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Catalytic Conversion of Heavy Oil Using Molybdenum Based Water-soluble Catalyst

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ABSTRACT

The growing global trend of putting heavy oil fields into development is a complex task that requires updating previously unprofitable approaches. One of such areas is cyclic steam stimulation of wells to stimulate the production of high-viscous and super-viscous oils. An increase in well flow rates and the depletion of reserves, a decrease in the time and financial costs of well servicing and workover can be achieved by combining thermal and chemical methods of inflow stimulation. This approach has proven itself successfully in the catalytic in-situ upgrading of oil. Within the framework of this article, a series of laboratory experiments were performed to confirm the effectiveness of ammonium molybdate as a water-soluble precursor of an aquathermolysis catalyst for upgrading super-viscous oil from one of the fields of the Timan-Pechora oil and gas province. The decrease in oil viscosity relative to the non-catalytic effect was 24%, which was also confirmed by the results of the SARA-analysis of oil: a decrease in the amount of asphaltenes by 15.9%, an increase in the amount of saturated and aromatic hydrocarbons by 3% and 4.6%, respectively. Based on a simple numerical model was predicted an increase in oil flow rate with a catalytic effect relative to a non-catalytic effect by 23%. The results obtained indicate the potential for using this catalyst precursor in field conditions.

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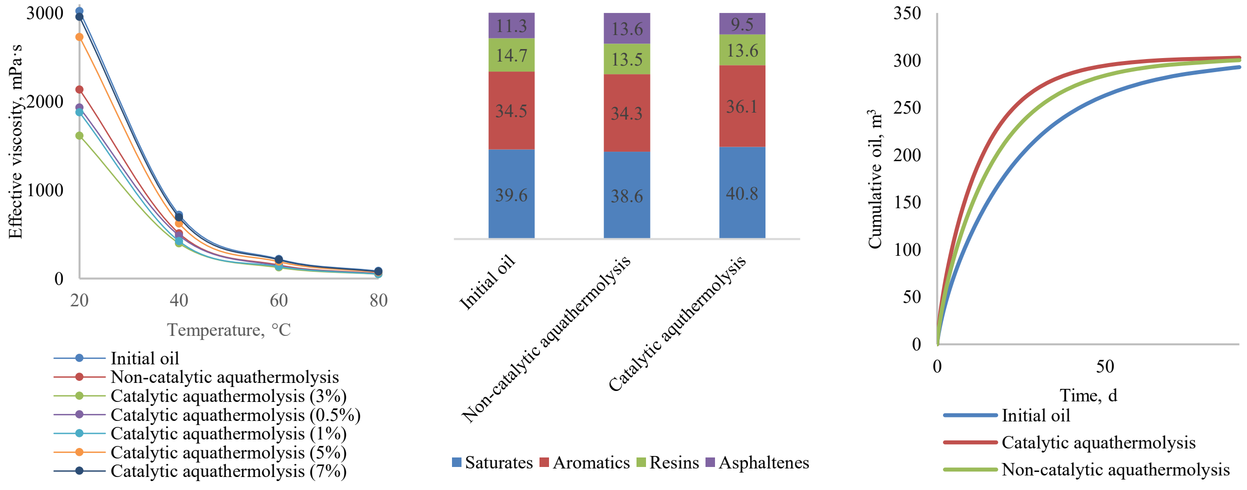
Graphical Abstract

Effective viscosity of oil (at a shear rate of 100 s^{-1}) (a), results of SARA-analysis (%) (b), cumulative oil with different viscosity characteristics (c)



