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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

ASPHALTENE-RESIN-PARAFFIN FOR INCREASING THE EFFICIENCY OF OIL FIELD DEVELOPMENT NEW COMPOSITIONS FOR ANTI-DEPOSIT DEVELOPMENT AND APPLICATION

Specialty: 2525.01 – “Oil and gas field development and operation”

Field of study: Technical sciences

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GENERAL DESCRIPTION OF WORK

The topicality and development degree of the subject.

The efficiency of exploitation of oil fields depends greatly on the condition of the bottom zone of production wells. During the development of the field, the permeability of the bottom zone decreases and the seepage indicators of the productive layers change.

As a result of the physico-chemical and thermodynamic changes occurring in the well bottom zone, the temperature and pressure drops created during the filtration of the multiphase system, the precipitation of the heavy components of the oil and the increase in the degree of water saturation of the rocks are observed. As a result of these processes, the fluid flow in the well weakens and the production rate decreases. In this regard, development and application of new multi-functional reagents in order to increase oil production in wells and restore permeability of the bottom zone is one of the urgent issues.

At the same time, the accumulation of asphaltene-tar-paraffin (ATP) compounds in the lift pipes of wells, discharge lines and other oil field equipment during oil extraction and transportation processes constantly creates technological problems, and prevention of ATP sediments or its cleaning is required.

In this regard, the effect of various surface-active substances and composite reagents created on their basis on interphase surface tension, wetting ability, adsorption of SAS in a porous medium, etc. research, development and testing of the technology of use of the composition are of great national economic importance.

Also, technological complications arise due to the formation of asphaltene-resin-paraffin (ATP), salt deposits and hydrate compounds in oilfield equipment during the transportation of oil and gas in our fields that have been in operation for a long time. In this regard, the prevention or treatment of ATP deposits is constantly required. Due to the changes in the composition of well products and technological parameters during operation, it is important to develop effective inhibitors against ATP sediments and their use technologies.

Thus, the development, research and application of new technologies against ATP and salt deposits during field development is characterized by its relevance and national economic importance.

The purpose of the study.

Against asphaltene-resin-paraffin and salt deposits formed on the surface of the equipment, it is the development of new, including nanocomposites, and the creation of more efficient technologies for increasing the efficiency of processing in oil fields, recovery of production and protection of operational equipment based on them.

Tasks of research.

- development and complex research of surface-active reagents in the fight against asphaltene-resin-paraffin and salt deposits;
- researching the mechanism of action of nanocompositions against asphaltene-resin-paraffin deposits based on analytical studies to increase the efficiency of processing of high-viscosity oil fields;
- development and research of a new ingredient inhibitor for the purpose of protecting equipment from salt deposits.

Research methods.

The issues were solved by analyzing the results obtained from laboratory studies and test works conducted in mining conditions.

Main clauses defended.

- development and research of new SAS reagents against asphaltene-tar-paraffin deposits;
- development and research of nanocompositions against complications;
- development of an inhibitor against salt precipitation and study of the mechanism of action.

Scientific innovations of research.

- new SAS reagents against asphaltene-tar-paraffin deposits were developed and studied.
- new nanocompositions have been developed based on nanotechnologies to regulate the rheotechnological properties of high-viscosity oils.
- an inhibitor of protection against salt precipitation was developed and its effectiveness was studied.

Theoretical and practical significance of research.

Based on the analysis of paraffin and salt deposition problems in the oil and gas fields of our republic, the possibilities of using new SAS reagents and nanocompositions were given.

The Patent of the Intellectual Property Agency of the Republic of Azerbaijan dated 12.05.2022 No. a 2022 0083 was obtained for the invention document "Paraffin Precipitation Inhibitor"/

As a result of the application of the developed technologies in wells No. 682, 683 and 607 of N.Narimanov Oil and Gas Research Center, an additional 51 tons of oil production was achieved.

Approval and application.

Dissertation materials were presented and discussed:

- at the VI International petrochemical conference dedicated to the 100th anniversary of Academician Y. H. Mammadaliyev, Baku NKPI, 2005;

- at the VIII Baku International Conference dedicated to the memory of Academician Y. H. Mammadaliyev, Baku NKPI, 2012;

- at the "Khazarneftgazyatag - 2016" conference, Baku ASSU, 2016;

- at the International conference dedicated to the 90th anniversary of Academician Azad Khalil oghlu Mirzajanzadeh, Baku, December 13-14, 2018;

- "Innervation technologies in the oil and gas field. Problems of sustainable development of territories" at the III International scientific-practical conference. Stavropol, December 2022.

Research publication rate. 15 scientific works have been published on the subject of the dissertation work. 9 of them are articles, 5 are thesis and conference materials, and 1 is a patent of the Republic of Azerbaijan. Four of the articles were published by a single author in periodical scientific publications included in international summarizing and indexing systems.

The name of the institution where the dissertation work was performed. Dissertation work was performed at SOCAR, "Oil and Gas Research Project" Institute.

The structure and scope of the dissertation, indicating the scope of the structural sections separately. The total volume of the dissertation work is 184738 marks, consisting of an introduction, three chapters, a conclusion, a list of used literature and appendices (introduction 5500 marks, chapter I - 68205, chapter II - 50924, chapter III - 58953 marks, conclusion 1156 marks). The dissertation includes 168 literature sources, 26 pictures, 32 tables, appendices.

THE CONTENT OF THE WORK

Brief information on the main propositions defended, scientific novelty, theoretical and practical significance of the research, approval, application, structure and scope of the work is provided.

The production of paraffin oils is observed with the occurrence of a number of complications. Formation of asphaltene-resin-paraffin (ATP) deposits in oilfield equipment and main pipelines in the well-bottom zone leads to complications such as reducing the productivity of pumping units. The presence of formation water in the oilaccelerates the formation of ATP sediments.

