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ASSESSMENT OF THE TECHNOLOGICAL EFFICIENCY AND POSSIBILITY OF IN-SITU REGENERATION OF A HETEROGENEOUS CATALYST IN THE COMPOSITION OF PROPPANT FOR HYDRAULIC FRACTURE OF OIL FORMATIONS

The object of this research is the process of in-situ catalytic upgrading of crude oil in a post-hydraulic fracturing environment using catalytically active proppant. The subject of research is the mechanism and conditions of in-situ catalysis of crude oil, as well as the efficiency and regeneration of the "zeolite + $CrCO_3$" heterogeneous catalyst introduced into a proppant mixture during hydraulic fracturing.

The research addressed the problem of improving the recovery of crude oils with high density, viscosity and a significant content of asphaltene-resin-paraffinic substances through in-situ catalysis using a heterogeneous catalyst injected into the formation together with proppant during hydraulic fracturing. The work aims to describe the technology for preparing catalytically active granules, justify the catalyst's operating cycle, investigate its regenerative capacity within the formation, and propose a regeneration technology.

The research analysed previous experiments aimed at determining the efficiency of the "zeolite + $CrCO_3$" heterogeneous catalyst and proposed a method for its preparation in the form of granules for subsequent addition to the proppant at a concentration of 20%. It was determined that one tonne of catalytically active granules provides catalytic treatment of approximately 7–8 thousand m^3 of crude oil until regeneration is required. Laboratory studies of catalyst regeneration on a filtration unit in dynamic mode were conducted, which showed that six cycles of washing with a solvent based on aviation kerosene with the addition of a surfactant are sufficient. The paper also describes a technology for washing the catalyst after a decline in well performance.

The obtained results can be effectively used in the design and implementation of hydraulic fracturing for oil extraction within a temperature range of 100–120°C. The proposed technology enables a significant increase in well production rates and the efficiency of field operations.

Keywords: *hard-to-recover hydrocarbons, hydraulic fracturing, proppant, in-situ oil catalysis, zeolite, chromium carbonate, production enhancement.*

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1. Introduction

In the context of depletion of light oil reserves, as well as the high degree of production of most of Ukraine's fields, the problem of involving highly viscous and difficult-to-recover oils in the development, as well as the extraction of residual oil reserves, is becoming particularly urgent. These oils are characterized by increased density, significant viscosity, high content of asphalt-resin-paraffinic substances (ARPS) and, accordingly, low efficiency of traditional extraction [1].

For the extraction of such hydrocarbons, hydraulic fracturing (HF) is becoming increasingly popular, which is one of the most effective methods of intensifying hydrocarbon production. The implementation of the ARPS operation is ensured by sequential injection of fracturing fluid and sand-bearing fluid, as a result of which a system of artificial fractures is created in the formation. In order for the cracks not to close, proppant is used. At the present stage, the following main types of proppants are used in industrial practice: natural quartz sand, medium-strength proppants and high-strength ceramic proppants based on bauxite. As a result of hydraulic fracturing, due to the increase in the permeability of a heterogeneous formation and the inclusion in the development of previously undrained zones and layers, a significant increase in hydrocarbon production is achieved [2].

Increasing the efficiency of hydraulic fracturing of the formation can be ensured by combining it with other methods of intensifying the flow of fluids [3], which leads to an increase in the well output and increases the technological and economic efficiency of the development of deposits.

An effective method of intensification is the use of catalysts for in-situ catalysis, i. e. the splitting of heavy hydrocarbons into lighter components. It is the catalysts that ensure the achievement of maximum reaction efficiency at the lowest possible temperature. One of the most important properties of a catalytic substance is its ability to maintain activity over time, as well as the ability to restore its properties by regeneration.

The essence of the method is that the productive reservoir is treated with a catalytically active mixture, a thermal effect is created on the reservoir at a temperature of 90–150°C to simulate the autocatalysis of the processes of decomposition of hydrocarbon molecules into radicals and their subsequent recombination, disproportionation, substitution and isomerization. As a result of these processes, simple hydrocarbons of the type of alkanes C_1–C_4 appear in the reservoir fluids, the yield of light gasoline fractions C_6–C_{10} and more mobile fractions of light gas oil C_{11}–C_{16} increases. The fractional composition of hydrocarbons changes, the density, dynamic viscosity, and average molecular weight decrease. An increase in hydrocarbon permeability is observed due to a change in the chemical composition, which in reservoir conditions will lead to an increase in the well output while simultaneously improving the quality of the hydrocarbon raw material [4, 5].

