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ABSTRACT

the dissertation for the degree
of Doctor of Philosophy

**INVESTIGATION OF THE JOINT HYDROTREATING
PROCESS OF DIESEL DISTILLATES AND VEGETABLE
OILS IN THE PRESENCE OF HALLOYSITE MODIFIED
WITH Ni, Co, Mo OXIDES**

Speciality: 3303.01 – Chemical technology and engineering

Field of science: Technical

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Baku – 2021

The work was performed at the Institute of Petrochemical Processes named after Yu.G. Mammadaliyev of Azerbaijan National Academy of Sciences at the «Renewable Fuels» Laboratory

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General description of work

Relevance of the topic. Large-scale use of petroleum fuels causes serious problems such as depletion of fuel and environmental pollution. Taking it into consideration the acquisition of various engine fuels using renewable raw materials plays a major role in the increase of reserve fund.

Depending on the amount of sulfur compounds contained in the oil products, there are two main areas of research to solve environmental problems caused by oil products: to increase the use of renewable energy sources for the replacement of petroleum fuels and to develop new highly active hydrotreating catalysts.

Biofuels obtained from biomass have great potential as an alternative to petroleum fuels. As an alternative raw material it is necessary to use various types of biomass, including processed vegetable oil which is not used in food industry. Currently, one of the ways to use plant-based raw materials as engine fuels is their processing with mineral oil fractions.

The production of high-qualitative diesel fuels as a result of hydrotreating process by adding vegetable products (cotton, sunflower, corn oils) of Azerbaijan to diesel fractions is directly related to the creation of new catalysts with high activity and selectivity.

There are two main disadvantages of the currently used industrial hydrogen catalysts: first, the catalysts have low activity, selectivity and stability due to the inactivation of the carrier and non-selectivity of the active component and the second is that most of the active metals are in narrow pores of the catalyst which is not suitable for catalysis. Therefore, the study and production of hydrostatic catalysts with optimal composition and the structure of the carrier localized only in pores suitable for catalysis of metals as an active component are extremely important.

From this point of view, it is reasonable to work out and develop new catalyst systems based on new types of catalysts - natural halloysite and their enriched transition metal (Ni, Co, Mo)

oxide for hydrotreating processes of mixture of vegetable oil and diesel fractions and it is of great practical importance.

Object and subject of research. In order to meet the perspective demand for the quality and quantity of diesel fuel extracted from oil, research has been conducted to obtain low-sulfur “green” diesel fuel by adding renewable raw materials to diesel fuel and conducting hydrotreating in a newly modified catalytic system with modified Ni, Co and Mo oxides.

The results of the research show that higher results can be achieved using a modified catalyst by the cavitation method.

The goal of the work. The goal of the work is to develop catalytic systems of natural aluminosilicate nanotube modified by transition metal oxides and to study the process of producing “green” diesel fuel of high-quality and meeting environmental standards as a result of joint hydrotreating by adding non-food products (cotton, sunflower and corn oil) of Azerbaijan plants to diesel fractions.

It is planned to carry out the following research for this purpose:

- The creation of a catalytic system modified by Ni, Co, and Mo oxides by absorption and ultrasonic cavitation of halloysite nanotube samples for highly-selective hydrotreating processes;
- Production of low - sulfur and environmentally friendly "green" diesel fuel as a result of hydrotreating process in the presence of newly developed catalysts by adding 10 % cotton oil to the diesel fraction of primary processing;
- Investigation of hydrotreating process in the presence of catalytic systems by adding catalytic cracking (15 %), light flexion of coking (15%) and vegetable oil (10 %) to the diesel fraction of initial processing.

Investigation methods. On investigation there were used the following methods: chemical (determination of qualitative measures of diesel fuel by ГOCT and ASTM), spectral (IR, EPR, NMR), X-ray phase (RF, DTA).

The main provisions submitted to the defense. Development of conditions for the production of environmentally safer diesel fuels, including:

1. Creation of catalytic system modified by Ni, Co and Mo oxides by the method of impregnation of nanotube halloisites under normal and ultrasonic cavitation.
2. Obtaining of "green" diesel by hydrotreating with the presence of a catalytic system created by addition of vegetable oils and light flexion to primary diesel fractions.

The scientific novelty is that for the first time:

Green diesel fuel was acquired as a result of the hydrotreating process of diesel fractions with natural halloysite nanotubes modified by transition metal oxides with the addition of new catalytic systems:

1. Halloysite nanotubes were modified by oxides Ni, Co, Mo using the ultrasonic cavitation method, and it was determined that the obtained catalytic system had more crystal structure.
2. It was found out when using samples modified by ultrasonic cavitation as a catalyst, unlike absorption method, the depth of sulfurization in DF-1^a and DF-2² fuels as a result of the mixture of 10% cotton oil with the initial diesel fraction in the hydrotreating process was 91,6-96,3% (mass) respectively.
3. It was found out that in the presence of a catalyst modified by ultrasonic cavitation, the depth of sulfurization of DF-3a and DF-4a fuels obtained from the hydrotreating process by mixing the diesel fraction of the initial processing with catalytic cracking and 30% light flexion of coking and 10% vegetable oil constitutes 94,8-96,2% (mass.) respectively.
4. It was found out that the low temperatures of diesel fuels obtained by adding up to 10% cotton oil meet the requirements of winter diesel fuel.

The practical value of the research is that modern diesel fuel compounds with less sulfur and aromatic compounds were obtained from the hydrotreatment process of diesel blends, which contain up to 10 % of plant oils with modified catalyst samples purchased and recommended for industry application.

