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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

**DEVELOPMENT AND APPLICATION OF NEW METHODS
FOR ISOLATION AND CONSOLIDATION OF THE
BOTTOMHOLE FORMATION ZONE BASED ON MODERN
METHODS OF TESTING WORKING AGENTS**

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

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GENERAL DESCRIPTION OF THE WORK

Relevance of the topic and degree of its development

Under present-day conditions of oil and gas production, one of the key factors for the efficient operation of wells is reliable water shut-off isolation and consolidation of the bottomhole formation zone. Water breakthroughs in wells leads to a decline in production rate, deterioration of the produced fluid quality, and an increase in operating costs.

Existing isolation and consolidation methods do not always provide sufficient durability and efficiency, particularly under complex geological and technical conditions.

The dissertation examines the improvement of downhole operations aimed at limiting water breakthrough and consolidating the bottomhole formation zone in the following directions:

- increasing the efficiency of chemical methods for consolidating the bottomhole formation zone through the use of nanoscale additives and polymer chemical compounds;
- increasing the efficiency of water shut-off technologies through the use of nanocomposite polymer compositions and expanding their range of applicability.

Although the use of various chemical compounds to consolidate weakly cemented formations in the bottomhole zone is a fairly widespread practice, the introduction of nanoscale additives makes it possible to significantly expand the range of their applicability.

Water shut-off limitation using various gel-forming compositions has become fairly widespread in the modern oil and gas industry; nevertheless, a number of technological problems remain unresolved by conventional technologies. In the opinion of specialists, viable solutions may lie in the area of nanocomposite materials or in the modification of the chemical compositions used.

Despite the fact that the above-mentioned issues are of considerable relevance to the oil and gas industry, many questions remain insufficiently studied:

1. the efficiency of nanoparticle application for consolidating the bottomhole formation zone has not been fully clarified;

2. the use of gel-forming compositions to combat sand production in wells has been little studied;

3. the issue of formation damage caused by the injection of gel-forming compositions has practically not been investigated;

4. the efficiency of nanocomposite gel-forming systems under high formation temperatures has not been studied.

The present work attempts to answer all of the above questions.

Object and subject of the research

The object of the research is the bottomhole formation zone of wells at fields that have been under long-term operation.

The subject of the research is the development of new water-shut-off compositions, temporary isolating compositions, and chemical methods for combating sand production in order to increase the operating efficiency of the wells in question.

Aim and objectives of the research

The aim of the research is the development and application of new methods for the isolation and consolidation of the bottomhole formation zone of wells based on modern methods of testing working agents.

The main objective of the research is the creation and application of new methods and technologies that increase the operating efficiency of wells at fields in the late stage of development.

Research methods

The tasks set in the dissertation were solved through theoretical, laboratory and field research. The following research methods were used: rotational viscometry, dynamic light scattering (DLS), pendant-drop method, differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and standard and special core studies.

Statements submitted for defense

The following main provisions are submitted for defence:

1. A formulation based on epoxy resin modified with graphitic carbon nitride ($\text{g-C}_3\text{N}_4$) nanoparticles, developed for consolidation of the near-wellbore zone.

2. A formulation based on urea–formaldehyde resin incorporating silicon dioxide (SiO_2) nanoparticles, developed for consolidation of the near-wellbore zone.

3. A gel-forming formulation developed to reduce sand production from the formation during well operation.

4. A gel-forming formulation developed for water shutoff in wells.

5. A gel-forming formulation based on colloidal silica, developed to limit water inflow into wells under high reservoir temperature conditions.

6. A nanocomposite polymer formulation developed for water shutoff in wells under high reservoir temperature conditions.

7. A method developed to mitigate formation permeability impairment caused by the application of silicate-based gel-forming formulations and to restore permeability.

8. A method for water shutoff in wells through the sequential application of a temporary plugging agent and a gel-forming formulation.

Scientific novelty of the dissertation

The scientific novelty of the dissertation is reflected in the following results:

1. An epoxy resin formulation modified with graphitic carbon nitride ($\text{g-C}_3\text{N}_4$) nanoparticles, a urea–formaldehyde resin formulation incorporating silicon dioxide (SiO_2) nanoparticles, and a polymer-based gel-forming formulation have been developed for near-wellbore formation consolidation and sand production control. The effects of nanoparticles on the mechanical strength and thermal stability of the resin-based formulations have been established. The feasibility of controlling the permeability of the consolidated porous medium by incorporating a gas-generating agent into the epoxy resin formulation has been demonstrated. Experiments have shown that the developed gel-forming formulation reduces sand particle migration and increases rock mechanical strength.

2. A polymer-based gel-forming formulation, a colloidal silica-based gel-forming formulation for use at high reservoir temperatures, and a nanocomposite polymer formulation have been developed for

water inflow control and water shutoff in wells. The dependence of gelation time in the colloidal silica-based system on temperature, silica concentration, and the concentration of activating salts has been established. Under the investigated conditions, increases in temperature, silica concentration, and salt concentration have been shown to shorten gelation time. Experiments have demonstrated that incorporating nanoparticles into the polymer formulation improves gel stability and increases the residual resistance to flow in the porous medium.

