the Center for Collective Use «Nano-materials and Nanotechnology» of the Kazan National Research Technological University.

### 3. Results and discussion

The process of hydrothermal upgrading of heavy oil in the supercritical aqueous fluid leads to an increase in the yield of the low-boiling fraction (i.b.p.-200  $^{\circ}\text{C}$ ), as well as saturated and aromatic hydrocarbons (HC) (fig. 1 and 2). The use of activated charcoal allows to reduce the heavy crude oil upgrading temperature from 420 to 375  $^{\circ}\text{C}$  (therefore, brings the process temperature closer to the critical point of water). For liquid products of upgrading in superheated steam under thermodynamic conditions close to the critical point of water in the presence of activated charcoal, a high content of hydrocarbons (HC) and a low content of asphaltenes are also characteristic.

The converted oils differ from the initial heavy oil by a large, 2.3-fold, yield of gasoline fraction (i.b.p.-200  $^{\circ}\text{C}$ ). The amount of asphaltenes in converted oil in the presence of charcoal decreases from 7.7 to 1.6 wt.%, resin content – from 37.8 to 22.3 wt.%. It can be assumed that asphaltenes are adsorbed on the active surface of charcoal, which has a large number of basic and acidic groups. When asphaltenes are adsorbed on the surface of activated charcoal, C – C bond energies in their molecules decrease, which

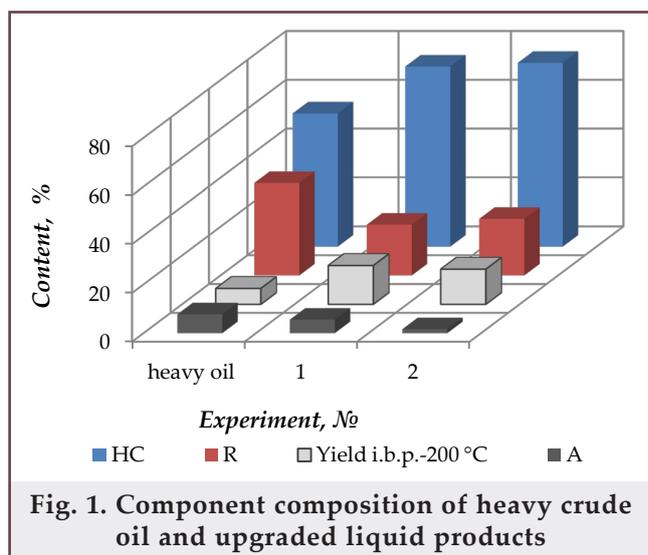


Fig. 1. Component composition of heavy crude oil and upgraded liquid products

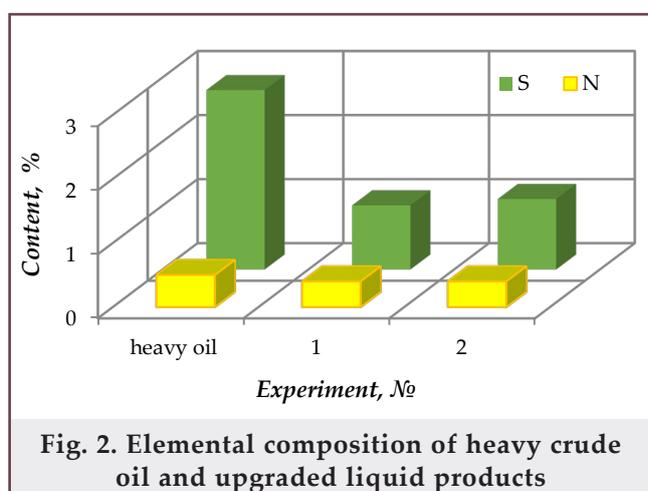


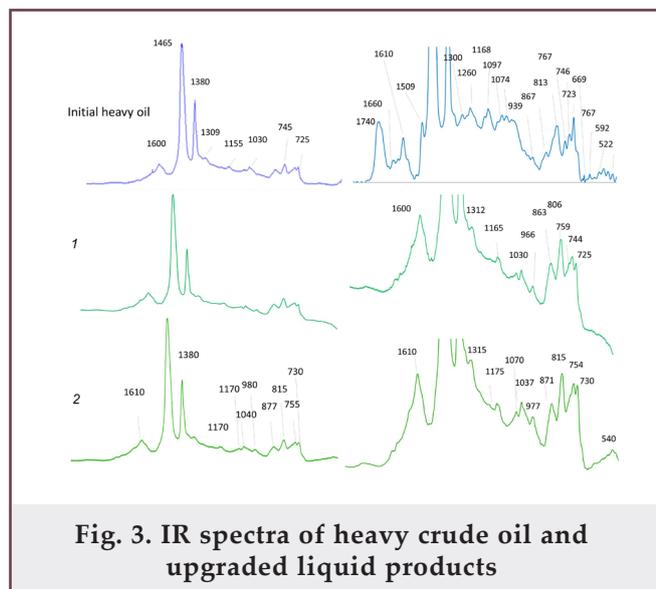
Fig. 2. Elemental composition of heavy crude oil and upgraded liquid products

leads to a decrease in their cracking temperature. As a result, the yield of the HC fraction (saturated and aromatic hydrocarbons) in oil increases from 54.5 to 73.7 wt.% and up to 75.2 wt.% during hydrothermal conversion in the presence of charcoal.

