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**OBTAINING HIGH-MOLECULAR HYDROCARBONS FROM
 SYNTHESIS GAS AND PHYSICO-CHEMICAL CHARACTERISTICS OF
 THE CATALYST**

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ABSTRACT

The article studied the reaction of obtaining high-molecular hydrocarbons from the synthesis gas and the physicochemical characteristics of the catalyst containing Co-Fe-Ni-ZrO₂/HSZ used in this process. The reason for the increase in selectivity in methane formation is, firstly, the acceleration of the hydrogenation reaction of CO in the methane formation centers with increasing synthesis temperature and secondly, the diffusion of CO and H₂ at different speeds through zeolite pores. In all fractions of hydrocarbons synthesized in the presence of hybrid catalysts, secondary changes: isoparaffins and olefins are formed due to cracking and isomerization. A large amount of isoparaffins is recorded for the composite catalyst - the iso/n parameter is 0.6, which indicates an increase in its activity relative to the catalysts absorbed in the hydrogenation reactions. For catalysts with low cobalt content, the concentrations of olefins will be high, which is due to their low hydrogenation ability compared to unsaturated hydrocarbons. In absorption catalysts, the hydrocarbon synthesis centers and the zeolite acid sites are in close contact with each other, which contributes to the intensification of secondary processes. However, the content of liquid hydrocarbons (C₅-C₁₈) in C₅+ hydrocarbons is about 83%, which is 12% less than in the composite catalyst. No oxide-oxide interaction was found for the composite catalyst prepared by mixing the components, the pores of the zeolite are not

blocked by cobalt, which allows the reagents to be efficiently delivered to the active part of the hydrocarbon synthesis.

KEYWORDS: *Synthesis Gas, High Molecular Weight Hydrocarbons, Catalyst, IR Spectrum, Diffractogram.*

INTRODUCTION

One of the most promising methods of obtaining motor fuel from carbon-containing sources, which is an alternative to oil, is GTL technology. Unlike petroleum products, synthetic fuels in practice do not contain aromatic mono and polycyclic compounds, organic sulfur and nitrogen compounds, and are environmentally friendly and high-quality consumer fuels [1-5]. In nature, the raw material base of synthetic fuels is widespread, consisting of carbon-containing materials - natural, petroleum gases, coal, biomass, etc. [6-11]. GTL technology involves the following steps: production of synthesis gas (a mixture of CO₂ and H₂); catalytic conversion of synthesis gas to hydrocarbons by the Fisher-Tropsh method; separation of products into fractions of gasoline (C₅-C₁₀), diesel (C₁₁-C₁₈), wax (C₁₉₊) [12-16].

The main stage of GTL technology is the catalytic conversion of synthesis gas to hydrocarbons by the Fisher-Tropsh method (FT-synthesis). In the catalytic conversion of synthesis gas to hydrocarbons, cobalt, nickel, ruthenium, iron, etc., from group VIII metals with variable valence, exhibit catalytic activity.

Cr, Mo, W from group VI metals, Mb, Te, Re from group VII metals and Cu, Ag, Au from group IV metals increase the activity level of the catalyst to some extent [17-21]. In practice, two types of FT-synthesis are used in industry: low-temperature and high-temperature variants [22-23].

Low-temperature synthesis (LTFT) is carried out in cobalt or iron catalysts at temperatures below 300 °C. It forms mainly high-molecular-weight n-paraffins, n-olifins and oxygen-containing components. High-temperature synthesis (NTFT) is carried out in an iron catalyst at temperatures above 300 °C. The product is mainly a mixture of hydrocarbons with a high content of olifin, which are used as the main raw material for many chemical processes. FT is characterized by a relative lack of isoparaffins for synthesis products, so the hydrocarbons being synthesized have a low octane number. The turbidity temperature and filtration norm of the diesel fraction are not very high, which makes it difficult to use these components as fuel.

Therefore, in both options it is necessary to change the composition of raw materials obtained in the production of motor fuel, which are hydrolysed by combining processes and cracking, isomerization and other processes according to the technological scheme [19-23]. It is advisable to intensify each stage of the process in order to demonstrate the workability of the GTL technologist in obtaining the motor fuel in the traditional way. One solution to this problem is to implement a single reactor by combining hydrocarbonar synthesized hydrolysis steps. This requires the development of new Effective Hybrid Catalysts. In the process of GTL technologist, blockchain-enabled mobile technology allows simple equipment to process petroleum gases and pastes pressurized gases of used gas cones in mining conditions. developed [15-22].

