STUDY OF THE ACTIVATING EFFECT OF INTRODUCING AIR ADDITIVES INTO THE REACTOR DURING FUEL OIL CRACKING ON COMPOSITE CATALYSTS

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ABSTRACT

In this research commodity fuel oil and fuel oil of the M-100 brand from the Amangeldy Gas Processing Plant (Kazakhstan) were used as cracking raw materials. The effect of adding air with different concentrations into the reaction mixture of fuel oil during the cracking on the composite catalysts was determined. Composite catalysts were prepared on the basis of natural Kazakhstan raw materials: zeolite of the Taizhuzgen deposit and clay of the Narynkol deposit. They were activated by ion exchange of sodium cations of the zeolite framework to cations of lanthanum and ammonium. It was found that the optimal for the process of cracking fuel oil at high feed rates to the reactor is a temperature of 470°C. The yield of the kerosene-gas oil fraction during cracking of fuel oil slightly decreases with increasing volumetric feed rate. At high concentrations in the reaction mixture of air, intense gas formation begins. Therefore, air additives contribute to a deeper destruction of the hydrocarbons of the cracked feed. In the presence of air, a secondary process of symmetrical decomposition of medium-molecular hydrocarbons begins, accompanied by cyclization of the resulting hydrocarbon molecules of the gasoline fraction. The results of the research on the use of activation of cracked fuel oil by dosed injection of air into the reaction zone indicate great prospects for a new direction in the catalytic processing of heavy hydrocarbon feedstocks.

KEYWORDS: Fuel Oil, Air Additives, Catalytic Cracking, Taizhuzgen Zeolite, Narynkol Clay

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INTRODUCTION

Among the most important tasks facing the oil refining industry is the deepening of oil refining in order to obtain the maximum yield of high-quality motor fuels and raw materials for petrochemical synthesis. Limited reserves of “traditional oil” leads to the need to involve heavy and bituminous oils in the processing. However, the yield of light fractions in such oils is an insignificant part. Heavy crude oil differs from light crude in the composition and temperature of the end of boiling, characterized by a significant content of polycycloalkanes, condensed aromatic hydrocarbons and asphaltene (Scott, 2002; Amghizar et al., 2017; Delattre et al., 2001; Occelli et al., 1984; Dadyburjor and Liu, 1993; Sassykova et al., 2019). Processing heavy hydrocarbons is significantly more expensive due to the high coking index > 8% (according to Conradson), a significant content of high molecular weight components - resins and asphaltene (up to 35%), which are enriched in heteroatoms (S, N, O) and metals, especially nickel and vanadium (up to 1000 g / t), which quickly deactivate catalysts for industrial catalytic and hydroprocessing processes, which imposes limitations on the quality of raw materials and eliminates its low cost (Baiseitov et al., 2017). In recent years, much attention has been paid to the development of the catalytic cracking process for processing residual types of raw materials. It is not only less expensive and more affordable, but also its

consumption in traditional areas, except for cracking, is decreasing (Gomez and Molina, 2019; Sassykova et al., 2020; Liu et al., 2004; Pandey, 2018). The need to use a more complete load of the available catalytic cracking capacities led to the intensification of research and improvement of the cracking process of high boiling oil fractions. With an increase in the temperature of the boiling point of vacuum distillates, oil consumption decreases, and when switching to fuel oil, in addition, capital and operating costs are reduced by eliminating the vacuum distillation of fuel oil. The most rational way to ensure the economical operation of catalytic cracking units is the special preparation of raw materials (Abul-Hamayel, 2002; Tashmukhambetova, et al., 2017; Guerzoni and Abbot, 1993). Such raw materials are residues of direct distillation of oil, residues of products of distillation of thermal cracking, pyrolysis, as well as high-boiling flavored concentrates and gas oils obtained on the basis of distillate products (Redwan and All, 1992; Akhmedzhanov et al., 2012; Tulepov et al., 2018; Wada et al., 2004; Lesin et al., 2017). The fundamental approach to solving the problems of processing heavy hydrocarbon feedstocks may be the transformation of resinous-asphaltene substances, organometallic, sulfur- and nitrogen-containing compounds into compounds of other classes due to directed chemical reactions with the simultaneous disaggregation of the highest molecular weight components of heavy petroleum feedstocks (Itoh et al., 1984; Yemelyanova et al., 2019; Lesin, 2018; Rigutto et al., 2007). Traditional processes of catalytic cracking of oil residues require continuous regeneration of expensive zeolite-containing catalysts and complex instrumentation of the process. Obviously, the most promising method for producing light hydrocarbon fractions is the catalytic cracking of oil residues in an oxidizing medium, i.e. in the presence of air additives in the reaction medium. Zeolite-containing catalysts with a unique microporous structure and acid-base properties are known to catalyze the conversion reactions of C_2 - C_7 paraffin hydrocarbons, turning them into valuable products of organic synthesis (Tashmukhambetova, et al., 2017; Sadeghbeigi, 2000; Ibrasheva and Zhubanov, 2000; Ibrasheva et al., 2001; Hamasaki et al., 2008; O'Connor, 2007; Omarova et al., 2019; Stocker, 2005; Tuktin et al., 2019; Brueva et al., 2001).