Approbation of work. The main results of the dissertation were reported and discussed at the following conferences. 90th Anniversary of Academic Togrul Shahtakhtinsky (Baku 2015), 3rd International Conference Catalysis For Renewable Sources: Fuel, Energy, Chemicals CRS-3 (Catania, Sicily, Italy 2015), International Scientific Week International Conference, ASW-2015, (Baku 2015), 4th International Scientific Conference of Young Researchers “Dedicated to the 93rd Anniversary of the National Leader of Azerbaijan, Heydar Aliyev (Baku-2016), International Conference “Current Problems of Contemporary Chemistry and Biology”, Oil Chemistry and Petrochemical Synthesis (Ganja 2016), Republican Scientific Conference dedicated to the 80th Anniversary of M. Nagiyev Institute of Catalysis and Inorganic Chemistry (Baku 2016), Baku International Mammadaliyev Conference on Petrochemicals (Baku 2016), International Conference «Renewable Plant Resources: Chemistry, Technology, Medicine» (Saint Petersburg, Russia 2017 International scientific and technical conference "Petrochemical synthesis and catalysis in complex condensed systems" dedicated to the 100th anniversary of Academician B.K. Zeynalov (Baku 2017), Republican Scientific and Technical Conference “Fuels, Fuel Components, Liquids, Oils and Additives”, dedicated to the 90th anniversary of SA Sultanov (Baku 2017), International Scientific Conference on the 95th Anniversary of National Leader H.Aliyev (Ganja-2018), Scientific Conference "Nagiyev's Readings", dedicated to the 110th anniversary of academician M. Nagiyev (2018), International Scientific and Practical Conference "Innovative Prospects for the Development of Oil Refining and Petrochemicals" dedicated to the 110th anniversary of Academician V.S. Aliyeva (Baku-2018), The International Scientific Conference “Actual Problems of Modern Chemistry” Dedicated to the 90th Anniversary of the Academician Y.H.Mammadaliyev IPCP (Baku 2019)

On the topic of the dissertation, 28 scientific papers were published: including 12 articles and 16 abstracts at conferences.

Place of dissertation. The work was performed in accordance with the work program of the IPCP ANAS 7/2013, registration number 0113 Az 2039

Personal participation of the author. The author outlines the main goals of the study and the tasks to achieve them, identifies areas of research, carried out processing, systematization and discussion of the results. The author also took a direct part in the formulation and conduct of laboratory and pilot tests.

The structure and scope of the dissertation. The dissertation consists of an introduction, 5 chapters, 52 illustrations, 9 charts, 53 tables, results and 184 literary references, introduction 12116, Chapter I 58099, Chapter II 20440, Chapter III 17925, Chapter IV 73505, V Chapter 39934 and the result 4248, with a total of 226267 characters (without tables, pictures and lists of literature) and published in 219 pages.

The introduction deals with the importance of new directions of the hydrotreating process and the problems of attracting alternative raw materials to the process, the purpose and objectives of the study.

The first chapter is devoted to the environmental demands for fuels in many countries around the world, hydrotreating plants capable of producing low-sulfur diesel fuel, different directions of high-quality diesel fuel production using vegetable oil as an alternative source, types of catalysts for the hydrotreating process, the structure of active components and methods of their modification, simultaneously, the role of halloysite nanotube cavities in catalysis is discussed, analysis of the research carried out with reference to the literature is given.

The second chapter presents the characteristics of raw materials, catalysts used in the research process, research methods of quality indicators, preparation methods of new catalysts, and operation scheme and the working principle of the hydrotreating laboratory equipment.

The third chapter presents the phase and elemental composition of halloysite nanotubes (A-1-A-8) modified by nickel, cobalt and molybdenum oxides with absorption and ultrasonic cavitation methods investigated by X-ray diffractometer (diffractometer XRD TD 3500, China), X-ray fluorescence microscopy (XGT 7000, Horiba, Japan), atomic absorption

spectrometry (AA ICE-3500, Thermo Scientific) IR, electromagnetic resonance (EPR) (radiospectrometer JES-PE-3X, Jeol). These samples were registered by Thermographic differential thermal analysis (TG / DTA / DTG) methods and the use of STA 449F3 NETZSCH Jupiter analyzer produced by the NETZSCH Company of Germany.

The fourth and fifth chapter presents material balance and quality indicators of the hydrotreating process with initial processing diesel fraction, 10 % vegetable oil, 30 % catalytic cracking and light flexion of coking in the presence of samples (A-1 – A-8) modified by the transition metal oxides of halloysite nanotubes, at pressure of 3,0-6,0 MPa and 330-380 °C, with the mass transfer rate of raw material 0.5-1 h⁻¹, IR, UV, NMR spectra was taken and the results of the studied process and their comparisons were given.

In summary and conclusions, general results are presented and the main achievements of the study are formulated. The dissertation is completed with main results and a list of literature references.

MAIN CONTENT OF WORK

In the dissertation, there were used straight-run diesel fuel (DF-1), straight-run diesel fuel with adding of 10 % of vegetable oil mixture (DF-2), straight-run diesel fuel with adding of 15 % of coking and 15% of catalytic cracking light gas oil (DF-3) and the last mixture with adding of 10 % vegetable oil (DF-4).

During hydrotreatin process of given diesel fraction and its mixtures there were researched two types of catalysts: industry catalysts (Цеокар-600, АГКД-400 БН) and Ni, Co, Mo oxides as nature nanotube alumosilicates. The process was realized by two methods.

1. The obtaining of halloysite based catalysts samples modified with Ni, Co, Mo oxides

In the presented dissertation, the samples of natural alumosilicate-halloysite nanotubes modified with 5-10 % nickel, cobalt and molybdenum oxides by ordinary (absorbing) and ultrasonic cavitation waves were prepared. The phase and element composition of obtained samples (A-1 – A-8) have been investigated

by using X-ray diffractometry (X-ray diffraction XRD TD 3500, China), X-ray fluorescence microscopy (XGT 7000, Horiba, Japan), atomic absorption spectrometry (AA ICE-3500, Thermo Scientific), IR, electromagnetic resonances (EPR) (radiospectrometer JES-PE-3X, Jeol). The thermal geographical differential thermosetting (TG / DTA / DTG) method and thermal properties of these samples were determined by using the NETZSCH Jupiter antennas produced by the German company NETZSCH.

It was found that the specific surface area of the initial halloisite sample is $67.7 \text{ m}^2 / \text{g}$, and the surface area of the halloisite in the prepared catalyst samples varied in the range of $60,3\text{-}62,4 \text{ m}^2 / \text{g}$. As you can see, the specific surface area of the obtained catalysts is considerably smaller than the specific surface area of the carrier. This result proves that the pores of the carrier are replaced by active metal oxides and that the pores that form part of the active surface area are blocked by crystals of these oxides.

