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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

DEVELOPMENT OF NEW METHODS TO ELIMINATE PARAFFIN OILS AND COMPLICATIONS CAUSED BY AGGRESSIVE ENVIRONMENTS DURING WELL OPERATION

Speciality: 2525.01 – “Development and exploitation of oil and gas fields”

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GENERAL NATURE OF THE WORK

Relevance of the topic and the degree of development. Excessive non productive time or turnaround has a negative impact on production performance. Complications leading to repairs include the formation of a solid layer of asphaltene-resin-paraffin deposits on the inner surface of the pump-compressor pipes and well equipment, salt deposition in oilfield equipment and corrosion of this equipment.

Numerous researches and laboratory researches have been devoted to the problem of paraffin deposition control, based on the results of these studies, the mechanism of paraffin deposition, sedimentation conditions have been studied, methods and measures to control it have been developed. Eliminating the problems caused by paraffin deposition during the movement of high-paraffin oil remains relevant. Therefore, the development of new chemical methods - reagent-depressants - is important in the asphaltene-resin-paraffin deposits control.

In the final stages of development due to the fact that the production of oil is carried out mainly in the conditions of high flooding of the fields, high-aggressive gases dissolved in the water produced, the presence of Ca^{2+}, Mg^{2+}, Na^{+}, $SO_4{}^{2-}$, Cl^{-} salt ions and calcium and magnesium carbonates, calcium, barium sulfates, chlorides and other salts causes corrosion of oil field equipment and salt deposition on its surface.

Dissolved aggressive gases and salt ions in the water cause corrosion of the oilfield equipment, and inorganic salts precipitate on the equipment surface, complicating the movement of the fluid and activating the equipment failure. Corrosion of equipment and deposition of salt on the surface are relevant due to the fact that the flooding of most oil fields of Azerbaijan exceeds the critical limit.

Despite the wide range of corrosion and salt control reagents, the development of new complex inhibitors and compositions not only weaken the corrosion and salt deposition processes, it is important to protect equipment from corrosion, mechanical spills and salt deposits, as well as the development of biocidal inhibitors against corrosive microorganisms.

Research goals and objectives Development of new methods to eliminate paraffin oils and complications caused by aggressive environments during well operation

Object and subject of research

1. Development of a new method on asphalt-resin-paraffin deposits control;
2. Systematic analysis of samples taken in technological environments and productive strata and monitoring of corrosion status of fields;
3. Assessment of the role of microbiological factor in the process of equipment corrosion;
4. Determining the composition of inhibitors based on the analysis of the causes of equipment corrosion and the development of a corrosion inhibitor with a complex effect and bactericides;
5. Development of a new method for the prevention of inorganic salt deposits.

Research methods

The issues were solved by analyzing the results of laboratory and experimental research and their application in the fields.

Scientific novelty of the research:

1. New agent against asphalten-resin-paraffin deposition in wells;
2. A new corrosion inhibitor and inhibitor-bactericide composition have been developed against equipment corrosion and microbiological erosion;
3. A new composition has been developed against inorganic salt sediments formed on well equipment;

The main provisions:

1. A new agent that plays a depressant role against asphaltene-resin-paraffin deposition;
2. A new corrosion inhibitor and inhibitor-bactericidal composition against corrosion and microbiological erosion.
3. A new composition against inorganic salt sediments formed on well equipment.

Theoretical and practical significance of the research:

The results obtained in the dissertation were applied in the oil industry.

"Neftgaz-2016 NS" corrosion inhibitor was tested at the production well No. 241 exploited in deepwater jacket No. 5 of "28 May" OGPD. The results of the tests: the corrosion rate decreased from 0.545 gm2 / h to 0.048 gm2 / h, the metal loss decreased from 1.273 g to 0.1098 g and the protection effect was 91.01%.

The new composition developed for the prevention of inorganic salt deposits on oilfield equipment and tubing was tested in the service area of formation water utilization equipment of the H.Z. Tagiyev OGPD. The results were satisfactory.

It was also decided to publish in the Industrial Property Bulletin of the Intellectual Property Agency of the Republic of Azerbaijan information on the claim document No. 20200019 on the invention "Composition for the prevention of inorganic salt deposits formed during oil and gas production in wells".

Approbation and application of the work. The main provisions of the dissertation were discussed at the following International and Republican conferences:

- 4th International Turkic World Conference on Chemical Sciences and Technologies, 7-10 September 2018, Kiev, Ukraine, 2018;
- International scientific-practical conference "Modern methods of development of fields with labor-intensive reserves and non-traditional collectors", September 5-6, 2019, Republic of Kazakhstan, Atyrau;
- 5th International Turkic World Conference on Chemical Sciences and Technologies, 25-29 October 2019, Sakarya, Turkey.
- Materials of the IV International scientific-practical conference; Bulatovskie readings Collection of articles of 2020.

Publications. The main content of the dissertation is reflected in 10 scientific works, including 5 scientific articles, 4 conference proceedings and 1 application for an invention.

The structure and scope of the work. The dissertation consists of an introduction, 3 chapters, results and suggestions, 143 references, 4 appendices, 21 figures and 36 tables and 235,143 symbols.

SUMMARY OF THE WORK

The introduction substantiates the relevance of the dissertation, gives a summary of the purpose and main issues, and indicates the practical significance of the work.

The dissertation is devoted to the methods of removing asphaltene-resin-paraffin deposits and complications arising from inorganic salt deposition and corrosion on oilfield equipment and tubing.

The first chapter of the dissertation provides an overview of the methods against complications in oil production.

The first paragraph of the first chapter is devoted to a review of the research conducted against the complications caused by asphalt-paraffin deposits.