Previous studies of the effects of homogeneous and heterogeneous catalysts on oil and condensate recovery have been reviewed in [4, 6]. Heterogeneous catalysts, unlike homogeneous ones, act through reactions at the interface between a solid catalytic carrier and a liquid petroleum feedstock [7]. For example, oxide catalysts Fe_2O_3 and Cr_2O_3 promote the decomposition of macromolecules, while zeolites provide selective reduction of molecular weight without significant coke formation [8]. An important advantage of such systems is the possibility of their regeneration and reuse, which increases their cost-effectiveness [7, 8].

Experimental studies [6] were conducted on four types of oil typical of Ukrainian deposits, characterized by increased density, viscosity and significant content of ARPS: Karakoyizivska, Zhuravlinivska, Bobrytska and Yarmakivska. Zeolites, metal oxides, clay minerals, as well as carbonates and chromium oxides were used as catalysts. The main factors that determined the choice of zeolites for research are their catalytic activity, acidity, thermal stability and ability to adsorb heavy hydrocarbons. According to the results of the studies, combined catalysts based on zeolite and chromium carbonate ("zeolite + $CrCO_3$") turned out to be the most effective. Fractional distillation of the studied oils showed that after the oil contract with this catalytic mixture, a pronounced decrease in boiling points is observed in the entire range of fraction selection, and this directly indicates an increase in the content of volatile components.

Thus, the introduction of a solid catalyst into the pore space is advisable to carry out together with the proppant during hydraulic fracturing of the formation. Unlike traditional hydraulic fracturing technologies, in which the proppant prevents the fracture from closing, the proposed approach combines hydraulic fracturing of the formation with in-situ catalysis, which was not previously used. This will allow producing companies to increase the recovery of high-viscosity oils by conducting hydraulic fracturing of the formation, since as a result of in-situ destruction, the oil becomes lighter and more mobile.

The object of research is the process of in-situ catalytic conversion of oil after hydraulic fracturing of the formation using a catalytically active proppant.

The aim of research is to assess the technological efficiency, duration of action and the possibility of regeneration in reservoir conditions of a heterogeneous catalyst based on the "zeolite + $CrCO_3$" composition, which is used as an additive to the proppant during hydraulic fracturing of oil formations.

To achieve the aim, the following objectives were set:

1) to determine the rational temperature range of operation of the "zeolite + $CrCO_3$" heterogeneous catalyst in reservoir conditions;

2) to develop a technology for preparing catalytically active granules intended for addition to proppant during hydraulic fracturing of the reservoir;

3) to estimate the resource of the "zeolite + $CrCO_3$" catalytic composition until the moment of regeneration;

4) to substantiate the possibility and develop a technology for catalyst regeneration in reservoir conditions.

2. Materials and Methods

First, an analysis of laboratory studies of the influence of the selected catalytically active mixture "zeolite + $CrCO_3$" on the physicochemical properties of oil (viscosity, density, fractional composition and boiling point) was performed.

During the experimental studies, the following instruments and equipment were used:

- oil viscosity – glass viscometers of the ВПЖ type (Ukraine);
- density – glass hydrometers with scales of 0.75–0.95 (Ukraine);
- fractional composition – atmospheric-type laboratory installation [9];
- static studies – bomb-type autoclave installation [9];
- filtration studies – atmospheric-type laboratory installation [9];
- laboratory scales ВЛР-200 (Ukraine);
- drying and other auxiliary equipment.

