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АКЦИОНЕРЛІК ҚОҒАМЫ

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# N-HEXADECANE HYDROISOMERIZATION IN THE PRESENCE OF AL-HMS - BASED BIFUNCTIONAL CATALYST

**Abstract.** In this article the synthesis of a nickel-promoted composite based on a mesoporous aluminosilicate of the Al-HMS type and activated bentonite from the Tagan deposit is provided. The physical and chemical characteristics of the Ni/Al-HMS-bentonite composite and the action of temperature on the group hydrocarbon-type content of the normal hexadecane hydroisomerization products in the presence of a synthesized aluminosilicate - based composite were studied. Adsorption studies show that the synthesized catalyst Ni/Al-HMS-bentonite with a silicon-to-aluminium ratio (Si/Al) = 20 is characterized by a higher specific surface area (570 $m^2/g$). It has been established that 300 °C is the optimum temperature for carrying out the process of hydroisomerization of normal hexadecane. Under these conditions the main direction of its transformation is the isomerization process. The total yield of isoparaffins was 45%.

**Key words:** Mesoporous aluminosilicate, hydroisomerization, dewaxing, diesel fuel oil, higher n-paraffins, template.

The process of hydroisomerization of oils' diesel fractions on aluminosilicate catalysts is the most promising low-tonnage production of low pour point diesel fractions.

Diesel fractions obtained during the primary oil refining usually contain a significant amount of normal paraffins and therefore are characterized by high temperature properties and are unsuitable for use as fuel during the winter period in cold regions without additional refining.

One of the most promising ways to improve the performance of diesel fractions is the process of catalytic dewaxing of diesel fractions. The process of catalytic dewaxing is based on the selective hydrocracking and hydroisomerization of higher n-paraffins of the diesel fraction on bifunctional catalysts [1-9]. The main feature of bifunctional catalysts is the ability of their active centers to accelerate reactions proceeding via different mechanisms. Most often, bifunctional catalysts are used, in which simultaneously exist: acidic active sites, which accelerate the reactions of breaking and isomerization of the hydrocarbon chain, and sites which are active in hydrogenation and dehydrogenation reactions.The selectivity of bifunctional catalysts depends on the ratio of their acidic and hydro-dehydrogenating functions. Mesoporous aluminosilicates with a specific surface area of more than 500 $m^2/g$ are most often used as the acid base of catalysts. Acidic activity is determined by the structural type of aluminosilicate and the composition of its active centers, which, in turn, depends on the chemical composition and aluminosilicate's modification conditions [10-18]. The hydro-dehydrogenating function is determined by three main factors: the chemical nature of the metal component of the catalyst, its specific surface area and process parameters – temperature, hydrogen pressure.

The purpose of this work is the synthesis of effective and stable composites based on mesoporous aluminosilicateand activated bentonite modified with nickel for the process of hydroisomerization of n-hexadecane.

