

## Thermocatalytic Cracking of the Natural Bitumens of Kazakhstan

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**Abstract**—The heat treatment of the natural bitumens of Kazakhstan was carried out in the presence of a catalyst of power-generating ash microspheres and di-*tert*-butyl peroxide. Changes in the substantial and fractional compositions of cracking products depending on process conditions were established. Characteristic differences in the composition of liquid cracking products in the presence of the catalyst and the additive from the cracking products of the initial bitumen were found.

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The basic trends in the development of current oil-refining industry are caused by the need for increasing the depth of petroleum conversion and by more rigid ecological requirements imposed on oil-refining processes and products [1]. The international oil-refining industry is currently characterized by a decrease in light oil reserves and an increase in the fraction of the production and processing of heavy petroleum residues and oil-bituminous rocks. Because of a decrease in the production and an increase in the prime cost of light oils, the production of raw materials for the manufacture of petroleum products becomes a problem of increasing current interest [2].

The processing of oil-bituminous rocks, which should be considered as a source of natural bitumens, promising hydrocarbon raw materials, is a developing branch of petroleum refining. One of the most important problems related to the processing of natural bitumens is a high concentration of high-molecular-weight compounds (tars and the asphaltenes, whose molecules concentrated a large part of heteroatoms present in the source material) in these bitumens. The amount of tars and asphaltenes is responsible for the properties of both a dispersion medium and a dispersed phase and the aggregative stability of natural bitumens under the conditions of thermolysis [3]. These compounds have high molecular weights; they are prone to condensation and coke formation upon processing, and they deactivate catalysts. The development of methods for the deep destruction of the

tar—asphaltene components of heavy oils and natural bitumens will make it possible to solve the main problem of the processing of heavy hydrocarbon raw materials and will reduce the scarcity of hydrocarbon fuel in the future.

The thermal destruction processes of heavy hydrocarbon raw materials make it possible to increase the yield of low-boiling liquid products with the formation of coke and gas as by-products. Cracking processes in the presence of different catalysts are of special interest. The thermocatalytic conversion of heavy hydrocarbon raw materials with iron oxide additives is a promising method for the production of synthetic oil. Microspheres, which can initiate the deep destruction of high-molecular-weight components, exhibited high efficiency in the processes of cracking [4, 5]. According to patent data, it is well known that the catalysts based on iron oxides, both synthetic and of technogenic or ore origin, exhibit activity in the processes of the steam cracking and hydrocracking of crude heavy oils [6].

The aim of this work was to perform the thermocatalytic cracking of the natural bitumens of Kazakhstan under varied conditions and to determine the group and fractional composition of the cracking products.

### EXPERIMENTAL

The samples of oil-bituminous rocks from the Munaily-Mola and Beke deposits (Kazakhstan) were

**Table 1.** Physicochemical characteristics of the initial bitumens

Characteristic	Bitumen	
	Beke	Munaily-Mola
Concentration in rock, wt %	12.1	16.0
Density, kg/m <sup>3</sup>	1112.2	992.1
Coking capacity, wt %	30.1	35.0
Elemental composition, wt %:		
C	84.79	84.69
H	11.68	11.39
O	1.45	2.12
S	1.50	1.42
N	0.58	0.38
H/C	0.14	0.13
Solidification point, °C	18.0	16.0
Bp, °C	116.8	96.5
Component composition, wt %:		
Oils (hydrocarbons)	49.17	47.58
Silica gel tars	44.89	46.37
Asphaltenes	5.94	6.05
Fractional composition, wt %:		
bp – 200 °C	5.1	2.2
bp 200 – 360 °C	20.2	15.6
> 360 °C	74.7	82.2

used as test materials. The extraction of natural bitumen was performed in a Soxhlet apparatus with chloroform as a solvent. The amounts of natural bitumen extracted from the oil-bituminous rocks of the Beke and Munaily-Mola deposits were 12 and 16 wt %, respectively (Table 1). The residual concentrations of organic matter in the rocks of the Beke and Munaily-Mola deposits were 0.05 and 0.08 wt %, respectively. The organic matter of the oil-bituminous rocks belongs to heavy oils in terms of density. The sulfur contents of the organic matter of both of the rocks are almost identical: 1.5 and 1.42 wt %, respectively. Both of the natural bitumens are characterized by a low content of the fractions that boil away to 200 and 350 °C. The organic matter contains a large amount of solid paraffins and tars (>40%).

The figure schematically shows the experiment on the cracking of bitumen and the analysis of the obtained products.