Asphalten-resin-paraffin (ARP) deposits in the well product cause changes in the viscosity of the well product (oil), deterioration of well performance, reduction of the inside diameters of the wellbore zone and pump-compressor pipes, and complications in well production.

Despite the fact that a lot of research work and effective methods have been proposed to eliminate the complications caused by asphaltene-resin-paraffin deposits, the problem remains actual.

There are various mechanical, chemical, thermal and physical methods to eliminate complications in pipelines and oilfield equipment during extraction of oil containing asphaltene-resin-paraffin compounds.

Special spherical separators are used when applying mechanical methods. These pigs can only be successfully used in supply pipelines. Pigging the deposits in variable diameter pipes and their bendings using spherical separators is not possible.

Thermal methods, including hot water, water vapour, hot gases, vapor-gas mixtures, and thermoelectric methods are widely used in paraffin-rich high-viscosity oil production. Despite a lot of works performed in this direction, the issue is still relevant.

In order to intensify asphalt resin paraffin deposits control by chemical reagents, the specialists of the Oil and Gas Scientific Research Institute have also carried out a number of new scientific and practical works.

Treatment with condensate and condensate-azolate reagent has been proposed as an effective method of chemical control of asphalt resin paraffin deposits. To this end, high efficiency was achieved by studying these reagents' effect on overhaul period of paraffin wells and the production of wells.

Chemical reagent Antipag has been developed to prevent the formation of asphalt, resin and paraffin deposits. Based on research, it has been found that the satisfactory results are achieved by the usage of the appropriate reagent when pigging lifting pipes.

İn the application of thermal methods, heating steam engines, percussion aggregates and tanks are required to dissolve the paraffin deposits accumulated on the inner surface of the tubing.

The application of these methods in asphalt-resin-paraffin sedimentary wells increases oil cost and creates some limitations. Although thermoelectric well treatment, one of the thermal methods, are widely used, electric heaters are used in production, and their operation is based on the principle of resistance and induction. These heaters are effective in wells with depth less than 600 meters. Paraffin oils can be heated up to 10-90 °C by the usage if this method.

However, research reveals that due to the different geological and technological conditions (flooding percentage, water circulation in annulus, formation thickness, etc.) in production wells, the temperature of the liquid increases much in one field and slightly in another. Research with high-viscosity oils reveals, the viscosity, shear stresses, elastic properties and electric conductance of the oils change during the thermoelectric treatment. Changes in the electrical conductivity and rheological properties of oil (the amount of asphaltene-resin-paraffin compounds) depend on the temperature fields' change rate and the frequency of heat treatment. Frequent failures of thermoelectric devices, cables, explosive fires and high power consumption restrict the application of the method.

The use of magnetic devices of various designs and voltages to control asphalt resin paraffin sediments in wells operated by the free flow -compression method is of practical interest. These facilities have been used with great success in the wells of Baku and Absheron archipelagos onshore and offshore providing the basis for improving technical and economic performance. Placing these devices at small and large depths, they prevented the formation of asphalt resin paraffin sediments by improving the working conditions of the wellbore zone. The effect of magnetic fields on oil transportation processes in pipelines has been studied. The hydraulic losses in oil flow through the magnetic field in the pipes are sharply reduced.

The solution of the problem was recognized as more expedient by regulating the structural, mechanical and rheological parameters of oils containing ARP, giving preference to combined methods in the ARP deposits' control measures and desingning new compositions based on cheaper chemicals produced in our Republic. Analyzes and classifications of the corrosion of oilfield equipment and the resulting complications is given in the second paragraph of the first chapter.

The failure of downhole equipment increases in the field in a short period of time. By the end of well operation, well production is flooded, and the amount of mechanical impurities increases. The effect of dissolved and free aggressive gases in the product on downhole equipment is obvious.. They have a positive effect on the corrosion of equipment and intensify its failure.

The main types of corrosion in the oil and gas industry are considered. The classification of corroded oilfield equipment has been carried out, the corrosion of casing, tubing, downhole pumps and rods, oil collecting and transporting equipment, equipment of the reservoir pressure maintenance system (RPM) has been studied.

The casing is exposed to internal and external corrosion, and corrosion of the outer wall of the pipe is more common.

This is due to the fact that the outer surface of the pipeline is in contact with layers of different structures, and the inner surface of the pipeline is in contact with oil, formation water and associated gases. Therefore, the nature of the corrosion and its rate, and, accordingly, the corrosion failure of the internal and external surface of the casing are different. In oil wells, the failure of the inner surface of the casing is particularly depend on the H_2S and CO_2 gases in the content of formation water.

The annulus between the internal part of the casing and the outer part of the tubing is filled with the well production from the formation, so the corrosion effect of this environment on the casing and the tubing is practically the same. However, the inner surface of the tubing is more corrodible.

When the aggressive components in the oil content is low, the corrosion of the tubing is very low, even in conditions of high flooding. The presence of mechanical impurities in the fluid flow, such as sand, causes corrosion and hydroerosion of the inner surface of the tubing. Acid retaining gas is used as a working agent in gaslift well operations. Therefore, the corrosion of downhole equipment of wells is significantly increasing. Tubing corrosion, hydrogen embrittlement and mechanical corrosion occur in the production of hydrogen sulfide-containing oil.

Most of the water extracted during the oil and gas fields development is used to maintain formation pressure. To maintain reservoir pressure, both natural (natural and less mineralized) and waste (drainage) water, containing reservoir (up to 85%), fresh (up to 10%) and rain (up to 5%) water is injected into the reservoir.

