










Catalytic cracking of M-100 fuel oil: relationships between origin process parameters and conversion products

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Abstract

The development of technologies for processing oil residues is relevant and promising for Kazakhstan, since the main oil reserves of hydrocarbons in the country are in heavy oils. This paper describes the study of the influence of technological modes on the yield and hydrocarbon composition of products formed because of cracking of commercial fuel oil and fuel oil M-100 in the presence of air in the reactor. For catalysts preparation, natural Taizhuzgen zeolite and Narynkol clay were used. It was found that the introduction of air into the reaction zone, in which oxygen is the initiator of the cracking process, significantly increases the yield of the middle distillate fractions. In the presence of air, the yield of diene and cyclodiene hydrocarbons significantly increases compared to cracking in an inert atmosphere. According to the data of IR spectral analysis of M-100 grade oil fractions, in addition to normal alkanes, the final sample contains a significant amount of olefinic and aromatic hydrocarbons. On the optimal catalyst, owing to oxidative cracking of fuel oil, the following product compositions (in %) were established: Fuel oil M-100: gas – 0.8, gasoline – 1.1, light gas oil – 85.7, heavy residue – 11.9, loss – 0.5 and total – 100.0%; commodity Fuel oil (M-100): gas – 3.3, gasoline – 8.4, light gas oil – 84.3, heavy residue – 4.0, loss – 0 and total – 100.0%.

Keywords

catalytic cracking
oxidative cracking
natural zeolite
Taizhuzgen zeolite
Narynkol clay
Amangeldy GPP
fuel oil M-100

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1. Introduction

Recently, there has been an increased interest in technologies for processing oil residues due to the growing difference in prices for light and heavy grades of oil. At present, the development of technologies for processing oil residues is relevant and promising, which is associated with an increase in the share of hard-to-recover oil reserves: heavy and high-viscosity oils in the world. This, in turn, forces refineries to select carefully the available technologies for processing oil residues and increase the share of heavy oil raw materials in the total volume of oil-

processing feedstock [1–20]. 40–45% of all high-octane gasoline is produced at catalytic cracking plants with a steamer. In the classical version, the cracking process of heavy hydrocarbons occurs in the vapor phase due to contact with a circulating fluidized catalyst consisting of particles ranging in size from 50 to 100 microns [4, 21–36]. Over the past decades, the world's leading companies have developed a number of new catalytic cracking technologies to maximize the yield of light olefins, for example: the use of promoted catalysts or increased rigidity of the technological mode; the addition of light hydrocarbon fractions to raw materials; reducing the contact time of raw materi-

als with the catalyst and, finally, the use of heavy fractions to ensure the thermal balance of the installation [33–49].

Fuel oil is a residual substance after a simple distillation of oil, which contains mainly hydrocarbons and oil resins of large molecular weight. Fuel oil M-100 belongs to the furnace types of fuel oil and can be used as a liquid fuel for combustion in boiler furnaces of thermal power plants. The fuel oil can be further processed to produce gas oil by vacuum distillation [54, 55]. Most scientists in the field recognize that the specific properties and complex composition of heavy oils and oil residues do not allow the use of classical processing methods for light oils; such schemes are ineffective or not suitable at all [1, 2, 4, 14–16]. One of the actual methods of intensification of thermal processes of refining of high-viscosity oils is wave action (ultrasonic, acoustic, ultra-frequency) [56–65].

Oxidative cracking is a process of cracking petroleum fractions carried out at atmospheric pressure in the vapor phase. A Soviet petroleum engineer, doctor of technical sciences, a specialist in the field of oil production and oil refining, one of the founders of thermal methods of oil production, A. B. Sheinman (1898–1979) with his colleagues (among them, in particular, a famous Soviet and Hungarian scientist of Hungarian origin Carl Dubrovay (1888–1957)) created the scientific and technical foundations of oxidative cracking [66, 67].