## EXPERIMENTAL PART

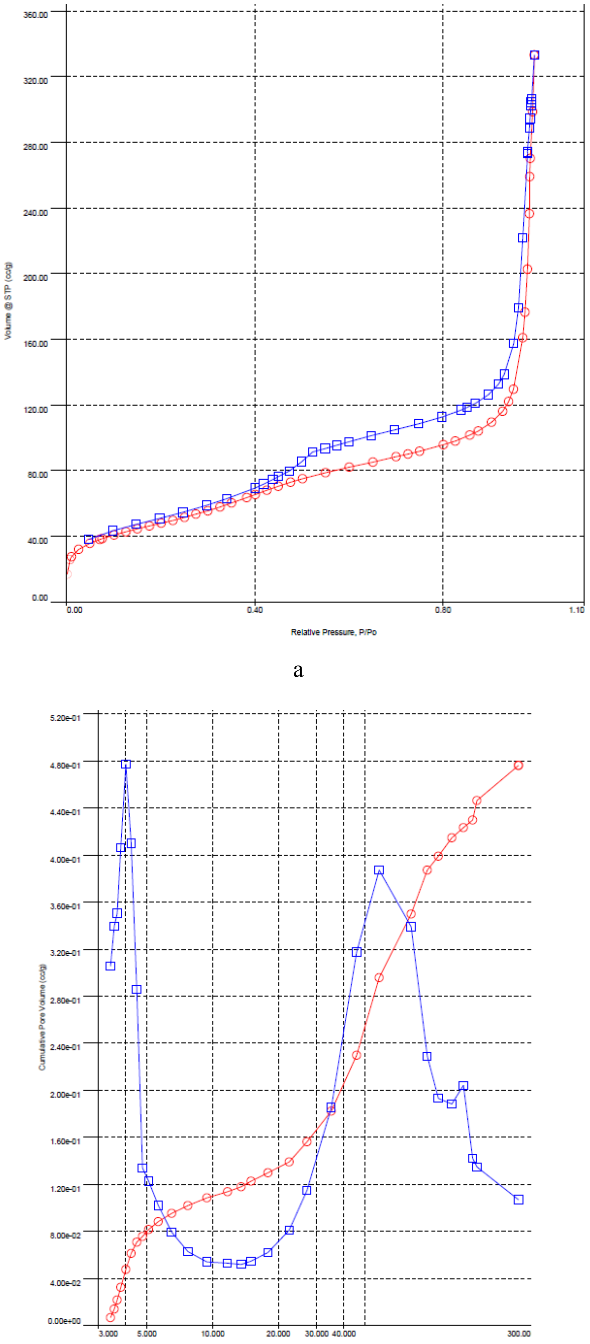
In this work, nickel-containing composite was synthesized usingAl-HMS type mesoporous aluminosilicate and bentonite from the Tagan deposit as the acidic part. Synthesis of mesostructured aluminosilicate was carried out according to the technique [19], based on the joint hydrolysis of $Si(OC_2H_5)_4$ and $Al(Oi\text{-}Pr)_3$ in an alkaline medium. Hexadecylamine was used as a template. In order to study the activity of the synthesized catalyst, n-hexadecane was used as a model compound,since it is a part of the diesel fraction.

The process of catalytic conversion of n-paraffin was carried out in a laboratory setup with a fixed-bed catalyst under hydrogen in the temperature range of 350-400 °C; feed rates were 1 $h^{-1}$, hydrogen/raw materials ratio equal to 1000 $nm^3/m^3$, hydrogen pressure in the reactor is of 3 MPa. Analysis of liquid products was carried out on the "Crystal 5000" chromatograph with linear programming of the temperature from 35 °C to 250 °C. "Crystal 5000" chromatograph is equipped with a DB-1 column 100 meters long, with a diameter of 0.25 mm, polymethylsiloxane is a liquid stationary phase. The carrier gas is helium.

Adsorption studies were carried out on $N_2$ adsorption isotherms at 77 K, which were measured on Micromeritics' (USA) ASAP-2400 installation after training the samples in vacuum at 1500 °C. These isotherms were used to calculate the total accesssible surface by the BET method, the total porosity $\sum V_{pores}$ with effective sizes up to 100-200 nm (according to the value of adsorption at a relative nitrogen pressure of ~ 0.99), the distribution of the volume of mesopores by characteristic sizes (according to the desorption curve of the isotherm using the BJHV, micropores volume $V_{micropores}$ and mesopores surface $S_{mesopores}$ remaining after micropores filling.

## RESULTS AND DISCUSSION

The results on the study of the porous structure and specific surface area of Al-HMS mesoporous aluminosilicates with a Si/Al ratio of 20 are presented in figure and table 1.



a

b

Adsorption/desorption isotherms (a) and pore size distribution (b) of Ni/Al-HMS-bentonite sample with a Si/Al ratio = 20

Table 1 – Physico-chemical characteristics of Ni / Al-HMS-bentonite catalysts with a Si/Al ratio = 20

| Catalyst | SSA, $m^2/g$ | $D_{pores}$, nm | $V_{pores}$, $cm^3/g$ |
|---|---|---|---|
| Ni/Al-HMS(20)-bentonite | 570 | 4,1 | 0,8 |