Similar to the composition, the converted oil viscosity also decreases quite strongly (figs.1 and 2). The most significant decrease in the viscosity of converted oil in the presence of activated charcoal from 2771 to 682 MPa·s is observed.

The IR spectra of heavy crude oil and upgraded oil products show intensive absorption bands characteristic for methyl (1380  $\text{cm}^{-1}$ ) and methylene (1465  $\text{cm}^{-1}$ ) groups, an absorption band with a maximum at 725  $\text{cm}^{-1}$ , reflecting the presence of long methylene chains ( $\text{CH}_2 > 4$ ), and C = C in the aromatic ring (1600  $\text{cm}^{-1}$ ) (fig. 3). A pronounced triplet of absorption bands at 900-730  $\text{cm}^{-1}$  is observed, reflecting the C = C bond of the aromatic ring with alkyl substituents. The diverse nature of the intensities of large and small peaks in this region indicates differences in the composition of polyaromatic structures in the samples.

The similarity of the IR spectra of liquid products of experiments (1) and (2) indicates that activated charcoal does not initiate new reactions leading to new products (low selectivity), however, it significantly accelerates the ongoing reactions. A narrow band at 1509  $\text{cm}^{-1}$  disappears in the converted oil spectra,



**Fig. 3. IR spectra of heavy crude oil and upgraded liquid products**

which indicates the presence of nitro-gen-containing heterocyclic compounds in the original oil. At the same time, in the spectrum of the initial oil, in contrast to converted oil, peaks in the region of 1200-1100  $\text{cm}^{-1}$  are observed, corresponding to vibrations of oxygen-containing groups. A significant peak of the absorption band at 1740  $\text{cm}^{-1}$  reflects the presence of a non-conjugated carbonyl group. In the products spectra, these peaks appear less intensely, however, the presence of an absorption band in the 1030  $\text{cm}^{-1}$  region is noticeable, characterizing the presence of a sulfoxide group (S = O).

The heavy oil converted in the presence of activated charcoal during hydrothermal upgrading near the critical point of water (375 °C) in terms of the component composition and molecular-mass distribution of n-alkanes is comparable to oil produced by upgrading in supercritical aqueous fluid at 420 °C. Analysis of gas chromatographic data showed a similar character of the molecular-mass distribution of n-alkanes of  $\text{C}_{10}$ - $\text{C}_{38}$  series in HC of converted oils (fig. 3). However, there are some differences in the content of n-alkanes in HC converted oil at 420 °C (exp. 1) and at 375 °C (exp. 2), which indicates a possible redistribution of the original heavy oil components in the pore space of activated coal, depending on pore diameter and

sorption properties.

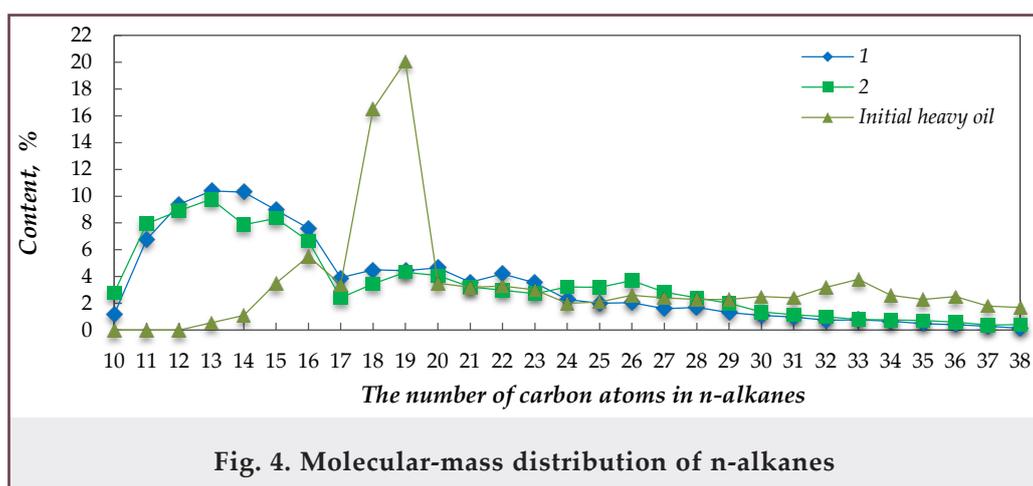
The highest concentrations in the initial heavy oil are found for n-alkanes of  $\text{C}_{19}$ - $\text{C}_{20}$  composition, and in HC of the converted oils – for low-molecular n-alkanes of  $\text{C}_{11}$ - $\text{C}_{16}$  composition (fig. 4). In HC of converted oils relative to the original heavy oil, the content of n-alkanes of the composition  $\text{C}_{30}$ - $\text{C}_{38}$  is reduced. A distinctive feature in the composition of n-alkanes of converted oil HC in the presence of active charcoal is a slight increase in the content of their high-molecular homologues of  $\text{C}_{24}$ - $\text{C}_{27}$  composition.