The studies were performed in static (autoclave tests) and dynamic (filtration experiments) conditions at pressures and temperatures corresponding to reservoir conditions. In static experiments, an autoclave reaction unit of the bomb type [9] was used, which ensured uniform heating and constant contact of oil with the catalytic system. Experiments on a filtration unit [9] reproduced the conditions of oil movement through a porous medium filled with catalyst and proppant, which made it possible to simulate real filtration conditions in the reservoir. The efficiency of the catalysts was evaluated by changes in density, viscosity, fractional composition, gas formation pressure and ARPS content. The studies were carried out by atmospheric distillation of oil on a laboratory atmospheric type unit [9], which allows determining the temperature range of boiling of fractions and their quantitative distribution. Catalysts that demonstrate high efficiency contribute to a decrease in the initial boiling point of hydrocarbons, which indicates their ability to activate the catalysis of heavy fractions. In addition to changes in fractional composition, one of the main parameters of the efficiency of catalysts is the effect on the density and viscosity of oil. High density and viscosity are key factors limiting the mobility of hydrocarbons in the reservoir, so an effective catalyst should contribute to their reduction.

In order to determine the effect of temperature on the effectiveness of catalysts on Karakoyizivska oil, a series of experiments were conducted under static conditions (in a bomb-type installation) using a "zeolite + $CrCO_3$" mixture. The experiments were carried out at temperatures of 90, 110 and 150°C, which corresponds to the conditions of thermocatalytic action in the bottomhole zone of wells in the Dnipro-Donets Basin, where at the depths of industrial oil occurrence, reservoir temperatures are usually within 60–120°C [10]. For each experiment, a chamber with a working volume of 300 ml was filled with oil, proppant and catalyst in a ratio of 90:10 (90 ml of proppant and 10 ml of catalyst). The tightness of the system was ensured by complete displacement of air from the working zone, which made it impossible to influence foreign oxidants. Heating to the set temperature was carried out gradually over 40–50 min, after which the samples were kept isothermally for 30 minutes. Cooling was carried out in two stages: first naturally to 80°C, and then by forced immersion of the reactor in a cold water bath. After the completion of the treatment cycle, the reaction products were subjected to fractional distillation to determine the boiling temperature intervals of individual fractions, as well as to measure the density by the areometric method. The pressure in the reaction chamber during the experiments fluctuated within 180–260 atm, which confirms the intensity of gas formation reactions with increasing temperature.

The methodology for experimental studies of the activity of the "proppant – zeolite + $CrCO_3$" catalytic mixture consisted in passing oil through a sample of catalytically active material. One catalyst cycle was assumed to pass 10 cm^3 of oil through an equivalent volume of the mixture, which consisted of 80% proppant (inert filler) and 20% catalyst, in which 80% was natural zeolite and 20% was chromium carbonate $CrCO_3$. The duration of one pumping cycle was 0.5 h. After 25 h (the research period), an oil sample was taken, for which the density, kinematic viscosity and boiling point were determined. To confirm the possibility of regenerating the catalytic mixture in the reservoir, laboratory studies were conducted on a filtration unit [9] in a dynamic mixture washing mode. Based on the obtained positive effect, a technology for in-situ regeneration of the catalytic mixture in proppant using a liquid based on aviation gas with a surfactant was proposed.

3. Results and Discussion

3.1. Determination of the rational temperature range of operation of the "zeolite + $CrCO_3$" heterogeneous catalyst

The results of the influence of the temperature regime on the operation of the catalyst are shown in Table 1 and on the acceleration curve of Karakoyizivska oil (Fig. 1).

As can be seen from Table 1, with increasing temperature, a gradual decrease in the density of the studied oil is observed, which indicates a decrease in the content of high-molecular components and heavy fractions.

The initial kinematic viscosity of the Karakoyizivska crude oil was 5.3 mm^2/s, which corresponds to typical indicators for medium-paraffinic oils with a high content of ARPS. After treatment with a catalyst, the viscosity decreased almost 2 times and amounted to 2.9 mm^2/s [6].

Thus, a decrease in density, redistribution of the fractional composition and a decrease in the viscosity of the oil after treatment indicate a comprehensive improvement in the rheological properties of the system, which potentially increases fluid mobility in the reservoir and recovery efficiency.