The nature of the corrosive aggression of the reservoir product has been established. The types of CO_2 -corrosion, acid corrosion, oxygen corrosion, galvanic corrosion, corrosion cracks, erosion corrosion, microbiological corrosion are investigated and briefly described. CO_2 is one of the most aggressive components in oil and gas production systems. Dry CO_2 itself is not corrosive in terms of the temperature at which oil and gas are produced. However, when dissolved in water, it causes an electrochemical reaction between the aqueous phase in contact with the steel.

Oxygen is a strong oxidizing agent and easily reacts with metals. Dissolved oxygen in the drilling fluid is one of the main causes of drill pipe corrosion.

Microorganisms can take a direct part in corrosion processes, using ions of structural materials of oil equipment for their vital activity. At the same time, many cases are known when the products of the vital activity of various microorganisms affect the materials of the equipment. Most microbiological corrosion leads to the development of local electrochemical corrosion.

Microorganisms can participate directly in the anodic process, forming wounds on metals, their metabolic products (for example, hydrogen sulfide), or the microorganisms themselves can participate in the cathodic depolarization process as a depolarizer. A fundamental analysis of the corrosion process was carried out.

The third paragraph of the first chapter is devoted to the precipitation and complications of calcium and magnesium carbonates, calcium sulfates, barium, chlorides and other salts in wells and oilfield equipment during the development and operation of oil fields.

The analysis and classification of complications caused by the deposition of salts in oilfield equipment has been carried out. The causes of deposition of calcium and magnesium carbonates, calcium sulfates, barium, chlorides and other salts in wells and oilfield equipment during the development and operation of oil fields are investigated.

If the amount of any sediment consists of 60-80% of inorganic salt compounds of the same composition for each deposit (for example, carbonates, sulfates, chlorides, etc.), then the deposit is characterized by this type of sediment. Deposition of inorganic salts occurs in all types of well operations; blowout, pump, gas lift. However, jack wells are more common, as salinity is 45% in rod pump wells and 35% in centrifugal electric submersible pumps (CESP). Since most of the oil fields in Azerbaijan are at the final stage of operation and are mainly operated by submersible pumps, the issue of scaling is relevant.

As a result of scaling, the inner diameter of the tubing narrows by 10-12 mm. That is why 50-60% of accidents occur at wells operated by the gas lift method. Deposits in the lower part of tubing are mainly composed of calcium and barium sulfate salts, while deposits at the wellhead and pressure lines are represented by calcium and magnesium carbonates.

Deposits of inorganic salts occur in wells, oilfield equipment, oil and water collection and treatment systems, as well as in the bottomhole zone of wells.

The most common deposits of inorganic salts are 60-80% calcium sulfate and 5-16% calcium carbonate and magnesium carbonate. Moisture and hydrocarbon compounds are 7-27%. Under certain conditions, each molecule of calcium sulfate ($CaSO_4$) combines two water molecules, resulting in the formation of a gypsum crystal. Thus, water itself is a source of salt formation. Since water is the best solvent for many substances, it retains a large amount of dissolved minerals. Since all natural waters are in contact with the environment, they retain dissolved components, which leads to the formation of complex solutions rich in ions, some of which are saturated with certain mineral phases.

The study examined the formation of salt deposits in pipes and oilfield equipment of oil wells and the work being carried out to combat it.

Methods for the chemical analysis of produced water are shown - methods for determining the total hardness, calcium, magnesium, carbonate and hydrocarbon ions, chloride ion by the sea method, the concentration of sulfate ion and dry residue by the gravimetric method.

The second chapter is devoted to the development of new methods to deal with complications in oil production.

The first paragraph of the second chapter is devoted to the development of a new reagent against the complications caused by asphaltene-resin-paraffin deposits. Numerous laboratory experiments have been carried out to create a new reagent for eliminating the consequences of asphaltene-resin-paraffin deposits.

When developing new methods to prevent paraffin deposition in wells, the reasons for the deposition of asphaltene-resin-paraffin compounds and hydrates on the inner surface of wells and mining equipment, as well as methods to combat them, were studied. The closest to the developed method is a 0.03-0.5% solution of the

Dissolvan 4411 demulsifier, which is highly soluble in water, and 0.1-1.5% solution of the dispersant XT-48, used in the precipitation of paraffins, an aqueous solution of ammonium, which is part of the composition. Although these compounds are highly soluble in AQP deposits in wells, they do not provide long-term protection of equipment and production wellheads, and the consumption of reagents is high. The goal is to develop a new, relatively inexpensive composition based on local raw materials, which makes it possible to reduce the consumption of inhibitors used in paraffin deposition, providing long-term protection of equipment and wells in operating wells from AQP deposits.

A new composition has been developed - the reagent "OKI-18" based on the demulsifier Alkan DE-202, isopropyl alcohol and alkaline waste from diesel fuel processing. Along with oil wells, this reagent cleans the bottomhole zone from ACP deposits and does not have toxic chemical components.

The effect of the new OKI-18 reagent on the deposition of paraffin on the basis of chemical products produced in the republic was investigated in the laboratory by the "cold fingers" method.

Initially, paraffin-containing oils are heated in a water bath to 60°C and paraffin particles are completely dissolved in the oil. Then the heated oils are poured into three liter nozzle refractory and placed on magnetic stirrers. Add OKI-18 reagent of various concentrations to two nozzles. No reagent is added to one of the cups; this cup performs a test function.

After the temperatures have equalized (t = 60°C), “brass fingers” are inserted into the cups. The liquid placed in the thermostat circulates and its temperature is maintained at -50°C.