The efficiency of processing raw materials with ozone to the depth of conversion of fuel oil under the conditions of its cracking process and the possibility of lowering the temperature to 425–450 °C are described [1, 2, 4, 58–75]. In the literature, studies are known of the processes of cracking vacuum distillates on a zeolite-containing catalyst and the reforming of gasoline fractions on a modified catalyst in the presence of atmospheric oxygen. The authors found that the presence of oxygen contributes to an increase in the yield of light fractions, but causes an increase in the yield of hydrocarbon gases and the formation of coke on the catalyst. The octane number of the obtained gasoline fraction in the presence of oxygen was higher (the increase in the number was within 1.5–2.3 units) than the octane number of the same fraction obtained in the absence of oxygen. This phenomenon can be explained by the presence of oxygen-containing hydrocarbon compounds in it, which was confirmed by IR spectroscopy. These results, obtained by various authors, indicate the feasibility of processing heavy oil fractions by oxidative cracking. An analysis of the data of various scientific schools allows us to conclude that one of the most promising ways to increase the depth of processing of heavy oil fractions is to carry out catalytic cracking in an oxidizing medium, for example, with controlled air supply to the reactor [58–75].

In this work, the influence of technological modes on the yield and hydrocarbon composition of products formed as a result of catalytic cracking of commercial fuel oil and fuel oil M-100 in the presence of air in the reactor on cata-

lysts synthesized on the basis of natural raw materials of Kazakhstan fields was investigated.

2. Experimental

Commercial fuel oil and fuel oil grade M-100 from the Amangeldy GPP (Kazakhstan) were used as initial products for studying cracking. Sulfur content is 0.7% in fuel oil M-100, and 2.1% – in commercial fuel oil.

For the preparation of catalytic composites, the fractions of 60–80 μm of natural zeolite from the Taizhuzgen deposit (Kazakhstan) and clay from the Narynkol deposit (Kazakhstan) were taken (Figure 1, Table 1).

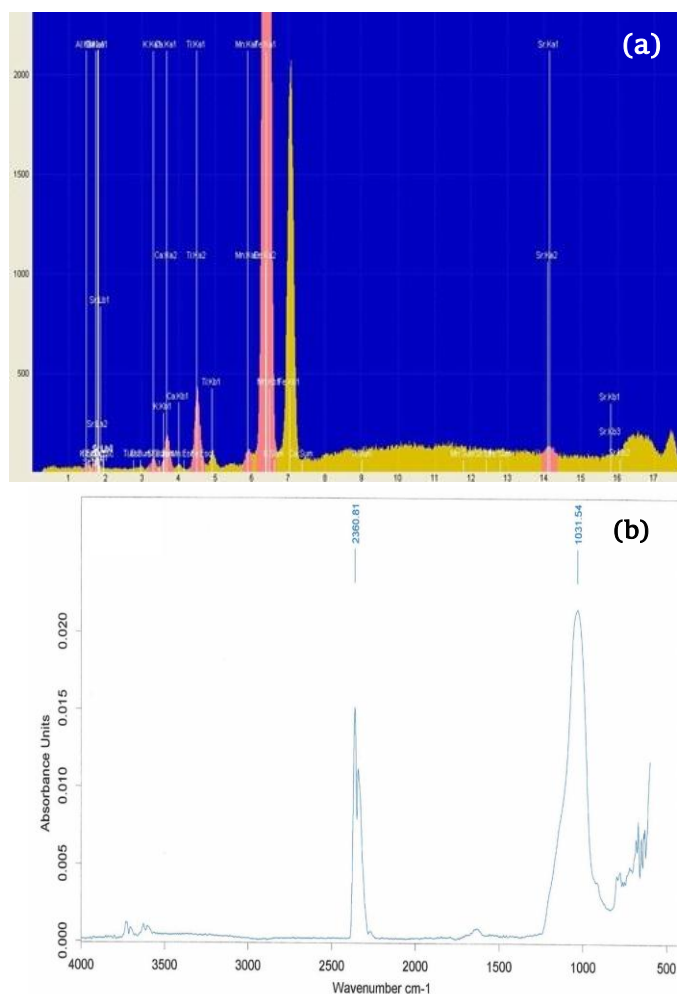


Figure 1 Study of physico-chemical properties of natural zeolite from the Taizhuzgen deposit (Kazakhstan): X-ray diffraction analysis (a), IR spectroscopy (b).