The results of thermal analysis show that the mass loss of ions ($\approx 10 \%$ mass) in halloisite samples modified by transient metal oxides is negligible. This loss is found in the temperature range of $500\text{-}550 \text{ }^\circ\text{C}$. The calcite particles of the catalyst samples heated to $400 \text{ }^\circ\text{C}$ do not change. At temperatures above $400 \text{ }^\circ\text{C}$, already catalyst samples lose their crystallinity and become amorphous.

Compared with the initial halloisite rock, valence dances between the peaks caused by the intense vibration of the metal-oxygen bonds in the IR spectrum of the modified catalysts suggest that the Me-HNB bond may be linked to the halloisite.¹

The main purpose of drawing EPR spectra of catalyst samples is to study their paramagnetic properties and the formation of radicals. Extensive EPR lines related to transition metal ions have been discovered in the EPR spectra of the catalysts removed²

It has been found that the pores of the carrier were replaced by active metal oxides and the pores that form part of the active surface

¹ Larkin P. Infrared and Raman Spectroscopy: Principles and Spectral Interpretation, 1st Edition. Stamford: Elsevier, 2011, p.230

² Weil J.A., Bolton J.R. Electron Paramagnetic Resonance: Elementary Theory and Practical Applications. New-York: John Wiley & Sons, Ltd., 2007, p. 664

area were blocked by crystals of these oxides. In addition, the surface area, sulfurization capacity and working duration of the catalysts, obtained during the modification by ultrasonic cavitation increase. Thus, each catalyst sample was processed for up to 400 hours with regeneration every 50 hours.

No changes were observed in the repeated analysis of the used catalyst samples after the hydration treatment process. This also demonstrates the resistance of the presented catalyst samples against the hydrotreating process carried out under high temperature and pressure. At the same time, despite each catalyst sample has been used repeatedly in the process of hydrotreating, it has not lost its catalytic activity and has a sufficiently long working duration. These indicators allow them to be used in hydrotreating processes as industrial catalysts.

2. The hydrotreatment process of a mixture of straight-run diesel fraction and vegetable oil (10%) in the presence of prepared catalyst samples

Greater use of oil-based fuels has led to serious problems, including national energy security, depletion of oil reserves, and environmental pollution. One of the main objectives is to increase the use of renewable energy sources to reduce the amount of these wastes that are harmful to the environment and to the human body and to replace fuels. It is known that biofuels obtained from biomass have great potential as an alternative to petroleum fuels. One of the biofuel manufacturing processes is the production of green diesel - the "green diesel" or the "second generation diesel".

This chapter investigates the process of hydrogenation of diesel fraction (DF-1) and 10 % (DF-2) oil in primary oil processing.

In the process of green diesel production, linoleic acid, a component of vegetable oils, was used to study the process of converting vegetable oils. As a catalyst, the original primary halloisite and its modified samples with Ni, Co and Mo oxides were taken. The hydrocarbon composition of the hydrogenation products

of the linoleic acid is presented in graphs 1 and 2, as compared to the industrial catalyst АГКД-400 БН .

As can be seen from the results, the use of samples modified with primary halloisite and transitional metals directed reactions to decarboxylation. Thus, when using the industrial catalyst АГКД-400 БН, the ratio of hydrocarbons C_{17} / C_{18} to 0.9-1,1, in catalytic systems based on halloisite, is 1.4-1.6, ultrasonic cavitation. in the samples prepared by the method, this figure is 1.5-1.7. This also confirms the process of decarboxylation.

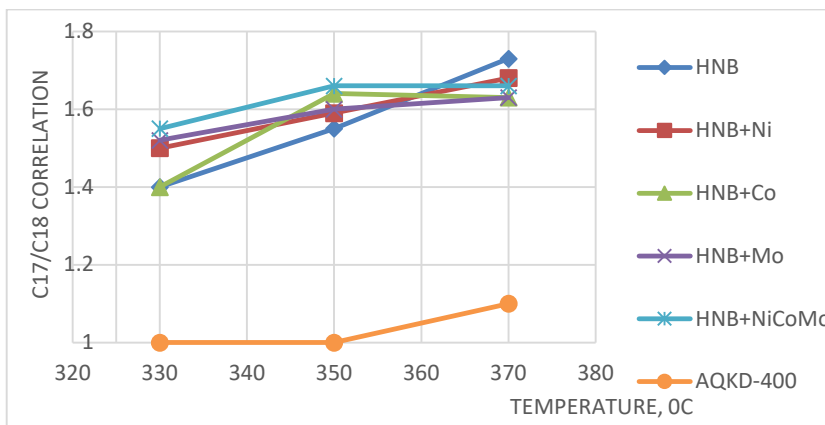


Chart 1. Change of hydrocarbons C_{17} / C_{18} , depending on the types of catalysts modified by impregnation

Mass spectroscopic analysis of the hydrocarbon composition of the hydrogenated diesel fractions showed that for all the used catalytic systems, the pressure increase was to direct the deoxygenation reaction of the triglyceride molecules of vegetable oil to the hydrodeoxygenation reaction. Thus, if the content of paraffins C_{17} and C_{18} is 3.0-6.0 MPa at 52.5 and 31.7% bulk for the A-6 catalyst. Whereas, the catalyst for A-7 is 53.1 and 30.9% mass, respectively, at the 6.0 MPa hydrogen pressure 35,6 and 48,1 % for the A-6 catalyst, respectively. % mass, 34,9 and 49,1 % mass for catalyst A-7. It is also undesirable. (Figure 1)

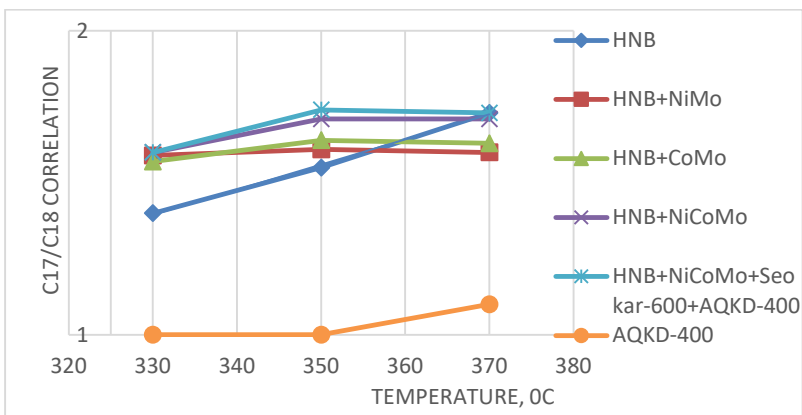


Chart 2. Change of hydrocarbons C_{17} / C_{18} , depending on the types of catalysts modified by ultrasonic cavitation.