At the same time, due to a decrease in temperature in the “brass fingers”, asphalt-resin-paraffin begins to settle on them. The oil is compressed with a “brass finger” for 20 minutes at the required temperature. At the end of the study, the asphalt-resin-paraffin deposits settled on the “brass fingers” are heated with hot water from the 2nd thermostat by turning the tap, poured into an empty glass of known weight and weighed on an analytical balance.

The oil extraction department named after N.Narimanov is the oil extraction department with the highest concentration of asphaltene-resin-paraffin sediments in the well products among the oil fields of Azerbaijan.

Laboratory researches were carried out on 4 samples taken from different wells of OGPD named after N.Narimanov. Prior to the start of the research, the physical and chemical properties of these oil samples and the solid compounds deposited on the inner surfaces of the pump-compressor pipes removed during the underground repair of the sampled wells were analyzed.

First of all, many experiments were conducted to optimize the amount of components in the reagent. During the experiments, the effect of paraffin oils on the freezing temperature was studied by preparing a reagent in the ratio of 1-3% of demulsifier DE-202, 2-5% of isopropyl alcohol and 92-97% of alkaline waste.

The following ratio of components of the newly developed reagent Alkan DE-202 demulsifier, isopropyl alcohol and alkaline waste from diesel fuel treatment was taken as the most effective ingredient:

DE-202 demulsifier	with 2-3% volume
Isopropyl alcohol	with 2-4% volume
Alkaline waste	the rest

"OKI-18" reagent also has a depressant function due to its low freezing point. This ingredient is yellow and has a freezing point of minus 25 °C.

A number of experiments were also performed to determine the optimal consumption rate of the proposed new reagent. These experiments were also carried out on 2 samples (No. 1 and 3) taken from the wells of the OGPU named after N.Narimanov. First, the freezing point of paraffin oil was determined, and then the freezing point was determined by adding "OKI-18" reagent to the oil, starting from 50 mg / l to 1000 mg / l.

As the amount of depressant increases, the freezing point of the oil decreases, and this value reaches such a level that the freezing point price does not change despite the increase in the amount of depressant. In the absence of OKI-18 reagent, paraffin-rich oil freezes at positive 23-25 ° C, while in case of OKI-18 reagent in the amount of 400-500 mg / l, the oil freezes at minus 5 °C. This value of the reagent is considered to be the optimal amount in the fight against ARP sediments.

High paraffin oils are non-homogeneous, unbalanced rheological compounds that undergo structural changes as the temperature drops.

In order to improve the extraction and transport properties of high-paraffin oils, polymer additives are widely used in oil, which prevent the formation of a paraffin space crystal lattice and, consequently, reduce the freezing point.

It is sometimes impossible to determine which of the additives is better because some reduce the freezing point, some reduce the static shear stress, and some lower the dynamic shear stress and dynamic viscosity. To determine this, the problem of selecting a reliable parameter arises, and the presence of a large amount of data at different temperatures makes it even more difficult to select this parameter.

The rheological parameters of the oils that make up the structure are usually studied with a rotary viscometer, which determines the appropriate shear stress at certain temperatures and shear rates. The graph of the dependence of the shear stress on the shear velocities gives the flow curves characterizing the rheological parameters of the fluid. It should be noted that in high paraffin oils, the flow curves obtained by successively increasing and decreasing the sliding speed at low temperatures do not overlap, forming a hysteresis loop.

Gradually increasing the sliding speed destroys the structure formed by paraffin crystals and balances the system. When the sliding speed is reduced backwards in a completely collapsed structure, balanced flow curves characterizing the movement of the fluid in the pipelines are obtained. At higher values of sliding speed, these curves are closer to each other. When the temperature is raised and an “OKI-18” reagent is added to the system, which acts as a depressant, no hysteresis process is observed, the system imbalance is reduced, and the oil approaches Newtonian properties.

The rheological properties of paraffin oil with the addition of a new reagent were studied. The rheological parameters of the structural oils were studied in a rotary viscometer.

The graph of the non-equilibrium coefficient of non-equilibrium of crude oil applied to high paraffin oil and oil with the addition of “OKI-18” depressant is given in Figure 1.

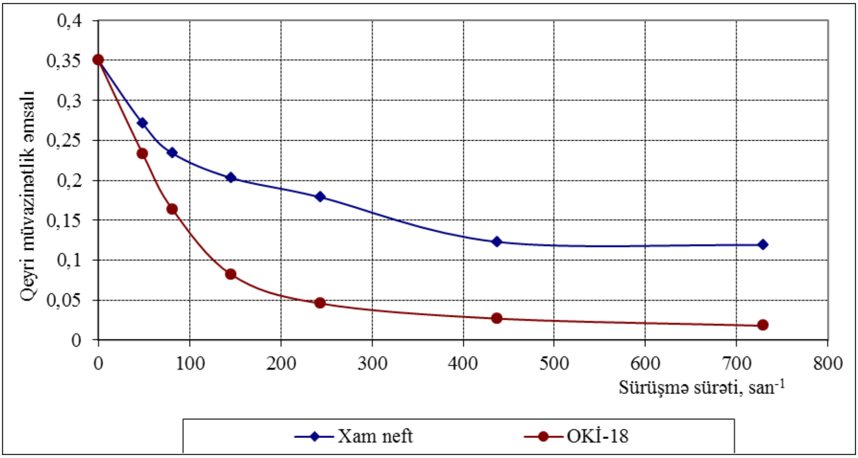

Figure 1. Graphs of the relationship between the non-equilibrium coefficients of crude oil and “OKI-18” depressant added oil and the sliding velocities (example 1)

Figure 1 shows that as the sliding speed changes, the value of the non-equilibrium coefficient decreases, i.e. the structure begins to collapse and the oil tends to become balanced. When the depressant is added to high-paraffin oils, it creates local crystalline centers that are poorly connected to each other, preventing the formation of crystalline structures of paraffin. The depressant reduces the degree of imbalance by lowering the system's unbalance coefficient.