According to X-ray structural analysis data, the initial active charcoal consists of amorphous carbon and crystallites with a size of 1.1 nm (table 1), which enables to assign it to the microcrystalline carbon group. The crystalline structure of activated charcoal comprises 97.3% of carbon, the rest is calcite (2.7%). In the diffraction patterns before and after the experiments, the ratios of the diffraction maxima from the crystallographic planes for the anthracite phase slightly changes, which may be the result of the destruction of the activated charcoal surface.

The diffractogram of the converted oil asphaltenes in the course of experiment (2) regularly demonstrates the presence of an active charcoal phase with a smaller crystallite size 0.9 nm, while the lattice parameters remain constant. The adsorption of amorphous carbon, being part of asphaltenes, on activated carbon after hydrothermal treatment is observed.

According to the data of X-ray fluorescence analysis of charcoal before and after the experiment (table 2), the ratio of the concentrations of elements is significantly different: the content of sulfur (S) increases from 0.06 to 6.66%, Si to 0.42%, iron (Fe) from 0.01 to 0.19%. A feature of charcoal after the experiment (2) is the content of valuable elements vanadium and nickel, 0.11 and 0.05%, respectively. It can be assumed that the processes of sorption on the developed surface of charcoal metals contained in heavy oil are predominantly in asphaltenes due to the destruction of the latter.

In the composition of charcoal, after the experiment, the content of calcium (Ca) decreases from 0.76 to 0.42% and potassium (K) from 0.48



**Fig. 4. Molecular-mass distribution of n-alkanes**

Table 1

## Parameters of the crystal lattice and size of crystallites in initial activated charcoal and asphaltenes of converted oil during the experiment (2)

Experiment	Phase	Content, %	Lattice parameters				Crystallite size, nm
			a (Å)	b (Å)	c (Å)	beta (°)	
Initial activated charcoal	Calcite	2.7	5.022	-	17.239	-	22.1
	Anthracite	97.3	8.506	6.608	11.215	124.023	1.1
Charcoal after the exp. (2)	Anthracite	100	8.753	6.528	11.430	123.844	0.9

Table 2

## Elemental composition of charcoal according to X-ray fluorescence analysis

Experiment	C	S	Ca	Si	Mg	Fe	K	Mn	Al	P	Zn	Cl	V	Ni	Na	Cu	Br
Initial activated charcoal	98.33	0.06	0.76	0.03	0.14	0.01	0.48	0.06	0.02	0.07	0.02	0.02	0	0	0	0	0
Charcoal after the exp. (2)	91.43	6.66	0.42	0.42	0.29	0.19	0.13	0.03	0.10	0.05	0.05	0.03	0.11	0.05	0.02	0.01	0.01

to 0.13%, which also indicates the occurrence of leaching processes in supercritical aqueous fluid.

During hydrothermal treatment of heavy oil, changes occurring on the surface of the activated charcoal are observed (fig. 5). On the surface of the original coal, small light areas of crystalline accumulations are noticeable: according to X-ray diffraction analysis, the phase of calcite used to activate the coal with calcium carbonate.

Hydrothermal treatment near the critical point of water leads to the transformation of the initial

structure of the coal surface in the direction of crushing the relief. On the surface of the coal after the experiment, coke-like products - carbonaceous substances are present, which indicates the ongoing processes of chemisorption of asphaltenes on the surface of the activated carbon.

The data obtained indicates the prospects for the use of activated charcoal during hydrothermal primary processing of heavy oil and can be used to select optimal parameters or during adaptation of existing technologies for their deep processing.

