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Determination of Optimal Conditions for Catalytic Hydrogenation of Oil Sludge (Atasu-Alashankou)

The optimal conditions of catalytic hydrogenation of oil sludge (Atasu-Alashankou) and the change in the kinematic viscosity of the fraction to 350°C from the studied factors using the method of probabilistic-deterministic planning were experimentally determined. During the hydrogenation process of oil sludge, the maximum total yield of light fractions reached 62.1 %, and the kinematic viscosity decreased from 2.2 to 1.2 mm²/s. It was established the initial hydrogen pressure and the amount of added nanocatalyst microsiliate with cobalt (catalyst 1) have the greatest influence on the yield of the middle fraction from oil sludge under experimental conditions. It was shown that catalyst 1 increases the yield of diesel fraction components during the hydrogenation process of oil sludge. For the first time, we established the individual and group chemical composition of the fraction up to 350°C before and after processing. The use of nanocatalyst 1 in amounts of 1.2–1.5 % led to an increase in the yield of the fraction up to 350°C and diesel fraction components. This is due to the yield of paraffinic hydrocarbons increasing from 57.6 (initial fraction) to 80.7 %, as well as a decrease in aromatic hydrocarbons from 14.1 to 12.9 % and polycyclic aromatic hydrocarbons from 9.56 to 4.3 %.

Keywords: optimization, oil sludge, catalyst, microsiliate, cobalt, nickel, viscosity, hydrogenation, optimization, correlation, matrix.

Introduction

Oil-producing and oil-refining manufactories, as well as oil transported through pipelines, are sources of environmental pollution. Oil-containing waste called oil sludge, the most common type of industrial waste. For every ton of oil produced, 7 tons of sludge are generated [1–2]. Its approximate composition is: water (from 30 to 80 %), oil products (from 10 to 50 %), and solid inclusions (from 1 to 40 %) [1–2]. The organic part of the waste in oil sludge is distributed as follows: 7–10 % are oil hydrocarbons, 5–10 % are in an emulsified and dissolved state, and the remaining hydrocarbons are on the surface of the oil sludge in the form of a film [1–2]. The inorganic part of the oil sludge contains silicon and iron oxides (sand, corrosion products), a small amount (less than 1 %) of aluminum, sodium, and zinc [3].

Currently, known technologies for oil sludge processing contain physical, mechanical, and biological methods [4]. However, none of the listed methods are efficient in processing and disposal of oil sludge and still causing damage to the environment. We propose a combined method for oil sludge processing, which includes preliminary fractionation in order to remove mechanical impurities and solid inclusions, followed by catalytic hydrogenation of the obtained wide fraction.

The oil-containing sludge (Atasu-Alashankou), which formed during the transportation of Kazakh oil through the pipelines, contains drilling mud, used oil, emulsified solid particles, and crude oil. In the studies [5–6], the results of the optimal conditions determination for electro-hydraulic treatment of oil are given.

Literature analysis showed there is no data available for catalytic hydrogenation of oil sludge. Previously, the optimal conditions for electrohydraulic treatment of oil sludge were determined in [5–6]. The data obtained in [5–6] were used for the process of hydrogenation of a wide fraction of oil sludge in the presence of a nanocatalyst.

The purpose of the work is to determine the dominant factors (temperature, initial hydrogen pressure, duration of the experiment, number of nanocatalysts added) for the yield and quality of the individual

chemical composition of the target hydrogenate during destructive hydrogenation of a wide fraction of oil sludge.

The literature does not contain data on the use of factor planning of the experiment to study the process of catalytic hydrogenation of oil sludge and the determination of parameters for hydrogenation of oil sludge, which makes this study relevant.

Experimental

The object of research for experimental work in order to determine the optimal conditions and kinetic parameters of oil sludge hydrogenation was the raw material obtained during the transportation of oil through the pipelines of the KazTransOil company in the West Kazakhstan oil fields of the Republic of Kazakhstan. The physicochemical characteristics of the oil sludge (Atasu-Alashankou) and the method of synthesis of the nanocatalyst are presented in [6].