The optimal consumption rate of the proposed new reagent was determined.

The optimal concentration of “OKI-18” reagent was selected by calculating the non-equilibrium coefficient by giving 300, 500 and 600 mg of “OKI-18” reagent per ton of oil. Based on the research, the optimal concentration of OKI-18 reagent is 500 mg / l, and 600 mg / l again reduces the effect of the depressant. The results have been confirmed by both laboratory studies and reports.

The second paragraph of the second chapter presents the results of the investigation of the causes of corrosion in the production wells and surface equipment of the "Guneshli" and " Neft Dashlari " fields and the development of new methods against it in order to protect oilfield equipment from corrosion.

In order to intensify oil production in the oil industry in Azerbaijan, the water of the Caspian Sea, rich in various groups of bacteria, sulfate ions and organic substances, is injected into the productive strata. This sharply raises the problem of microbiological corrosion of oilfield equipment.

For this reason, it is important to study the amount of various microorganisms in the products of irrigated production wells that affect the corrosion process of oilfield equipment.

Initially, microbiological pollution of the mining environment was studied, sulfate-reducing bacteria were identified, and a methodology was selected for the determination of sulfate-reducing bacteria. The method is based on laboratory processing of the reagent to prevent biological corrosion, determination of bactericidal properties, selection of bactericidal inhibitors, and determination of microbiological contamination in the water from the wells to be applied, cultivation of microbes in appropriate nutrient media by extreme dilution. The bactericidal properties of plankton bacteria were determined in the laboratory. Product samples were taken from production wells operating in the Gunashli and Neft Dashlari fields and their chemical and microbiological research was carried out, and the rate of corrosion wear of oil drilling equipment was studied in that environment. Corrosion aggression was determined by analyzing the corrosion aggressiveness of produced waters, sulfate-reducing bacteria, hydrogen sulfide content, chloride and sulfate ion content.

Gas samples were taken from the gas lift lines of wells No 111, 117, 66, 292, 245, 3, 43, 160, 172, 177, 218, 314, 259, 305 and 283, operated in Deep Water Jacket No. 3, 4, 5, 9, 10, 11, 13, 14 and 15 of "28 May" OGPD , and the amount of corrosive components was determined by chromatographic analysis in the laboratory.The content of carbon dioxide in the gas samples is 0.8-3.0%, the amount of oxygen is 0.05-1.1%.

The amount of hydrogen sulfide (H_2S) and mercaptans in the gas samples taken by iodometric method was determined. Hidrogen-sulfidin miqdarının 0.0051-0.0051 q/m^3, merkaptanların miqdarının isə 0.0064-0.0096 q/m^3 olması müəyyənləşdirilmişdir. The amount of hydrogen sulfide is determined within 0.0051-0.0051 g/m^3, and the amount of mercaptans is within 0.0064-0.0096 g/m^3.

It is shown that increase in the amount of H_2S and CO_2 in the downhole environment and increase in the partial pressure in the string and flow rate, respectively, lead to the intensification of the corrosion process.

Chemical and microbiological analysis of water were conducted according to standard methods to determine the aggressiveness of produced water taken from wells 111, 117, 66, 292, 245, 3, 43, 160, 172, 177, 218, 314, 259, 305 and 283 operated in Deep Water Jacket No. 3, 4, 5, 9, 10, 11, 13, 14 and 15.

It was determined that the majority of produced water samples are sodium hydrocarbonate. These water density varies from 1,009-1,024 g / cm^3. the total mineralisation varies in the range of 14.57-36.82 g / l. In these samples, the price pH of the environment is 8.0-8.8. The amount of chloride ions (CL^-) is 6026.50-19143.00 mg / l, sulfate ions ($SO_4^{2-)}$ - 18.11-493.80 mg / l, carbonated ions- 45,00-750,00mq/l, The amount of hydrocarbonate ions (HCO_3^-) - is 854.00-622.00 mg / l. The amount of calcium ions (Ca^{2+}) is 20,04-40.08 mg / l, magnesium ions (Mg^{2+}) - 12.16-608.00 mg / l. The amount of iron (Fe^{3+}) ions is within 64.26-1075.84 mg / l.

The amount of sulfate reducting bacteria (SRB)that are corrosion bacteria,is respectively 10^1 – 10^4 cells/ml, iron bacteria (FeB)- 10^5-10^7 cells/ml, carbohydrogen oxidative bacteria (KOB) - 110^4-10^7 cells/ml during water microbiological analysis.The amount of hydrogen sulfide of biogenic origin is in the range of 11.7-25.56 mg / l depanding of the well, in 3 days of trial periods, corrosion velocity is changed respectively 0.118 q/m^2·saat - 0.275 g / m^2·saat (Table 1).

The results of produced water content study provide extensive information about the intensity of electrochemical and biological corrosion processes in oil mining equipment. Although a large number of corrosion inhibitors are currently developed, hydrogen sulfide and microbiological environments are limited to the number of universal bactericide-inhibitors, which ensures steel corrosion. “Oil-gas 2016 NS” corrosion inhibitor has been developed on basis of acids and polyamines to prevent possible corrosion processes.