In this paper the effect of adding air with different concentrations into the reaction mixture of fuel oil during the cracking on the composite catalysts was researched. As cracking raw materials commodity fuel oil and fuel oil of the M-100 brand from the Amangeldy Gas Processing Plant (Kazakhstan) were used. Composite catalysts were prepared on the basis of natural Kazakhstan raw materials: zeolite of the Taizhuzgen deposit and clay of the Narynkol deposit.

EXPERIMENTAL

Commodity fuel oil and fuel oil of the M-100 brand from the Amangeldy Gas Processing Plant (Kazakhstan) were used as cracking raw materials (Table 1). The fractional composition of fuel oils was determined on the basis of distillation at atmospheric and reduced pressures. The following fractions were selected: gasoline, light gas oil, vacuum gas oil and a heavy residue with a boiling point >500 °C. The content of each fraction was determined by the gravimetric method and was expressed in % of the mass. The sulfur content in fuel oils and vacuum gas oils was determined by high-temperature oxidation of samples followed by acid-base titration of the resulting sulfur dioxide. In the initial fuel oils, as well as in gasoline and kerosene-gas oil fractions obtained as a result of cracking, the presence of oxygen- and sulfur-containing compounds was determined by the IR spectral method, which, in addition, makes it possible to establish the qualitative composition of hydrocarbons in the analyzed samples. 84.5 wt.% of the weight of the fuel oil is constituted by the vacuum distillate and heavier fractions, in addition, the fuel oil contains more sulfur compared to the studied vacuum distillates (Table 1).

Table 1: Fractional Composition and Sulfur Concentration in Commercial Fuel Oil

Content, wt. %	Fuel Oil	Gasoline	Light Gas Oil	Vacuum Gas Oil	Heavy Residue
Fractions	100.0	0.5	15.0	44.8	39.7
Sulfur	2.1	-	-	-	-

Figure 1 shows the scheme and main flows of reagents for oxidative catalytic cracking of fuel oil.

Figure 1: Schematic diagram of the catalytic conversion of fuel oil : I-catalyst preparation unit: 1-mixing unit; 2-sieves; 3-thermostatic container with agitator; 4-drying and calcination oven; 5-electromagnetic homogenizer; II - block for preparation of composite suspension in fuel oil: 1-a thermostatically controlled container with a stirrer; 2 - a reactor for electromagnetic activation of fuel oil; III-oxidative cracking unit: 1-thermostatically controlled flow reactor with systems for supplying inert gas, air and catalyst suspension in fuel oil, 2-cooled flow capacity for condensation of liquid cracking products; 3-removal of tar residue in a mixture with spent catalyst; 4 - system for dumping and burning cracking gases; IV - block for rectification of cracking products: 1-evaporation column; 2-rectification of cracking products.

The calculation of heat consumption for heating fuel oil in a cracking reactor was carried out as follows. Heat balance equation:

$$Q_1 = Q_2,$$

Where Q_1 is the amount of heat given off by the induction heating unit per unit of time, W;

Q_2 is the amount of heat perceived by fuel oil per unit of time, W.

The heat transfer capacity is calculated using the formula:

$$Q = G \Delta h,$$

Where G is the coolant flow rate, kg / s; Δh -is a change in specific enthalpy, J / kg.

The change in specific enthalpy is equal to:

$$\Delta h = c_p \Delta T;$$

The specific heat capacity of fuel oil c_p is found in the reference book:

$c_p = 2.7 \text{ kJ/kg}^\circ\text{C}$ at average temperature:

$$(300^\circ\text{C} + 470^\circ\text{C})/2 = 385^\circ\text{C}$$

Volume of a cylindrical cracking reactor:

$$V = \pi r^2 h = 3.14 \cdot 0.36 \cdot 2.4 = 2.713 \text{ m}^3$$

Fuel oil feed rate to the cracking reactor=2.713m³/h or 0.0007536 m³/s.