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1. INTRODUCTION

Development of hard-to-recover reserves (HTR) represented by complex geological conditions and heavy oil (1-3) is one of the most important strategic tasks of the oil and gas industry. The solution to this problem lies in sufficient investment in new technologies (4), conducting comprehensive laboratory studies (5, 6), determining an integrated approach to reduce the negative impact on the reservoirs and planning operations to restore productivity (7), building complex numerical compositional models (8), as well as substantiating the most effective approach to development. Stimulation of productive reservoirs with steam is one of the key approaches to the development of deposits of super-viscous oils (9, 10). The choice of optimal conditions for the implementation of in-situ upgrading of super-viscous oil is an important task when planning and conducting cyclic steam stimulation (CSS) of the well. The in-situ upgrading of oil is based on aquathermolysis reactions. It is the reaction of transformation of high-molecular oil components in the presence of water at a temperature of 200–320 °C (11) as a result of destruction of carbon bonds with sulfur, oxygen and nitrogen. In addition, changes in pressure can also influence the nature of aquathermolysis reactions (12, 13). The carbon-sulfur bond is most prone to rupture due to its relatively low energy (14). Increased efficiency of these processes is achieved by including precursors of catalysts based on transition metals in the aquathermolysis reactions (15). The functioning of the catalyst begins after its transition to the active form: decomposition of catalyst precursors with formation of the active form: sulfides and oxides of transition metals (16). Today, physical modeling of the conditions of in-situ oil conversion in the presence of steam at high-pressure and high-temperature cells (autoclaves) is one of the key methods for evaluating aquathermolysis catalysts. Based on previous research, it can be noted that molybdenum ranks first among transition metals in catalytic activity: Mo>Cu>Ni>Fe>Co>Zn>Mn (17, 18). At the same time, molybdenum contributes to a higher yield of the liquid fraction and a decrease in the amount of coke, which was confirmed by the results of tests under severe conditions (460 °C). It is also confirmed by the significant effect of molybdenum on the destruction of asphaltenes and resins (19, 20). They proved that molybdenum sulfides formed in-situ are almost 10 times smaller than sulfides of other transition metals. It indicates their smaller specific surface area and, as a consequence, lower activity. Molybdenum as an active metal demonstrates a higher decrease in the molecular weight of oil and sulfur in resins than Fe and Ni using the same ligand due to its higher ability to hydrogenate. It contributes to further opening of aromatic rings (21). Deng et al. (22) found that the presence of nickel in oil after in-situ upgrading can lead to coking of oil refining catalysts.

Thus, nickel and molybdenum arylsulfonates, as well as some metals alloyed with molybdenum (23-25), were used as oil-soluble catalyst precursors during studies of super-viscous oil from the Shengli oil field (China). After holding the mixture at a temperature of 200 °C for 24 hours, the decreases in viscosity of 97% and 72.7%, respectively, were achieved. After holding an oil sample from the Liaohe oil field (China) in an autoclave at a temperature of 240 °C for 24 hours in the presence of molybdenum oleate, a decrease in the molecular weight of the oil sample by 1.5 - 1.7 times was noted (26). In this case, Zhao et al. (27) showed that the hydrocarbon ligand "oleate" has greater catalytic activity in the reactions of in-situ upgrading of oil relative to "octanoate, decanoate and stearate". High efficiency (reduction of oil viscosity by 82.26%) was demonstrated by the precursor of aquathermolysis catalyst based on zeolite, molybdenum and zirconium oxides as a result of holding an oil sample from the Shengli oil field (China) in an autoclave at a temperature of 280 °C for 24 hours in the presence of a hydrogen donor 1,2,3,4-tetrahydronaphthalene (28). Tajik et al. (29) conducted autoclave studies of high-viscosity oil in the presence of a nanohybrid of silicon dioxide and graphene together with molybdenum disulfide. The aging was carried out with the addition of phenanthrene in decalin as a hydrogen donor at a temperature of 200 °C for 24 hours. The viscosity reduction was 80.8%. A high degree of methyl palmitate conversion was achieved in a high-pressure and high-temperature reactor in the presence of an oil-soluble ionic liquid based on molybdenum at a temperature of 320 °C for 3 hours (30). The use of water-soluble, oil-soluble precursors of aquathermolysis catalysts, as well as solid catalysts based on molybdenum, made it possible to reduce the viscosity of Mackay River bitumen as a result of holding in an autoclave for 24 hours at a temperature of 250 °C (31). The range of viscosity reduction was 52.6% - 95.1%. Wang et al. (32) carried out experimental studies of molybdenum and nickel catalysts applied to proppant in an experimental setup for in-situ upgrading of oil. Aquathermolysis reactions occurred in cells filled with rock mixed with activated proppant. Holding was carried out at a temperature of 240 °C for 24 hours. The viscosity of oil from the Liaohe oil field (China) was reduced by 84%. Also Wang et al. (32) note better distribution of the catalyst in oil when using oil-soluble precursors. However, there are also significant disadvantages: the complexity of separation from oil and further processing, the high cost of synthesis. In this case, the use of Mo_2C as an aquathermolysis catalyst demonstrated a better ability to activate water compared to the bimetallic catalyst NiMo (33). Successful field tests of a water-soluble precursor of the ammonium tetrathiomolybdate catalyst $(NH_4)_2MoS_4$ were also carried out for in-situ upgrading of super-viscous oil from the Shengli oil field (China) (34). First, 1 ton of catalyst precursor was injected, then 2500 tons of steam were injected at a rate of 10 t/h. The soaking period was 7 days, production was carried out for 4 months. The average decrease in oil viscosity was 85.7%, which is confirmed by a change in the group composition of the oil: a decrease in the amount of asphaltenes by 12%, an increase in the amount of saturated compounds by 3%. It is also worth noting that the removal of the catalyst together with the oil was recorded only in the third month of production, which indicates its effective adsorption on the reservoir rock. During physical modeling of in-situ upgrading of oil from one of the oil fields in China, the addition of a precursor of aquathermolysis catalyst containing MoO_3 contributed to the inhibition of coke formation. It has a positive effect on the preservation of the reservoir properties of the bottomhole formation zone (35).