Table 1

Results of hydrogen-sulfide (H_2S) and corrosion microorganism analysis in the produced water samples taken from the Gunashli field

DWJ	Well	H_2S,mq/l	Corrosion velocity,q/m^2·hour	SRB, cells/ml	FeB, cells/ml	KOB, cells/ml
DWJ -3	111	12.3	0.177	10^2	10^6	10^7
	117	15.7	0.141	10^3	10^7	10^6
DWJ -4	66	12.64	0.141	10^2	10^6	10^7
	292	11.7	0.200	10^3	10^7	10^7
DWJ -5	241	15.7	0.149	10^2	10^6	10^6
DDÖ-9	3	17.04	0.242	10^2	10^6	10^7
DWJ -10	43	25.56	0.144	10^4	10^7	10^7
	160	17.04	0.118	10^3	10^6	10^7
DWJ -11	172	0.0	0.275	10^1	10^5	10^4
	177	0.0	0.200	10^1	10^4	10^4
DWJ -13	218	15.336	0.21	10^2	10^5	10^7
	314	17.04	0.121	10^2	10^6	10^7
DWJ -14	259	12.64	0.149	10^2	10^5	10^7
	305	11.7	0.18	10^2	10^5	10^6

Fatty acids were obtained from SOAPSTOK, as a result of vegetable oils with of alkaline treatment from Azersun Holding's Baku Oil Refinery. In order to separate free fatty acids from SOAPSTOK, it was distilled by processing with mineral acids and alkalis (for saponification of neutral oils).The chemical reaction of condensation of aminoethylethanolamine (AEEA) with fatty acids was selected for the synthesis of the inhibitor. Based on the experimental results of the reaction under different conditions and at different molar ratios of reagents, more optimal parameters of condensation were selected. Under intensive mixing conditions and at a temperature of 140° C, the mixture is stirred for 2 hours at a ratio of AEEA: YT = 1.0: 2.0 mol. Carboxamide obtained at 240° C is converted to imidazoline by the separation of water. It should be noted that imidazoline is used in water-soluble form as an inhibitor. Therefore, salts of imidazoline in a 1: 1 ratio with acetic acid were obtained.

The composition of the obtained compound in isopropyl alcohol was prepared and its physical and chemical parameters were determined in accordance with the relevant standards. The composition consisted of 10-25% imidazoline acetate, 20-50% isopropyl alcohol and the rest was water.

Initially, the protective effect of the inhibitor composition with optimal content against hydrogen sulfide and carbon dioxide corrosion was studied in the model water in the laboratory.

In the range of 20-120 mg / l model water concentration, protective effect at a concentration of 120 mg / l of synthesized inhibitor against hydrogen-sulfide corrosion was 90-98%, and the protective effect against CO_2 corrosion at a concentration of 200 mg / l was 96-97%.

The effect of the determined concentration of "Oilgas-2016NS" corrosion inhibitor on the number of bacterial cells and the degree of reduction of their vital activity was also determined. Imidazolines and amidoamines, which are part of the inhibitor composition, clearly inhibit the enzymatic reactions responsible for the reduction of sulfates.

As a result of conducted investigation to clarify the causes of corrosion of oilfield equipment operated in flooded production wells of "Oil Rocks" field it was found that the main factor is related to the life of corrosive microorganisms.

Based on the results of microbiological analysis of various well samples taken from "Oil Rocks" field, SRB-10^3-10^6 cells/ ml, iron bacteria -10^5 – 10^7 cells / ml and KOB 10^6-10^8cells / ml were identified (Table 2).

From this point of view, an inhibitor-bactericide composition with high biocidal properties at low consumption rates has been developed for corrosion protection of oilfield equipment operated in flooded production wells in "Oil Rocks" field.

Table 2

Results of microbiological analysis of produced water taken from the flooded production wells of OGPD No. 1 of "Oil Rocks" OGPD

Well	Flooding %	The amount of microorganisms, cells/ml			Corrosion velocity, mm/y
		SRB	FeB	KOB	
2210	66	10^5	10^7	10^6	0.017
1729	57	10^7	10^7	10^6	0.7535
819	59	10^8	10^8	10^7	1.2985
795	90	10^4	10^6	10^7	1.3069
2180	50	10^4	10^7	10^7	1.3756
2416	50	10^6	10^7	10^7	1.4337
2570	60	10^5	10^7	10^7	1.4594
2167	68	10^4	10^6	10^5	1.5778
2114	67	10^4	10^6	10^7	1.6451
2571	68	10^6	10^7	10^6	1.7365
2425	64	10^7	10^7	10^6	1.8578
966	64	10^4	10^7	10^7	1.9222
2346	56	10^5	10^7	10^7	2.1094
2568	50	10^6	10^7	10^7	2.4749
2350	56	10^5	10^6	10^8	2.5190
2065	68	10^6	10^6	10^7	2.6342

A new inhibitor-bactericidal composition "NEFTGAZ-2016 NS1" has been obtained, which is polyamides synthesized on the basis of fatty acids and polyamines with the addition of glutar aldehyde and quaternary ammonium salts containing 1-10% by weight of polyamide, 5-10% by weight of glutaric aldehyde, 5-10% by weight of non-alkonium chloride, 20-30% by weight of methanol and the rest from water. Synergism is observed during the modification of corrosion inhibitors based on nitrogen compounds with these compounds. In this case, their biocidal properties are observed at higher concentrations than individual effectiveness. The observed synergism allows to reduce the amount of materials to be used to achieve the required high biocidal properties, thus achieving high efficiency in terms of environmental impact and material costs.

Laboratory tests were conducted to assess the biocidal and corrosion protection properties of the oil-gas-2016 NS1 inhibitor-bactericide. The tests were carried out on samples of produced water brought from irrigated wells 2568, 2350, 2065 operated by Neft Dashlari OGPD.