First of all, the causes of complications caused by ATP sediments in oil extraction were analyzed, and modern methods of their elimination were considered. Measures to combat ATP sediments are carried out in two directions. The first direction is related to reducing the possibility of sediment formation. In other words, prevention of sedimentation is ensured as a result of preventive measures. Technologies related to smooth protective coatings, methods of chemical action through chemical regents such as depressors, dispersants, and methods of physical action with vibration, ultrasound, electric and electromagnetic fields are widespread. The second direction of struggle includes a complex of technological measures aimed at eliminating ATP sediments. Thus, the washing of the well bottom zone with hot oil and water, the application of steam, and the use of various exothermic reactions are obvious examples of this. Mechanical cleaning of arsenics, removal and cleaning with chemical reagents are still used. Experience shows that the more favorable of these two methods are those aimed at preventing the formation of ATP deposits, rich in preventive effects. Minimizing the possibility of formation of ATP sediments, reducing the risk of formation of these sediments has a special role for ensuring the normal operation of oil field facilities as a whole. A complex approach to the development of the method of combating ATP deposits should be taken into account, the characteristics of the field, the properties and composition of the produced oil. At present, the wide application of chemical methods in the fight against ATP sediments is of particular interest. Most studies have shown that the use of chemical reagents in solving existing problems creates the basis for maintaining the effectiveness for a long time. The main principle of using chemical methods is that as a result of the introduction of the reagent processed from the produced oil in doses, the possibility of ATP deposits forming in the pipelines is drastically reduced. The use of chemical reagents with inhibitory properties is very important for solving the current problem in order to reduce the possibility of ATP deposits. It should be noted that the selection of optimal methods of combating ATP sediments depends on many factors. Thus, the method of oil extraction, thermobaric characteristics of flow regimes, the composition and properties of the produced hydrocarbon product have an important role in this process. ATP sediments are mainly formed in wells with a low degree of dilution (32% of the total studied wells). Wells with 50-90% dilution rate are in the second place according to the frequency of formation of ATP sediments. The characteristic difference of ATP sediments in these wells is that the sediments accumulate both in the pumping equipment and in the pump-compressor pipes. ATP sediments formed in different fields differ from each other in terms of their chemical composition, and this difference depends on the hydrocarbon content of the oil produced in the corresponding wells. Despite the diversity of the composition of ATP sediments, it was determined that the amount of asphaltene-resin and paraffin components in the sediments varies inversely. As the amount of asphaltene-resin compounds in ACP sediments is high, the amount of paraffin decreases, and this ratio is determined by their amount in oil. Depending on the amount of asphaltene-resin and paraffin compounds in the sediments, ATP sediments belong to asphaltene, mixed and paraffin group ATPS. This property of ATP deposits is based on the nature of interaction of paraffin, tar and asphaltenes in oil before the formation of deposits. Studies have shown that paraffin crystals form a complete network similar to a wide ribbon before settling on the surface of the well equipment. In this form, the adhesion property of paraffin increases many times, its accumulation on solid surfaces is significantly intensified. However, when the amount of asphaltenes in oil is high, their depressant properties show their effect. Asphaltenes participate in the crystallization process of paraffin as crystallization centers, and as a result, paraffin crystals are distributed in numerous centers, the number of sediments on the surface of the equipment is reduced because the ribbon network structure is not formed. Despite the development of many methods in the fight against ATP sediments, the solution to the problem still remains and highlights the need to develop new methods and tools in the field of oil extraction, which is the most difficult area of oil industry.

The first chapter is devoted to the analysis of the current state of development of paraffin oil deposits and the justification of research. First, the reasons for the complications caused by ATP sediments in oil extraction were analyzed, and modern methods of their elimination were considered. At the same time, the current state of the development of hydrophobic compositions was analyzed to increase the permeability of WBZ.

In production, special importance is attached to measures to combat paraffin deposits. The main idea is that the paraffin precipitates should not have the opportunity to crystallize, thereby minimizing their complication.

Methods such as high-pressure systems, depressor additives, special coating technology, and heating systems are currently widely used. In addition to the methods listed above, limiting the formation of paraffin crystals using a magnetic field has also been widely applied in industry. There are also methods of controlling paraffin with SAS reagents. However, increasing the variety of these reagents remains relevant.

It should be noted that the selection of optimal methods of combating ATP sediments depends on many factors. Thus, the method of oil extraction, thermo baric characteristics of flow regimes, the composition and properties of the produced hydrocarbon product have an important role in this process.

In the second chapter, the problem of development of new compositions against asphaltene-tar-paraffin sediments was considered to increase the efficiency of oil field development[1].

Primary components were selected against ATP sediments. In the first approach, naphthenic acid, polypropylene glycol (PPG), KOH alkali and Al metal nanoparticles were proposed as primary components, and in the second approach, naphthenic acid, triethanolamine (TEA), KOH alkali and Al nanoparticles with dimensions of 50-70 nm were proposed. The properties of the named components and the effects they can create have been taken into account:

1.Primary components have high surface activity;

2. Creation of favorable conditions for observing exothermic reaction in alkaline environment;

3. Both surface activity and temperature effect of metal nanoparticles.

Thus, the proposed composition against asphaltene-resin-paraffin deposits consists of Acidol separated from naphthenic acids, Polypropylene glycol and potassium hydroxide, in the following % proportions of the components:

Naphthenic acid (Acidol)	55,6 – 54,695
Polypropylene glycol (PPG)	44,3 – 45,1
KOH	0,1 – 0,2
Al nanoparticle	0,003 – 0,005

This compound developed against ACP is conventionally called IKA-25.