The object of the study is super-viscous oil in the Timan-Pechora oil and gas province in Russia. In accordance with the results of the above studies, commercially available ammonium molybdate was used as a water-soluble catalyst precursor during physical modeling of in-situ upgrading of oil. This reagent does not require additional costs for the synthesis process and, presumably, will not require its separation from the oil due to its solubility in water.

The relevance of this work is due to the fact that the use of classical CSS in super-viscous oil fields stimulates the deposition of high-molecular components of oil (asphaltenes, resins) in the bottomhole formation zone. It significantly reduces the reservoir properties. In addition, molybdenum-based aquathermolysis catalysts have not previously been considered for possible use in conducting the CSS at one of the oil fields of the Timan-Pechora oil and gas province. This catalyst can have a technological effect even in the most accessible form. Thus, the main objective of this study is to confirm the catalytic properties of molybdenum in relation to the oil under study.

The Methods and Materials section presents the stages of laboratory research. During the work, oil was prepared for the study, the rheological properties and group composition of oil were determined before and after the experiment. Physical modeling of aquathermolysis reactions in an autoclave was also carried out. Further in the article, the results of numerical modeling are presented and the corresponding conclusions are made.

2. METHODS AND MATERIALS

2. 1. Preparation of Oil and Determination of its Properties

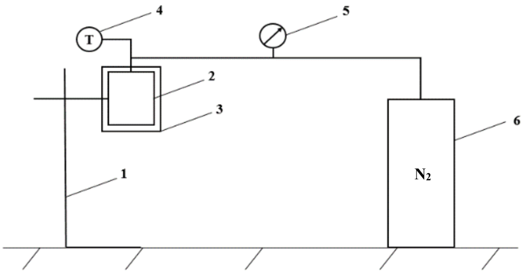
The studied oil was super-viscous oil from one of the fields of the Timan-Pechora oil and gas province with a viscosity of more than 3000 mPa·s at a temperature of 20 °C. The selected oil sample was pre-treated to separate water by adding a demulsifier to 1 liter of oil and keeping it in a heat chamber at a temperature of 70 °C for 8 hours. After that, the water was drained using a separatory funnel (Figure 1). The viscosity of the oil was measured using a Rheotest RN 4.1 rotational viscometer at different shear rates (1, 10, 50, 100 s^{-1}) for 5 min at each speed. The analysis of the group composition of the oil (SARA-analysis) was carried out in accordance with ASTM D4124-09 and GOST 32269-2013.