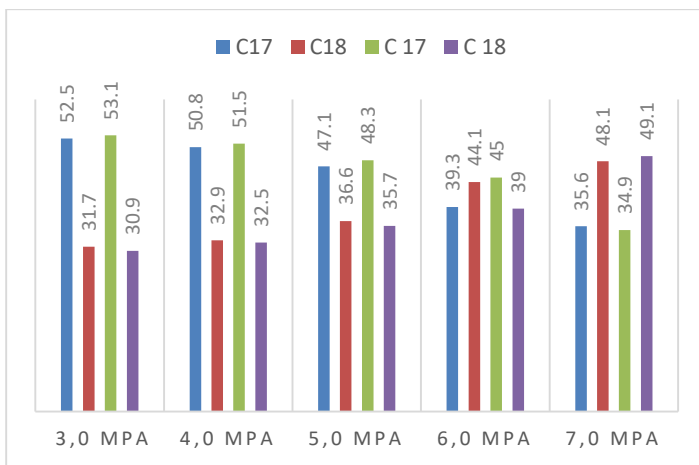


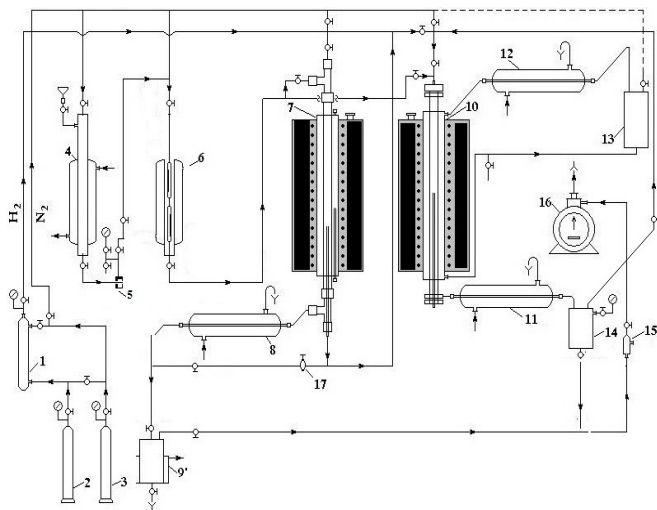
Figure 1. Influence of hydrogen pressure on change of hydrocarbon content of C_{17} and C_{18} in diesel fraction in hydrolysis process with catalyst A-6 and A-7

It has been shown that the salinity of the modified metal oxide oxides is reversible, as they increase the hydrogenation function of the process and also reduce the secondary cracking reactions.

Experimental experiments in the dissertation were carried out on a pilot device created in the laboratory. Optimal test conditions: at a pressure of 3.0-6.0 MPa and temperature 330-380 ° C, the mass transfer rate is 0.5-1 h⁻¹, the hydrogen / raw ratio is 1: 700. The technological scheme of the device is shown in Figure 2.

Sulfur containing fuels obtained with the use of catalysts A-1-A-8, prepared by various methods used in hydrolysis process of 3.0-3.5 and 5.0-5.5 MPa of the initial processing diesel fraction. the effect of the number of compounds was investigated.

The best results were obtained by the effects of samples prepared by ultrasonic cavitation. As can be seen from Figure 3, the results of the modification of the transition metals by the solubilization method of halloisite nanopores (A-1 – A-3) are satisfactory.



1-gas collector; 2, 3-gas cylinders; 4-D raw material capacity; 5-regulating pump; 6-heater-mixer; 7, 10 -medium and high pressure reactors; 8,11,12-refrigerators; 9-separator-cooler; 13-dropper; 14- separator; 15-pressure regulating valve; 16-gas meter; 17-rossel valve.

Figure 2. Technological scheme of the pilot unit of the hydrotreating process

The HNB + NiMo (USK) (A-4) and HNB + CoMo (USK) (A-5) samples were prepared by ultrasonic cavitation and yielded higher results compared with the other three catalysts. Although the composition of the HNB + NiCoMo (A-6) and HNB + NiCoMo (US-7) catalysts is the same, the preparation methods are different and the results of the A-7 sample developed by ultrasonic cavitation meet the Euro-4 standard. The latter catalyst is a multifunctional catalytic system that combines several catalysts, and the best results have been obtained with the presence of HNB + NiCoMo + АГКД-400БН + Цеокap-600 (USK) (A-8).

Fuels (DF-1^a and DF-2^a) with the use of different methods A-1-A-8 catalysts used for hydrolysis under pressure of 3.0-6.0 MPa of the initial processing diesel fraction and its 10% vegetable oil mixture.) influence on the amount of aromatic hydrocarbons in the structure. The HNB + NiMo (USK) and HNB + CoMo (USK) samples can be considered relatively positive compared to the other three catalysts.