[1]Shahbazov, E.K., Management of reotechnological properties of highly viscous oils based on nanotechnologies / Mursalova M. A., Ahmadova İ.K // Scientific Israel - Technological Advantages" The Journal "Scientific Israel - Technological Advantages" - 2018, №6 pp.85-90.

It should be noted that on the basis of the conducted scientific and research work, a newer composition (conditionally IKA-30) was developed based on Acidol separated from naphthenic acids. Unlike IKA-25, the components of this composition are triethanolamine (TEA), which is another surface-active reagent, instead of PPG) was organized from.

The composition components of IKA-30 reagent were in the following % proportions.

Naphthenic acid (Acidol)	59,8 – 59,2
TEA	39,9 – 40,5
KOH	0,2 – 0,3
Cu nanoparticle	0,003 – 0,005

Thus developed IKA-25 and IKA-30 anti-ATP compositions have aroused interest due to their low primary components and economic benefits. Based on the conducted studies, the following physico-chemical constants of the IKA-25 reagent were determined:

Density at 20 °C, kg/m^3	978 – 980
Viscosity at 20 °C, sSt	135 – 140
color	dark brown
it is dissolved	in oil

To prepare 1 liter of the used IKA-25 reagent, 55,6% acidol is alkalized with 0,1% KOH alkali for 30 minutes by intense mixing. The 44,3% PPQ to be used should be enriched with Al nanoparticles in the amount of 0,003 – 0,005%. The nano PPG composition formed in 20 – 25 minutes is added to the alkalized acidol in parts and mixed in the reactor for one hour.

59,8% Acidol taken for the preparation of IKA-30 reagent is alkalized with 0,2% dry KOH and an alkaline solution is obtained. In the second stage, intensive mixing of 50 –70 nm Al particles with triethanolamine allows preparation of NanoSAS composition. The next stage allows the IKA-30 reagent to be presented as a finished product in the reactor by adding and mixing NanoSAS to the alkalized Acidol in portions. Based on research, it was determined that IKA-25 reagent reduces the viscosity of oils and has a protective effect against paraffin deposits.

At the same time, a number of new paraffin inhibitors were synthesized and conventionally named as LPTI, LPPI and LPFK. The composition of all three compositions consists of a constant weight of surface-active substance. Other components include organic solvents and stabilizers. Synthesized new paraffin inhibitors were applied to multi-paraffin oil samples in laboratory conditions, and the obtained results were comparatively analyzed. The results obtained during the conducted studies were compared with the indicators of the well-known "СНПХ-2005" and Laprol 3603-2-12 reagents. For this purpose, oil samples taken from well No.607 of N.Narimanov Oil and Gas Production Union of "Azneft" Production Union were used as a research object. The indicators of used oils are given in table 1.

Table 1

Indicators of the product taken from well No.607

№	Indicators	Well number
		607
1	20 °C -Density at , kq/m^3	868,4
2	Freezing temperature, °C	+22
3	Viscosity at 30 °C, mm^2/s	59,15
4	Amount of combined water, %	No
5	Wetting angle, cos Θ, during time: 30" 1" 3"	 0,8748 0,8823 0,8995

For this purpose, three oil samples were taken in laboratory conditions and each sample was heated separately at 45 - 55 °C and then cooled to room temperature (20 - 22 °C). Then each of the cooled oil samples was individually dosed with LPTI, LPPI and LPFK inhibitors at a consumption of 200 g/t. Oil samples with inhibitor added are sampled after mixing in a magnetic stirrer for 5-7 minutes and physical parameters are determined[2]. The physical research was carried out on the SVM-3000 stapinger of the "AntonPaar" company. Freezing temperature was determined according to known methodology. The physical parameters of free and inhibitor added oil samples are given in table 2.

Table 2

Product change indicators taken from well No.607

№	No. Nameof NQCI and number of the well	Reagent	Temperature, °C	°C Amount of reagent, q/t	q/t Dynamic viscosity, mPa.s	Kinematic viscosity,	Density, g/cm³	FreezingTemperature °C	AQPCH,%
N. Narimanov Well 607		reagent-free	20		317,8	364,75	0,871	+22	10,8
1		LPTİ	20	200	90,64	103,72	0,874	-15	
2		LPPİ	20	200	31,80	36,414	0,873	-20	
3		LPFK	20	200	70,48	80,642	0.874	-13	

As can be seen from the table, the dynamic and kinematic properties of the oil sample with LPTI added decreased by 3,5 (87,9%) times, the freezing temperature decreased to minus 15 °C. When treated with another inhibitor, LPPI, the dynamic and kinematic properties decreased 10 times (90%), and the freezing

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[2]Əhmədova, İ.K. Asfalten-qətran-parafin çökmələrinə qarşı yeni effektiv ingibitorların işlənməsi / Əlsəfərova, M.E., Həsənov, X.İ. // Azərbaycan Neft Təsərrüfatı, - Bakı: - 2019. №12, - s. 38 - 41.

temperature decreased to minus 20 °C. The indicators of the LPFK inhibitor are weak compared to the indicators of the first two inhibitors. That is, the dynamic and kinematic strength decreased by 4,5 times (77,8%), and the freezing temperature decreased to minus 13 °C.

The change graph of viscosities, density and freezing temperature of oil samples with depressant added is given in figure 1, 2 and 3.