In all experiments at different catalyst operating temperatures, a decrease in the boiling point compared to crude oil is noted. At 90°C, the process is stable, but less efficient, while at 150°C the maximum yield of light fractions is achieved, but the process is too intensive. This allows to conclude that for practical application it is advisable to use the "zeolite + $CrCO_3$" catalyst mixture precisely within the range of average temperatures (100–120°C), which ensures high efficiency and stability of catalytic processes. This temperature range corresponds to the most common depths of hydraulic fracturing in Ukraine.

Table 1

Change in parameters of Karakoyizivska oil at different temperatures of treatment with the "zeolite + $CrCO_3$" mixture

Parameter	Crude oil	Experiment temperature		
		90°C	110°C	150°C
Density, g/cm^3	0.830	0.814	0.812	0.808
*BP**, °C	85	60	60	55
Distillation volume	Boiling point, °C			
10%	90	75	75	70
20%	115	100	100	95
30%	140	130	125	120
40%	180	165	155	155
50%	240	225	205	195
60%	275	270	240	230
70%	320	–	–	–

Note: * *BP* – boiling point

3.2. Technology of preparation of catalytically active granules for addition to proppant

Within the framework of this work, a modification of the classical hydraulic fracturing technology is proposed by introducing catalytically active granules into the proppant mixture, which combine the mechanical function of supporting the fracture with catalytic activity towards oil components.

To implement the technology of hydraulic fracturing using a catalytically active mixture, a technological scheme for preparing a granular catalyst (Fig. 2) intended for injection into the productive formation together with the proppant was used. The resulting granules perform a dual function: on the one hand, they mechanically fix the hydraulic fracturing cracks, and on the other hand, they provide catalytic activation of the hydrocarbon phase directly in the pore space of the formation.