The cracking of bitumen was carried out in 12-cm<sup>3</sup> autoclave reactors; the weight of a bitumen sample was 7 g. According to the results of previous studies [7, 8], the temperature of the heat treatment of bitumens for 60 min was 450 °C (at a cracking duration shorter than 60 min, the required degree of conversion of high-molecular-weight components was not reached and

the bitumens cannot be heated for a longer time because of extremely high coking characteristics). In the course of the experiment, the weight of a reactor without a sample and the weight of the reactor with a sample prepared for the experiment were measured. After conducting the heat treatment of heavy oil, the yield of gaseous products was determined based on the weight loss of the reactor with the sample after the removal of gaseous products from the reactor. The sampled gaseous products were analyzed on a gas chromatograph. After the removal of liquid products, the reactor was washed with chloroform and weighed. The obtained difference between the reactor weights before and after the experiment was referred to as coke.

The narrow fractions of magnetic microspheres, which were isolated from fly ash after the combustion of coals at heat and power plants by a set of separation and hydrodynamic and granulometric classification processes, were used as an initiator of the cracking of bitumens [9]. The samples of magnetic microspheres were separated under laboratory conditions from magnetic concentrates obtained upon the dry magnetic separation of fly ash from the combustion of powdered brown coal of the Berezovo section of the Kansk-

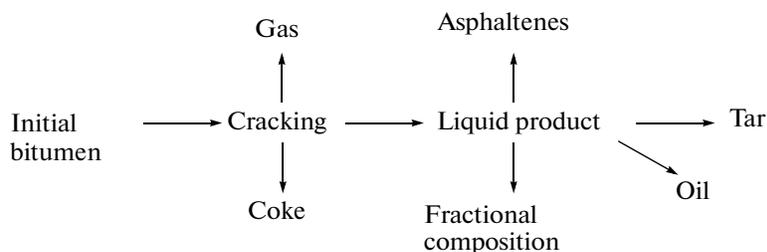


Diagram of the experiment

Achinsk Basin at Berezovo GRES-1 (State Regional Electric Power Plant) [10]. Table 2 summarizes the chemical composition of the investigated fractions of magnetic microspheres. The catalytic properties of microspheres are determined by the oxide forms of iron [9]; based on published data [11], iron compounds are converted from a catalytically inactive magnetite phase into an active hematite phase upon thermal treatment. Therefore, for the subsequent experiments, the microspheres were preliminarily calcined in air at 800°C for 2 h. Table 3 summarizes the composition and the concentrations of crystalline phases in the calcined samples. The diffractograms of the samples were measured on an X'Pert Pro MRD PANalytical powder X-ray diffractometer with the use of  $\text{CuK}\alpha$  radiation. It was experimentally established [5, 12, 13] that an optimum quantity of microspheres for an increase in the degree of destruction of the tar–asphaltene components of heavy hydrocarbon raw materials is 10 wt %.

Because the amount of liquid product obtained in the experiments on the thermal degradation of bitumens was small, it was impossible to analyze the fractional composition of liquid products by a standard procedure (GOST [State Standard] 2177-99, method B, or GOST 11011-85). Therefore, the concentrations of light fractions in the above products were evaluated based on thermogravimetric analysis data. Thermogravimetric analysis was carried out in an atmosphere of air on a MOM derivatograph (Hungary), which makes it possible to measure the weight loss of an analytical test sample with increasing the temperature to 350°C at a heating rate of 10 K/min.

The gaseous hydrocarbons formed upon the cracking of bitumens were analyzed by gas chromatography on a Khromatron chromatograph. The method is

based on a combination of gas–liquid and gas-adsorption chromatography with the use of a thermal conductivity detector [14]. The  $\text{C}_1$ – $\text{C}_6$  hydrocarbons were separated by gas–liquid chromatography, and the nonhydrocarbon components (hydrogen, nitrogen, and oxygen), by gas-adsorption chromatography. Analysis was conducted under isothermal conditions simultaneously on two columns.

The group composition of the initial bitumen and the liquid products of cracking was determined in accordance with a traditional procedure: first, the asphaltene content of the sample was determined by the cold Holde method. The concentration of tars in the resulting oils was determined by an adsorption method: the analyzed product was applied to activated ASK silica gel; the mixture was placed in a Soxhlet extractor, and the hydrocarbon components (oils) and tars were sequentially extracted with *n*-hexane and an ethanol–benzene mixture in a ratio of 1 : 1, respectively (STP SZhShI 1217-2005 procedure, Institute of Petroleum Chemistry, Siberian Branch, Russian Academy of Sciences).