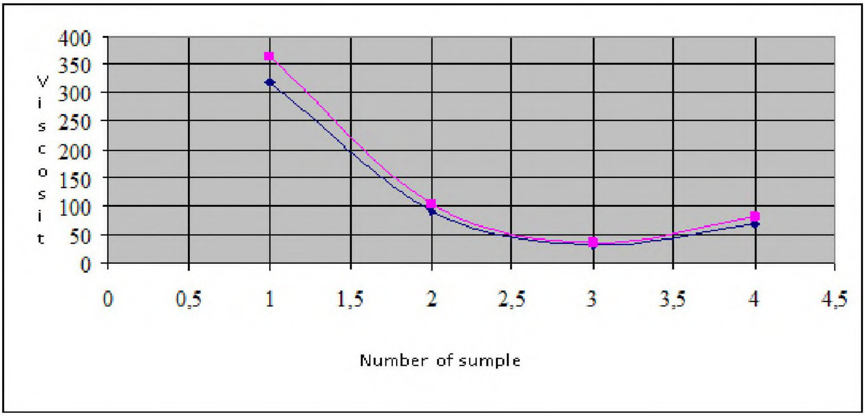


Figure1. Results of the effect of reagents on the dynamic and kinematic viscosities of the oil sample

Newly acquired inhibitors dramatically lower the viscosities and freezing points of high-paraffin oils.

Thus, the new paraffin inhibitor purchased on the basis of local raw materials has a very high effectiveness against ATP - sediments in the oil samples taken from well No.607 of N.Narimonov Oil and Gas Research Institute, and their wide application is inevitable in the future.

The proposed inhibitor was tested in wells No. 680, 433, 673, 603, 635, 699, 717 and 671 of N.Narimanov Oil and Gas Research Institute with high paraffin. The results of the tests show that the proposed paraffin precipitation inhibitor is much better than the known inhibitor in terms of technical and economic indicators.

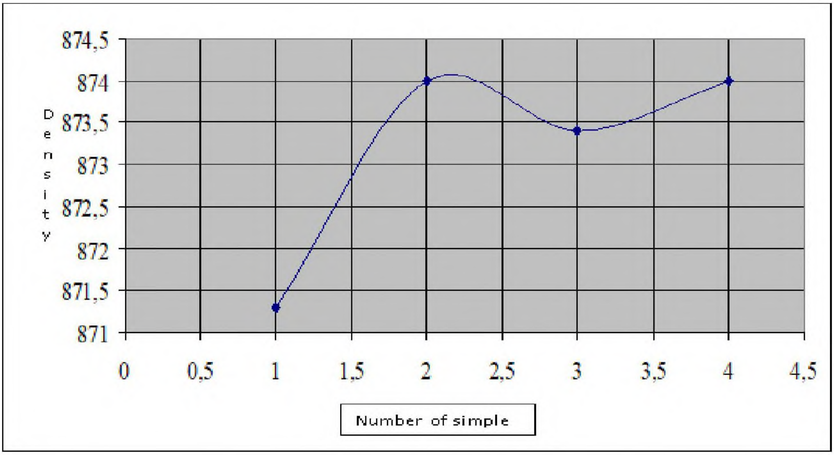


Figure2. Results of the effect of reagents on the density of N.Narimanov oil sample

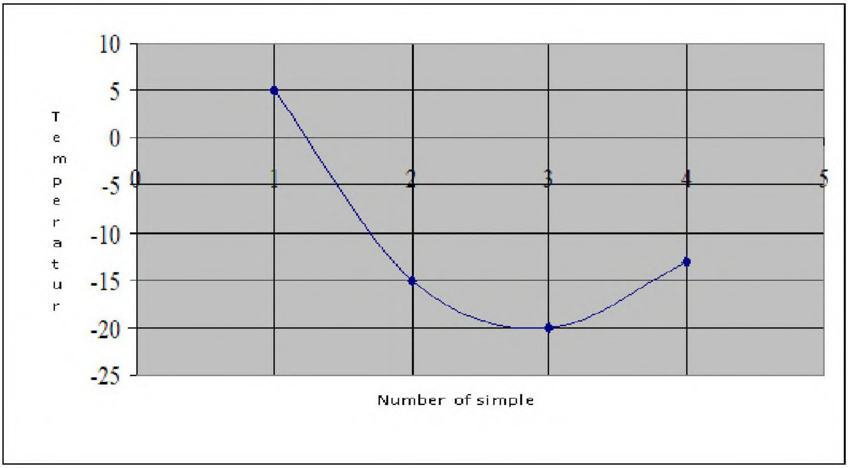


Figure 3. Results of the effect of reagents on the freezing temperature of the oil sample

The efficiency of the proposed paraffin precipitation inhibitor is due to the low amount of consumption, the reduction of the number of repairs without paraffin precipitation in oil wells, and the increase of the time between repairs.The IKA-25 reagent and naphthenic acids produced in oil refineries were used in the research as an oil-soluble SAM.

The indicators of these reagents are given in table 3.

Table 3

Physico-chemical indicators of oil-soluble reagents

№	The name of indicators	IKA-25	Crude naphthenic acids
1	External appearance	Dark colored homogeneous liquid	Brown homogeneous liquid
2	Density at 20 °C, kg/m^3	940 - 960	945 - 960
3	acid number,mg KOH/g	1 - 5	240 - 320
4	Interfacial surface tension of 0,1% SASsolution in kerosene at 20 °C at the distilled water boundary, mN/m	2 - 3	15 - 18

The effect of SAM on the rate of wetting of the porous medium was studied in quartz sand with a fraction of 0,1 – 0,25 mm.The research work was carried out in a laboratory model consisting of a glass tube with a size of d = 20 mm, filled with quartz sand dried by preheating.

Oil samples taken from well No. 483 of "Absheronneft" NQCI were used in the research. The indicators of the used oil are given in the previous sections of this topic.