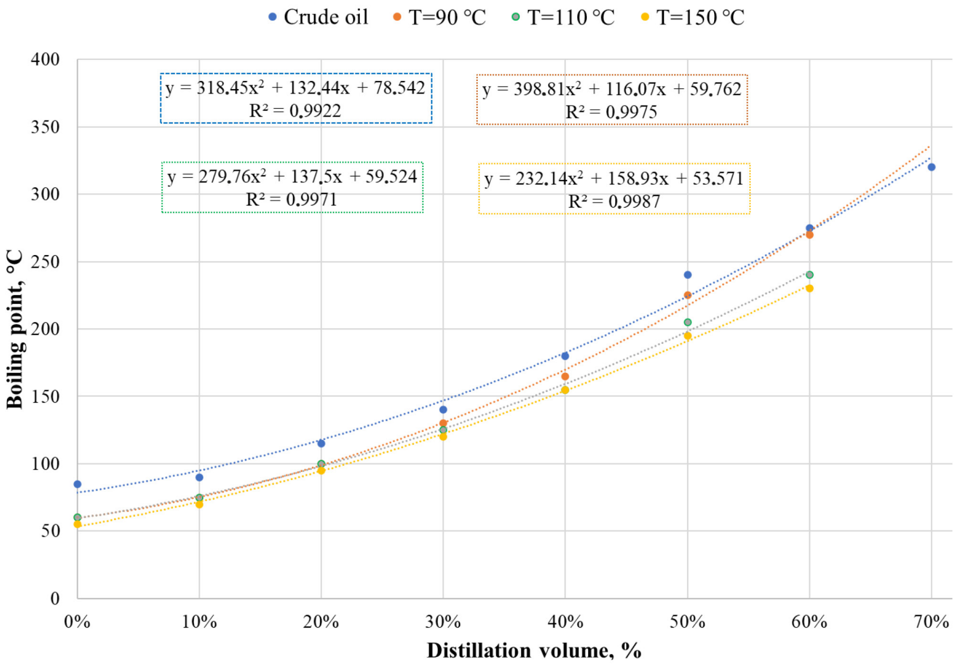

Fig. 1. Karakoyizivska oil acceleration curves at different temperatures

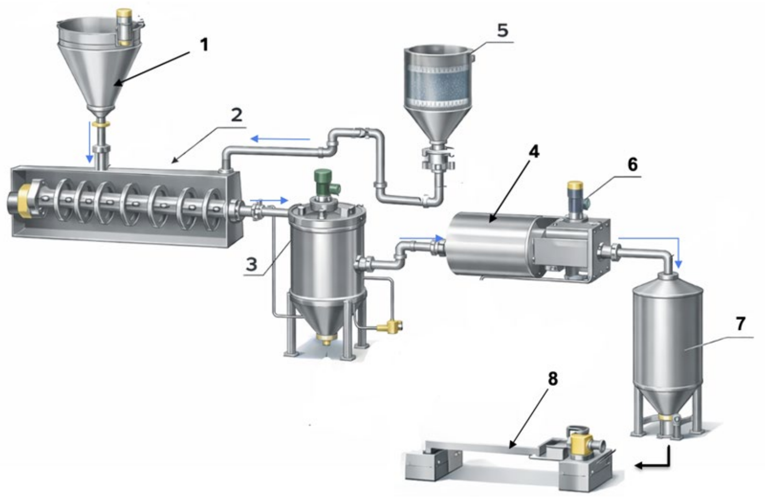

Fig. 2. Technological scheme for the preparation of catalytically active granules: 1 – bulk material hopper; 2 – screw mixer; 3 – granulator; 4 – heat exchanger; 5 – liquid component hopper; 6 – sieve; 7 – storage hopper; 8 – fraction calibration sieve

The preparation of the catalytically active mixture is carried out according to a continuous technological scheme, which includes the stages of dosing, dissolution, mixing, heat treatment and granulation.

The starting materials used for the synthesis of the catalyst are fed from separate dosing hoppers. The process begins with the feeding of the components into the bulk material hopper 1, from where they enter the screw mixer 2, where preliminary mechanical mixing of the solid phases is carried out. Then the mixture is transported to the granulator 3, equipped with a heating system.

In parallel, a solvent and acid additives are fed from the liquid component hopper 5 through the dosing system. In the reactor, a homogeneous reaction mass is formed with the formation of catalytically active centers due to the coordination of transition metal ions with acid groups.

To ensure the stability of the catalyst structure, the reaction mixture is fed into the heat exchanger 4, where the specified temperature regime is maintained, necessary for the polymerization processes and the formation of a strong matrix of future granules.

After heat treatment, the mixture is fed to a calibration sieve 6, which allows separating fractions suitable for use as a proppant in hydraulic fracturing.

The finished catalytically active granules are accumulated in a storage hopper 7, after which, if necessary, additional screening of the granules occurs through a mesh with a diameter of 1.5 mm 8 and then transported to the well for further use as part of the hydraulic fracturing fluid.

During hydraulic fracturing, the catalyst in the form of granules is injected into the cracks together with a traditional proppant in a ratio of 0.2:0.8, respectively. This ratio was chosen based on previous stationary experiments, in which at these concentrations the optimal balance between acidity, catalysis rate and catalyst structure stability was observed. Excessive catalyst concentration (>20%) led to an increase in the hydraulic resistance of the filtration zone, and a lower one (<20%) to a decrease in the efficiency of catalytic processes. Due to their high mechanical strength, they provide stabilization of cracks, preventing their closure after pressure relief.

At the same time, heterogeneous catalysis of hydrocarbon transformations is implemented on the surface of the granules, in particular:

- catalysis of heavy oil fractions;
- reduction of formation fluid viscosity;
- activation of hydrocarbon desorption processes from the rock surface.

Thus, the proposed technology combines mechanical and chemical-catalytic effects on the productive reservoir, which leads to increased permeability, intensification of inflow and an increase in the final hydrocarbon recovery coefficient.

Experimental studies on the development of the catalyst formulation have shown that the most effective composition was a composition of 80% natural zeolite and 20% chromium carbonate. When using compositions with a lower chromium content (from 5 to 15%), the efficiency of catalysis decreased, which was assessed by viscosity indicators (the decrease in value was only 20–40% of the initial one) and the initial boiling point of oil (the initial boiling point was 10–15°C higher than in the most effective formulation). The use of compositions with a chromium content of 25–30% led to excessive coke formation and, as a result, loss of catalytic activity – inactivation of the catalyst occurred within 3–4 cycles of its use. It is the composition of 80% natural zeolite and 20% chromium carbonate that ensures equalization of the rates of the external and in-situ diffusion stages of the process of catalytic destruction of heavy hydrocarbons. This effect is fundamentally important for the reservoir conditions, since it is diffusion limitations that most often cause local supersaturation of active centers and intensification of the formation of coke-like products.