As a result of the thermal cracking of natural bitumens, liquid products and coke were formed, and small quantities of gas appeared (Table 4). The yield of the liquid products of the cracking of bitumen from the Munaily-Mola deposit was found higher than that upon the processing of bitumen from the Beke deposit by 6%, whereas the yield of coke was lower by 4.7 wt %. Heat treatment leads to an increase in the oil content, and the total high-molecular-weight components of bitumen decrease. This is likely due to an increase in the formation of coke and the destruction of tar components with the formation of lighter products. In this case, the concentration of oils in the composition of the liquid products of the cracking of Munaily-Mola was 22% higher than that of bitumen from the Beke deposit, and the concentrations of tars and

**Table 2.** Chemical composition of the magnetic microspheres

Fraction, mm	Apparent density, g/cm <sup>3</sup>	Chemical composition, wt %								
		SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	SO <sub>3</sub>
0.4	1.87	4.00	1.90	85.20	8.69	1.00	0.25	0.07	0.18	0.25

**Table 3.** Composition of the samples of calcined magnetic microspheres and the concentrations of crystalline phases

Fraction, mm	Phase concentration, wt %				
	spinel	hematite	quartz	CaSO <sub>4</sub>	vitreous phase
0.4	13.05	62.98	1.65	3.87	18.45

**Table 4.** Material balance and substantial composition of the cracking products of natural bitumens

Process	S <sub>total</sub> in oil, wt %	Yield, wt %			Composition of liquid products, wt %		
		gas	liquid	coke	oil	tar	asphaltenes
Natural bitumen from the Beke deposit							
Initial bitumen	0.30	0.00	100.00	0.00	49.17	44.89	5.94
Cracking	0.43	1.40	67.70	30.90	41.49	19.14	7.07
Cracking with the microspheres;	0.34	1.30	63.00	35.70	37.83	20.31	4.86
Cracking with the radical-forming additive	0.35	1.10	70.30	28.60	44.48	17.44	8.38
Natural bitumen from the Munaily-Mola deposit							
Initial bitumen	0.7	0.00	100.00	0.00	47.58	46.37	6.05
Cracking	0.57	0.20	73.60	26.20	61.54	9.85	2.21
Cracking with the microspheres;	0.64	0.50	64.80	34.70	50.24	9.32	5.24
Cracking with the radical-forming additive	0.65	1.50	87.60	10.90	67.51	13.34	6.75
Cracking product of ozonized bitumen	0.78	0.10	97.65	2.25	61.49	25.84	10.32

asphaltenes were lower by 15 and 7 wt %, respectively. The experimental data show that the high-molecular-weight components of bitumen from the Munaily-Mola deposit more easily undergo cracking than bitumen of the Beke deposit.

The presence of microspheres has a negative effect on the amount of liquid products of the cracking of bitumens; the yield of coke increased by 6–8 wt % (Table 4). The oil content decreased by 4 and 9 wt % for bitumens from the Beke and Munaily-Mola deposits, respectively, whereas the total tar–asphaltene component content remained almost unchanged (in comparison with the composition of thermal cracking products). Apparently, the microspheres intensify the condensation reactions of bitumen cracking products, which occur on the surface; this was confirmed by the greatest coke formation in these experiments.

The addition of radical-forming components, which are the initiators of the radical-chain processes of low-temperature cracking, is a method that makes it possible to reach a deeper conversion of the tar–asphaltene components of bitumens into target products and, as a result, to increase the yield of distillate fractions in the cracking process. We used di-*tert*-butyl peroxide as a radical-forming additive. Based on earlier obtained data [15], the amount of di-*tert*-butyl

peroxide was 3 wt %. It was found that this organic peroxide initiates the degradation reactions of the high-molecular-weight compounds of natural bitumens and ensures the yield of light cracking products.

The peroxide additive favorably influenced the thermal destruction process: the yield of the liquid products of the cracking of Munaily-Mola bitumen increased by 14% in comparison with thermal cracking, and the amount of coke decreased by 15 wt %. The concentration of oil components in the liquid cracking products of bitumen from the Beke deposit increased by 2%, and the amount of tars decreased by 3.4%. However, upon the processing of bitumen from the Munaily-Mola deposit, an increase in the concentration of tar–asphaltene substances was observed, although the yield of liquid cracking products and the oil content of these products considerably increased. It is likely that coke formation processes are considerably inhibited on cracking with the radical-forming additive, which leads to the accumulation of tars and asphaltenes in the composition of the products.