3. An alkaline solution treatment method has been proposed to mitigate permeability impairment caused by silicate-based gel-forming formulations and to restore formation permeability. The ability of a sodium hydroxide solution to restore porous-medium permeability by breaking down silicate gel plugs has been demonstrated experimentally. A water shutoff method based on the sequential application of a temporary plugging agent and a gel-forming formulation has also been developed. The method involves creating a temporary filter cake in oil-saturated zones, directing the gel into water-conducting fractures, and subsequently breaking down the filter cake with enzymes to restore permeability in the oil-saturated zones.

Theoretical and practical significance of the research

The propositions developed in the dissertation can serve as a basis for further scientific research aimed at improving water shut-off control technologies, preventing sand production, and increasing the profitability of hydrocarbon production. They can also be integrated into educational programs for training specialists in the field of oil and gas field development, which contributes to improving the quality of engineering education and the introduction of modern technologies into industry.

Field trials of the developed gel-forming composition for consolidating the bottomhole formation zone in order to prevent sand production were carried out at the Verkhne-Shapshinskoye field. As a result of implementing these technological measures on wells No. 1004, 1007 and 1009, an additional oil recovery of 125 tonnes was achieved.

Selective water shut-off isolation operations using the developed gel-forming composition were carried out on wells No. 1325 and 1350 of the Urengoy oil-and-gas-condensate field (OGCF). Monitoring results showed that the volume of produced water decreased by 3,659 m³, while oil production increased by 460 tonnes over the observation period.

Approbation and implementation of results

The materials of the dissertation were presented and discussed at the following international and republican conferences:

- Ashirov Readings. Samara: Samara State Technical University, 02.12.2020;
- V International Scientific-Practical Conference, Bulatov Readings. Krasnodar, 31.03.2021;
- V International Scientific Conference of Young Researchers. Baku: Baku Engineering University, 29–30.04.2021;
- SPE Annual Caspian Technical Conference. Virtual conference, 5–7.10.2021.

The main content of the dissertation has been published in 9 scientific works, including 4 abstracts and 5 articles.

Organization at which the dissertation research was carried out

The dissertation research was carried out at NIPi “NefteGaz”, SOCAR.

Volume and structure of the dissertation

The dissertation consists of an introduction, four chapters, the main conclusions and recommendations, a list of references, and appendices. It comprises 154 pages and contains 1 table, 8 figures, 31 graphs, and 117 references.

The total number of characters is 171,839. The Introduction contains 11,821 characters; Chapter I – 65,594 characters; Chapter II – 23,243 characters; Chapter III – 54,519 characters; Chapter IV – 11,778 characters; Conclusions and Recommendations – 4,884 characters.

SUMMARY OF THE DISSERTATION CONTENT

The introduction substantiates the relevance of the dissertation, defines the aim and main objectives of the research, its scientific novelty, and indicates its practical value.

The first chapter (literature review) presents an analysis of existing methods for testing working agents used for the isolation and consolidation of the bottomhole formation zone of wells. The relevance of developing and implementing innovative technologies for preventing sand production and limiting water breakthrough is substantiated. A critical review is conducted of theoretical and experimental studies devoted to mechanical, chemical, microbiological and nanotechnological methods of rock consolidation, as well as selective and non-selective water isolation technologies. Promising research directions aimed at increasing the efficiency and environmental safety of the technologies applied are considered.

Sand production is the process of rock particle migration from the formation into the wellbore under the action of the flow of formation fluids. This problem can substantially reduce both well productivity and casing-string stability.

Conventional sand-control methods include the installation of various filters, hydraulic-fracturing technologies combined with gravel-pack completions (frac-pack), and gravel packing.

The parameters affecting a rock's propensity to produce sand can conventionally be divided into two main groups: the influence of formation-fluid flow, and the mechanical strength of the formation.

Tectonic stresses in the formation have the most significant effect near the crests of structural traps and fault zones.

Rock cementation characterizes its ability to preserve structural integrity and resist failure. This parameter is determined by the degree of grain bonding and the strength of intergranular contacts.

Heterogeneity of the productive formation has a significant effect on stress distribution as well as on the filtration and storage characteristics of the rock. The key factors influencing sand production in this case are the stress distribution in remote zones and permeability variations.

One of the key factors contributing to active sand production from the productive formation may be the intensification of the hydrocarbon production process. This effect is caused by an increase in the pressure drawdown, i.e., the difference between the formation pressure and the bottomhole pressure of the well.

At high pressure gradients in the productive-formation zone, hydrodynamic loads on the rock increase, which can lead to destabilization of its structure and the entrainment of sand particles into the fluid flow.

An increase in the water–oil ratio also has a substantial effect on sand-production processes. This effect is explained by two main mechanisms: the first is related to a change in reservoir wettability characteristics, and the second is caused by a change in the relative permeability of the rock.

Hydrodynamic effects on the rock are determined not only by the fluid flow velocity but also by its viscosity characteristics. As hydrocarbon viscosity increases, the friction force exerted on the porous medium increases, which raises the likelihood of rock failure and sand-particle entrainment.