Physical and chemical characteristics of oil sludge (Atasu-Alashankou): density — 0.87 g/cm³, viscosity — 30 cSt, ash content A — 0.36 %, C — 72.3 %, H — 11.1 %, N — 0.1 %, O — 16.1 %. The atomic ratio of hydrogen to carbon in oil sludge is 1.8.

The microsilicate is used as a carrier and catalyst — a product of the Karaganda silicon plant Tau-Ken.temir LLP.

The individual and quantitative chemical composition of the starting microsilicate was determined by X-ray spectral analysis. Content of components, %: SiO₂ — 95.5; TiO₂ — 0.02; Al₂O₃ — <0.95; Fe₂O₃ — <1.0; CaO — 0.5; MgO — 0.4; MnO — 0.04; P₂O₅ — 0.06; K₂O — <0.1; Na₂O — 0.3.

The starting microsilicate was pre-ground, then samples with a particle size of 0.1 mm were taken by sieve analysis. The initial microsilicate was leached using a 20 % hydrochloric acid solution to remove alkali and alkaline earth metals. Chemical composition of the initial microsilicate after leaching by X-ray — spectral analysis is following, %: SiO₂ — 97.439; TiO₂ — 0.023; Al₂O₃ — <0.95; Fe₂O₃ — <1.0; CaO — 0.414; MgO — 0.304; MnO — 0.033; P₂O₅ — 0.057; K₂O — <0.1; Na₂O — 0.276.

The given data demonstrate the chemical composition of the starting microsilicate and after leaching changed almost slightly.

For the preparation of nanocatalysts, we used the method of impregnating the catalyst support with the active substance followed by calcination. Impregnation was carried out by immersing the support (leached microsilicate) in a 1.5 % salt solution of Ni(NO₃)₂·6H₂O and Co(NO₃)₂·6H₂O. Impregnated support (microsilicate) with solutions of nickel nitrate and cobalt nitrate of microsilicate (20 g) was heated for 2 hours at 80–90 °C temperature, and the prepared samples were dried at 105 °C temperature for 2 hours. Then, the obtained catalysts were calcined in a muffle furnace at 650 °C temperature for 2 hours. 2 catalyst samples were prepared to study the effect on thermal decomposition of nickel oxide, cobalt oxide and iron oxide: Sample 1 (microsilicate + Co 1.5 % + oil sludge, particle size 0.1 mm), Sample 2 (microsilicate + Ni 1.5 % + oil sludge, particle size 0.1 mm). Previously, we carried out work [7] with a nanocatalyst obtained by the impregnation method; the particle size of the starting microsilicate and prepared catalyst samples was established using a laser *Nano-S90* particle size detector.

After the oil sludge processing, the hydrogenate was fractionated up to 350 °C. The individual and group chemical composition of the fraction up to 350 °C was determined by the chromato-mass-spectrometry on the HP 5890/5972 MSD device (Agilent, USA).

Identification of substances was performed by the NIST98 mass spectral database.

Chromatography conditions:

Column: DB-5, 30 m × 0.2 mm × 0.5 μm.

Carrier-gas: helium, 0.8 ml/min.

Thermostat: 50 °C — 4 min;

50–150 °C — 10 °C/min

150–300 °C — 20 °C/min

300 °C — 4 min

Atomizer: 250 °C

Atomizer: 200 °C

The determination of the kinematic viscosity of the fraction up to 350 °C, obtained in the process of hydrogenation of oil sludge of the studied samples was carried out at a temperature of 30 °C by the automatic viscometer SYD-265B-1 (P.R. China).