2. 2. Determination of Rheological Parameters of Oil

In order to simulate the reactions of catalytic aquathermolysis, distilled water and a water-soluble precursor of the catalyst for aquathermolysis reactions, commercially available ammonium molybdate $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$, chemically pure, were used. The experiment was carried out in a high-pressure and high-temperature reactor is an autoclave without a stirrer. The pressure in the autoclave was maintained by pumping nitrogen. Before loading with oil, water and a reagent, the reactor was purged with nitrogen to remove air. To minimize heat loss, a thermal jacket was put on the autoclave. The basic diagram of the laboratory bench is shown in Figure 2.

The autoclave was loaded with 33 g of oil and 10 g of water in accordance with the available capacity of the reactor (36-38). When modeling the reactions of catalytic aquathermolysis, ammonium molybdate was dissolved in water with constant stirring at a speed of 100 rpm for at least 15 min. In accordance with the experimental experience of conducting similar works (17, 26), an assumption was made about the effective concentration of the catalyst precursor of 3 wt.% by oil. To confirm this, the experiment was repeated for four additional precursor concentrations of 0.5%, 1%, 5%, 7%. Before loading the components into the autoclave, the oil and water with the dissolved catalyst precursor were subjected to active stirring at 100 rpm for at least 15 min to create a stable

Figure 1. Dehydrated oil sample



1 – chemistry stand; 2 – autoclave; 3 – thermal jacket; 4 – temperature gage; 5 – pressure gage; 6 – nitrogen bottle
Figure 2. Schematic diagram of a laboratory setup for modeling aquathermolysis reactions

emulsion (monitored visually). The formation of the emulsion in this case was important due to the need to increase the volume of interaction of oil with the potential catalyst, since it is known that at high temperatures the water-oil emulsion begins to collapse. The reaction mixture was held in the autoclave at a temperature of 250 °C and 7 MPa for 24 h. The reaction time was selected in accordance with the data reported by Baygildin et al. (39). After holding the samples, the heating was switched off, the pressure was gradually reduced to atmospheric, and the samples were cooled to room temperature for 8 hours.

3. RESULTS AND DISCUSSION

The results of measuring the viscosity of oil after catalytic and non-catalytic aquathermolysis are presented in Figure 3. The most informative results were obtained at a shear rate of 100 s^{-1}.

At a temperature of 20 °C, the decrease in oil viscosity after catalytic aquathermolysis in the presence of ammonium molybdate relative to the non-catalytic reaction is about 24%, and the overall effect of reducing viscosity relative to the initial oil is 46.6%. The viscosity values of oil in the case of adding the catalyst precursor in concentrations of 0.5% and 1% are close to the viscosity values resulting from the non-catalytic process. In this case, the process of destruction of high-molecular compounds does not reach maximum efficiency due to the low concentration of the catalyst. Increasing the concentration of the reagent to 5% and 7% has a negative effect on the viscosity values and brings its values closer to the parameters of the initial oil. This result is presumably associated with the aggregation of catalyst particles and the formation of large complexes with asphaltenes, which leads to an increase in the viscosity of the system (26). The reaction conditions relative to the conditions described by Qu et al. (31) are more stringent, since the reaction occurs without constant stirring and without an emulsifier, which affects the contact area of the oil and the resulting catalyst. It is assumed that the primary destruction of the emulsion is determined in the temperature range of 50-70 °C after 5 hours of reaction. Due to the fact that oil was sampled from the autoclave for viscosity measurement using a syringe, there is a possibility of water getting into the sample, which could also negatively affect the results obtained. Kadieva et al. (40) noted that as a result of aquathermolysis reactions, molybdenum-based precursors form catalysts in the form of the following molybdenum compounds: MoO_2, MoO_3, MoS_2. In their work, it was assumed that the above-mentioned compounds were also formed as a result of the experiments. This assumption is planned to be confirmed by SEM, XRD, FT-IR or other methods in subsequent studies. In addition, it is worth assessing the nature of the adsorption of catalysts in the pores of the rock containing the studied oil in order to exclude the likelihood of formation damage. At the same time, regardless of what specific compound was formed during the aquathermolysis reactions, simplified holding conditions and sampling errors, it is possible to unambiguously assert the achievement of a technological effect from the use of ammonium molybdate, and also with a high degree of probability speak about the effectiveness of oil-soluble molybdenum-based precursors. In this case, the water-soluble precursor eliminates the need for complex synthesis processes and can potentially be more easily separated from the produced oil, which speaks to the commercial advantages of the reagent.