As follows from the data of Figure 1, the nitrogen adsorption/desorption isotherm for the Ni/Al-HMS (20)-bentonite sample is characterized by a slightly wide hysteresis loop. Nickel promotion of catalysts based on mesoporous aluminosilicate leads to significant changes in the structural characteristics of the catalyst compared to the nitrogen adsorption/desorption isotherm for the Al-HMS (20) sample [20]. There are three peaks observed on the pore effective diameters distribution curve: one of which corresponds to the mesoporous aluminosilicate, the second - to bentonite and the third - to the promoting additive. The sample under study is characterized by a high specific surface area, which is of 570 $m^2/g$.

Hexadecane was used in order to establish the mechanism of heavy petroleum residues hydroisomerization in the presence of a composite based on the mesoporous aluminosilicate and bentonite, promoted by nickel.

Process of hexadecane transformation on the compositeon a basis of the promoted mesoporous aluminosilicatewas studied in a temperature interval of 300-450 °C, at a feed rates – 1 $h^{-1}$, under hydrogen pressure and at a hydrogen/raw materials ratio of 100 (vol.). The experimental data are provided in tables 2, 3.

Table 2 – The action of the temperature on the group hydrocarbon-type content of the products of hexadecane transformation on the Ni/Al-HMS(20)-bentonite composite, Wfeed rate = 1,0 $h^{-1}$, $P_{H2}$= 3 MPa

| № | Hydrocarbon-type content | Content by mass % | | |
|---|---|---|---|---|
| | | 300 °C | 350 °C | 450 °C |
| 1 | N-paraffins | 31,98 | 36,39 | 43,02 |
| 2 | Isoparaffins | 45,23 | 39,76 | 12,41 |
| 3 | Arenes | 5,20 | 8,25 | 7,16 |
| 4 | Naphthenes | 8,66 | 8,41 | 15,83 |
| 5 | Olefins | 5,23 | 4,10 | 14,98 |
| 6 | Dienes | 0,70 | 0,80 | 5,30 |
| 7 | Cycloolefins | 2,25 | 2,04 | 0,67 |
| 8 | Cyclodienes | 0,75 | 0,25 | – |

From the experimantal data obtained (table 2) it is seen that isomerization process is the main direction of hexadecane transformation under the conditions of low-temperature alumiosilicate catalysis (300 °C). It also should be noted that isometric structures of hexadecane are obtained due to isomerization process, without cracking participation. As the temperature rises from 300 to 350 °C parallel with isomerization reactions, there are also decomposition, dehydrogenating and hydrogen disproportionation reactions. Marginally there are dehydrocyclization reactions with the formation of aromatic hydrocarbons, which mostly presented by alkylaromatics (polysubstituted alkyl benzenes). Destruction of hexadecane goes on to the n-decane.

The increase in the concentration of n-paraffinic hydrocarbons by 5% is clearly due to a slight shift of equilibrium state paraffin ↔ isoparaffin to paraffins. However, a significant part of the isoparaffins under these conditions is undergone a decomposition with the formation of olefinic and diene hydrocarbons. At the same time, the released hydrogen is participated in the hydrogenation reactions of cycloolefine and cyclodiene hydrocarbons and partially of arenes with the formation of naphthenes.

Table 3 – Hydrocarbon-type contentof gaseous phase, obtained during n-hexadecane hydroisomerization on Ni/Al-HMS(20)-bentonite composite, T = 450 °C, $P_{H2}$ = 3 MPa

| № | Content | Amount by mass % |
|---|---|---|
| 1 | $H_2$ | 37,30 |
| 2 | $CH_4$ | 23,70 |
| 3 | $C_2H_4$ | 2,00 |
| 4 | $C_2H_6$ | 2,80 |
| 5 | $C_3H_6$ | 2,70 |
| 6 | $C_3H_8$ | 1,30 |
| 7 | i-$C_4H_{10}$ | 9,80 |
| 8 | $C_4H_{10}$ | 8,00 |
| 9 | $C_4H_8$trans | 0,20 |
| 10 | $C_4H_8$cis | 4,00 |
| 11 | $C_5H_{10}$ | 1,50 |
| 12 | i-$C_5H_{12}$ | 1,40 |
| 13 | $C_5H_{12}$ | 5,30 |

The deepening of the cracking of hexadecane during the process at 450 °C is also indicated by the qualitative and quantitative composition of the gas-formation products (table 3), the hydrocarbon composition of which is represented mainly by $C_1$-$C_5$ fragments and hydrogen, but the main components of the cracking products are methane and hydrogen, and content of $C_4$-$C_5$ isocompoundsis of 12, 4 by mass %.