Results and Discussion

A probability-deterministic design of experiment has been used in order to determine the optimal conditions for the hydrogenation of oil sludge [5]. The dependence of the yield of the middle fraction and the changes in the kinematic viscosity of the wide fraction in of oil sludge on the studied factors usually is nonlinear. Hence, it is more rational to use design methods that are based on the Protodyakonov-Malyshev nonlinear multiple correlation to process statistical data [8–10]. The authors [8–10] proposed an empirical formula for multiple nonlinear correlations in the form of a power one less than the number of partial functions:

$$Y_p = \frac{\prod_{i=1}^n P Y_i}{Y_{mid}^{n-1}}, \tag{1}$$

where Y_{mid} — the total average of all considered values of the generalized function; Y_p — generalized functions of $X_1, X_2 \dots$ factors; Y_i — partial function; n — number of partial functions (factors); $P_{i=1}^n$ — product of all the partial functions.

Experimental work was conducted in order to develop a mathematical model of the process of catalytic hydrogenation of oil sludge using a matrix of the design of experiment compiled by the Protod'yakonov-Malyshov method [8–12]. The obtained results were processed by the probabilistic-deterministic design of experiment program (PDDoE) [10–12]. The sequence of program operations is reduced to finding the partial dependence of the yield of the middle fraction and the change in the viscosity of the middle fraction obtained from the oil sludge.

As a result equations of the multiple correlation R [8–9] and tr coefficient [8–9] have been obtained. The selected intervals and levels of changes in factors, the matrix of the design of experiment of oil sludge hydrogenation are given in Tables 1 and 2.

Table 1

Levels and intervals of the variation of the studied factors

Factors	Levels			
	1	2	3	4
X_1 , amount of the catalyst 1 added to the oil sludge (catalyst 1 — microcatalyst coated with 1.5 % Co)	0.001	1.0	1.5	2.0
X_2 , initial hydrogen pressure, P=MPa	0.0	3.0	4.0	5.0
X_3 , temperature, T °C	350	370	390	410
X_4 , duration, min	0.0	3.0	45	60
X_5 , amount of the catalyst 2 added to the oil sludge (catalyst 2 — microcatalyst coated with 1.5 % Ni)	2.0	1.5	1.0	0.001

Table 2

Matrix of the 5-factor 4-level design of experiment

№	X_1	X_2	X_3	X_4	X_5	$Y_{exp.}$ yield of the fraction up to 350 °C, %	Y_p , yield of the fraction up to 350 °C, %	$Y_{exp.}$ kinematic viscosity of the fraction up to 350 °C, mm*s ⁻²	Y_p , kinematic viscosity of the fraction up to 350 °C, mm*s ⁻²
1	2	3	4	5	6	7	8	9	10
1	1	1	1	1	1	32.2	34.4	1.38	1.38
2	2	2	2	2	1	68.4	61.4	1.34	1.34
3	3	3	3	3	1	62.05	66.4	1.335	1.33
4	4	4	4	4	1	74.8	71.4	1.37	1.37
5	1	2	3	4	2	72.13	63.58	1.33	1.34
6	2	1	4	3	2	64.85	62.12	1.44	1.51
7	3	4	1	2	2	55.64	49.96	1.29	1.25
8	4	3	2	1	2	48.12	50.86	1.291	1.25
9	1	3	4	2	3	48.72	58.93	1.345	1.37

Continuation of Table 2

1	2	3	4	5	6	7	8	9	10
10	2	4	3	1	3	48.54	50.78	1.26	1.21
11	3	1	2	4	3	61.36	66.68	1.28	1.24
12	4	2	1	3	3	51.29	50.40	1.4	1.46
13	1	4	2	3	4	51.63	50.33	1.18	1.14
14	2	3	1	4	4	48.22	53.58	1.25	1.23
15	3	2	4	1	4	64.87	59.36	1.42	1.50
16	4	1	3	2	4	61.76	62.83	1.29	1.27

Table 3 and 4 show the calculated values of the partial functions for the yield of the fraction up to 350 °C from oil sludge in the process of hydrogenation and the viscosity changes caused by various conditions of the process.