To assess the potential change in well flow rate as a result of using $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$, the obtained viscosity characteristics were incorporated into a numerical model built in the software product for geological and hydrodynamic modeling. Modeling was carried out for a horizontal well to obtain the maximum possible inflow. The model dimension is 30x30x5 m (Figure 4). The parameters of the modeled object are presented in Table 1. The boundary conditions of the

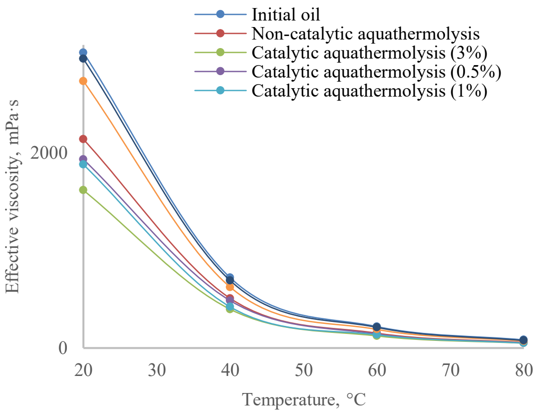


Figure 3. Effective viscosity of oil before and after the experiments (at a shear rate of 100 s^{-1})

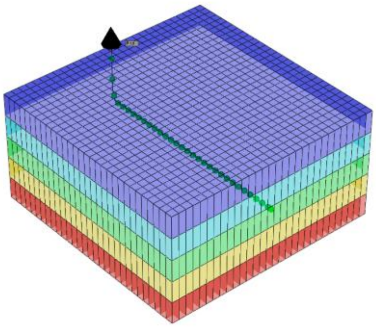


Figure 4. Model in software product

TABLE 1. Parameters of the simulated object

Propertie	Unit of measurment	Value
Absolute permeability	μm^2	0.9
Porosity	%	18
Initial reservoir pressure	MPa	7
Initial reservoir temperature	°C	28
Net reservoir thickness	m	5

model are the absence of heat and mass exchange between the object and the environment, as well as the same initial properties in each of the blocks. The constructed model does not take into account possible geological heterogeneity, the actual trajectory of the horizontal well, possible heat losses. In addition, only the process of oil production after conditionally completed cycles of CSS is modeled. The construction of a full-fledged model is an important task for future studies, requiring a detailed study of the geological structures of the target object, the inclusion of all necessary cycles of steam injection and chemical composition, the inclusion of all necessary chemical reactions and their kinetic parameters.

Well flow rate and cumulative oil production were calculated for three variants of oil viscosity characteristics: initial, after non-catalytic and catalytic in-situ upgrading (Figures 5 and 6) with catalyst precursor of 3 wt.% by oil. Oil flow rate after catalytic action increased relative to non-catalytic action by almost 23%, while the overall effect relative to the initial oil is about 61%, which is also reflected in the cumulative production graphs. The obtained results allow us to predict the technological effect of catalytic in-situ upgrading of oil using $(NH_4)_6Mo_7O_{24}\cdot4H_2O$ as a catalyst precursor in field tests.