The density of fuel oil at 300°C is 797 kg / m³, then the fuel oil consumption:

$$G = 0.6 \text{ kg / s}$$

Power of the induction heating unit:

$$Q = G \cdot c_p \cdot \Delta T = 0.6 \cdot 2.7 \cdot 170 = 275.4 \text{ W.}$$

Composite catalysts were prepared on the basis of natural Kazakhstan raw materials: zeolite of the Taizhuzgen deposit (Figure 2) and clay of the Narynkol deposit.

Figure 2: X-Ray Diffraction Analysis of Taizhuzgen Zeolite.

According to X-ray diffraction analysis, the presence of eight different components in the composition of Taizhuzgen zeolite (concentration, % intensity) was detected: Fe (49.939/739.15); Ca (1.715/9.16); Sr (0.270/1.98); Mn(0.129/1.81); Al (21.955/0.31); Si (23.114/0.98); Ti (1.903/20.87) and K (0.976/1.96).

The starting zeolite and clay were crushed and sieved with a selection of fractions of 63-80 microns. The zeolite was activated by ion exchange of sodium cations of the zeolite framework to cations of lanthanum and ammonium. The sorption of lanthanum ions on zeolites is accompanied by an increase in the pH of the equilibrium solution as a result of cationic exchange of the solution and hydrolysis of zeolites. Zeolites are subjected to the processes of decationization and dealumination. The chemical composition of activated zeolite and clay, as well as zeolite-containing composites was determined by the X-ray fluorescence method. The concentration of lanthanum in the catalysts was measured on a DFS-13 spectrograph using calibration graphs constructed using a reference sample. The phase composition of zeolite-containing composites was determined by X-ray spectral method. The measurement was carried out on a DRON-4 X-ray diffractometer equipped with an Iskra-330 control microcomputer. The value of the specific surface of the catalysts was determined by the method of thermal desorption, which allows surface measurements in the range of 0.1-500 m²/g. The individual hydrocarbon composition of kerosene-gas oil fractions was determined using a HP 5890/5972 gas chromatography mass spectrometer.

In the Table 2 data on composition of catalysts made of Taizhuzgen zeolite and Narynkol clay are shown.

Table 2: Chemical Composition of Catalysts made of Taizhuzgen Zeolite and Narynkol Clay

Chemical Composition	Narynkol Clay	14% Zeolite-Containing Composite	20% Zeolite-Containing Composite	Taizhuzgen Zeolite
SiO ₂	38.05	40.00	47.07	67.93
CaO	20.40	19.77	15.14	1.97
Al ₂ O ₃	8.49	9.97	11.51	14.28
MgO	6.15	6.27	4.72	1.39
Fe ₂ O ₃	4.07	3.75	4.01	1.79
K ₂ O	1.80	2.29	3.02	4.47
Na ₂ O	1.10	<0.50	0.55	1.11
TiO ₂	0.44	0.23	0.41	0.29
P ₂ O ₅	0.11	0.15	0.14	0.01
MnO	0.10	0.15	0.11	0.04
Calcination Losses	19.47	17.43	13.52	7.04
La	0	0.20	0.20	0.24

The spectra of the sample were registered on the JNN-ECA 400 spectrometer of the company “Jeol” (Japan). The operating frequency of the spectrometer is 400 and 100 MHz on ¹H and ¹³C cores, respectively. The survey was performed at room temperature using CDCl₃ solvent. Chemical shifts are measured relative to the signals of residual protons or carbon atoms of deuterated chloroform. The proton spectrum of Amangeldy fuel oil contains ¹H signals of paraffinic, naphthenic and aromatic compounds, which are the main components of the mixture (Figure 3). Signals of methyl protons of saturated hydrocarbons (HC) are noted as a broadened singlet at 0.90 ppm. High-intensity signal with a chemical shift of 1.28 ppm indicates a high content of protons of methylene groups of aliphatic cyclic and acyclic hydrocarbons. A small amount of CH₃ groups was noted in the α-position to the aromatic ring (δ = 2.30-2.63 ppm). In the range of 2.70-4.50 ppm probably the resonance of protons of the CH₂ and CH groups in the α-position of aromatic systems.

Figure 3: ¹H Spectrum of the Fuel Oil from the Amangeldy Gas Processing Plant.

RESULTS AND DISCUSSIONS

The highest yield of gasoline and light gas oil is observed when air is supplied at a speed of 0.15 h^{-1} , and an increase in the concentration of cracked raw materials suppresses the destruction of high molecular weight hydrocarbons that are part of fuel oil to light hydrocarbons of gas and gasoline fractions. The optimum temperature for fuel oil cracking at high feed rates to the reactor is 470°C (Table 3).