Table 3

Calculated values of the partial functions for Y_p

Functions	Levels				Total average	R	t_r
	1	2	3	4			
$Y_1 = -3.192x^2 + 10.615x + 51.005$	51.17	57.5025	60.98	59.005	57.164	0.95	13.6
$Y_2 = -0.5993x^2 + 3.0321x + 55.444$	55.44	59.22	57.874	55.66	57.05	0.99	69.64
$Y_3 = 0.266x - 43.911$	46.83	57.37	61.12	63.32	57.164	0.88	5.6
$Y_4 = 55.619x^{0.0203}$	48.43	58.63	57.455	64.14	57.164	0.8594	6.63
$Y_5 = 3.4762x^2 - 4.756x + 56.214$	56.2	57.48	59.51	60.61	58.45	0.48	0.87

Table 4

Calculated values of the partial functions for Y_p

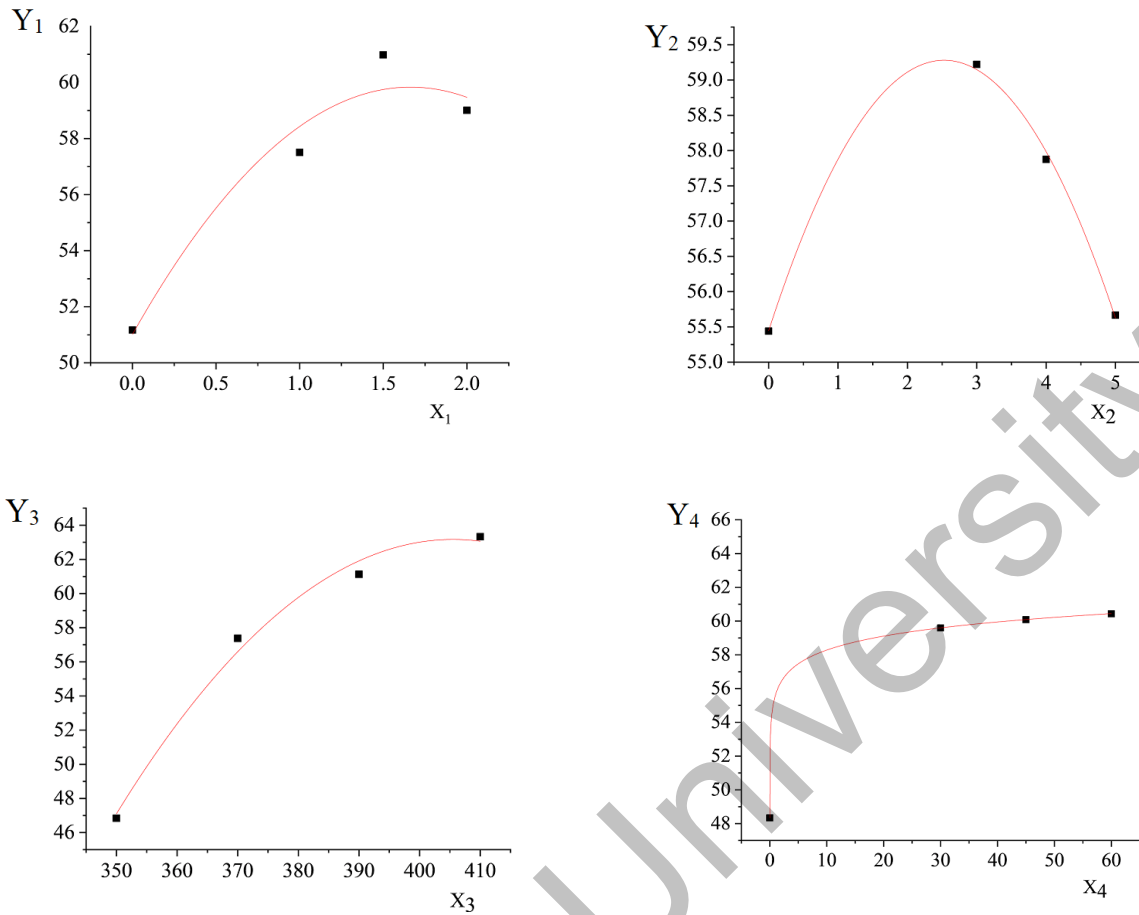
Functions	Levels				Significance		
	1	2	3	4	Total average	R	t_r
$Y_1 = 1.309 + 0.01467X_1$	1.31	1.32	1.33	1.34	1.325	0.99	441.5879
$Y_2 = -0.01524X_2^2 + 0.07196X_2 + 1.3$	1.30	1.37	1.35	1.27	1.325	0.98	45.5597
$Y_3 = 6.081 \cdot 10^{-5} X_3^2 - 0.04461X_3 + 9.464$	1.30	1.27	1.32	1.39	1.325	0.96	22.2321
$Y_4 = 6.122 \cdot 10^{-5} X_4^2 - 0.00555X_4 + 1.413$	1.41	1.31	1.26	1.30	1.325	0.95	14.0683
$Y_5 = 1.285 + 0.03549X_5$	1.35	1.33	1.32	1.28	1.325	0.99	3535.1803