The chemical composition of the original Narynkol clay is (in %): SiO₂ – 38.05; CaO – 20.40; Al₂O₃ – 8.49; MgO – 6.15; Fe₂O₃ – 6.15; K₂O – 1.80; Na₂O – 1.10; TiO₂ – 0.44; P₂O₅ – 0.11; MnO – 0.10.

The zeolite was activated by ionic exchange of zeolite framework sodium cations for lanthanum and ammonium cations. Then, the zeolite suspension was stirred for two minutes with a suspension containing a certain amount of clay in distilled water, which was changed in each experiment in order to prepare composites with different concentrations of components.

Table 1 Composition of natural zeolite from the Taizhuzgen deposit (Kazakhstan) according to X-ray diffraction analysis.

No.	Chemical element	Concentration, %	Intensity
1	Fe	49.94	739.15
2	Ca	1.71	9.16
3	Sr	0.270	1.98
4	Mn	0.130	1.82
5	Al	21.955	0.30
6	Si	23.115	0.98
7	Ti	1.902	20.86
8	K	0.975	1.97

After that, the mixture was evaporated and molded to form a 0.05–0.25 mm fraction. The resulting microspherical catalyst was dried at 100 °C for 10 hours and hardened at 550 °C for 5 hours. Ion exchange for NH_4^+ and La^{3+} cations was carried out at 80 °C for 3 hours in a solution of ammonium sulfate and lanthanum nitrate at a ratio of 5 g-eq. $(\text{NH}_4)_2\text{SO}_4$ to 1 g-eq. Na_2O and 2 wt.% lanthanum. Then the samples were heat-treated and ion exchange was repeated in a solution of a mixture of ammonium sulfate and lanthanum nitrate. The final residual sodium oxide content in the zeolite was about 0.5 wt.%.

Figure 2 shows the methodology for the catalyst preparation used in our study.

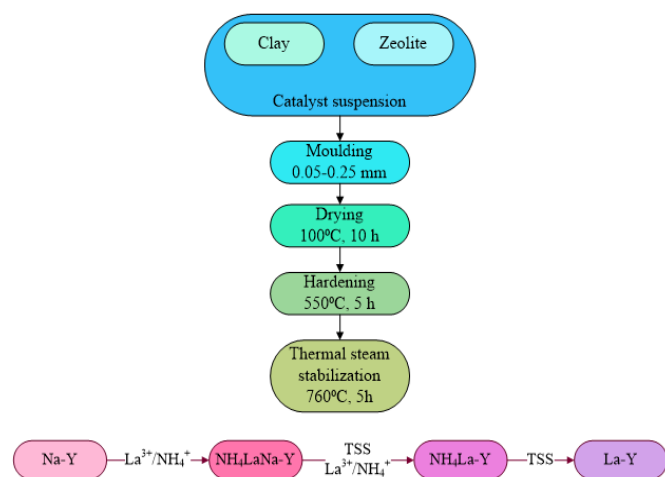


Figure 2 The scheme of preparation of the catalyst.

The procedures for selection and adjustment of process conditions were also described in more detail in our previous works [21, 30, 32, 58, 59].

The process was carried out in four versions: in an inert atmosphere in the absence of a catalyst (thermal cracking), with air supplied to the reactor (oxidative thermal cracking), in an inert atmosphere in a 0.2 wt.% suspension of a fine catalyst (catalytic cracking), and, finally, with the simultaneous supply of a catalyst suspension in fuel oil and air to the reactor (oxidative catalytic cracking). Figure 3 shows a principal scheme for implementing the process under study.