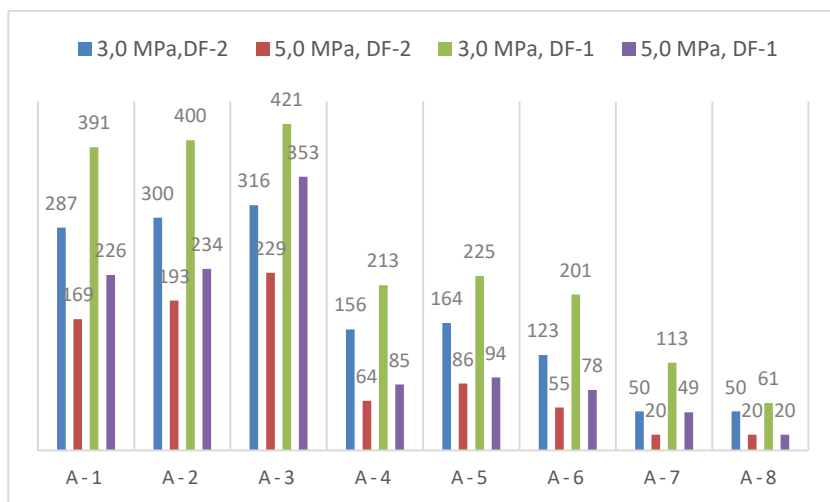


Figure 3. The effect of sulfur compounds on the composition of fuels obtained under different pressures (ppm) with the presence of catalysts A-1 – A-8

As can be seen in Chart 3 and 4, the results of A-1 – A-3 are considered satisfactory. It has been found that the effects of hydrogenated fuels are much better than those of other models, which are made by ultrasonic cavitation.

The results of sample A-7 from the HNB + NiCoMo (A-6) and HNB + NiCoMo (USK) (A-7) catalysts with the same composition meet the Euro 4 standard requirements.

The latter catalyst is a multifunctional catalytic system that combines several catalysts and the best results have been obtained with the presence of HNB + NiCoMo + АГКД-400БН + Цеокар-600 (USK) (A-8). Thus, the amount of aromatic hydrocarbons in DF-1a fuels is 11-11.6%, and in DF-2a fuels is 10.3-11.2%. contane.

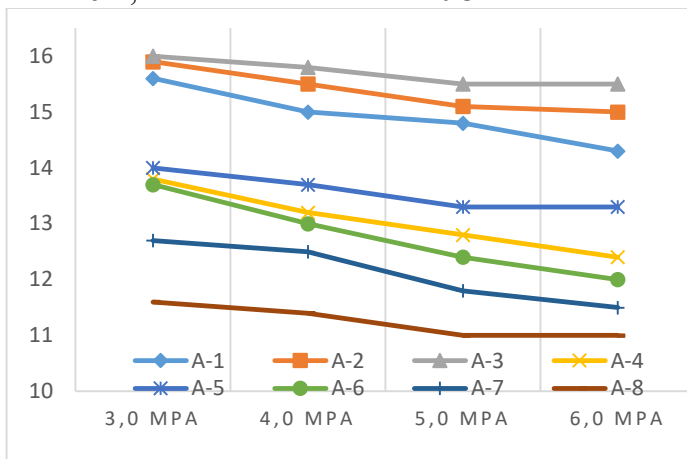


Chart 3. The quantity of aromatic hydrocarbons (% mass) in hydrotreated fuels (DF-1a) in the presence of catalysts A-1-A-8

The initial processing of diesel fraction and its 10% vegetable oil mixture at pressure of 3.0-6.0 MPa, with the use of catalysts A-1-A-8 and their hydrogen purification (KT-1) catalyst (АГКД-400БН catalyst). The effect of the amount of aromatic hydrocarbons on DF-1a and DF-2a) was investigated.

It has been found that the effects of hydrogenated fuels are much better than those of other models, which are made by ultrasonic cavitation. The results of A-1 – A-3 / KT-1 are satisfactory, HNB +

NiMo (USK) (A-4 / KT-1), HNB + CoMo (USK) (A-5 / The KT-1) samples gave comparatively superior results compared to the other three catalysts.

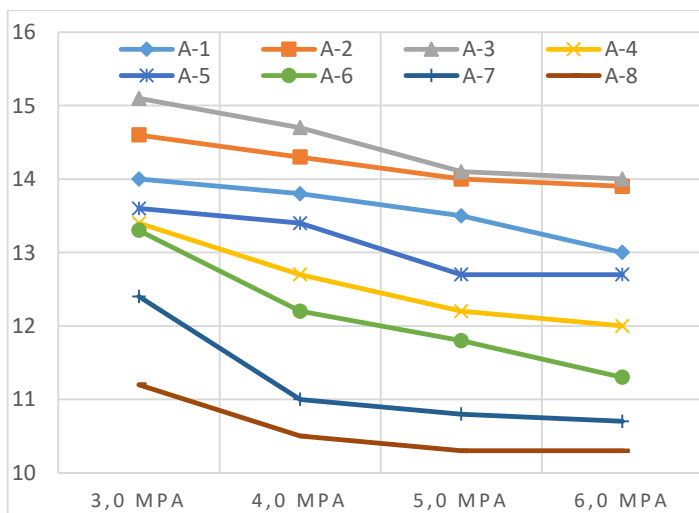


Chart 4. The quantity of aromatic hydrocarbons (% mass) in hydrotreated fuels (DF-2a) with the presence of catalysts A1-A-8

The results of the HNB + NiCoMo (A-6 / KT-1) and HNB + NiCoMo (USK) (A-7 / KT-1) catalysts meet the Euro-4 standard requirements. Thus, the amount of aromatic hydrocarbons in DF-1a fuels is 11.5 - 11%, and in DF-2a - 10.5-11%. contane.

It can be seen from the results that the initial processing is 10% bulk in the diesel fraction. Diesel fuel reserves obtained from hydrotreating process of adding 10% (wt) of vegetable oil to the composition of the initial processing diesel fractions by using halloysite modified metal oxides as hydrotreating catalysts with ordinary and ultrasonic cavitation waves and absorption method, increased 5.9% and 6.3% respectively. When the use of HNB+NiCoMo+ Цеорак-600 +АГКД-400БН catalytic system as a catalyst, it was observed that due to the increase of the yield gasoline fraction the yield of hydrogenated diesel fraction decrease.

This also proves that these catalysts have more cracking capabilities than the pure halloysite sample.

At the same time, the amount of water in the catalyst is reduced by 50 %. As a result of the repeated intensification of cracking process when using a hydrocarbon catalyst based on Цеокар-600, the amount of coke formation was 37.5 % - 57.1 % bulk compared to the АГКД-400БН and halloysite rocks. increased. In the process of hydrotreating the initial diesel oil fraction and its mixture with vegetable oil, the calculated amount of coke is 1.0-1.2 % bulk. in the presence of the catalysts studied, the amount of coke reduced by 0.5-0.6%. formed. This is also one of the reasons for the increase in the output of hydrogenated diesel fuel.