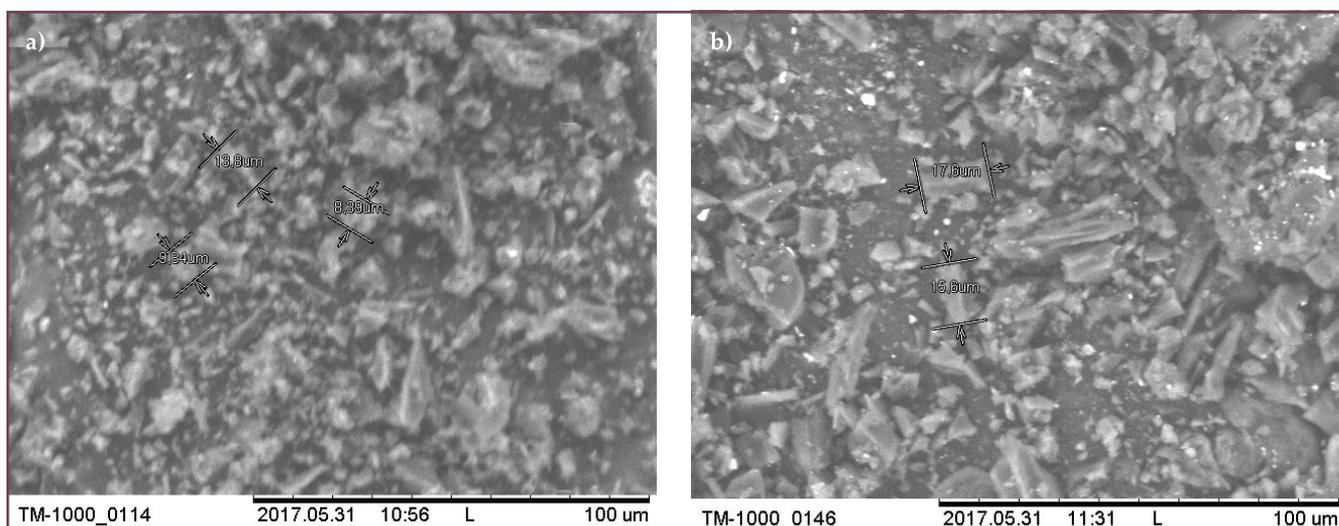


Fig. 5. Surface morphology of activated charcoal before (a) and after experiment (2) (b)

### Conclusion

1. An increase in the temperature and pressure of the superheated steam to the state of a supercritical aqueous fluid ( $T > 375\text{ }^{\circ}\text{C}$ ,  $P > 22.1\text{ MPa}$ ) in a closed system leads to the highest yield of light fractions (i.b.p.- $200\text{ }^{\circ}\text{C}$ ) and hydrocarbon components of oil (HC), desulfurization (the sulfur content is reduced from 2.8 to 1 wt.%) and a decrease in viscosity by more than 80% compared to the original heavy oil due to the destruction of resinous-asphaltene components.

2. The optimal conditions for hydrothermal upgrading of heavy oil in the presence of charcoal have been identified. The use of activated charcoal for hydrothermal upgrading of heavy crude oil near the critical point of water resulted in significant reduction of the process temperature from 420 to  $375\text{ }^{\circ}\text{C}$ , while obtaining hydro-carbon feedstock with a lower viscosity, with a low content of resins and asphaltenes, enriched in low-boiling fractions.

3. The research carried made it possible to consider super-critical fluid technology as an efficient way of primary processing of extra heavy oil.

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## Преобразование тяжелой нефти в сверхкритическом водном флюиде в присутствии активного угля

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### Реферат

В статье представлены результаты глубокой переработки тяжелой нефти в сверхкритическом водном флюиде, которая позволяет значительно снизить содержание в продуктах серы и смолистых асфальтеновых соединений, а также увеличить выход легких топливных фракций. Показана возможность снижения температуры облагораживания тяжелой нефти за счет присутствия в реакционной среде активного угля. Предлагаемая технология обеспечивает экологически безопасную безостаточную переработку тяжелой нефти и дальнейшее получение высококачественного углеводородного сырья, обогащенного низкокипящими фракциями.

**Ключевые слова:** превращение; тяжелая нефть; сверхкритический водный флюид; активный уголь.

## Aktiv kömürün iştiraki ilə yüksək kritik su flüidində ağır neftin dəyişməsi

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### Xülasə

Məqalədə yüksək kritik su flüidində məhsulun tərkibindəki kükürd və asfalten-qətran birləşmələrinin miqdarının əhəmiyyətli dərəcədə azalmasına, həmçinin yüngül yanacaq fraksiyalarının ayrılmasının artmasına imkan verən ağır neftin dərindən emalının nəticələri təqdim edilmişdir. Reaksiya mühitində aktiv kömürün iştirakı hesabına ağır neftin təmizlənmə temperaturunun azaldılmasının mümkünlüyü göstərilmişdir. Təklif olunan texnologiya ağır neftin ekoloji cəhətdən təhlükəsiz qalıqsız emalını və aşağı temperaturda qaynayan fraksiyalarla zənginləşdirilmiş yüksək keyfiyyətli karbohidrogen xammalının əldə edilməsini təmin edir.

**Açar sözlər:** dəyişmə; ağır neft; yüksək kritik su flüidi; aktiv kömür.