The preliminary ozonization of bitumen is another method for the intensification of the destruction reactions of tar–asphaltene components. Treatment with an ozone–oxygen mixture was performed on a laboratory installation equipped with a bubbling-type reac-

**Table 5.** Fractional composition of the natural bitumens and liquid cracking products

Sample	$T_b$ , °C	Concentrations of fractions, wt %		
		bp–200 °C	200–360 °C	>360 °C
Natural bitumen from the Beke deposit				
Initial bitumen	116.8	5.1	20.2	74.7
Cracking product	77.9	1.6	12.8	53.3
Cracking product with the microspheres	73.0	10.1	16.9	36.0
Cracking product with the radical-forming additive	77.4	3.4	10.3	56.6
Natural bitumen from the Munaily-Mola deposit				
Initial bitumen	96.5	2.2	15.6	82.2
Cracking product	92.0	6.8	25.2	41.6
Cracking product with the microspheres	75.0	4.6	15.1	45.1
Cracking product with the radical-forming additive	82.7	5.7	18.7	63.2
Cracking product of ozonized bitumen	90.1	6.7	24.7	66.2

tor; the space velocity of the supplied gas mixture was 31.15 dm<sup>3</sup>/h. The weight of raw material loaded into the reactor was 20 g. The quantity of absorbed ozone was monitored using a Tsiklon 5.11 instrument. The method consists in the selective absorption of UV radiation with a wavelength of 254 nm by ozone. The consumption of ozone upon the treatment of bitumen from the Munaily-Mola deposit with the ozone–oxygen mixture was 10.9 (g ozone)/(kg bitumen) [8]. The composition of the cracking products of ozonized bitumen differed significantly from the results of other experiments. An increase in the amount of oils in the liquid cracking products was observed (by 14% greater than that in the initial bitumen); the tar content decreased by 20%; the asphaltene content increased by 4 %, and the total amount of gas and coke was no greater than 2.5 wt %.

After conducting the thermal and thermocatalytic processing of natural bitumen from the Munaily-Mola and Beke oil-bituminous rocks, we studied the fractional composition of the resulting products. Data on the fractional composition of liquid cracking products make it possible to judge the depth of conversion of the bitumens.

An analysis of the fractional composition of bitumen cracking products (Table 5) showed that the initial boiling points decreased in all of the experiments in comparison with that of the initial bitumen. Upon the cracking of bitumen from the Beke deposit, an increase in the concentration of bp–360°C fractions was noted with the use of microspheres (the amount of bp–200°C fractions increased by 11%, and the amount of fractions that boiled away in a range of 200–360°C, by 6.6 wt %). The cracking of bitumen from the Munaily-Mola deposit, in all cases, led to an increase in the concentration of bp–200°C fractions, which is indicative of an increase in the fraction of

destructive processes in the reaction medium. A maximum increase in the concentration of distillate fractions was found in the products of the thermal cracking of bitumen and the products of bitumen ozonization: the concentrations of a bp–200°C fraction and a fraction with boiling points of 200–360°C increased by 4.5 and 10 wt %, respectively. The presence of a catalyst or an additive of di-*tert*-butyl peroxide led to an increase in the concentration of the bp–360°C fractions to a lesser degree.

The gaseous hydrocarbons are formed upon the thermolysis of bitumens were analyzed by gas-chromatography on a Khromatron chromatograph. The main gas products of cracking include methane; its concentration was higher than 30% upon the cracking of bitumen from the Beke deposit or higher than 20 wt % upon the cracking of bitumen from the Munaily-Mola deposit (Table 6). Ethane, propane, and hydrogen also occurred in considerable amounts in the composition of the gas cracking products. The presence of increased isobutane amounts in the composition of the gas cracking products of the bitumens with a radical-forming additive was caused by the thermal degradation of the additive.

The molecular weights of the tars and asphaltenes of natural bitumens and their cracking products (Table 7) were measured by cryoscopy in naphthalene on a Krypton instrument, which was developed at the Institute of Petroleum Chemistry, Siberian Branch, Russian Academy of Sciences. The values of the molecular weights of the tars and asphaltenes of the initial bitumens are typical of the materials of this kind. We found that the thermal cracking of bitumen from the Beke deposit leads to a decrease in molecular weights of high-molecular-weight compounds by 30%, as compared with the initial values. The introduction of additives leads to the insignificant coarsening of tar mole-