As a result of processing the experimental data, partial functions for the catalytic hydrogenation of oil sludge and the change in viscosity of the middle fraction were obtained. Point graphs are presented in Figures 1 and 2. They are generalized by the Protoiakonov-Malychev equation. The significance of partial and generalized dependencies was tested using the coefficient of nonlinear multiple correlation R [8–9] and its significance t_r for the 95 % confidence level [8–9]. It turned out that out of all the partial dependencies, only 5 (on the amount of added catalyst 2 (X_5)) was insignificant. Therefore, it was not included in the generalized equation (2).

$$Y_p = 57.14^{-3} y_1 y_2 y_3 y_4 ; R = 0.70 \quad t_r = 5.0. \quad (2)$$

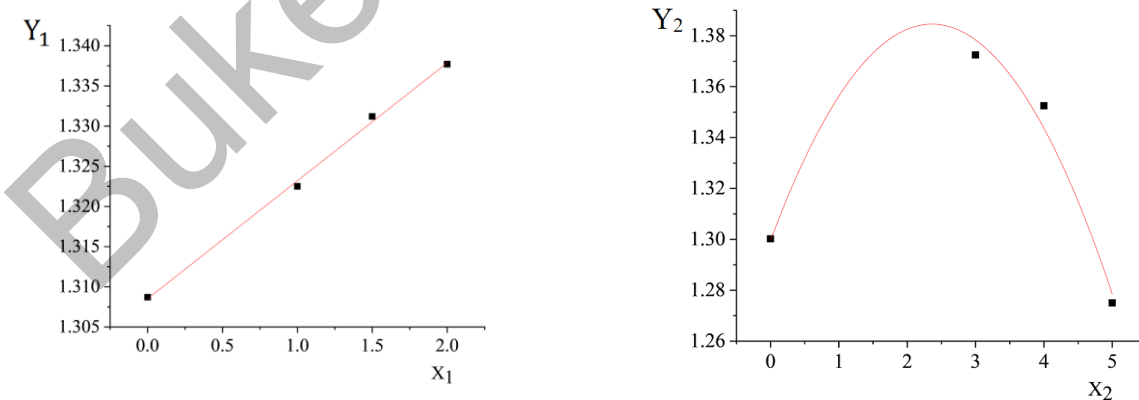
$$Y_p = \left((1.309 + 0.01467X_1) + (-0.01524X_2^2 + 0.07196X_2 + 1.3) + (6.081 \cdot 10^{-5} X_3^2 - 0.04461X_3 + 9.464) + (6.122 \cdot 10^{-5} X_4^2 - 0.00555X_4 + 1.413) + (1.285 + 0.03549X_5) \right) - 5.3004; \quad (3)$$

$$R = 0.66; \quad t_r = 3.7.$$



x_1 — amount of catalyst 1 added to the oil sludge; x_2 — initial hydrogen pressure, MPa;
 x_3 — temperature, T°C; x_4 — duration, min.)