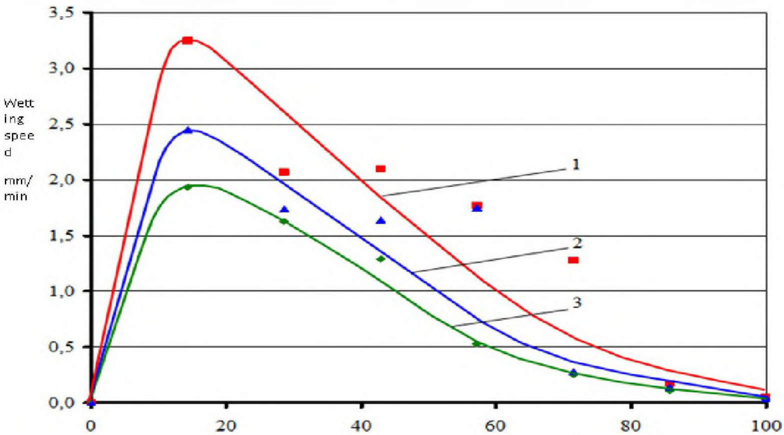
Table 4 and Figure 4 show the dynamics of quartz sand wetting rate changes when 0,1% solutions of IKA-25 reagent and naphthenic acid are given from the bottom of the model in the studied oil.

The analysis of experimental data shows that the rate of wetting of quartz sand of oil samples mixed with oil-soluble SAM is greater than that of pure oil, and this difference is visible at all stages of the studied process.

Table 4

Quartz sand with oil and oil-soluble IKA-25 reagent and naphtheneacid soaking rate

Height of sand soaking in oil, volume %	Wetting speed, mm/min		
	oil	1% IKA-25 reagent in oil	1% naphthenic acid in oil
14,3	1,934	3,250	2,450
28,6	1,629	2,070	1,740
42,9	1,290	2,100	1,640
57,2	0,528	1,770	1,750
71,5	0,254	1,280	0,279
85,8	0,108	0,176	0,140
100	0,039	0,050	0,040
Average speed	0,830	1,530	1,150



Rise height of oil in porous media, in % of total volume

Figure 4. Variation of wetting rate of porous media with oil-soluble IKA-25 and naphthenic acid

1 - oil + 1% IKA - 25 reagent; 2 - oil + 1% naphthenic acid; 3 - pure oil

As mentioned, the sharp decrease of formation pressures and temperatures in fields that have been in operation for a long time significantly affects the performance indicators of the fields. As a result, there is a decrease in the oil production coefficient of the layers, and a decrease in the rate of production. Contamination of the well bottom zone (WBZ), watering of wells, retention of high-viscosity oils in formation reservoirs, etc. such complications occur that, in order to combat them, it is necessary to use considerable material costs and invest a large amount of capital. Based on the problems highlighted above, a new approach methodology was proposed.

It should be noted that some nanoparticles, especially nanoparticles of metallic origin, cause an increase in the temperature of surface-active substance solutions used as a carrier. Thus, experiments conducted by US scientists have shown that it is possible to achieve aggregate change of even technical water from freezing temperature to vapor state by means of nanoparticles.

As a result of our experiments, it was determined that the addition of Al nanoparticles to water, which is a clear example of metal nanoparticles, increases the temperature. Note that the reaction of Al nanoparticles with water is distinguished by its individuality. As is known, Al is one of the active metals under normal conditions and is covered by an oxide layer. This layer protects the metal from contact with oxygen or water vapor. As a result of the damage of the corresponding oxide layer, serious changes are observed as a result of contact of Al metal with alkali and acids, as a result of which Al reacts with water. In the usual case, the reaction can be written as follows:

$$2Al+6H_2O = 2Al(OH)_3 + 3H_2\uparrow$$

As can be seen from the reaction, the contact of 2 atoms of Al with water produces 3 molecules of H2 gas. In other words, 6 g of H2 and 156 g of Al(OH)3 are formed in the reaction of 54 g of Al with water. The speed of this reaction in room conditions is not very high. The main reason for this is that there is enough oxygen dissolved in the water, which in turn causes the passivity and inactivity of Al Metal. However, at high temperatures, the corresponding reaction rate increases.

As a result of X-ray structural analysis of Al nanoparticles, it was determined that they have X-ray densities with small threshold values, which in turn is 0,2%. Such a density is observed when Al nanoparticles are heated to 70 °C. Under the conditions of the heating regime, Al nanoparticles create a basis for obtaining new effects with water. Based on research, it was determined that the speed of H2 gas released as a result of contact of Al nanoparticles with water is 1,38·ml/s·q. If we carry out this study for a temperature of 60 °C, the rate of H_2 gas release is 3 ml/s·g, and a temperature of 80 °C is 9,5 ml/s·g. As a result of these studies, it is clear that the rate of H_2 gas separation increases significantly with the increase in temperature.

A new approach for increasing the reaction rate has been proposed. The essence of this approach is that after water molecules are treated with caustic soda, a well-known exothermic reaction occurs, the temperature of alkaline water increases. After the caustic soda solution is treated with SAS, it is treated with Al nanoparticles. The proposed approach is proven by the fact that the rate of H2 gas release increases up to 18 ml/s·g as a result of water treatment with caustic soda, as the pH increases. The Nano composition is well soluble in water, has surface-active and wetting properties in highly mineralized waters.

The nanocomposition was studied using it as an inhibitor against ATP deposits. The amount of dry matter in the sample was 40%.

The technical characteristics of the nano composition are as follows:

- mass fraction of dry residue, %, not less than4
- density at 20 °C, kg/m3, not less than1205
- hydrogen indicator pH, not less than5
- freezing temperature, °C   minus 19
- kinematic viscosity at 20 °C, mm^2/s4,95

Due to its chemical properties, the presented nanocomposition creates a layer on the surface of the metal as a result of chemisorption with an inhibitor, and also has the property of corrosion protection. During the application of inhibitors with complex properties, deposits are prevented from accumulating in the lift pipes of the well as well as in the transport lines, and as a result, the viscosity of the emulsion oils decreases and the movement and transportation of the fluid inside the well is simplified[3].