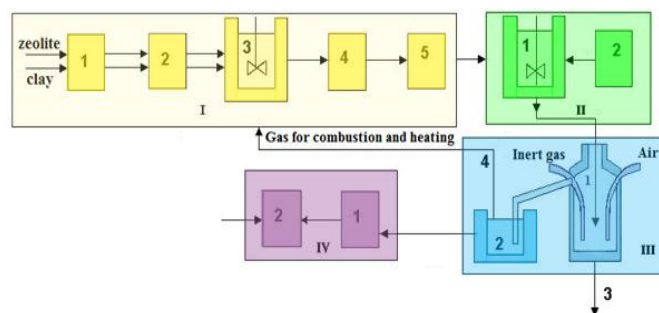


Figure 3 Schematic diagram for the implementation of catalytic processing of commercial fuel oil and fuel oil grade M-100 from the Amangeldy GPP (Kazakhstan): I – catalyst preparation section: 1 – section for mixing; 2 – sieves; 3 – temperature-controlled container with a stirrer; 4 – kiln for drying and calcining; 5 – electromagnetic homogenizer. II – section for preparation of a catalyst suspension in the feedstock (fuel oil): 1 – a thermostatically controlled vessel with a stirrer; 2 – reactor for electromagnetic activation of the feedstock-fuel oil. III – section for the implementation of the oxidative cracking process: 1 – thermostatically controlled flow reactor, equipped with devices and valves for dosed supply of inert gas, air, as well as a composite catalyst in the feedstock – fuel oil, 2 – flow tank with cooling for condensing liquid cracking products; 3 – output of the tar residue mixed with the spent catalyst; 4 – system for discharge and combustion of gaseous products formed as a result of cracking. IV – section of rectification of cracking products: 1 – evaporation column; 2 – rectification of cracking products.

The analysis of gaseous cracking products was carried out on a chromatograph with a flame ionization detector: on a 2-meter column with an inner diameter of 2 mm, filled with Poralac sorbent (fraction 8.2–8.3 mm) – for the analysis of hydrocarbon gases; on a 1-meter packed column filled with NaX zeolite (fraction 0.25–0.5 mm) – for the analysis of non-hydrocarbon gases. Argon was used as the carrier gas.

The programmed temperature sweep was carried out for hydrocarbon gas in the range of 25–165 °C with exposure at 25 °C for 7 minutes; and for non-hydrocarbon – 40–100 °C, at a speed of 8°/min. Determination of the hydrocarbon composition of gasoline fractions was carried out by gas chromatography on a chromatograph with a flame ionization detector on a 50 m stainless steel capillary column (internal diameter 0.2 mm) filled with Squalane sorbent. The carrier gas was argon. The programmed temperature sweep was carried out in the range of 40–110 °C at a speed of 2°/min.

3. Results and discussion

The content of components in catalysts affects the activity of catalytic composites. The chemical composition of the starting materials and catalysts based on them was determined. Two samples of catalysts were taken for comparison; in the 1st sample the content of zeolites was less than 15%, and in the 2nd – above 15% (Table 2). The $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio in the natural zeolite from the Taizhuzgen deposit is 4.5, and in the synthesized catalysts it is about 4. It should be noted that the content and ratio of silicon and aluminum oxides in the catalysts varies in proportion to their

concentration in the initial components and their content in the catalysts under study. When Narynkol clay is activated by the ion exchange method, the sodium cations are completely replaced by lanthanum cations. The natural zeolite of the Taizhuzgen deposit has a higher ion exchange activity compared to the used Narynkol clay. For this reason, the content of lanthanum varies.

Table 2 Chemical composition of initial materials-Taizhuzgen zeolite and Narynkol clay and samples of catalysts based on them.

Chemical component	Activated Taizhuzgen zeolite	Initial Narynkol clay	Zeolite-containing composite catalysts	
			1	2
SiO ₂	67.93	38.05	40.00	47.07
CaO	1.97	20.40	19.77	15.14
Al ₂ O ₃	14.28	8.49	9.97	11.51
MgO	1.39	6.15	6.27	4.72
Fe ₂ O ₃	1.79	6.15	3.75	4.01
K ₂ O	4.47	1.80	2.29	3.02
Na ₂ O	1.11	1.10	0.30	0.55
TiO ₂	0.29	0.44	0.23	0.41
P ₂ O ₅	0.01	0.11	0.15	0.14
MnO	0.01	0.10	0.15	0.11
Calcination	7.04	19.47	17.43	0.20
La	0.24	0.0	13.52	0.20

The effect of the catalyst and air on the process of cracking commercial fuel oil was studied at $w_{\text{suspension}} = 0.1 \text{ h}^{-1}$ and reaction temperature of the process is 450 °C; in the presence of 0.2 wt.% - catalyst based on Taizhuzgen zeolite and more than 80 wt.% of Narynkol clay. The ratio of the catalytic cracking products is shown in Figure 4.