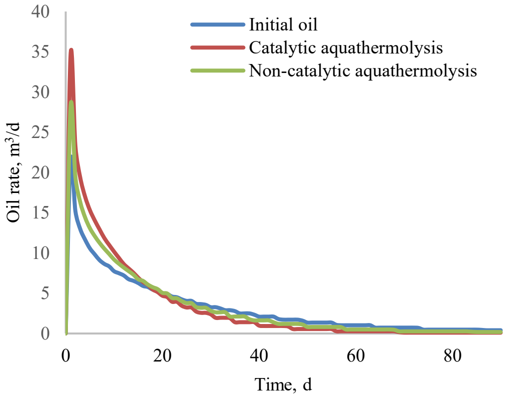


Figure 5. Oil rate with different viscosity characteristics

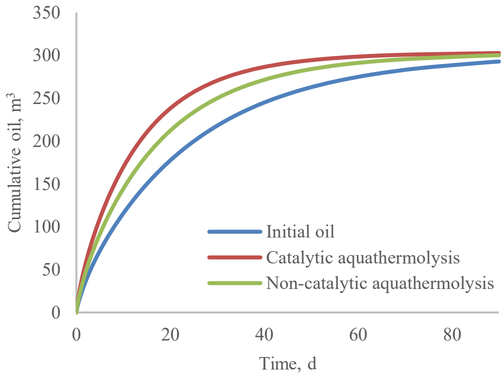


Figure 6. Cumulative oil with different viscosity characteristics

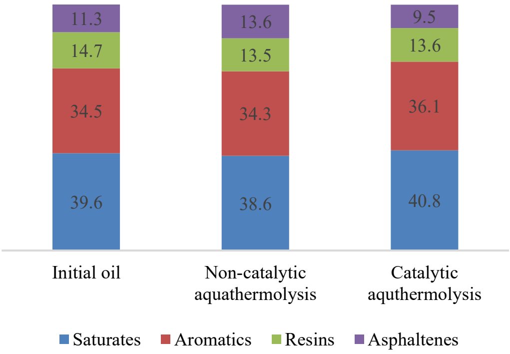


Figure 7. Results of SARA-analysis (%)

The results of SARA-analysis of oil before and after the experiment are presented in Figure 7. In non-catalytic aquathermolysis, a decrease in the amount of saturated, aromatic hydrocarbons, and resins in oil by 2.5%, 0.6% and 8.2%, respectively, was observed. But the amount of asphaltenes in oil, on the contrary, increased by 20.4%, which confirms the high probability of formation damage during a large number of cycles of CSS. At the same time, if we draw a parallel with viscosity characteristics, we can say that the viscosity of the oil under study is most sensitive to changes in the amount of resins in oil. The inclusion of ammonium molybdate in the reaction model confirms the assumption that the molybdenum-based catalyst contributes to an increase in the yield of light components of oil: an increase in saturated hydrocarbons by 3%, aromatic - by 4.6%. The resin content remains virtually unchanged relative to the non-catalytic aquathermolysis reaction, but a significant decrease in asphaltenes by 15.9% is an additional stimulus for a downward shift in the viscosity curve as a result of more active destruction of C-S bonds. The mechanism of destruction of this bond has been described in detail by Varfolomeev et al. (17), it corresponds to the temperature range of aquathermolysis reactions and can be conditionally illustrated by the following reaction:

$$RCH_2CH_2SCH_3+2H_2O \rightarrow RCH_3+CO_2+H_2+H_2S+CH_4$$

The obtained values once again confirm that even small changes in the group composition of oil can significantly affect its rheological characteristics. Such a change in the group composition of oil fully correlates with the obtained viscosity characteristic of oil and allows us to conclude that ammonium molybdate is effective as a water-soluble precursor of the aquathermolysis catalyst.

To assess the reproducibility of the viscosity measurement and SARA-analysis results obtained at a certain effective precursor concentration of 3 wt.% of oil, two additional viscosity and SARA-analysis tests were performed to calculate standard deviations. The calculation results are presented in Table 2.

The obtained values confirm the reproducibility and reliability of the constructed viscosity characteristic and SARA-analysis.

Li et al. (41) have noted that aquathermolysis reactions contribute to a change in the structure of paraffins crystals. It was a dense structure in the form of a "snowflake" before aquathermolysis. After which their "needle-like" stacking is observed. Thus, it is possible to prove the decrease in the aggregation of paraffins crystals and an increase in the degree of their dispersion, and, consequently, their transformation into lighter fractions. This fact is confirmed by the decrease in paraffins in the studied oil from 3.5% to 3.32% (with non-catalytic aquathermolysis) and to 3.21% (with the use of a catalyst precursor). In this regard, it can be argued that the in-situ formed molybdenum-based aquathermolysis catalysts not only improve the rheological properties of oil, but also reduce the likelihood of paraffin deposits in the bottomhole formation zone and downhole equipment.

In this study, the autoclave was not connected to a gas chromatograph, so the gas composition was not determined. Varfolomeev et al. (17) described aquathermolysis reactions in detail, suggesting that the main share of gases is H_2S and CO_2. Therefore, when selecting downhole equipment, preference should be given to corrosion-resistant materials.