Thus, a nanocomposition that allows controlling the rheotechnological properties of high-viscosity non-Newtonian oils whose composition consists of ATP sediments was developed and studied in laboratory conditions. Properties of the appropriate composition were studied, applied in well conditions and efficiency was determined.

The study of the mechanism of action of the new anti-paraffin inhibitor, developed on the basis of research, is of great importance. As it is known, analytical methods are used to study the nature of many phenomena and processes in our modern times. In my research, I used an X-ray diffractometer device. The Miniflex 600 X-ray phase XRD X-ray diffractometer has a wide range of functions and allows to perform qualitative phase analysis and quantitative analysis. At the same time, the definition of the crystal lattice, the determination of the size of the crystallites and the level of distortions of the crystal lattice, the specification of the parameters of the lattice, and the determination of the molecular structure are solved within the capabilities of this device.

At the same time, information about the internal structure of the research object was obtained through a Skyner electron microscope. For this, a JEOL JSM 6610-LV device, a Skyner electron microscope, was used. Using this device, it is possible to carry out the following studies:

- studying the shapes and sizes of grains at the same time;
- determination of the distribution and phase composition of chemical elements on its own area and the area of the studied samples, as well as;
- chemical inhomogeneity on the transparent cut area;

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[3] Ахмедова, И.К. Исследование нового ингибитора парафиноотложений Доклады НАНА том LXVI / И.К. Ахмедова // Socar Proceedings - Баку: -2022, №1, - с. 73 - 76.

-acquisition of object images in a wide range of magnification (from 5 x to x 300000) in derived and reflected electrons;

- quantitative x-ray microanalysis by selecting the area to be analyzed: acquisition of spectra with a given step at a point, by area, by lines;

- synthesize spectra of complex or complete standard samples;

- compare current spectra with those in memory;

- obtaining the phase distribution map from the element maps, performing quantitative and qualitative X-ray microanalyses by selecting the analysis point on the screen of the microscope and microanalyzer;

- segmenting transition tone images by providing area information;

- acquisition and preservation of spectra from the sample area or individual points;

- obtaining the distribution map profile of elements along the line;

- X-ray spectroscopic (X-ray spectrometer RFA, universal S8 Tiger). Accurate measurements, speed, sensitivity and accuracy of received data in a short period of time;

- The amount of elements of different types (powder, liquid, solid) up to B ÷ U (excluding gases and inert gases) n 10-4–100% is determined.

IKA-25 reagent was added to the well in order to determine the extraction rate of the residual mineral (oil) in the decommissioned oil well. According to the measurements made at this time, it was possible to produce an additional 51 tons of oil. In order to clarify the mechanism of this process, some properties of (relatively) additional extracted oil (viscosity, density, surface tension, freezing temperature, paraffin precipitation) were studied. Elemental and phase analysis was performed after washing the residue from the organic part (toluene, acetone) to clarify which change corresponds to the added reactant.

The results of the conducted studies are presented in figure 5.

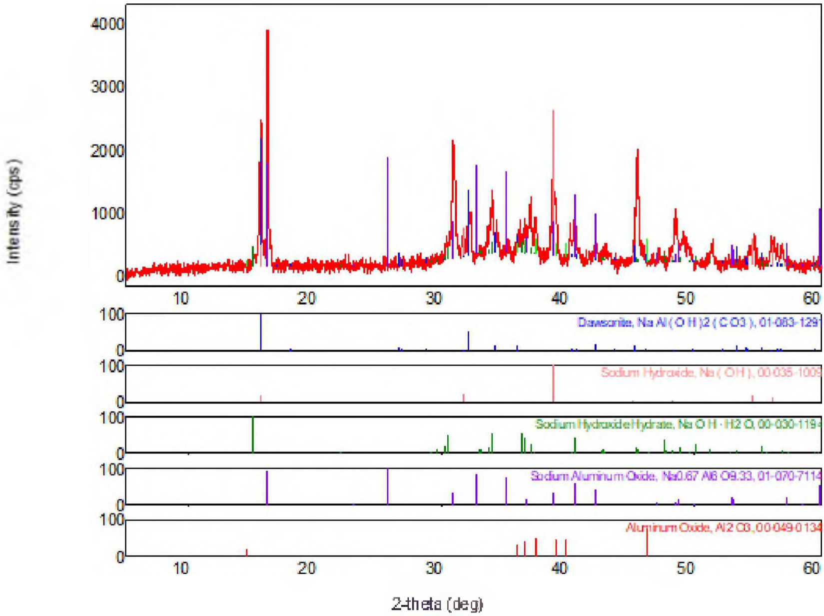

Figure 5. X-ray phase spectrum of Al nanoparticle

In addition, the X-ray phase spectrum of Al nanoparticle was also obtained. This analysis shows that the Al nanoparticle indeed has a crystalline structure (picture 5).

As can be seen from the radiograph, Al nanoparticles form a characteristic peak at $2(\theta) = 39°$ and $45°$. This proves once again that the Al nanoparticles are pure and crystalline.

Although there are various means of preventing salt precipitation in oilfield equipment, the main method is the method of application of special chemical reagents as inhibitors.

When choosing an inhibitor, its effectiveness in the applied technological process, its ability to coexist with other chemical reagents in the system, its effect on the efficiency and reliability of the main process, and the availability of the reagent are taken into account.

The effect of inhibitors on formation water can also be determined by comparing the amount of mineral salt deposits obtained during solution evaporation with and without inhibitors.

The organization of experiments when using real reservoir waters for inhibitor testing is associated with a number of difficulties. Components to create salt precipitation inhibitors were selected from reagents produced in the Republic or brought to the Republic for use in various technological processes.

For this purpose, besides Sulfanol, other SAS- lignosulfonates and the cubic residue of propylene glycol production - polypropylene glycol (PPG) were used.