The most adverse consequences of sand production include rock subsidence and formation degradation, accumulation of sand without being carried to surface, erosive wear of downhole and surface equipment, accumulation of sand in surface gathering systems, and its disposal.

The most common sand-consolidation technologies currently in use can be classified as follows:¹

4. Chemical consolidation methods:

- Resin injection: this method involves injecting resin into the bottomhole zone, where it hardens, consolidating the formation and preventing the migration of sand particles into the well.

- Injection of gel-forming compositions: this method involves injecting polymer gels into the bottomhole zone to consolidate it.

¹ Alakbari, F.S. Chemical sand consolidation: from polymers to nanoparticles / M.E. Mohyaldinn, A.S. Muhsan [et al.] // Polymers. – 2020. – Vol. 12, No. 5, – 1069.

- Injection of foam systems: in this case, sand production is blocked by a barrier created by the foam system.

- Injection of various chemical compounds: this group includes a fairly wide range of chemical compounds often used to create a barrier against sand production.

5. Application of electromagnetic effects for consolidating weakly cemented rocks: this group of methods is based on the use of a high-frequency electric field to create strong bonds between sand grains. The advantage of this technology is that it does not require the use of chemicals, which reduces environmental impact.

6. Microbiological consolidation: the use of bacteria to create a natural binding agent that holds sand grains together. This technology is distinguished by its environmental cleanliness and the possibility of application in deep formation zones.

7. Application of nanoparticles: technologies in this group are based on the introduction of nanoparticles no larger than 100 nanometres in size to bind sand grains together.²

An increase in the water cut of well production is one of the most pressing problems of the modern oil and gas industry. In general, an increase in the water cut of produced fluid creates numerous economic problems for oil-producing companies. First, it negatively affects the productivity of producing wells and shortens their service life, increasing operating costs. For example, in gas-lift production, the amount of gas injected increases proportionally with an increase in water cut. An equally important factor is the increase in various types of deposits and instances of corrosion, and, as a consequence, failure of both downhole and wellhead equipment.

The costs of primary treatment of the produced oil also increase substantially.

All water shut-off limitation technologies are divided into two large groups — non-selective and selective — according to their

² Mishra, S. Nanoparticle induced chemical system for consolidating loosely bound sand formations in oil fields / K. Ojha // Journal of Petroleum Science and Engineering. – 2016. – Vol. 147, – pp. 15–23.

ability to act selectively on water-saturated zones without affecting oil-saturated zones.³

Non-selective methods block water breakthrough without distinguishing between water- and oil-saturated zones, which may reduce oil production. These methods are effective when the water breakthrough is catastrophic in nature and rapid intervention is required. An example is the use of various cement- and synthetic-resin-based plugging compositions.⁴

The second chapter is devoted to the development of new compositions for consolidating the bottomhole formation zone of wells and preventing sand production. Innovative methods of chemical consolidation of weakly cemented rocks using nanofluids, nanoparticles and gel-forming compositions are considered. An analysis of conventional mechanical and chemical sand-control methods is presented, and their limitations are identified.⁵ Particular attention is paid to chemical technologies involving the injection of polymer- and resin-based compositions to increase the strength of the bottomhole zone and reduce permeability. Promising modifications based on epoxy and urea-formaldehyde resins with nanoparticle additives, as well as gel-forming compositions, are considered.

Although the use of resins with nanostructure additives increases the effectiveness of these compositions in increasing the mechanical strength of the formation, it has a negative effect on permeability. Therefore, additional additives are required to preserve formation permeability or to reduce this negative effect. This study presents a new technology using the addition of a gas-generating agent and carbon nitride to epoxy resin. In addition, a series of experiments on unconsolidated (sand-pack) formation models was carried out to

³ Al-Muntasheri, G.A. A review of crosslinked polymeric gels for water shut-off treatments / H.A. Nasr-El-Din, I.A. Hussein // *Journal of Petroleum Science and Engineering*. – 2010. – Vol. 73, No. 3–4, – pp. 138–147.

⁴ Du, J. Nanocomposite gels for water shut-off and temporary plugging in the petroleum industry: a review / Q. Wang, P. Liu [et al.] // *Petroleum Science and Technology*. – 2022. – pp. 1–36.

⁵ Ben Mahmud, H. Sand production: A smart control framework for risk mitigation / V.H. Leong, Y. Lestario // *Petroleum*. – 2020. – Vol. 6, No. 1, – pp. 1–13.

evaluate the effect of the developed nanofluid on the permeability of treated zones of the formation [5].

In the first series of experiments, various resin-to-solvent ratios were investigated. Two parameters were studied: the dependence of the degree of consolidation of treated samples on the volume of the working agent, and the effect of resin concentration in the working agent.

To assess the consolidating capacity of the developed compositions, they were injected into a sand-pack formation model and sealed for conditioning at a temperature of 62 °C and a pressure of 3,000 psi for 48 hours, corresponding to reservoir conditions of the Azeri-Chirag-Guneshli (ACG) field. After conditioning, the permeability of the sand-pack model was measured again. The sample was then removed from the core holder and its compressive strength was measured. Uniaxial compressive strength (UCS) is assessed by applying an axial force to a cylindrical sample of a given diameter until failure.