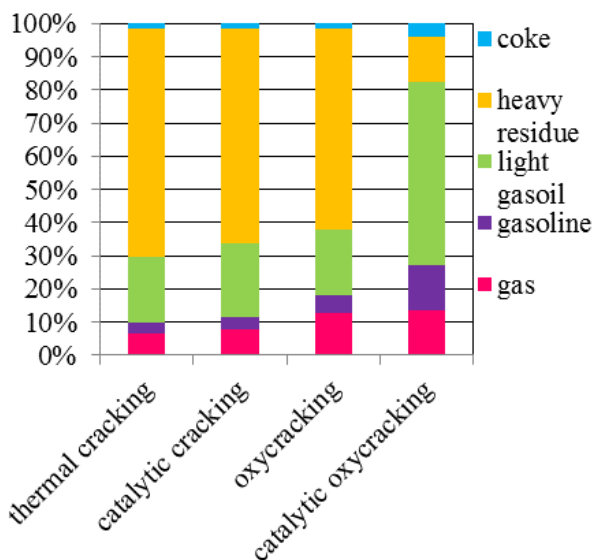


Figure 4 Catalytic cracking product ratio at different cracking conditions.

According to the results of chromatographic analysis, the quantitative composition of the gaseous products of thermal and oxidative thermal cracking is almost identical (Table 3, Figure 5).

Table 3 Composition of gaseous products (%) of M-100 fuel oil cracking ($w_{\text{suspension}} = 1.0 \text{ h}^{-1}$, $T = 470 \text{ °C}$)

Composition of gases	Cracking conditions			
	without catalyst and air	without catalyst, $w_{\text{air}}=0.15 \text{ h}^{-1}$	0.2 wt.% zeolite-containing catalyst, without air	0.2 wt.% zeolite-containing catalyst, $w_{\text{air}}=0.15 \text{ h}^{-1}$
Hydrogen	2.8	3.4	10.7	4.3
Methane/Ethane	16.2/15.5	20.1/18.4	68.0/4.9	21.8/20.1
Ethylene/Butylene	25.4/9.2	25.7/6.1	11.7/0.2	29.3/2.8
Propane/Propylene	5.6/17.6	5.6/14.0	0.1/0.1	5.6/12.2
Carbon Monoxide/Carbon Dioxide	6.3/1.4	6.7/0	4.9/0	3.8/0.1

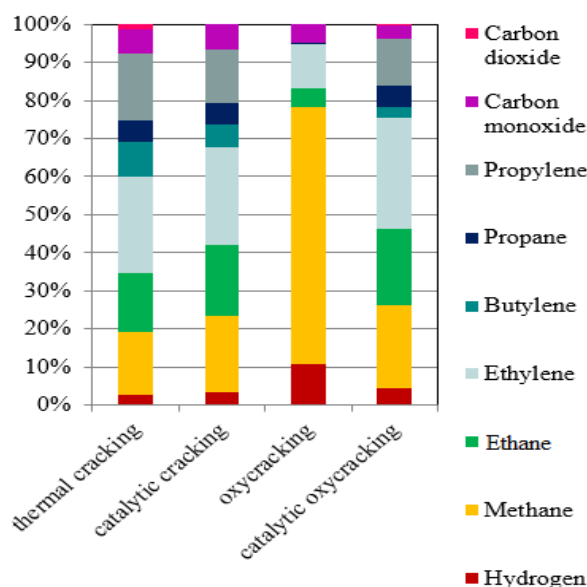


Figure 5 Composition of gaseous products of M-100 fuel oil cracking.

The only difference is that in the presence of air slightly more methane is formed, and the content of propylene and butylene is reduced, i.e. the depth of destruction of gaseous hydrocarbons increases.

During catalytic cracking, the yield of methane is maximum, the resulting ethane is dehydrogenated to ethylene, and there are practically no C₃-C₄ hydrocarbons.

Consequently, the reactions of destruction and dehydrogenation of hydrocarbons proceed on the catalyst. The presence of the latter reaction is confirmed by the maximum hydrogen concentration in the gaseous products compared to the other cracking conditions.