The aim of the work is to obtain high molecular weight hydrocarbon from synthesis-gas and to organize the physicist-chemical characteristic of catalyting.

EXPERIMENTAL PART

In the synthesis of hydrocarbons from CO and H₂, the catalytic properties of the catalysts were tested under the following optimal conditions in a flow isothermal reactor filled with 30 cm³ of quartz (catalyst layer 10 cm³): temperature range 280-300 °C, 0.5 MPa and volumetric velocity of gas flow (GHT) 1000 h⁻¹. Balanced experiments were performed for at least 150 h, the incoming and outgoing gas composition was analyzed, and the amount of hydrocarbons and reaction water obtained was recorded. The activity of hydrocarbon synthesis catalysts was evaluated on the following indicators: CO conversion, selectivity and productivity for hydrocarbons. The calculation error did not exceed 2.5%.

CO conversion was calculated according to the following formula:

$$X_{CO} = \frac{V_{in} \cdot c(CO)_{in} - c(CO)_{out}}{V_{in} \cdot c(CO)_{in}} \cdot 100\%$$

where V_{in}, V_{out} - gas consumption at the inlet and outlet of the reactor, dm³/h;

c(CO)_{in}, c(CO)_{out} - Co concentration at the inlet and outlet of the reactor, unit fraction. Methane selectivity was calculated according to the following formula:

$$S_{CH_4} = \frac{V(CO)_{CH_4}}{V(CO)_{np}} \cdot 100\%$$

where V(CO)_{CH₄} is the volume of CO used to produce methane, dm³; V(CO)_{conv} is the volume of CO converted to methane, dm³. Selectivity for C₅ + hydrocarbons was calculated using the formula:

$$S_{C_{5+}} = \frac{V(CO)_{C_{5+}}}{V(CO)_{conv}} \cdot 100\%$$

bu erda V(CO)_{C₅₊} - C₅₊ uglevodorodlarni hosil qilish uchun sarflangan CO hajmi, dm³.

C₅₊ uglevodorodlari uchun unumdorlik quyidagi formula bo'yicha hisoblab chiqildi:

$$G_{C_{5+}} = \frac{m_{C_{5+}}}{V_{cat} \cdot \tau}$$

where m_{C₅₊} - C₅₊ - mass of hydrocarbons, kg; V_{cat} - catalyst volume, m³; t - time, hour.

Determination of the composition of synthesis products. The composition of gaseous synthesis products was analyzed on a chromatograph "Crystal 5000" (Chromatek, Russia) equipped with a thermal conductivity detector with gas adsorption chromatography and two columns with active Haysep R phase and NaX molecular sieves. Analysis mode The temperature can be programmed with a heating rate of 8 °C/min. The composition of C₅₊ hydrocarbons was determined by capillary gas-liquid chroma-mass spectrometry on a gas chromatograph (Agilent, USA) equipped with a mass-selective detector MSD 5975C.

RESULTS AND DISCUSSION

The study of the catalytic properties of the samples during the synthesis of hydrocarbons from CO and H₂ was carried out at a temperature of 290 °C, a pressure of 0.5 MPa, a volumetric

velocity of the gas at 1000 h^{-1} and a ratio of $\text{H}_2/\text{CO}=2$. High-silicon zeolite derived from Navbahor bentonite was used as the catalyst holding agent [24-28].

The experimental data obtained are presented in Table 1. Analysis of the dependence of the conversion rate of CO on the synthesis temperature shows that the composite catalyst exhibits higher activity than the absorption catalysts in FT-synthesis. Despite a 3-fold decrease in cobalt concentration, an increase in FT-synthesis temperature to $290 \text{ }^\circ\text{C}$ in the presence of a composite catalyst allows CO conversion rates to be increased by 74.2% and hydrocarbon $\text{C}_5 +$ by 91.7 $\text{kg}/\text{m}^3_{\text{cat}}\text{-hour}$. The FT-synthesis catalyst is comparable to similar values for Co-Fe-Ni-ZrO₂/HSZ (Table 1).