Figure 1. Partial dependencies of catalytic hydrogenation of the oil sludge
 ($Y_{1,2,3,4}$ — yield of the reaction up to 350 °C, %)



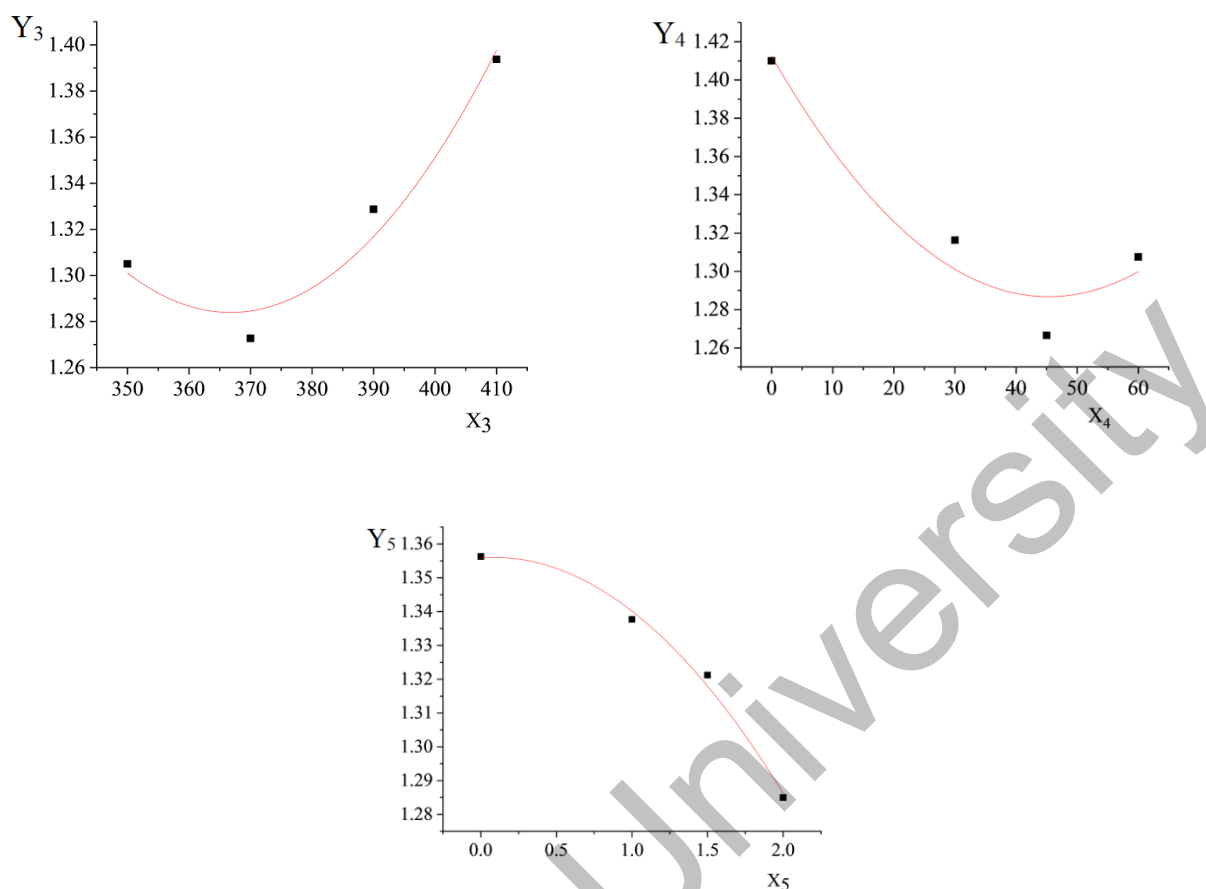


Figure 2. Partial dependencies of changes in kinematic viscosity of the fraction up to 350 °C on the various factors (x_1 — amount of catalyst 1 added to the oil sludge; x_2 — initial hydrogen pressure, MPa; x_3 — temperature, T°C; x_4 — duration, min; x_5 — amount of catalyst 2 added to the oil sludge)