In the presence of air additives, the yield of diene and cyclodiene hydrocarbons significantly increases compared to cracking in an inert atmosphere if the process is carried out at low volumetric feed rates to the reactor. The conclusions obtained agree with the data that the yield of the reaction of oxidative dehydrogenation of olefins increases in the presence of air [5, 12, 22, 26]. The hydrocarbon composition of cracking gasolines depends on the conditions of cracking in a similar way (Table 4).

Table 4 Influence of process conditions on the hydrocarbon composition of gasoline cracking fuel oil M-100 ($w_{\text{suspension}}=1.0 \text{ h}^{-1}$, $T=470 \text{ }^{\circ}\text{C}$).

Composition of hydrocarbons, %	Cracking conditions			
	without catalyst and air	without catalyst, $w_{\text{air}}=0.15 \text{ h}^{-1}$	0.2 wt.% zeolite-containing catalyst, without air	0.2 wt.% zeolite-containing catalyst, $w_{\text{air}}=0.15 \text{ h}^{-1}$
n-paraffins/isoparaffins	23.6/46.2	24.9/45.6	7.15/5.8	17.8/40.2
Naphthenes	14.8	15.8	15.8	22.8
Olefins/ Cycloolefins	4.9/0	5.2/0	6.3/1.1	3.9/0
Arenes/ Dienes	10.1/0	8.2/0	12.7/0.9	15.1/0
Octane Number (RM)	71.3	70.5	76.6	76.2

In the absence of a catalyst, the ratio of hydrocarbons of different classes does not change when moving from an inert to an oxidizing atmosphere. In the presence of a catalyst, the process of isomerization of n-alkanes sharply intensifies and, to a much lesser extent, so does the dehydrogenation of the formed light hydrocarbons. The oxygen from air changes the course of catalytic cracking reactions: the proportion of naphthene and arene cyclization reactions increases and the isomerization process decreases.

However, the isomerization of hydrocarbons that make up gasoline under all the conditions studied prevails over other reactions occurring during cracking. According to the data of the individual hydrocarbon composition of the obtained gasolines, their formation is most likely by the carbocation mechanism. This is evidenced by the presence of a large number of isomeric hydrocarbons with a substituent at the tertiary carbon atom.

The results obtained suggest the mechanism of oxidative cracking of fuel oil on a low-percentage suspension of an activated catalyst synthesized from natural zeolites. The catalytic destruction of hydrocarbon molecules proceeds through the formation of free radicals, so the introduction of air into the reaction zone, in which oxygen is the initiator of this process, significantly increases the yield of the middle distillate fraction. Since the symmetrical decomposition of heavy fraction hydrocarbon molecules occurs during cracking, the main product is light gas oil. The resulting less reactive light hydrocarbons are practically not cracked on the relatively weak acid sites of the natural catalyst, so the total yield of gas and gasoline fraction does not exceed 4 wt.%. According to the IR spectral analysis of M-100 fuel oil fractions, in their composition, along with normal alkanes, a significant amount of olefinic and aromatic hydrocarbons was also noticed.

The composition of the products according to the optimal composition of catalysts for oxidative cracking of fuel oil (in %) was as follows: fuel oil M-100: gas – 0.8; gasoline – 1.1; light gas oil – 85.7; heavy residue – 11.9; loss – 0.5; total – 100.0%. Commodity fuel oil (M-100): gas – 3.3; gasoline – 8.4; light gas oil – 84.3; heavy residue – 4.0; loss – 0; total – 100.0%.

4. Conclusions

This study showed that the introduction of air into the reactor during the catalytic cracking of M-100 fuel oil on natural zeolites increases the yield of the middle distillate fractions. Since cracking involves symmetrical decomposition of heavy fraction hydrocarbon molecules, the main product is light gas oil. The resulting light hydrocarbons are almost not cracked on the relatively weak acid centers of the natural catalysts. The identical catalysts based on natural zeolites were used in this work. It is obvious that the differences found in the rates of catalytic cracking can only be associated with a change in the process conditions (without air additions or in the presence of air).

Supplementary materials

No supplementary materials are available.

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Conflict of interest

The authors declare no conflict of interest.

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