The effect of resin concentration and working-agent volume on sand-consolidation efficiency was studied first. Based on the results obtained, a resin concentration of 60% and a working-agent volume of 2 pore volumes were selected for further experiments.

Next, the effect of nanoparticles and the gas-generating agent on the compressive strength and permeability values of the tested samples was studied. Graph 1 shows the effect of nanoparticles and the gas-generating agent on the compressive strength and permeability of the sand-pack formation model, respectively.

As shown in Graph 1(a), at low concentrations of the gas-generating agent (0–2%), compressive strength initially increases with increasing nanoparticle concentration. However, once the nanoparticle concentration reaches 0.5%, the rate of increase in compressive-strength values slows down.

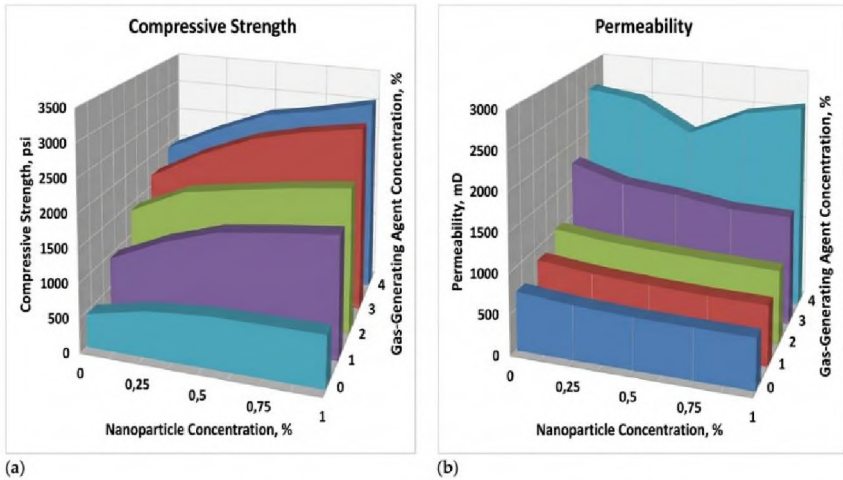
Figure 1(b) shows that the permeability value of the samples at a constant gas-generating-agent concentration remains relatively stable with increasing nanoparticle concentration, indicating a minimal effect of nano-g-C₃N₄ on permeability.

The experimental study of the new nanofluid for chemical sand consolidation produced promising results. The incorporation of graphitic carbon nitride nanoparticles led to a significant increase in the compressive strength of the core sample. In addition, the use of a gas-generating agent (a magnesium compound) in the epoxy-resin-based nanofluid led to a noticeable increase in absolute permeability.

Overall, the results obtained indicate that the developed composition can become an effective solution for sand consolidation in oil-and-gas reservoirs, providing increased compressive strength together with permeability control.

Chemical sand-control methods are mainly based on various polymer- and resin-based compositions.

In the study conducted, silica nanoparticles (nano-SiO₂) and urea-formaldehyde resin (UF) were used. Sodium hydroxide, hydrochloric acid and sodium chloride were added as needed to regulate pH and salinity. Commercial crude oil was used as the oil phase [7].



Graph 1. Effect of nanoparticles and the gas-generating agent on the compressive strength and permeability of the sand-pack formation model

To confirm the effectiveness of the developed composition, a series of experiments on sand-pack formation models was carried out. The maximum permeability reduction of treated samples (16.1%) was observed upon injection of a 12% UF + 0.5% nano-SiO₂ solution. In this case, the compressive strength value was also at its maximum, at 2,457 psi. Overall, a decrease in permeability and an increase in compressive strength were observed with increasing working-agent concentration (Table 1).

Thus, a composition was proposed for combating sand production that provides fairly high compressive-strength values for treated samples with only a slight drop in permeability compared with conventional methods. The presence of bentonite in the formation significantly increases the effectiveness of the cementing solution.

The advancement of science and new scientific developments are improving year on year and showing increasingly promising results in the field of hydrogel applications. This study presents an analysis of the possibility of using a gel-forming composition to combat sand production, based on the results of laboratory studies [9].

Table 1. Effect of nanoparticle concentration on compressive strength and permeability reduction of treated samples

UF solution, %	Nano-SiO ₂ , %	Compressive strength, psi	Permeability reduction, %
6	0.1	819	4.3
9	0.1	1378	9.8
12	0.1	1685	13.0
6	0.2	875	5.2
9	0.2	1426	11.3
12	0.2	1794	14.8
6	0.5	1127	5.8
9	0.5	1864	12.6
12	0.5	2457	16.1

To study the effectiveness of the proposed composition, the experimental part was divided into the following three main directions:

- Studying the effect of crosslinker concentration on gelation time at various concentrations of the polymer component.
- Studying the rheological properties of the proposed gel-forming composition.
- Studying the isolating properties of the proposed gel-forming composition using sand-pack formation models as an example.