The generalized equations (2) and (3) were tested for significance by comparing the results of that with experimental data. The generalized Protodyakonov-Malyshev equations (2), (3) are adequate for all R and t_r response functions. The obtained particular equations can be used both for optimizing the process and for technological modeling of the process of catalytic hydrogenation of oil sludge in enlarged laboratory conditions. It follows from these data that under experimental conditions the initial hydrogen pressure and the amount of catalyst 1 added have the greatest effect on the average fraction yield from the oil sludge. The change in the kinematic viscosity of the middle fraction is affected by the amount of catalyst 1 added to the oil sludge and the process temperature.

The generalized multivariate equation of fraction yield before 350 °C from oil sludge is

$$Y_p = \frac{(-3.192x^2 + 10.615x + 51.005)(-0.5993x^2 + 3.0321x + 55.444)}{(0.266x - 43.911)^{-1}(55.619x^{0.0203})^{-1}57.14^3}, \quad (4)$$

at standard deviation of 1.81 %. The calculation of this equation (4) showed at 410 °C temperature, the process time was 60 minutes, the amount of nanocatalyst 1 (cobalt-supported microsilicate 1.5 %) to be added was 2 %; initial hydrogen pressure 5.0 MPa, the amount of nanocatalyst 2 added (nickel-supported microsilicate 1.5 %) — 2 % is respectively 71.4 %.

The generalized multivariate equation changes in the kinematic viscosity of the middle 350 °C fraction from oil sludge is

$$Y_p = \frac{(1.309 + 0.01467X_1)(-0.01524X_2^2 + 0.07196X_2 + 1.3)6.081 \cdot 10^{-5} X_3^2 - 0.04461X_3 + 9.464}{(6.122 \cdot 10^{-5} X_4^2 - 0.00555X_4 + 1.413)^{-1}(1.285 + 0.03549X_5)^{-1}1.325^4} \quad (5)$$

at standard deviation of 0.014 %. Calculation of this equation (5) showed that at 410°C temperature, process time was 0.0 min, the amount of nanocatalyst 1 (cobalt-supported microsilicate 1.5 %) to be added

was 1.5 %; initial hydrogen pressure 3.0 MPa, the amount of nanocatalyst 2 added (nickel-supported microsilicate 1.5 %) — 0.000 % was respectively 1.50 %.

Based on the conducted experimental research, the following optimal conditions have been established:

a) Yield of the middle fraction of oil sludge: amount of added nanocatalyst 1 — 1.2–1.5 %; initial hydrogen pressure 2.5–3 MPa; temperature 400–410 °C; process duration 50–60 minutes.

b) Change in the kinematic viscosity of the middle fraction obtained from oil sludge: amount of added nanocatalyst 1 — 0.5 %; initial hydrogen pressure 4.5 MPa; temperature — 370 °C; process duration — 45 minutes; amount of added catalyst 2 — 2.0 %.

During hydrogenation of oil sludge, the maximum total yield of light fractions of 62.1 % and a decrease in the kinematic viscosity value from 2.2 to 1.2 mm²/s were achieved.

This is explained by changing the content of aromatic hydrocarbons in the initial fraction to 350 °C — 14.08 % (individual chemical composition is presented: toluene — 3.07 % and others), paraffinic hydrocarbons — 57.6 (individual chemical composition is presented: heptane — 3.02, 1-nonadecene — 1.52 %, decane — 7.59, dodecane — 4.51, heptadecane — 3.19, hexadecane — 2.37, nonane — 5.96, octadecane — 6.87 and others), unsaturated hydrocarbons — 15.7 % (individual chemical composition is presented: 1-decene — 1.09, 1-hexadecene — 1.1 and others), naphthenic hydrocarbons — 35.97 % (individual chemical composition is presented: 1.3-dimethylcyclohexane — 2.57 %, 1-ethyl-3-methylcyclohexane — 1.25 %, cyclododecane — 0.56 %, cyclohexane, (1-methylpropyl) — 4.89 % and others), polyaromatic hydrocarbons — 9.56 % (individual chemical composition is presented: naphthalene, 1,4,6-trimethyl — 1.33, naphthalene, 2,7-dimethyl — 2.59 %, naphthalene, decahydro-2-methyl — 1.8 % and others).