During the study of the dynamics of optical density changes due to the precipitation of previously formed salt deposits after intensive mixing of formation water treated with different inhibitors and without inhibitors, a similar change of indicators was observed. Researches were performed by photoelectrocolorimetric method.

The analysis of the optical density indicators of formation waters every 10 minutes for an hour allows us to make an opinion about the precipitation rate of calcium carbonate salt. It was determined that the optical density was 0,06 after 10 minutes of mixing in the formation water without inhibitors. In addition, the optical density in inhibited formation water was 0,17 after 10 minutes and decreased to 0,15 within 60 minutes.

In order to study the indicated dependences in more solid salt solutions, studies were continued in model-containing systems.

The formation water model is based on calcium carbonate, because carbonate rocks are typical for oil fields.

Supersaturated calcium carbonate solutions were prepared by mixing equal amounts of 0,8 N calcium chloride and sodium carbonate solutions. Precipitation inhibitor is dissolved in calcium chloride solution. The results of the effect of various inhibitors and their concentrations on the dynamics of the optical density of over-beaten solutions of calcium carbonate are shown in table 5.

It was determined that as the concentration of SAS - lignosulfonates and sulfanol increases, their effect on the optical density of the investigated solutions, in other words, on the degree of dispersion of the obtained salt crystals, becomes stronger.

Table 5

Dependence of the composition on the optical density

Composition of inhibitors	Optical density as a function of time (min), D					
	10	20	30	40	50	60
Control - no inhibitor	0,60	0,45	0,32	0,25	0,20	0,17
Lignosulfanate, %						
0,05	0,86	0,77	0,70	0,61	0,53	0,45
0,01	0,67	0,63	0,58	0,50	0,38	0,30
0,005	0,60	0,53	0,46	0,40	0,32	0,24
Sulfanol, %						
0,05	0,88	0,81	0,75	0,65	0,57	0,48
0,01	0,70	0,65	0,59	0,50	0,42	0,35
0,005	0,66	0,58	0,50	0,41	0,36	0,28
PPQ– 0,01 %	0,70	0,63	0,48	0,41	0,35	0,23
Sulfanol – 0,01 % PPQ– 0,01 %	0,76	0,68	0,59	0,51	0,40	0,36
Sulfanol – 0,01 % PPQ–0,01 %	0,80	0,70	0,63	0,50	0,45	0,34
Lignosulfanate – 0,01 % PPQ– 0,01 %	0,76	0,65	0,57	0,51	0,40	0,32
Sulfanol – 0,01 % Lignosulfanate, – 0,01 %	0,81	0,72	0,67	0,58	0,41	0,35
Sulfanol – 0,01 % Lignosulfanate, – 0,01 % PPQ– 0,01 %	0,82	0,75	0,69	0,58	0,49	0,38

As a result of the conducted laboratory studies, it was determined that the composite composition consisting of SAS and glycols is more effective due to the degree of reduction of salt precipitation and its effect against corrosion. The protection effect of such composition against salt deposits is 61 - 71%, and the effect of protection against corrosion is 64 - 75%.

In the third chapter, the collection and analysis of the mining indicators of the wells where the complications related to AQP sediments were observed were carried out.

According to mining data, paraffin wells are operated from the QUQ, QA and VIII horizons. These wells are located in DSO No. 9, 13, 14, 15 and 19. The amount of water in the product of the wells is different. The fluid of wells 208 and 412 is approximately 50% oil and 50% water, while the oil of well 314 is anhydrous.

The amount of water in the production of heavy oil wells 222, 225 and 257 located in DDÖ-14 varies from 1,6% to 65%. The production of oil extracted from the wells, as well as the composition of the well fluid, are different.

The wells are highly productive, the daily amount of extracted oil varies from 31 to 105 t. The analysis of mining data shows that the most maintenance works related to the cleaning of paraffin deposits were performed in well No. 208. Cleaning of elevator pipes from paraffin deposits is carried out with gas condensate every three days.

The repair period of the well is determined based on the rise of pressure in the annular space. Well No. 314, located in DSB-13, has the highest production of paraffin wells. The oil of the well is anhydrous, the production is 105 t/day, and the well is operated from the QA horizon. Since the pressures in the well are high (Pg.a. - 57/61 atm, Ph.f. - 105 atm, Pb.a - 148 atm), the cleaning of the lift pipes from accumulated sediments is carried out according to the daily oil production changes, according to the mine data. The same situation is observed in well No. 412 located in DSB-14. In order to ensure a stable production mode of the well, the amount of extracted oil is measured every day and, if required, the lift pipes are treated

with gas-condensate. The fluid of well No. 412 consists of 50% oil and 50% formation water, 181 thousand m3 of gas is extracted from the well per day. Since the well product is emulsified, the movement speed of the liquid on the surface of the lift pipes is low and conditions for the accumulation of sediments are created. The product of the well No. 431 operated from the QA horizon in DSB-15 is very wet, dilution is about 70%. Pressures in the well are high - Pq.a. - 38/42 atm, Ph.f. - 50 atm, Pb.a .- 180 atm.
According to mining data, although the oil of the well is paraffinic, there are no complications due to paraffin deposits in the well. This is due to the fact that the heat capacity of the product of the well, which consists of ~ 70% by mass of formation water, is high, so its temperature at the wellhead is 35 - 40 °C.