TABLE 1. CATALYST TEST RESULTS

Catalyst	C _{CO} , %	T, °C	X _{CO} , %	selectivity, %				G _{C₅₊} , $\text{kg}/\text{m}^3_{\text{cat}} \text{ h}$
				CH ₄	C ₂ -C ₄	CO ₂	C ₅₊	
Co-Fe-Ni-ZrO ₂ /HSZ	20,8	225	76,2	16,1	10,4	1,4	72,1	115,0

Note: X_{CO} is the conversion rate of CO; Productivity for G_{C₅₊} - C₅₊ hydrocarbons.

Samples of hybrid catalysts are characterized by a decrease in the selectivity of C₅₊ hydrocarbon formation to 58-66% relative to the Co-Fe-Ni-ZrO₂/HSZ catalyst. The intensification of these intermediate processes - the formation of methane and hydrocarbons C₂-C₄, is associated with the reaction of water gas. The increase in selectivity in methane formation is due, on the one hand, to the acceleration of the hydrogenation reaction of CO at the methane formation centers and the diffusion of CO and H₂ at different rates through zeolite pores due to the increase in synthesis temperature. As mentioned above, a Co-Fe-Ni-ZrO₂/HSZ catalyst of FT synthesis has been developed for the synthesis of high molecular weight hydrocarbons (C₁₉₊). Therefore, their content in C₅₊ hydrocarbons reaches 46.7% (Table 2). The group composition of C₅₊ hydrocarbons is characterized by paraffins of normal content, the content of isoparaffins and olefins does not exceed 3%. The molecular mass distribution of hydrocarbons is shown in Figure 1 below. In the diagram showing the distribution of the amount of hydrocarbons obtained according to the number of carbon atoms in the molecule, the main part of the obtained hydrocarbons includes up to 25 carbon atoms, a significant part - 25-35. The fractional composition of C₅₊ hydrocarbons obtained in the presence of hybrid catalysts is mainly represented by liquid hydrocarbons (C₅-C₁₈), their content is 83% for absorbing catalysts and 95% for composite catalysts. This is mainly due to the cracking of C₁₉₊ hydrocarbons in the acid center of the zeolite. Against the background of a decrease in the content of hydrocarbons C₁₉₊ and partly C₁₁-C₁₈, an increase of C₅-C₁₀ hydrocarbons by 24-34%, redistribution of products of synthesis of products obtained in the presence of FT-synthesis catalyst Co-Fe-Ni-ZrO₂/HSZ compared to their content.

TABLE 2. THE COMPOSITION OF THE OBTAINED S₅ + HYDROCARBONS

Catalyst	Hydrocarbons	Content, %			Total	iso/n
		C ₅ -C ₁₀	C ₁₁ -C ₁₈	C ₁₉₊		
Co-Fe-Ni-ZrO ₂ /HSZ	n-paraffins	17,2	34,7	45,5	97,4	0,02
	isoparaffins	0,4	0,6	0,8	1,8	
	olefins	0,1	0,3	0,4	0,8	
	Total	17,7	35,6	46,7	100	

Note: iso/n - isoparaffins: paraffin ratio

In all fractions of hydrocarbons synthesized in the presence of hybrid catalysts, a large amount of products of secondary transformations (cracking, isomerization) - isoparaffins and olefins were found. A large amount of isoparaffins is recorded for the composite catalyst - the iso/n parameter is 0.6, which indicates an increase in its activity relative to the catalysts absorbed in the hydroimprovement reactions. High concentrations of olefins have been found for catalysts with low cobalt content, which is due to their low hydrogenation ability compared to unsaturated hydrocarbons.

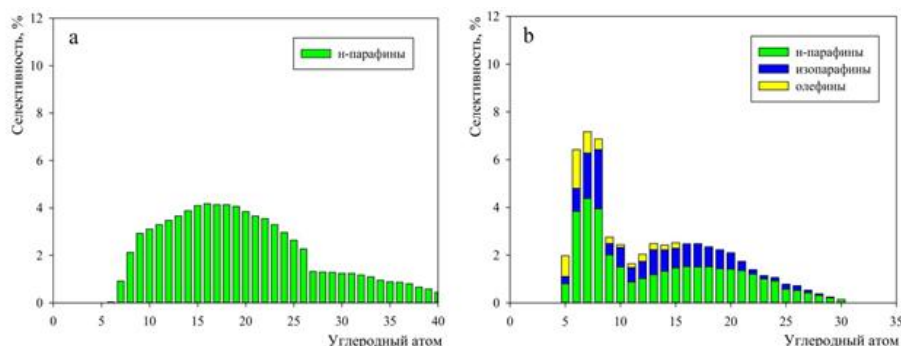


Figure 1. Molecular-mass distribution of C_{5+} hydrocarbons obtained in the presence of catalysts: a - Co-Fe-Ni-ZrO₂/HSZ; b - absorbent, containing 6.5% cobalt.