**Table 6.** Composition of the gaseous products of the cracking of natural bitumens (wt %)

Gas composition	Natural bitumen from the Beke deposit			Natural bitumen from the Munaily-Mola deposit		
	process					
	cracking	cracking with the microspheres	cracking with radical-forming additive	cracking	cracking with the microspheres	cracking with radical-forming additive
H <sub>2</sub>	3.69	4.08	3.11	5.45	5.16	5.88
CH <sub>4</sub>	41.28	49.29	40.78	29.34	30.93	31.50
C <sub>2</sub> H <sub>6</sub>	13.82	15.97	9.99	20.45	16.54	13.13
CO <sub>2</sub>	32.40	19.54	20.91	29.82	35.43	27.50
C <sub>3</sub> H <sub>8</sub>	7.03	8.71	6.47	10.68	8.87	9.75
C <sub>3</sub> H <sub>6</sub>	0.03	0.10	0.10	0	0	0
<i>i</i> -C <sub>4</sub> H <sub>10</sub>	1.04	1.33	17.88	2.47	1.75	11.37
<i>n</i> -C <sub>4</sub> H <sub>10</sub>	0.04	0.05	0.04	0.10	0.06	0.06
$\Sigma$ <i>i</i> -C <sub>5</sub> H <sub>12</sub>	0.41	0.49	0.49	1.15	0.86	0.46
<i>n</i> -C <sub>5</sub> H <sub>12</sub>	0.26	0.44	0.22	0.55	0.40	0.34

**Table 7.** Molecular weights (amu) of the tars and asphaltenes of the initial bitumens and their cracking products

Sample	Natural bitumen from the Beke deposit		Natural bitumen from the Munaily-Mola deposit	
	asphaltenes	tars	asphaltenes	tars
Initial bitumen	2044	751	1803	566
Cracking product	1304	499	677	437
Cracking product with the microspheres	1003	550	1045	586
Cracking product with the radical-forming additive	1042	564	869	552

cules in the course of cracking; on the contrary, the molecular weight of asphaltenes continues to decrease under these conditions (by a factor of 2, as compared with that of the initial asphaltenes). The molecular weight of asphaltenes in the thermal cracking products of bitumen from the Munaily-Mola deposit is lower by a factor of almost 3 than that in the initial bitumen, and the molecular weight of tars decreased by 130 amu. The initiated cracking of bitumen leads to an increase in the molecular weight of the high-molecular-weight compounds of bitumen (as compared with the thermal cracking). Upon the cracking in the presence of microspheres, the molecular weights of tars and asphaltenes are somewhat higher than those with the use of radical-forming additives.

## CONCLUSIONS

We found that the heat treatment of bitumen from the Beke deposit impaired the fractional and substantial composition of the liquid products of cracking,

whereas, on the contrary, the quality of the cracking products of Munaily-Mola bitumen was improved (the amount of oils increased; the amounts of gasoline and diesel fractions increased by a factor of 3 and by 10 wt %, respectively). In both cases, the tar content was decreased and significant coke formation was noted.

The addition of microspheres as an initiator of the process of bitumen cracking led to an increase in coke formation, a decrease in the amount of oils, and a decrease in the initial boiling temperature of liquid products (in comparison with the composition of thermal cracking products). The total concentration of distillate fractions (bp–360°C) in the cracking products of bitumens with the additive of microspheres was almost the same as the initial values with an increase in the quantity of gasoline fractions by 2–5 wt %. The amounts of oils increased by 3 and 6 wt %, respectively.

The presence of a radical-forming additive decreased coke formation on the cracking of bitumens (by 2.3 and 15.3 wt % for the Beke and Munaily-Mola

deposits, respectively), as compared with thermal cracking products. The concentration of distillate fractions in the products of the initiated cracking of bitumen from the Beke deposit was minimal; it is likely that di-*tert*-butyl peroxide initiates the condensation reactions of bitumen components to a larger degree. Oils (more than 67 wt %) predominated in the liquid products of the initiated cracking of Munaily-Mola bitumen. The amount of bp–360°C fractions in the products of the initiated cracking of Munaily-Mola bitumen was greater than the concentration in initial bitumen and in the products of cracking with microspheres, but it was lower than that upon the thermal cracking of bitumen. Note that a decrease in coke formation led to an increase by 20 wt % in the concentration of residual fractions (>360°C), which are raw materials for obtaining gas oils and oil distillates.

The preliminary treatment of bitumen from the Munaily-Mola deposit with ozone (6 g/kg of bitumen) with the subsequent cracking led to a considerable decrease in coke formation and the production of considerable amounts of liquid cracking products (more than 97%) with a smaller tar content (by 20.5 wt %). The yield of distillate fractions was no lower than that in the thermal cracking products of bitumen, but the concentration of residual fractions was higher by 25 wt %.

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