According to mining data, the viscosity of the product of wells No. 245 and 222, 225, 257 operating from the VIII horizon in DSB-13 and DSB-14, as well as well No. 91 located in DSB-19, is high. High viscosity makes it difficult to extract oil from wells. These wells are treated periodically to keep the production stable and to clean the sediments accumulated in the lift pipes. For this purpose, a schedule for the processing of wells has been drawn up in OGED. According to the results of the physico-chemical composition of the studied oils, as well as the amount of asphaltene-resin-paraffin substances in these oils, samples of oils with high paraffin, freezing at high temperature and water in a combined state were selected for the selection of the inhibitor. On the basis of previous studies of the physical and chemical composition of oils, as well as the determination of the amount of ATP substances contained in the oil, and based on these indicators, for the selection of an inhibitor against paraffin precipitation, from wells No. Oils produced from well No. 314 were selected. From the results of our previous studies, we see that the oil produced from well No. 208 is very diluted, so that its emulsion contains 50% combined formation waters. In the selection of the inhibitor for this oil, the purpose of preventing the dispersion of the emulsion in the well pipe was set. First of all, the physico-chemical indicators of the product of paraffin wells were studied based on laboratory research. The laboratory research was carried out according to the traditional "cold finger" method. The temperature of the cold surface was maintained at +15 ºC. The contact of the tested oil with the cold cylinder was continuously continued for 30 minutes by means of a magnetic stirrer. The concentration of the reagent in oil (dry matter content 40% by volume) changed from 0,05% to 0,20%. It was determined that the protective effect of the reagent increases with the increasing consumption (based on commodity product), and the degree of concentration of the reagent in oil was 79% at 0,2% by volume. To determine the effectiveness of the protective effect of the reagent in emulsion watered oil taken from well No. 314, which freezes at a higher temperature (analogous to oil taken from well No. 208) and oil taken from well No. 412, which contains combined and also free formation waters interested. Researches were carried out by the above methods, but on a cold surface at three times lower temperature (+ 5 ºC against + 15 ºC in the previous test). The following conclusions can be drawn from the conducted experiments: the combined or free presence of reservoir water in the oil does not affect the effectiveness of the reagent against paraffin deposits; in order to obtain effective results against paraffin precipitation in oil that freezes at low temperature (well No. 314), it is required to increase the reagent consumption; in the studied wells, the effect of protection against paraffin precipitation was 61,9% (well No. 412) and 47,3% (well No. 314). It is known that UPB is a by-product obtained as a result of fermentation of sugary substances. Therefore, pH = 4 - 5 in the reagent in commodity form All known surfactants have an alkaline reaction above pH = 7. In this regard, it has been interesting to study the effectiveness of the protection effect of the reagent against paraffin precipitation at higher values (pH = 9 and pH = 11). The study of the protective effect of the pH reagent of the reagent against paraffin deposits was carried out in the oil of well No. 208. The temperature of the cold surface of the cylinder was + 15 ºC, the duration of the study was 30 minutes. The concentration of the reagent (in commodity form) in oil was 0,025-0,050% by mass. First of all, based on laboratory research, the physico-chemical indicators of the product of paraffin wells were studied, and the mechanism of the physico-chemical action of the reagents used against ATP sediments was studied, and the proposed technology was used in the wells of "N. Narimanov" and "Neft Daşlari" OGED. and the possibility of using IKA-30 reagents was considered. The physico-chemical mechanism of action of the reagents used against ATP sediments was studied, as well as the feasibility of using the IKA-25 and IKA-30 reagents in the wells.

The evaluation of test program items is determined by the extent to which the approved work plan is fulfilled. Failure to comply with the requirements of the work plan or the impact of its violation on the final result of the test is assessed in advance; The components of the composition are brought to the well site in the required amount and the reagent is prepared in the required concentration according to the drawn up work plan (with the condition that it is 1 m3 per 1 m interval); Lift pipes are delivered to the bottom of the well by adding new PCPs; the prepared composition is delivered to a one-time layer; after the composition is pressed into the formation, the well is kept closed for 24 hours, after which time it is assimilated. After the normal operating mode of the well is restored, daily registration of its operating parameters is carried out.

In 2022, the new IKA-25 inhibitor against asphaltene-tar-paraffin deposits was applied in operational wells No. 682, 683 and 607 of the "N.Narimanov OGED". Due to the successful implementation of the event, 51,0 tons of additional oil was produced.

CONCLUSION

1. The current situation of combating asphaltene-tar-paraffin deposits was analyzed and research directions were determined;

2. New surface-active compositions against asphaltene-resin-paraffin deposits have been developed. The following were determined in laboratory conditions:

- The rate of wetting of quartz sand of oil mixed with IKA-25 reagent is 1,68 times higher than the rate of wetting of the environment with pure oil;
- The rate of wetting of quartz sand of oil mixed with IKA-25 reagent is 1,27 times higher than that of oil containing naphthenic acid;
- rheological indicators of asphaltene-resin-paraffin sediments are regulated up to 25% by means of IKA-25 and IKA-30 reagents;

The composition of IKA-25 was successfully applied at N.Narimanov National Research Institute. The composition "Paraffin Precipitation Inhibitor" has received the Patent of the Republic of Azerbaijan No. I 2004 0109.

3. Nanocompositions were developed to regulate the rheotechnological properties of high viscosity oils. The following were determined in laboratory conditions:

- when the density of the nanocomposition in oil is 0,2%, the protective effect against ACP deposits is 78,9%;
- the protective effect of the composition prepared from nanocomposite and Sulfanol in a ratio of 9:1 against ACP deposits increases from 61% to 80%.

4. An inhibitory composition has been developed against salt deposits that occur during operation. The following were determined in laboratory conditions:

- effect of protection against salt precipitation 61 - 71%;
- the corrosion protection effect is 64-75%.

5. As a result of the application of IKA-25 composition against processed asphaltene-resin-paraffin sediments in wells No. 682, 683 and 607 of N.Narimanov Oil and Gas Plant, an additional 51 tons of oil production was achieved.

The main results of the dissertation were published in the following theses and articles:

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Candidate's personal contribution

[7, 12 - 14] were performed freely,

participation in conducting research and summarizing results in [1 - 6, 8, 9, 15],

[10, 11] reports, analysis of results, participation in conducting laboratory tests and generalization of results.

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