When using samples modified by the absorption of Ni, Co and Mo oxides with natural aluminosilicates as catalysts, the sulfurization depth of DF-1 fuel as a result of hydrotreating process of the mixture - initial processing diesel fraction (DF-1) with 10 % cotton is 53.5-65,6 % (wt) and of DF-2 fuel is 85,7-89,8 % (wt).

In the hydrotreating process with the catalyst modified by ultrasonic cavitation, the sulfurization depth in DF-1 and DF-2 fuels is 95.4 % and 96.3 % (wt) respectively.

The amount of total sulfur in the fuel content obtained from the hydrotreating process under the influence of the new catalytic system was reduced to ≤ 20 ppm. In the meantime, the sulfurization depth is 96.3% (wt), respectively.

It was also revealed that the amount of aromatic hydrocarbons in DF-1 fuels obtained from the hydrolysis process with each prepared catalyst is 11.6-11.0% (wt), and the amount of aromatic hydrocarbons in DF-2 fuels is 11.2-10.3% (wt), respectively.

As shown from the results, the quality indicators of fuels obtained from the hydrotreating processes involving catalysts modified by transition metal oxides meet the requirements of EN-590 (2009).

Comparison of the results showed that the catalysts were modified together with the Ni, Co, and Mo oxides by the presence of hydrogen catalysts and a new catalytic system (A-8), which was

created by adding a catalyst for the АГКД-400БН + Цеокар-600. Fuel quality indicators meet the requirements of EN-590 (2009) (Table 1).

Table 1.

The quality indicators of fuels obtained from hydrolysis process with catalysts A-7 and A-8.

Göstəricilər	EN-590 (2009) standards		HNB+NiCoMo (USK) (A-7)		HNB+NiCoMo+ АГКД-400 БН+Цеокар-600 (USK) (A-8)	
	Min	Max	DF-1 ^a	DF-2 ^a	DF-1 ^a	DF-2 ^a
Density at 15 °C, kg/m ³	820	845	844,4	845,0	844,4	845,0
Fraction composition, °C						
% Vol Rec 250°C	-	65	25	17	20	14
% Vol Rec 350°C	85	-	90	92	89	91
95%	-	360	360	360	360	360
Flash point, °C	56	-	69	72	70	72
Freezing point, °C	-30	-45	-40	-41	-41	-42
Cloud Point	-	-5	-30	-31	-31	-31
Kinematic viscosity at 20 °C, mm ² /s	2,00	4,50	3,31	3,32	3,17	3,23
The content of total sulphur, % mass	350	10	45	≤20	≤20	≤20
Coking of 10% mixture, % mass	-	0,30	0,0012	0,0018	0,0012	0,0018
Aromatic hydrocarbons, % mass	-	11,0	11,5	10,8	11,0	10,3
Cetane number, no less than	51	-	51	52	51	52

The amount of aromatic hydrocarbons varies from 10 to 11% (mass), while sulfur compounds vary between 0.0045 and ≤0.0020. The low-temperature properties of the obtained diesel fractions have improved (-40 / -42⁰ C) and the winter type corresponds to diesel fuels.

3. The hydrotreating process of the mixture of recycled fractions (30%) of the straight-run diesel fraction and vegetable oil (10%) in the catalytic system.

The hydrotreating process of the mixture - the recycled fractions of the straight-run diesel fraction (30%) and vegetable oil (10%) in the well-known hydrotreating process was carried out in the presence of halloysite samples modified with Ni, Co, Mo oxides.

Preliminary processing of diesel fraction of 15% catalytic cracking and 15 % coking of light (DF-3) and its mixture with 10% vegetable oil (DF-4) 3.0-3.5 and 5.0-5.5. The effect of sulfur compounds on the composition of fuels obtained with the use of catalysts A-1 – A-8 prepared by various methods used in hydrogen purification under pressure of 5,0 MPa was investigated. It turned out that the best results were obtained by the effects of samples prepared by ultrasonic cavitation.

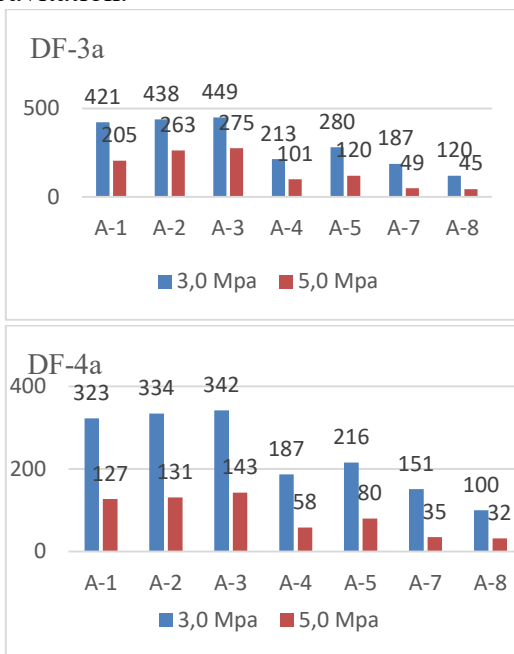


Figure 4. The amount of sulfur compounds in the fuel (DF-3^a and DF-4^a) with the presence of catalysts A-1 – A-8 (ppm)

As can be seen from Figure 4, the comparison of hydrodynamic processes carried out in four different conditions with single-phase modification of transition metals by halloisite nanopores (A-1-A-3) with the presence of catalysts A-2 and A-3 the quality of hydrogenated fuels has changed to a positive, but does not meet the requirements of EN-590 (2009). The results of the A-1 catalyst meet the requirements of this standard as a result of increased temperature and pressure.

The HNB + NiMo (USK) (A-4) and HNB + CoMo (USK) (A-5) samples were prepared by ultrasonic cavitation and yielded higher results than the other three catalysts. The HNB + NiCoMo (A-6) and HNB + NiCoMo (US-7) catalysts were investigated in the previous chapter and used only A-7 here because the results of the A-7 sample meet the requirements of EN-590 (2009).