The group and individual chemical composition of the hydrogenate of the fraction with the end of boiling 350 °C obtained during catalytic hydrogenation from oil sludge is characterized by the following composition: aromatic hydrocarbons — 12.94 % (individual chemical composition is represented by: toluene — 1.97 %, benzene, 1-ethyl-3-methyl — 1.68 % and others); paraffinic hydrocarbons — 80.72 % (individual chemical composition is next: 1-heptadecene — 0.36 %, 2-Bromo dodecane — 0.22 %, Decane — 9.89 %, Dodecane — 11.47 %, Heptadecane — 5.27 %, Heptane — 2.65 %, Hexadecane — 5.36 % and others); unsaturated hydrocarbons — 9.31 % (individual chemical composition is represented by: 1-Decene, 5-methyl- — 0.19 %, 1-Tridecene — 0.65 %, 2,4-Hexadiene — 2.33 %, 2-Heptene — 0.14 %, 2-Tetradecene — 0.66 %, 3-Dodecene — 1.16 % and others); naphthenic hydrocarbons — 34.48 % (individual chemical composition is following: Cyclodecane — 0.4 %, Cyclodecane — 1.18 %, 0.33 Cyclododecane, ethyl- — 0.8 %, Cyclohexane, (1-methylpropyl)- — 0.12 %, Cyclohexane, (2-methylpropyl)- — 1.36 %, Cyclohexane, 1,1,2,3-tetramethyl- — 0.34 %, Cyclohexane, 1,2,3-trimethyl- — 2.18 % and others); polyaromatic hydrocarbons — 4.27 % (individual chemical composition is represented by: Naphthalene, 1,2,3,4-tetrahydro-6- — 0.25 %, Naphthalene, 1,4-dimethyl- — 0.26 %, Naphthalene, 2,3,6-trimethyl- — 0.39 %, Naphthalene, 2,7-dimethyl- — 0.84 %, Naphthalene, decahydro-2-methyl- — 2.58 % and others); iso-Alkanes — 41.19 % (individual chemical composition is presented: Octane, 2,6-dimethyl- — 0.23 %, Nonane, 4-methyl- — 0.22 %, Nonane, 2,6-dimethyl- — 0.57 %, Decane, 5-methyl- — 0.33 %, Decane, 4-methyl- — 0.18 %, Decane, 2-methyl- — 0.41 %, Heptane, 3-ethyl-2-methyl- — 0.58 %, Undecane, 2-methyl- — 1.78 %, Undecane, 2,6-dimethyl- — 1.87 %, Tridecane, 2-methyl- — 1.23 % and others).

The obtained fraction composition of the hydrogenate up to 350 °C shows that the use of the nanocatalyst 1 in small amounts of 1.2–1.5 % allows increasing the yield of the fraction to 350 °C and components of the diesel fraction.

Conclusions

Thus, by the use of probabilistic-deterministic design of experiment, optimal conditions for the process of the oil sludge hydrogenation in the presence of a nanocatalyst (cobalt-coated microsilicate) and the change in the viscosity of the middle fraction from various factors were determined. In our hydrogenation conditions, the maximum total yield of light fractions (62.1 %) was achieved. It was established in the experimental conditions, the initial hydrogen pressure and the amount of added nanocatalyst, cobalt-coated microsilicate (catalyst 1), have the greatest effect on the yield of the middle fraction from oil sludge. It has been shown the catalyst 1 also increases the yield of diesel fraction components.

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