TABLE 2. Standard deviations

Standard deviations in viscosity					
Propertie	**Unit of measurment**	**Value**			
Temperature of measurment	°C	20	40	60	80
Standard deviation	mPa·s	0,328	0,534	0,487	0,405
Standard deviations in SARA-analysis					
Hydrocarbon groups	-	S	A	R	A
Standard deviation	d.f.	0,179	0,454	0,475	0,351

4. CONCLUSION

Ammonium molybdate can be used as a water-soluble precursor of an aquathermolysis catalyst for in-situ upgrading of super-viscous oil from the fields in the Timan-Pechora oil and gas province in Russia. The decrease in the viscosity of the original oil under laboratory conditions during catalytic aquathermolysis was 46.6%, which is 24% more than in the case of non-catalytic action. A numerical experiment for various viscosity characteristics made it possible to predict an increase in the flow rate in the case of catalytic aquathermolysis relative to non-catalytic by 23%.

The results of the SARA-analysis of super-viscous oil after non-catalytic and catalytic aquathermolysis correspond to the viscosity characteristics of the oil. The significant decrease in asphaltenes in the oil confirms the minimization of contamination of the bottomhole formation zone with high-molecular components of oil in the case of using an aquathermolysis catalyst. Reducing paraffins in oil can also potentially increase the time between overhauls (TBO) and, accordingly, reduce operating costs.

Thus, current research is the starting point for studying the effect of molybdenum-based aquathermolysis catalysts on the reaction mechanisms of super-viscous oil aquathermolysis and defines the following tasks for future research:

- assessing the impact of molybdenum catalytic activity in the presence of a rock sample and the nature of catalyst deposition on this rock;
- assessing the effect of the deposited catalyst on the rock on the reservoir properties;
- assessing the degree of catalyst dispersion in oil after the experiment;
- assessing the nature of catalyst removal under conditions of high fracturing as part of filtration experiments;
- selecting the most effective hydrogen donor.

The obtained results can be used by interested parties and representatives of oil companies seeking to increase the technological potential of the CSS at high-viscosity and super-viscous oil fields.

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Persian Abstract

چکیده

روند رو به رشد جهانی توسعه میادین نفتی سنگین، کاری پیچیده است که نیاز به به‌روزرسانی رویکردهای پیشین غیرسودآور دارد. یکی از این حوزه‌ها، تحریک چرخه‌ای چاه‌ها با بخار برای تحریک تولید نفت‌های با ویسکوزیته بالا و فوق ویسکوزیته است. افزایش نرخ جریان چاه و تخلیه ذخایر، کاهش زمان و هزینه‌های مالی سرویس چاه و تعمیرات اساسی را می‌توان با ترکیب روش‌های حرارتی و شیمیایی تحریک جریان ورودی به دست آورد. این رویکرد خود را در ارتقای کاتالیزوری نفت درجا با موفقیت ثابت کرده است. در چارچوب این مقاله، مجموعه‌ای از آزمایش‌های آزمایشگاهی برای تأیید اثربخشی مولیبدات آمونیوم به عنوان پیش‌ساز محلول در آب یک کاتالیزور آکواترمولیز برای ارتقای نفت فوق ویسکوزیته از یکی از میادین استان نفت و گاز تیمان-پچورا انجام شد. کاهش ویسکوزیته نفت نسبت به اثر غیرکاتالیستی ٢٤٪ بود که با نتایج آنالیز SARA نفت نیز تأیید شد: کاهش مقدار آسفالتین‌ها به میزان ١٥.٩٪، افزایش مقدار هیدروکربن‌های اشباع و آروماتیک به ترتیب ٣٪ و ٤.٦٪. بر اساس یک مدل عددی ساده، افزایش ٢٣٪ در سرعت جریان نفت با اثر کاتالیزوری نسبت به اثر غیرکاتالیستی پیش‌بینی شد. نتایج به‌دست‌آمده نشان‌دهنده پتانسیل استفاده از این پیش‌ساز کاتالیزور در شرایط میدانی است.