The critical polymer concentration required for the formation of a continuous gel structure was determined by tracking the viscosity dynamics of two groups of gel samples with crosslinker-to-polymer mass fractions of 0.25 and 0.5 wt.%, at various polymer concentrations from 2,000 to 14,000 ppm (Table 2). All measurements were carried out at a shear rate of 5 s^{-1} . As the results show, regardless of the crosslinker concentration, a polymer concentration of 5,000 ppm is the minimum required for gelation to begin.

Table 2. Dynamics of viscosity change of the gel solution

Polymer concentration, ppm	Crosslinker concentration – 0.25 wt.%	Crosslinker concentration – 0.5 wt.%
2000	3.2	2.1
4000	3.0	4.1
5000	10	9
8000	12	11
10000	13	12
12000	14	13
14000	15.6	14.2

To study the effect of various polymer and crosslinker concentrations on the rheological properties of the gels under investigation, four samples were selected (Table 3).

Absolutely all of the samples studied demonstrated non-Newtonian, or more precisely pseudoplastic, behavior.

It should be noted that pseudoplasticity is a desirable outcome when developing a hydrogel composition.

Table 3. Dynamics of viscosity change as a function of shear rate

Shear rate, 1/s	Viscosity – Sample 1, Pa·s	Viscosity – Sample 2, Pa·s	Viscosity – Sample 3, Pa·s	Viscosity – Sample 4, Pa·s
25	8.16	9.09	8.37	8.45
50	4.36	3.89	4.28	5.26
75	2.48	2.83	2.39	2.58
100	2.17	2.41	2.22	2.12
125	1.97	2.08	2.13	2.05
150	1.84	1.95	1.86	1.68
200	1.79	1.88	1.84	1.66

At the next stage, the gel was displaced at various pump-rate values while simultaneously measuring sand-production values. The results obtained showed a substantial reduction in sand production — by almost a factor of 10. The mechanism of action of the hydrogel is apparently based on the formation of a thin polymer film on the rock surface, which increases its cementation and resistance to erosion. An additional positive effect is a more significant drop in water permeability compared with oil permeability, which also reduces the water cut of the produced fluid. Thus, laboratory experiments showed a reduction in water permeability of more than 80-fold, and in oil permeability of no more than 8-fold (Table 4).

In this study, a composition was developed for consolidating weakly cemented rocks and reducing sand production with the following formulation: 0.5 mass fraction of crosslinker, 9,000 ppm polymer concentration.

The developed compositions exhibit pseudoplastic behaviour and can reduce sand production by up to a factor of 10, while showing a greater reduction in water permeability compared with oil permeability. For the developed gel composition, these values were 80-fold and 8-fold, respectively.

Experimental studies confirmed the effectiveness of incorporating SiO₂ and g-C₃N₄ nanoparticles, which increase compressive strength without significantly impairing permeability.

Table 4. Effect of the hydrogel on permeability and sand production

Stage	Relative water permeability, mD	Relative oil permeability, mD	Sand production at 1 pore volume of water injected, g	Sand production at 2 pore volumes of water injected, g
Before hydrogel injection	91	108	1.36	3.28
After hydrogel injection	1.1	12.6	0.12	0.31

The effect of nanoparticle and gas-generating-agent concentration on the filtration and storage properties of sand reservoirs was studied. It was demonstrated that the use of nanofluids based on epoxy resin and carbon nitride promotes the formation of a strong structure with controllable permeability.

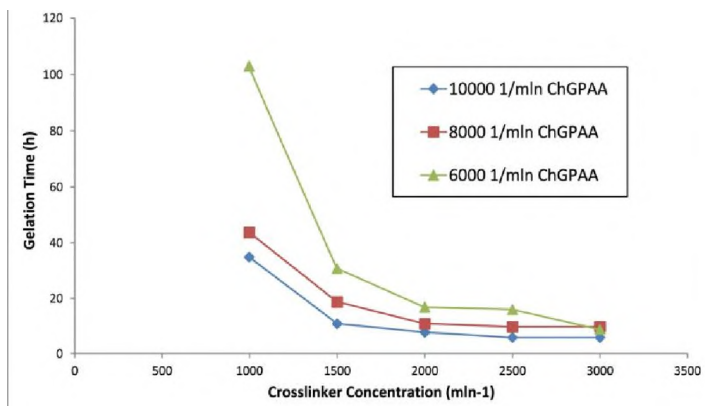
Hydrogel compositions with controllable rheological properties, used for sand-production isolation, were additionally studied. Experiments on formation models showed that the use of gel-forming systems can substantially reduce sand production while providing selective reduction of water permeability, which helps reduce the water cut of produced fluid.

The research results demonstrate the promise of the proposed compositions for application in weakly cemented reservoirs, providing effective stabilization of the bottomhole zone and extending well service life.