3.3. Assessment of the resource of the "zeolite + $CrCO_3$" catalytic composition until the moment of regeneration

The efficiency of in-situ catalysis is directly determined by the duration of preservation of the activity of the catalytically active mixture consisting of proppant, natural zeolite and chromium carbonate.

Experimental studies of the activity of the catalytic mixture "proppant – zeolite + $CrCO_3$" were carried out on Karakoyizivska oil with the following initial characteristics of crude oil: density – 0.83 g/cm^3, kinematic viscosity – 5.3 mm^2/s; boiling point – 65°C.

In the course of the research, it was established that the catalytic mixture retains activity for 52 periods of oil pumping. Table 2 shows the results of the research at the final stages of the experiment.

Table 2

Results of studies of the activity of the catalytic mixture at the final stages of the experiment

Measurement No. (period)	Indicators		
	Density, g/cm^3	Kinematic viscosity, mm^2/s	Boiling point, °C
49	0.808	3.09	72
50	0.814	3.23	74
51	0.817	3.42	78
52	0.822	3.50	82

It was experimentally determined that the effective activity of the catalyst is maintained for 1100 kinetic cycles (52 periods), and then it decreases. This was characterized by an increase in the boiling points of oil fractions by 10–15°C, the density of the studied Karakoyizivska oil from 0.799 to 0.822 g/cm^3 and higher, as well as an increase in kinematic viscosity from 2.9 to 3.5 mm^2/s. This led to the inefficiency of the catalysis process as a whole.

The balance of diffusion stages allows minimizing surface coking, reducing the rate of catalyst deactivation and stabilizing the reaction rate at a high level. The experimentally established rate of catalytic destruction is 12–16 mol per second. For further calculations, the average value of 14 mol per second is taken.

To calculate the kinetic resource, the average molecular weight of heavy oil fractions was assumed to be 226 g/mol (equivalent to C_{16}–C_{18}), which corresponds to 4420 mol in 1 ton of oil.

Thus, approximately 17.2 hours are required to process 1 ton of oil, which corresponds to one regeneration cycle. The total resource of 1100 operating cycles is 18920 hours, or approximately 788 days of continuous operation until regeneration.

Given an average well flow rate of 10 m^3/day, the total volume of oil for the calculation period will be 7880 m^3. Therefore, one ton of catalytically active granules provides catalytic processing of approximately 7–8 thousand m^3 of oil until regeneration is necessary.

3.4. Justification of the possibility and technology of catalyst regeneration in reservoir conditions

During operation in the bottomhole zone of the productive reservoir, heavy paraffin and resin-asphaltenic deposits gradually accumulate on the surface of the catalyst. This causes a deterioration in the performance of the catalytic transformation of oil, namely: a change in the fractional composition (an increase in the temperatures of the beginning and end of boiling), as well as an increase in its viscosity and density. ARPS screen active centers, complicate the access of hydrocarbon molecules to the pore structure and reduce the efficiency of catalytic conversion. In view of this, the regeneration of the catalytically active mixture is considered as a full-fledged technological operation integrated into the well operation cycle.

For regeneration by dissolving and subsequent removal of these deposits, a developed composition of the regenerating fluid for injection into the reservoir based on aviation kerosene and a composition of surfactants (surfactants) was proposed.

A cycle of laboratory studies was conducted to assess the effectiveness of the proposed technological solutions.

Laboratory studies of catalyst regeneration were conducted on a filtration unit in dynamic mode using Karakoyizivska oil as a model hydrocarbon medium. The reaction bomb was completely filled with a mixture of proppant and catalyst without the presence of air layers, which allowed to adequately simulate the porous medium of the productive reservoir and the conditions of fluid filtration in the bottomhole zone.

The studied mixture consisted of proppant (80% of the total volume), zeolite (15%) and chromium carbonate (5%). To simulate the process of contamination of the working surface of the catalyst, the mixture of zeolite and chromium carbonate was pre-mixed with paraffin in a ratio of 1:1. This approach allowed to recreate the real conditions of accumulation of ceresin and resin-asphaltene deposits, characteristic of long-term operation of the catalyst in reservoir conditions.