The best results were obtained in the presence of HNB + NiCoMo + Цеокап-600 + Цеокап-600 (USK) (A-8).

When analyzing the results of the hydrogen purification process under different temperatures and pressures of the impurities, there is a decrease in the amount of aromatic hydrocarbons along with the reduction of sulfur compounds. Here, results of A-1 – A-3 samples are considered satisfactory.

Of these three catalysts, A-1 gives better results than the other two. The A-4 and A-5 absorbed by the effects of ultrasonic waves produced positive results compared to the first three catalysts.

The results of the HNB + NiCoMo (USK) and HNB + NiCoMo + АГКД-400БН + Цеокап-600 (USK) catalyst samples are quite good and meet the requirements of EN-590 (2009) standards. Thus, the amount of aromatic hydrocarbons in DF-3a fuels is 17.6-18.4 %, and in DF-4a fuels is 17.0-17,8 %. (Chart 5-6)

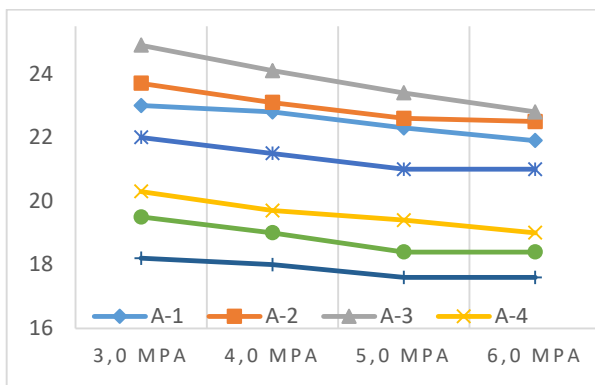


Chart 5. The amount of aromatic hydrocarbons (% mass) in hydrotreated fuels (DF-3^a) in the presence of catalysts A-1-A-8

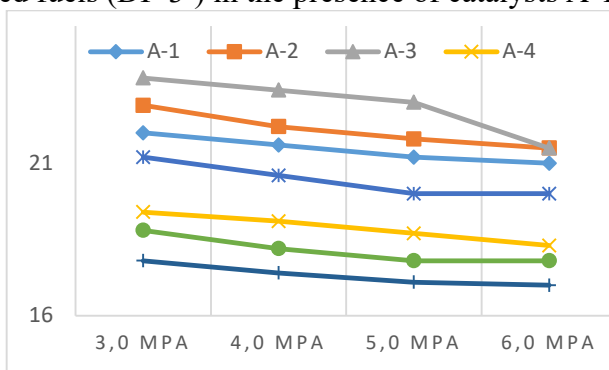


Chart 6. The quantity of aromatic hydrocarbons (% mass) in hydrotreated fuels (DF-4^a) in the presence of catalysts A-1-A-8

Based on our results, it is clear that the hydrogenation of the above-mentioned diesel fractions (DF-3a and DF-4a) was prepared by ultrasonic cavitation, HNB + NiMo (USK), HNB + CoMo (USK), HNB + NiCoNo (USK). The use of catalysts + NiCoMo + АГКД-400БН + Цеокап-600 (USK) is more appropriate.

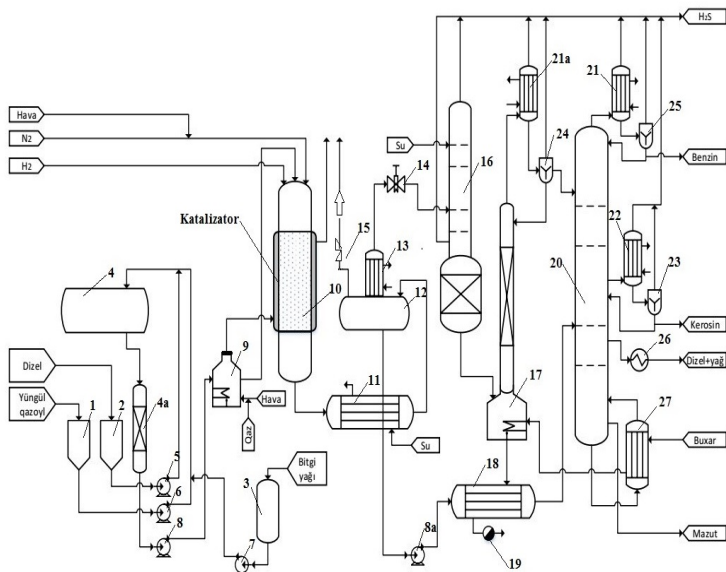
It should be noted that the yields of diesel fractions, obtained from the hydrotreating process of addition 30 % recyclable fractions (DF-3) and 10 % wt (DF-4) vegetable oil to the composition of initial processing diesel fraction by using modified halloysite transition metal oxides as a hydrotreating catalyst in the ultrasonic cavitation

waves in conjunction with Цеокар-600 + АГКД-400БН as industrial catalysts – are 34,5 % and 45,2-48,1 % (wt).

By using samples modified by the USK method of Ni, Co, Mo oxides with natural aluminosilicates and Цеокар-600 + АГКД-400 БН as catalysts, the sulfurization depth of recyclable fractions the primary processing diesel fraction (30 %) (DF-3) and vegetable oil mixtures(10 %) (DF-4) as a result of the hydrotreating process is 92.6-94.7 % (wt) and 93.2-95.2 % (wt), respectively.

As can be seen from the results, the quality indicators of fuels obtained from the hydrotreating process involving catalysts modified by transition metal oxides meet the requirements of EN-590 (2009).

The simplified principle scheme of the proposed technology for the process of hydrotreating of raw materials DF-1 - DF-4 with the catalytic system A-7 and A-8 under optimal conditions ($P = 3.0-6.0$ MPa and $T = 330-3800C$, $0.5-1$ hr⁻¹, hydrogen / raw ratio 1: 700) and obtaining the results meeting the requirements of EN-590 (2009) standards is given below. (Figure 5).