The third chapter examines the development of new compositions for water shut-off isolation in oil and gas wells. Various isolation methods, including mechanical and chemical ones, are investigated. Particular attention is paid to gel systems possessing high selectivity of action. A gel composition based on partially hydrolyzed polyacrylamide (HPHA) with adjustable gelation time, providing isolation of water-saturated zones, is presented. The adjustable

gelation time allows this composition to be used both for near-wellbore treatments and as a diverting agent in deep formation zones [1, 6]. Gelation time is controlled by varying the concentrations of the polymer and the crosslinking agent. There is a direct relationship between crosslinker concentration and gelation time, i.e., a decrease in crosslinking-agent concentration reduces the rate of gelation, while an increase raises it.

The mechanism of action of the crosslinker is based on the fact that potassium dichromate and thiourea form a complex structure with the carboxylate groups of the partially hydrolyzed polyacrylamide, forming three-dimensional gel networks. Polymer concentration also has a significant effect on the physical properties of the gel and on gelation time (Graph 2). In this case, however, the relationship is inverse: as polymer concentration decreases, gelation time increases. This effect is apparently related to a decrease in the number of crosslinking points in the solution. A thermogravimetric analysis of the gel samples was carried out, revealing parameters of their thermal stability and bound-water content.



Graph 2. Effect of crosslinking-agent concentration on gelation time

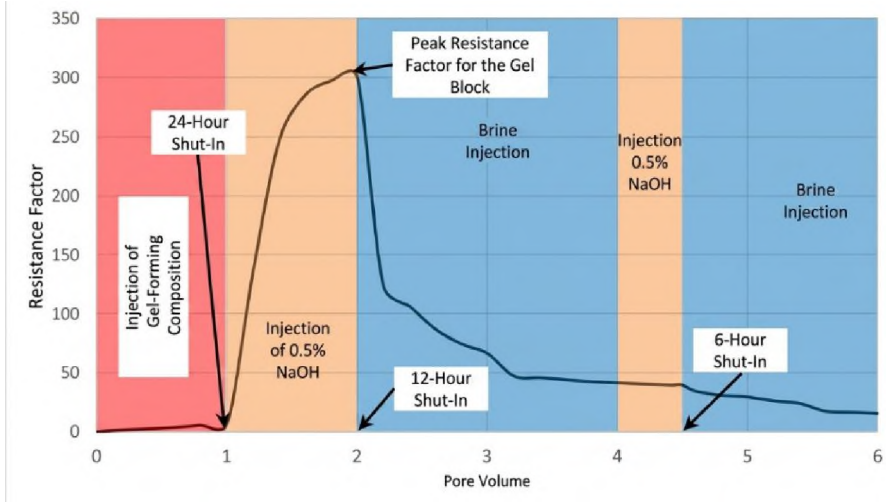
Experiments on core material from the Azeri-Chirag-Guneshli field confirmed a permeability reduction of 80.5%, proving the effectiveness of the composition for water shut-off. Thus, the resulting gel system is a fairly effective agent for reducing the water cut of

produced fluid and sealing water-saturated zones of the formation. The residual resistance factor obtained, together with the ability to control gelation time over a wide range, allows this system to be considered not only as a water-shut-off composition but also as an agent for redirecting filtration flows in deep zones of the formation.

A new water shut-off isolation technology has been developed using a temporary plugging agent together with a gel-forming composition. The proposed method involves a three-stage process: creating a filter cake, injecting the gel, and subsequently restoring the permeability of the oil-saturated zone. The technology involves the development and injection of three different fluids through a fractured core in a specific sequence. The first fluid consists of suspended microfibrinous particles in a viscous solution. It is injected to create an impermeable filter cake on low-permeability oil-bearing zones while allowing fluid to pass through the fractures. The second fluid is a crosslinked polymer gel, which is injected immediately after the first fluid at a pressure below the fracturing pressure of the formation. The gel selectively penetrates and seals only the fractures, since the low-permeability zones of the core are already protected by the filter cake. The third fluid contains an enzyme that breaks down the filter cake, restoring the permeability of the oil-saturated zones. The proposed method significantly reduces the risk of damage to oil-saturated zones of the formation and ensures the injection of a sufficient amount of gel into the fractures to provide long-term effectiveness of the gel barrier. The developed water shut-off control technology presented in this study is a low-cost and effective solution for fractured reservoirs. The technology is particularly effective where there is a significant permeability contrast between the oil-bearing zone and the water-conducting fractures.

The mitigation of formation damage caused by silicate gels used for water shut-off isolation was investigated [4]. Weight-loss and sand-pack-model waterflooding experiments showed that a 1% NaOH solution effectively dissolves bulk gel plugging, restoring the permeability of the porous medium. The effectiveness of strong alkalis for permeability restoration was confirmed through filtration tests carried out under reservoir conditions (permeability of 1,200 mD and

temperature of 60 °C). Instead of directly measuring permeability, the ratio of injection pressure to the injected pore volume of fluid was monitored (Graph 3).



Graph 3. Results of experiments on the sand-pack formation model

The final results show that approximately 95% of the gel plug was ultimately removed, and the final RF was 15.7. It should also be noted that, under field conditions, alkali injection can be repeated several times until formation damage is completely eliminated. This technology demonstrates numerous advantages, including the use of cost-effective chemicals, ease of implementation, and no need for complex backwashing procedures. The developed method can be used to adjust gel systems and eliminate their residual effects in the formation.