At the first stage, oil was pumped through the system at a rate of 5 ml/min, while a deterioration in the fractional composition and an increase in density were recorded, which indicated a decrease in catalytic activity due to surface contamination. To restore activity, a washing stage was carried out with the injection of a special washing solution. After that, oil was pumped again at a similar speed.

Washing was performed in several cycles. Analysis of the regeneration efficiency was carried out after 3 and 6 cycles, which corresponded to pumping volumes equivalent to 3 and 6 volumes of the reaction bomb. After each stage, density and atmospheric distillation curve measurements were performed to assess the degree of recovery of catalytic activity.

The research results are given in Table 3.

The experimental data obtained show that contamination of the catalyst with paraffin leads to a significant deterioration of the main physicochemical parameters of oil. In particular, the density of the sample after contamination increased from 0.830 to 0.835 g/cm^3, which indicates an increase in the proportion of heavy fractions in the product composition. Despite a slight decrease in the boiling point, the fractional composition shifted towards higher boiling points, which is confirmed by an increase in temperatures for 50% and 60% distillation to 250°C and 295°C, respectively.

Carrying out six stages of washing gave a clearly pronounced positive effect. After only 3 cycles, the density of the oil decreased to 0.828 g/cm^3, and the fractional composition began to approach the parameters of crude oil. Further washing up to six cycles led to even more pronounced recovery: the density reached a value of 0.820 g/cm^3, and the boiling point decreased to 60°C. At the same time, a decrease in temperatures was observed for 50% and 60% distillation to 205°C and 240°C, respectively, which indicates intensive removal of heavy components and increased formation of light fractions.

Table 3

Results of washing the catalytically active mixture with a solvent

Parameter	Crude oil	After contamination (before washing)	After washing (3 cycles)	After washing (6 cycles)
Density, g/cm^3	0.830	0.835	0.828	0.820
*BP**, °C	85	80	80	60
Distillation volume	Boiling point, °C			
10%	90	90	90	75
20%	115	120	110	100
30%	140	140	136	125
40%	180	180	170	160
50%	240	250	220	205
60%	275	295	255	240

Note: * *BP* – boiling point

Thus, the results of laboratory studies confirm that contamination of the catalytically active mixture with paraffin deposits leads to a decrease in the efficiency of in-situ catalysis and a deterioration in the quality of hydrocarbon products, but is also subject to regeneration.

In field conditions, regeneration begins with well preparation. At this stage, the well is stopped and its flushing is performed to remove mechanical impurities, free paraffins and corrosion products. The flushing is carried out in a circulation manner using a high-pressure pump unit, process tanks, a pipeline system and wellhead equipment. The purpose of the preparatory stage is to create hydrodynamic conditions that ensure uniform penetration of the regenerating fluid into the area of the catalytically active mixture.

After preparation, the regenerating solution is prepared. A composition based on aviation kerosene with the addition of a surfactant is used as a solvent. Kerosene acts as an organic solvent for paraffins and partially resinous components, while surfactants reduce interfacial tension, improve the wettability of the granule surface and promote the desorption of asphaltenes from the active centers of the catalyst. The solution is prepared in a mixing tank with mechanical stirring until a homogeneous composition is obtained, which ensures the stability of properties during injection.

The prepared solution is injected into the formation through the tubing or through the annulus using pumping equipment that allows maintaining a controlled pressure. The injection pressure is selected in such a way as to ensure the penetration of the solvent into the bottomhole zone and the area of the catalytically active mixture, without exceeding the hydraulic fracturing pressure of the formation. The injection volume is determined taking into account the capacity of the pore space and the radius of the treated area. After the injection is completed, the well is sealed and left for a technological hold lasting an average of 6 to 12 hours. During this period, the solvent diffuses into the porous structure of the proppant-catalytic mixture, dissolves paraffin and resinous deposits, and gradually cleanses the active surface of zeolite and chromium carbonate. The hold duration is determined by the formation temperature, the degree of catalyst contamination, and the rheological properties of the solvent.