Table 3: Effect of Temperature on the Yield of Cracked Fuel Oil Product After Cracking
($w_{\text{suspension}} = 1.0 \text{ h}^{-1}$, $w_{\text{air}} = 0.15 \text{ h}^{-1}$, 0.2 wt. % in Raw Materials of 14% Composite)

T, $^\circ \text{C}$	The Yield of Cracking Products, wt. %						Total
	Gas	Gasoline	Light Gas Oil	Heavy Residue	Coke	Losses	
450	1.1	3.0	42.7	51.7	1.5	0	100.0
470	3.3	8.4	84.3	2.8	1.2	0	100.0
500	2.7	6.4	73.8	15.5	1.6	0	100.0

With an increase in the cracking temperature from 450°C to 470°C , the yield of light gas oil doubles. Thus, in the oxidative cracking of high sulfur fuel oil in a low percentage suspension of a composite from natural raw materials, the conversion to gasoline and light gas oil is 92.7 wt.%. This significantly exceeds the performance of known technologies for catalytic cracking of heavy oil residues (Tanimoto et al., 1998; Wang, 1979; Zhao et al., 1993). A synergistic effect is observed under conditions of the simultaneous presence of a composite catalyst and small amounts of air in the reaction zone (Table 4).

Table 4 :The Effect of the Catalyst and Air on the Cracking Process of Commercial Fuel Oil (a) and Vacuum Gas Oil Obtained from it (b) ($w_{\text{suspension}} = 1.0 \text{ h}^{-1}$, 0.2 wt. % in the Feedstock of a 14% Composite, $T = 470^\circ \text{C}$)

No	Catalyst	$w_{\text{air}}, \text{h}^{-1}$	The Yield of Cracking Products, wt. %								Total
			Gas	Gasoline	Light Gas Oil 185-340 $^\circ \text{C}$	Light Gas Oil 340-350 $^\circ \text{C}$	the total amount of light gas oil	Heavy Residue	Coke	Losses	
a	Without catalyst	0	1.8	0.8	-	-	12.3	83.7	1.4	0	100.0
	With catalyst	0	2.2	2.1	11.8	8.4	20.2	73.9	1.6	0	100.0
	Without catalyst	0.15	2.6	1.2	12.4	6.8	19.2	74.5	1.4	1.1	100.0
	With catalyst	0.15	3.3	8.4	42.5	41.8	84.3	2.8	1.2	0	100.0
b	With catalyst	0.15	8.6	0.2	28.3	12.9	41.2	50.0	0	0	100.0

It should be noted that when cracking vacuum gas oil obtained from fuel oil, the yield of the kerosene-gas oil fraction is reduced by half (Table 4, b) compared to cracking under identical conditions of the original fuel oil (Table 4, a). As can be seen from Table 4(a), the composition of the kerosene-gas oil fraction of cracking products, depending on the conditions of its implementation, includes from 35.6 to 49.6 wt.% of the fraction boiling off in the temperature range of $340\text{--}350^\circ \text{C}$. The catalytic destruction of hydrocarbon molecules proceeds through the formation of free radicals, therefore the introduction of air, the oxygen of which is the initiator of this process, significantly increases the yield of the middle distillate fraction.

CONCLUSIONS

- The effect of air additives in the reaction mixture on the cracking of fuel oil on a composite catalyst at a high feed rate of fuel oil has been studied.
- Commodity fuel oil and fuel oil of the M-100 brand from the Amangeldy Gas Processing Plant (Kazakhstan) were used as cracking raw materials.
- Composite catalysts were prepared on the basis of natural Kazakhstan raw materials: zeolite of the Taizhuzgen deposit and clay of the Narynkol deposit.
- It was shown that the technological parameters of the fuel oil cracking process determine the hydrocarbon composition of the resulting products.
- The air additives contribute to a deeper destruction of the hydrocarbons of the cracked feed. The highest yield of gasoline and light gas oil is observed at a speed of air 0.15 h⁻¹. An increase in the concentration of cracked raw materials suppresses the destruction of high molecular weight hydrocarbons (that are a part of fuel oil) to light hydrocarbons of gas and gasoline fractions.
- With the simultaneous action on the cracked molecules of the active centers of the optimal catalyst and atmospheric oxygen, a synergy was established in the yield of the middle distillate fraction.
- The results of the research on the use of activation of cracked fuel oil by dosed injection of air into the reaction zone indicate great prospects for a new direction in the catalytic processing of heavy hydrocarbon feedstocks.

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