A colloidal-silica-based gel system adapted for high formation temperature conditions has been developed [3].

To activate the process, common inorganic chlorides and salts were used, including NaCl, KCl, NH₄Cl, CaCl₂, NaNO₃ and Na₂SO₄. Gelation parameters were studied as a function of salt concentration, temperature, and anion type. Laboratory studies showed that the composition possesses high stability and controllable gelation time,

allowing its application under complex reservoir conditions. The concentration of silica (SiO_2) significantly affects gelation time at 50 °C. At a constant temperature, a clear trend of increasing gelation time with decreasing SiO_2 concentration is observed, while the salinity of the medium remains constant. The study also examined the effect of various cations on the gelation process of colloidal silica using different inorganic chlorides.

At a temperature of 50 °C, all of the chloride salts tested initiated the gelation of the silica gel. Notably, gelation time decreased with increasing chloride-salt concentration. This trend arises from the interaction between the negatively charged surface of the alkaline colloidal-silica nanoparticles and the introduced inorganic chlorides. These chlorides act as counter-ions (Na^+ , K^+ , Mg^{2+} and Ca^{2+}), penetrating the electrical double layer of the nanoparticles, reducing the zeta potential and destabilizing the colloidal system. This destabilization leads to gelation, an effect that is enhanced at higher salt concentrations.

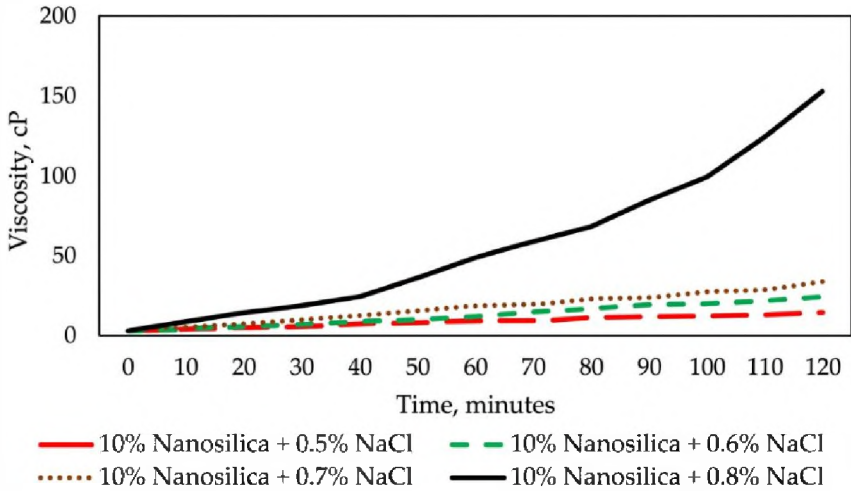
Different cations have different effects on gelation time. Divalent cations, particularly Ca^{2+} and Mg^{2+} , cause “instantaneous” gelation, distinguishing them from other monovalent cations. This behaviour can be explained by the ability of multivalent cations, such as divalent Ca^{2+} , to precipitate surface SiO_3^{2-} ions. This reduction in surface charge, together with the formation of insoluble particles as a result of OH^- ion reactions, significantly weakens the repulsive forces that stabilize colloidal systems, thereby shortening gelation time.

Temperature plays a decisive role in determining the gelation time of colloidal nano-silica. As temperature rises, the system's zeta potential decreases, making the colloidal system less stable and triggering the gelation process. Analysis of the test results revealed a clear pattern: gelation time decreases substantially with increasing temperature.

The main goal of this study was to develop a water shut-off-limiting composition based on colloidal nano-silica, adapted for high-temperature formations. A relatively modest increase in temperature (from 20 to 80 °C) resulted in a substantial reduction in gelation time.

In addition, activator concentration proved to be another important factor influencing the gelation kinetics of colloidal silica.

In the context of applying this composition under high-temperature reservoir conditions, sensitivity to the salinity of the medium is of paramount importance. To this end, rheometric tests were carried out at a temperature of 110 °C using NaCl as an activator. The results obtained are presented in Graph 4.



Graph 4. Gelation dynamics at elevated temperatures

Notably, even small changes in NaCl concentration, such as 0.1%, have a substantial effect on gelation time at high temperatures such as 110 °C. These results underscore the exceptional importance of a careful approach when introducing activators during the preparation of these compositions under field conditions.

The study focused on developing a working agent for water shut-off limitation based on colloidal silicon dioxide, intended for application in high-temperature reservoir conditions. The developed system has low viscosity, ensuring deep penetration into the formation and high application efficiency.

As a result of the study, the following conclusions were drawn:

- higher temperatures, increased salt concentration, and higher silica concentration accelerated gelation;
- the concentration of the activating salt had a significant effect on gelation time, underscoring its role in the gelation process;
- different activator ions affect gelation differently, primarily due to their charge density and size;
- the study revealed the system's sensitivity to even small changes in salt concentration, particularly at elevated temperatures.

Based on these results, it was proposed that, when using silica gels in formations characterized by high formation-water salinity, it is advisable to inject a low-salinity water pad to prevent premature gelation.