After holding, reverse circulation or well development is carried out in order to remove dissolved deposits to the surface. The process is accompanied by sampling of the spent solvent for further analysis. At this stage, pumping units, separation tanks and equipment for collecting and temporarily storing the spent liquid are used.

The efficiency of regeneration can be controlled by various methods. One of such methods is fractional distillation of the selected solvent. The appearance of heavy fractions in it, an increase in the initial boiling point or an increase in the residue after distillation indicate the removal of paraffin and resin deposits from the surface of the catalyst. Additionally, the cold rod method is used, which consists in immersing a cooled metal element in the selected solvent. The deposition of a layer of crystallized paraffins on its surface confirms the presence of removed deposits and the effectiveness of the cleaning process.

If after one cycle the full recovery of the flow rate and fractional composition of the oil is not achieved, the regeneration can be repeated.

Thus, the implementation of this technology ensures the restoration of active catalyst centers, the extension of its interregeneration resource and the stabilization of the quality indicators of the produced oil.

3.5. Limitations and directions of research development

The obtained research results can be used as a theoretical and practical basis for the implementation of the method of in-situ catalysis using a "zeolite + $CrCO_3$" heterogeneous catalyst as an additive to the proppant during hydraulic fracturing of the formation with the possibility of its subsequent regeneration with solutions based on aviation kerosene with the addition of surfactants.

The practical significance of the work lies in the possibility of implementing the technology of intensification of oil production, which ensures an increase in the well output, the inclusion in the development of previously undrained zones and layers and increasing the economic feasibility of field development.

The limitation of research is that there are no field studies and implementation of the use of the proposed catalyst as an additive to proppant during hydraulic fracturing of an oil reservoir. Prospects for further research are to conduct experimental and field studies of a combination of catalytic and thermal effects for the extraction of difficult-to-extract oils at temperatures below 90–100°C. Catalysts that are activated at temperatures of 50–70°C can significantly improve the efficiency of oil extraction in conditions where reservoir temperatures do not exceed 100°C. The use of thermal methods is aimed at starting the autocatalysis process. Another direction of research is to study the effect of catalysts with the simultaneous use of surfactants, acidic hydraulic fracturing fluids, etc.

4. Conclusions

1. It has been established that the use of the "zeolite + $CrCO_3$" catalyst improves the physicochemical and rheological properties of oil: the boiling point of fractions, density and viscosity decrease, which increases the mobility of oil in the formation. The rational temperature range of the catalytic mixture is 100–120°C, within which the highest efficiency of the thermocatalytic conversion of oil is ensured.

2. A technology for preparing catalytically active granules with subsequent addition to the proppant has been proposed. The resulting granules perform a dual function: they fix cracks and provide catalytic activation of the hydrocarbon phase directly in the formation. The rational ratio of catalytic granules and proppant is 0.2:0.8, respectively, while the ratio between granular zeolite and chromium carbonate is 0.8:0.2, respectively.

3. The resource of one ton of catalytically active granules of the "zeolite + $CrCO_3$" composition until the need for regeneration has been determined, which is approximately 7–8 thousand m^3 of processed oil.

4. Laboratory experiments on washing the catalytically active mixture with a solvent confirmed the possibility of catalyst regeneration. Six-stage washing of the catalyst ensured the restoration of oil density from 0.835 to 0.820 g/cm^3, a decrease in the initial boiling point from 80 to 60°C, as well as the boiling point at 50 and 60% distillation – by 45 and 55°C, respectively, which indicates the effective removal of heavy components (paraffins). According to the results of these studies, it was proposed to use a solvent based on aviation kerosene with the addition of a surfactant for washing in reservoir conditions.

Conflict of interest

The authors declare that they have no conflict of interest regarding this research, including financial, personal, authorship, or other, that could influence the research and its results presented in this article.

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The research was conducted without financial support.

Data availability

The manuscript has no associated data.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the presented work.

Authors' contributions

Ivan Zezekalo: Conceptualization, Methodology, Validation, Formal analysis, Writing – review and editing, Supervision; ***Mykola Podoliak***: Investigation, Formal analysis, Writing – original draft, Visualization, Project administration; ***Iryna Lartseva***: Writing – review and editing, Visualization.

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