Studies of the biocidal properties of the developed bactericidal inhibitor show that the inhibitor PostgateB is effective in reducing the number of SRBs in the nutrient medium (Figure 2).

If the concentration of the inhibitor at a concentration of 50 mg / l is reduced by 2 times after 7 days in a closed system, the protective effect of the inhibitor at a concentration of 100 mg / l reaches 90%, and after 200 mg / l exceeds this value.

Laboratory tests have shown that the inhibitor Neftgaz-2016 NS1 at a concentration of 100 mg / l allows 99-100% destruction of KOB, FeB and SRB (Figure 3).

The third paragraph of the second chapter deals with the development of a new composition to prevent inorganic salt deposits formed during the extraction of oil and gas from wells.

Layer waters were studied and the concentration of roughness, calcium, magnesium, carbonate and hydrocarbon ions and hydrochloric acid in the formation water was determined by the mor method and the concentration of sulfate ions.

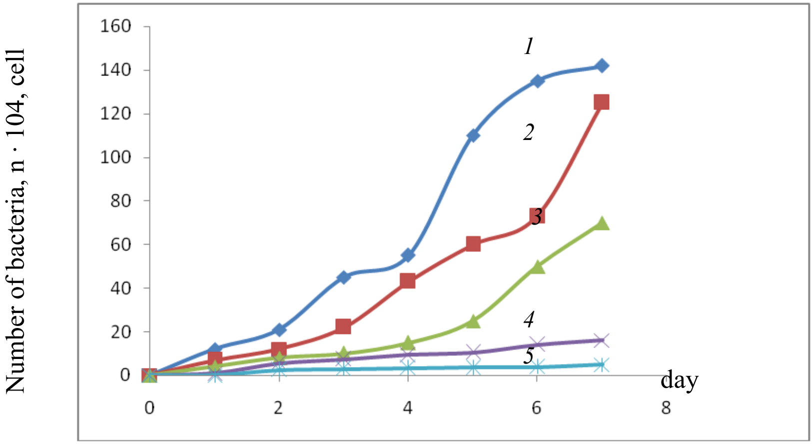

Figure 2. Variation of SRB number depending on inhibitor-bactericidal concentration (mg / liter): 1 - 0; 2 - 25; 3 - 50; 4 - 100; 5 – 200.

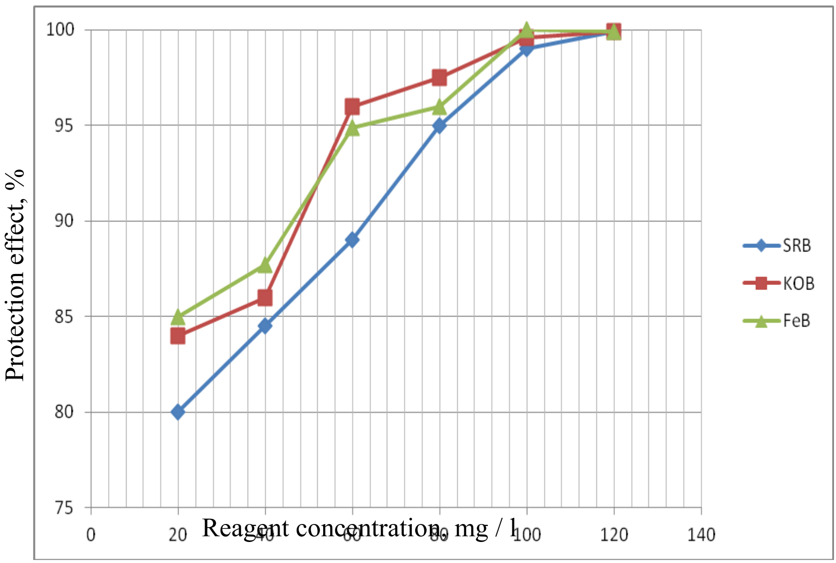

Figure 3. Protective effect of bactericidal inhibitor "Neftgaz-2016NS1" against bacteria

It is known to contain nitrilotrimethylenephosphon (NTF) acid, hydrochloric acid and water to prevent salt deposits. However, this composition is mainly used in the processing of productive strata with carbonate rocks.

NTF, hydrochloric acid, silicon hydrogen fluoride acid and water are used to prevent inorganic salt deposits during oil and gas extraction from productive stratified carbonate and terrigenous wells. These compounds are prepared using phosphorus, nitrogen-fixing complexes and silicon hydrogen fluoric acid.

The disadvantage of these compositions is the relatively high efficiency of difficult-to-find and expensive reagents.

For this purpose, a composition has been developed to prevent inorganic salt deposits in oil and gas production equipment and pump-compressor pipes during oil and gas production.

By maintaining hydrochloric acid and water to prevent inorganic salts formed during oil and gas extraction from carbonate and terrigenous wells to achieve technical results during the preparation of the new composition, additionally, the components of the dispersant EC 9660A and laprol 4202-2B-30 are stored in the following mass ratios, % by mass:

Hydrochloric acid	25-30
Dispersant EC 9660A	18-22
Laprol 4202-2B-30	8-12
Water	the rest

21% inhibited hydrochloric acid was used as hydrochloric acid, EC 9660A 35% as a dispersant, and laprol 4202-2B-30 as polyester.

The protective effect tests of the developed inhibitors were carried out in the formation water model to prevent calcium-sulfate precipitation. As a result of tests conducted in the produced water model, it was determined that the protective effect of the inhibitor varies from 90.4% to 100%.

The methods developed in the third chapter deals with mining testing.