1 Light gasoline tank, 2 diesel fuel tank, 3 vegetable oil tank, 4 mixed raw tank, 4 filter, 5,6,7,8,8a-raw pumps, 9-pipe, 10-reactor, 11-cooler, 12-seperator, 13-seperator refrigerator, 14-pressure dross-ventilator, 15-pressure washer, 16-scrubber, 17-absorber, 18-heater, 19-

riboyer-condensate separator, 20-fraction -21a, 22,26 cooling capacitors, 23,24,25 intermediate flange and irrigation capacities, 27-cube evaporator.

Figure 5. Proposed scheme of hydrotreating of crude compositions with oil refining products (catalytic cracking and lightweight coking flux) to diesel fuel from primary processing with vegetable oils (10%, mass)

At the same time, mathematical modeling and feasibility studies of the process were conducted.

Matlab-6 was used to solve the optimization problem, which incorporates all modern computational algorithms. The results obtained are consistent with the translation of the process ($F_{1\max} \rightarrow 97.0\%$; $F_{1\min} \rightarrow 0.49\%$) and can be used to predict the process.

The cost of the catalyst, the cost of catalyst losses (2.4 g) during the process and regeneration, as well as economic calculations, including the life of the catalyst, were calculated. Thus, the production of diesel fuel on the proposed catalyst contributes to lower tonnes of raw materials and catalyst costs (3.64 manats / 1.0 versus 1.09 man./t compared to АГКД-400БН). This also confirms the effectiveness of the catalyst proposed for the process.

RESULTS

1. Halloysite samples modified by Ni, Co, Mo oxide absorption and ultrasonic cavitation methods were prepared and the study revealed that catalyst samples modified by ultrasonic cavitation had a higher crystalline structure [3,5,6].
2. In the process of hydrotreating the triglyceride decomposition reactions were directed towards decarboxylation when using the initial halloysite and their samples modified by transition metals as catalysts. Thus, while the ratio of hydrocarbons C_{17}/C_{18} is 0.9-1.1 using industrial catalyst АГКД-400БН , in halloysite-based catalytic systems made with absorption this

- ratio is 1.4-1.6, whereas in the samples prepared with ultrasonic cavitation this indicator is 1.5- 1.7 [7,11].
3. When using initial halloysite as a catalyst, hydrotreating diesel fraction with 10% cotton oil (hydrogen pressure- 3.5 MPa, temperature -350 °C and the mass transfer rate of raw is $-0.5-1 \text{ s}^{-1}$) the sulfurization depth (decrease from 0.0538% to 0.0497%) in the obtained diesel fuel is 37.07% (wt). Increasing the pressure (from 3.5 to 6.0 MPa) in the hydrotreating process leads to an increase in sulfurization depth to 41.87%.
 4. When using samples modified by the absorption of Ni, Co and Mo oxides with natural aluminosilicates as catalysts, the depth of sulfurization of DF-1 fuel as a result of hydrotreating process of the mixture - initial processing diesel fraction (DF-1) with 10 % cotton oil (with hydrogen pressure 3.0-6.0 MPa, temperature 350-380 °C and the mass transfer rate of raw is $0.5-1 \text{ s}^{-1}$) is 53.5-65,6% (wt) and of DF-2 fuel is 85,7-89,8 % (mass). The depth of sulfurization in DF-^{1b} and DF-^{2b} fuels obtained from the hydrotreating process of the new catalyst sample with the catalyst АГКД-400БН is 92.6-93.9% (mass) respectively [1,2,4].
 5. It was found out that in the presence of a catalyst modified by the absorption of transition metal oxides of halloysite nanotubes, the depth of sulfurization increases from 36.7% to 81.2% (mass) in DF-3^a and DF-4^a fuels obtained from hydrotreating process (increasing the pressure from 3.5 to 6.0 MPa) of the initial processing diesel fraction with 30% catalytic cracking and light flexion of coking [10,11].
 6. It was found out that when using Ni,Co,Mo oxides modified by the natural aluminosilicates with ultrasonic cavitation as catalysts, the depth of sulfurization in DF-1^a and DF-2^a fuels in the hydrotreating process of the mixture of initial processing diesel fraction (DF-1) and 10% cotton oil (the hydrogen pressure 3,0-6,0 MPa, temperature 350-380 °C and the mass transfer rate of raw is $0.5-1 \text{ s}^{-1}$), constitutes 95,4 % (mass) and 96,3% (mass), respectively. The sulfurization depth

of DF-1^a and DF-2^a fuels obtained during the hydrotreating process of the new catalyst АГКД-400БН is 91.1 % (wt) and 96.3 % (wt), respectively [11].

7. It was found out that in the presence of a catalyst modified by the transition metal oxides with ultrasonic cavitation method, the depth of sulfurization in DF-3^a and DF-4^a fuels obtained from hydrotreating process of the initial processing diesel fraction with catalytic cracking and light flexion of coking and 10 % vegetable oil, constitutes 93,2-95,2 % (mass) respectively.
8. It was found out that when using samples of Ni,Co,Mo oxides modified by the absorption of natural almosilicates as catalysts, as a result of hydrotreating process of the mixture-initial diesel fraction (DF-1),10% cotton oil, 30% catalytic cracking and light flexion of coking (hydrogen pressure - 3,0-6,0 MPa, temperature- 350-380 °C ν XVKS 0.5-1 s⁻¹),the amount of aromatic hydrocarbons in DF-1a constitutes 11,6-11,0 % (mass.), and in DF-2^a fuel 11,2-10,3 % (mass) respectively. The amount of aromatic hydrocarbons in DF-1^b fuels obtained from the hydrotreating process of new catalyst sample in the presence of АГКД-400БН was 11.8-11.0 % (mass), in DF-2^b fuels was 11.6-10.5 %. (mass). Accordingly, the amount of aromatic hydrocarbons in DF-3^a fuels varies between 18.2-17.6 % (mass), and the amount of aromatic hydrocarbons in DF-4^a fuels varies between 17.8-17.0 % (mass) [11,12].

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The defense will be held on «07» May 2021 at 10⁰⁰ at the meeting of the Dissertation council ED 1.17 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at the Institute of Petrochemical Processes of Azerbaijan NAS

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Abstract was sent to the required addresses on: «05» April 2021

Signed for print: 02.04.2021

Paper format: 60×90 1/16

Volume: 36632

Number of hard copies: 20 copies