The use of zeolite in hybrid catalysts allows the production of C_{5+} hydrocarbons whose molecular-mass distribution does not obey the AShF equation. Maximum MMD corresponds to C_5 - C_{10} hydrocarbons. The products mainly consist of liquid hydrocarbons. For selective catalysts, the selectivity in the formation of C_5 - C_{18} hydrocarbons is 46–49%, and for the composition, 62.6% (Fig. 1). Thus, the use of cobalt mixing and cobalt precipitation methods by absorption into the resulting carrier was found to have a significant effect on the physicochemical and catalytic properties of hybrid catalysts. The use of the absorption method to obtain hybrid catalysts creates a system that exhibits lower catalytic properties than a composite catalyst during the synthesis of hydrocarbons. This is probably due to both diffusion factors, the blockage of pores by sedimentary cobalt, and the formation of compounds of cobalt and aluminum oxides that are difficult to recover from the oxide-oxide interaction. In absorber catalysts, the hydrocarbon synthesis centers and the zeolite acid sites are in close contact with each other, which should contribute to the intensification of secondary processes.

However, the content of liquid hydrocarbons (C_5 - C_{18}) in C_{5+} hydrocarbons is about 83%, which is 12% less than in the composite catalyst. No oxide-oxide interaction was found for the composite catalyst prepared by mixing the components, the pores of the zeolite are not blocked by cobalt, which allows the reagents to be efficiently delivered to the active part of the hydrocarbon synthesis. Such a catalyst has a high activity in the synthesis of hydrocarbons - the conversion rate of CO is 74.2%, the yield and selectivity for C_{5+} hydrocarbons is 91.7 kg/m³_{cat} · h and 66.5%. In a composite catalyst localized in silicon oxide, the acid center is not blocked by cobalt located above the silicon oxide in the Co-Fe-Ni-ZrO₂/HSZ catalyst. There are both external and internal centers for hydrocarbons, as a result of which the composite catalyst increases the activity in hydrolysis reactions - the amount of liquid hydrocarbons reaches 95%.

In this regard, the preferred method of preparation of hybrid catalysts is to mix the active components using a binder. Structure and phase composition of catalysts. The phase composition and structure of the catalysts were studied using RFA and IR spectroscopy. The radiographic image is the same for all samples (Figure 2)

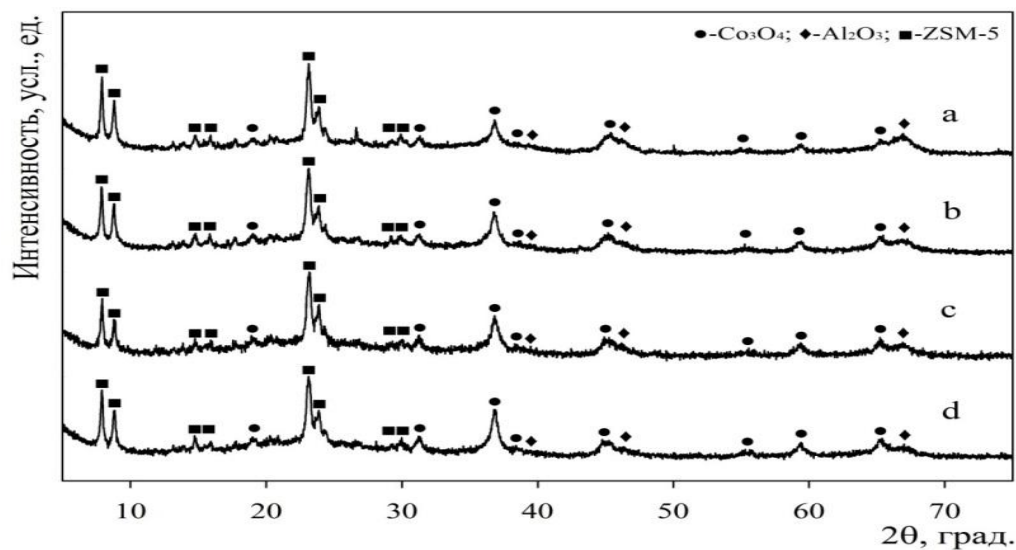


Figure 2. Diffractograms of catalysts containing different amounts of cobalt, %: a - 4.2; b - 6,3; c - 7,4; d - 8.4

The size of the Co_3O_4 particles varies in the range of 10–12 nm, with no deviations recorded at the elemental cell parameter 8.088 Å in the Co_3O_4 structure associated with the change in the components in the catalyst. According to IR spectroscopy data, no interaction of cobalt oxide with the catalyst components was observed (Fig. 3).