The first item of the third chapter shows the results of the application of anti-corrosion inhibitors-bactericides. With the consent of Azneft PU, oil extraction well No. 241, operated on the deep water jacet No. 5 of the 28 May OGPD, was selected for the test. Information on the operating condition and technological parameters of the equipment of the selected well for the last year was obtained and analyzed on the basis of mining documents. The effect of Oilgas-2016 NS corrosion inhibitor was determined by complete chemical and microbiological analysis of produced water taken before and after injection of the reagent into the system and by determining the corrosion rate in steel "witness" samples placed on the well discharge line. During 30 (thirty) days before the reagent was injected into the well, the metal loss was 1,273 grams and the average corrosion rate was 0.545 gm^2 / hour.

The amount of corrosion inhibitor is calculated according to the volume of the well product and is injected into the well continuously for 30 days. During the first 5 days, the inhibitor was injected continuously into the well for 3 days at a shock dose of 3 g / l per 1 liter of produced water.

After that, for 25 days, the consumption of the inhibitor was adjusted to 300 mg / l per day, and the injection was continued continuously by dosing pump. The effect of the bactericidal inhibitor Neftgaz-2016 NS was determined by complete chemical and microbiological analysis of produced water taken before and after the injection of the reagent into the system and by determining the corrosion rate in steel "witness" samples placed on the well discharge line. The results of the inhibitor's mining tests are given in Table 3 below.

The results of the tests showed that the corrosion rate decreased from 0.545 gm^2 / h, 0.048 gm^2 / h, the metal loss decreased from 1.273 g to 0.1098 g and the protection effect was 91.01%.

The second item of the third chapter shows the results of mining tests of the anti-salinity composition developed at the OGPU named after H.Z. Tagiyev

Mining tests were carried out in the service area of produced water utilization of OGPD named after H.Z. Tagiyev.

Table 3

Results of mining tests of corrosion inhibitor Neftgaz-2016NS

Examples number	Corrosion rate, qm2 / h	Delay factor, times	Metal loss, grams	Protection effect,%
1	0.0470	11.5957	0.1097	91.39
2	0.05	10.9	0.1102	90.82
3	0.0473	11.5221	0.1098	91.32
4	0.0475	11.4736	0.1110	91.28
5	0.05	10.9	0.1087	90.82
6	0.05	10.9	0.1095	90.82
Average indicator	**0.048**	**11.21**	**0.1098**	**91.07**

Utilization of produced waters in OGPU named after H.Z has shown that the increase in the amount of ions as a result of the application of a new compound developed against inorganic salt deposits in order to eliminate salt deposits in the equipment has shown that salt deposits are prevented under the influence of the processed composition.

CONCLUSIONS AND RECOMMENDATIONS

1. With a view to avoiding complications caused by paraffinic oil the OKI-18 chemical acting as a depressant has been produced. The rheology of paraffinic crude was studied using the proposed OKI-18 chemical which helped to demonstrate that the chemical converts paraffinic oil into Newtonian liquids, reducing their viscosity by 48-50%. The effect of the OKI-18 chemical on the pour point of paraffinic oil was tested through an experiment allowing to record its decline by 15-20°C. Driven by laboratory studies, the effectiveness of the OKI-18 to combat paraffin is 100% whereas the optimal consumption rate is 500 mg/l.

2. The amount of corrosion active aggressive components and corrosion-active microorganisms in the formation water taken from the production wells of “the Oil Rocks” and “28 May” OGPD was analyzed, and the amount of hydrogen chloride, sulfate bicarbonate and iron ions was established at a level that can lead to corrosion.

3. To protect well equipment against corrosion, a universal anti-corrosion inhibitor "Neftegaz-2016NS" was generated using polyamides which are condensation product of fatty acid fractions with polyamines and acetic acid of imidazolines with salts in isopropyl alcohol and aqueous solution in the course of which the consumption rate of the inhibitor was determined.

4. A new inhibitor-bactericidal composition "NEFTGAZ-2016 NS1" was produced based on polyamides, glutaraldehyde and quaternary ammonium salts synthesized on the basis of fatty acids and polyamines.

5. The protective action of the generated inhibitor composition "Neftegaz-2016NS" at a concentration of 100 mg/l against H2S and CO2 caused corrosion was 98-99% and the protective action of the inhibitor-bactericidal composition "NEFTGAZ-2016 NS1" was 99-100% against corrosion active bacteria.

6. A composition was worked out to prevent deposits of inorganic salts during oil and gas production, and the protective action of the composition was tested on a model of formation water,

and the protective action of the inhibitor fluctuated between 90.4% and 100%.

7. The corrosion inhibitor composition "Neftgaz-2016NS" was tested in the production wells at the Gunashli field of “28 May” OGPD of "Azneft" PU and based on the outcomes, the inhibitor is recommended for use in water-cut production wells to prevent corrosion.

8. In the disposal site of formation water of the OGPD under H.Z. Tagiyev, tests of the produced descaling composition were carried out. The increase in the number of ions in all cases as a result of injection of the new composition developed against inorganic salt deposits with an aim to avoid salt deposits on the equipment in the disposal site of formation water proved effective to prevent salt deposits due to the injected composition.

The main concept and conclusions of the thesis have been published in the following works.

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Candidate's Personal Contribution

Works [1, 7, 9] were carried out independently, support was provided for works [2, 3, 4, 5, 6, 8, 10] to enable formulation of the problem, to run studies and summarize the conclusions.

The defense will be held on 31 May, 2022 at 11:00 at the meeting of the Dissertation council ED 2.03 of Supreme Attestation Commission under the President of the Republic of Azerbaijan operating at Azerbaijan State Oil and Industry University.

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