Thus, a nickel-promoted composite based on a mesoporous aluminosilicate of the Al-HMS type and activated bentonite from the Tagan deposit was synthesized for the process of hydroisomerization of n-hexadecane. On the basis of experimental data, it may be said that the process of transformation of normal paraffinic hydrocarbons in the presence of a composite based on mesoporous aluminosilicate and bentonite occurs as follows: paraffins → olefins →naphthenes → aromatic hydrocarbons. It has been established that the optimal temperature of the process for the hydroisomerisation of n-hexadecane is of 300 °C. The yield of the total amount of isoparaffins is of 45%.

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## Резюме

*Р. М. Мойса, Г. К. Василина, К. М. Умбеткалиева, Т. С. Абильдин*

## Al-HMS НЕГІЗІНДЕГІ БИФУНКЦИОНАЛДЫ КАТАЛИЗАТОРДЫҢ ҚАТЫСУЫМЕН Н-ГЕКСАДЕКАНДЫ ГИДРОИЗОМЕРЛЕУ

Мақалада Al-HMS мезокеуекті алюмосиликаты мен Таған кенорнының белсендірілген бентониті негізіндегі никельмен промоторланған композит синтезделген. Синтезделген композиттің қатысында н-гексадеканды гидроизомерлеу процесі өнімдерінің топтық көмірсутегі құрамына температураның әсері және Ni/Al-HMS-бентонит композитінің физика-химиялық сипаттамалары зерттелді. Адсорбциялық зерттеулер Si/Alқатынасы 20-ға тең синтезделген катализаторы (Ni/Al-HMS-бентонит) үлкенменшікті бетімен (570 м$^2$/г) сипатталатыны көрсетті. н-Гексадеканды гидроизомерлеу процесін жүргізу үшін оңтайлы температуралық режим 300 °С екені анықталды, оның осы жағдайда айналуының негізгі бағыты изомерлеу процесі болып табылады. Изопарафиндердің жалпы шығуы 45% құрады.

**Түйін сөздер:** мезокеуекті алюмосиликат, гидроизомерлеу, депарафиндеу, дизель отыны, жоғары қ-парафиндер, темплат.

## Резюме

*Р. М. Мойса, Г. К. Василина, К. М. Умбеткалиева, Т. С. Абильдин*

# ГИДРОИЗОМЕРИЗАЦИЯ Н-ГЕКСАДЕКАНА В ПРИСУТСТВИИ БИФУНКЦИОНАЛЬНОГО КАТАЛИЗАТОРА НА ОСНОВЕ Al-HMS

В статье синтезирован промотированный никелем композит на основе мезопористого алюмосиликата Al-HMS и активированного бентонита Таганского месторождения. Исследованы физико-химические характеристики композита Ni/Al-HMS-бентонит и влияние температуры на групповой углеводородный состав продуктов процесса гидроизомеризации н-гексадекана в присутствии синтезированного композита. Адсорбционные исследования показывают, что синтезированный катализатор Ni/Al-HMS-бентонит с соотношением Si/Al = 20 характеризуется большей удельной поверхностью (570 $м^2/г$). Установлено, что 300 °С – оптимальный температурный режим для проведения процесса гидроизомеризации н-гексадекана, основным направлением превращения которого в этих уловиях является процесс изомеризации. Суммарный выход изопарафинов составил 45%.

**Ключевые слова:** мезопористый алюмосиликат, гидроизомеризация, депарафинизация, дизельное топливо, высшие н-парафины, темплат.