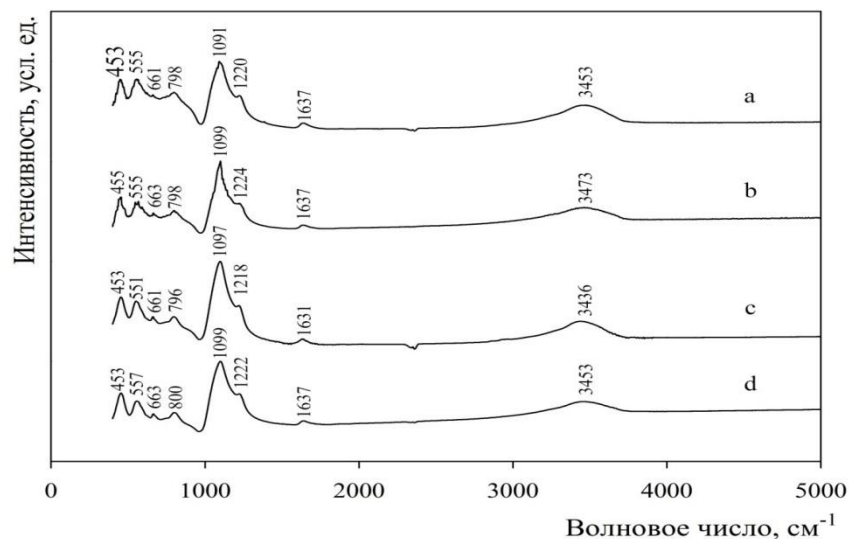


Figure 3 - IR spectra of catalysts of different cobalt content, %: a - 4.2; b - 6,3; c - 7,4; d - 8.4

By increasing the cobalt content in the catalysts, the intensity of the absorption bands corresponding to the Co^{2+} vs Co^{3+} oscillations increases. Between $3000\text{-}3700\text{ cm}^{-1}$, $1600\text{-}1650\text{ cm}^{-1}$ and $750\text{-}850\text{ cm}^{-1}$ the intensity of the absorbing bands varies with respect to the vibrations of the surface hydroxyl groups, the crystallization water and the Al-O bond, which is the aluminum content of the composite catalysts. associated with a decrease in the amount of oxide.

CONCLUSION

Thus, the reaction of obtaining high-molecular hydrocarbons from the synthesis gas and the physicochemical characteristics of the catalyst containing Co-Fe-Ni-ZrO₂/HSZ used in this process were studied. The reason for the increase in selectivity in methane formation is, firstly, the acceleration of the hydrogenation reaction of CO in the methane formation centres with increasing synthesis temperature and secondly, the diffusion of CO and H₂ at different speeds through zeolite pores. Secondary changes in all fractions of hydrocarbons synthesized in the presence of hybrid catalysts: isoparaffins and olefins are formed due to cracking and isomerization. A large amount of isoparaffins is recorded for the composite catalyst - the iso/n parameter is 0.6, which indicates an increase in its activity relative to the catalysts absorbed in the hydrogenation reactions. For catalysts with low cobalt content, the concentrations of olefins will be high, which is due to their low hydrogenation ability compared to unsaturated hydrocarbons. In the absorption catalysts, the hydrocarbon synthesis centres and the zeolite acid sites are in close contact with each other, which contributes to the intensification of secondary processes. However, the content of liquid hydrocarbons (C₅-C₁₈) in C₅₊ hydrocarbons is about 83%, which is 12% less than in the composite catalyst. No oxide-oxide interaction was found for the composite catalyst prepared by mixing the components, the pores of the zeolite are not blocked by cobalt, which allows the reagents to be efficiently delivered to the active part of the hydrocarbon synthesis.

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