A nanocomposite polymer composition for high-temperature water shut-off has been proposed. Experiments confirmed that adding nano-silica increases gel strength, reduces its syneresis, and increases bound-water content, which improves its long-term stability. Studies on core material demonstrated high efficiency of the composition in blocking water at a temperature of 105 °C.

The results obtained confirm the promise of the developed compositions for water isolation in oil and gas fields, making it possible to substantially reduce the level of produced-fluid water cut and increase the operating efficiency of wells.

The fourth chapter of the dissertation presents the results of implementing the developed compositions for consolidating the bottomhole formation zone and isolating water breakthrough.

Field trials of the gel-forming composition for consolidating the bottomhole formation zone were carried out at the Verkhne-Shapshinskoye field. The problem of sand production from the formation into producing wells is one of the most pressing issues. This process leads to a decline in well production rate, equipment damage, and increased operating costs. The application of bottomhole-zone consolidation technologies, such as polymer systems, makes it possible to minimize sand production and increase the operational stability of wells. Field trials conducted at the field confirm the effectiveness of using new approaches, including polymer systems and combined methods of formation treatment.

Field trials of the developed composition were carried out in August 2023 at pad No. 5 of the Verkhne-Shapshinskoye field on wells No. 1004, 1007 and 1009. The purpose of this technology was to prevent sand production, increase rock stability, and increase the oil recovery factor. As a result of implementing these technological measures on wells No. 1004, 1007 and 1009, an increase in additional oil production was achieved. The developed composition, based on polyacrylamide and chromium triacetate, made it possible to reduce sand production and increase oil recovery by 125 tons.

At the Urengoy OGCF, a selective water isolation technology using polyacrylamide, polyethyleneimine and thiourea has been implemented. Pilot field tests of the developed composition for selective water isolation were carried out on gas-condensate wells No. 1325 (pad 1-57) and No. 1350 (pad 49, UKPG 1-AB) of the Urengoy OGCF. The operations were carried out in June 2023 in connection with the need for selective isolation of water-bearing horizons following an unsuccessful hydraulic fracturing treatment (HF). As a result of implementing the technology, by February 2024 an increase in oil production of 460 tons had been achieved, together with a reduction in water production of 3,659 m³. The tests showed a reduction in water production of 3,659 m³ and an increase in oil production of 460 tons. The developed compositions demonstrated high efficiency in controlling filtration flows and reducing produced-fluid water cut.

CONCLUSIONS AND RECOMMENDATIONS

1. Compositions for consolidating the bottomhole formation zone of wells:

- An epoxy resin composition modified with graphitic carbon nitride nanoparticles (particle size 45–75 nm) has been developed to increase mechanical strength and thermal stability. Laboratory tests showed that the introduction of 0.5% nanoparticles increases the elastic modulus by 37% and thermal stability by 21% compared with the unmodified composition. Tests on porous media with a permeability of 120–160 mD confirmed a 59% reduction in the migration of mechanical particles, demonstrating the effectiveness of the composition for consolidating the bottomhole zone of wells. [5]

- A urea-formaldehyde resin composition containing silica nanoparticles (size 20–50 nm) has been developed to improve adhesive properties and resistance to mechanical loads. Laboratory studies showed that the addition of 0.5% nanoparticles increases material strength by 42% and resistance to thermal degradation by 18%. Experiments on sandstone samples with a permeability of 150–200 mD confirmed a 67% reduction in porous-medium erosion after treatment with this composition. [7]

- A polymer composition based on a copolymer of 2-acrylamido-2-methylpropane sulfonic acid (AMPS) with a molecular weight of 8 million Da, crosslinked with chromium triacetate (concentration 5 g/L), has been developed. Tests were carried out on cores with a permeability of 150–200 mD. It was determined that injecting a 15% gel-forming solution reduces solid-particle migration by 64% and increases core mechanical strength by 29%. [9]

2. Compositions for limiting water shut off into wells:

- A gel-forming composition for water isolation based on partially hydrolyzed polyacrylamide (PHPA) with a molecular weight of 5–6 million Da has been developed and optimized. A potassium dichromate/thiourea system was used as the crosslinking agent ($K_2Cr_2O_7$ concentration – 3,000 ppm). Laboratory tests on cores from the Azeri-Chirag-Guneshli (ACG) field showed a reduction in sample permeability from 181.5 mD to 35.4 mD (by 80.5%). The residual

resistance factor (RRF) was 5.1. Gelation time varied from 6 to 103 hours depending on crosslinker concentration. [1]

- A high-temperature gel system based on colloidal silica, stable at temperatures up to 110 °C, has been developed. It was determined experimentally that increasing the SiO₂ concentration from 15% to 35% reduces gelation time from 26 hours to 1 hour. At a NaCl content of 5%, gelation occurred instantaneously, whereas at 2% NaCl it took 5.5 hours. Studies on a porous medium with a permeability of 500–700 mD confirmed a high residual resistance factor (RRF = 15.7) after a 24-hour holding period at 110 °C. [3]

- A nanocomposite polymer composition based on polyacrylamide (0.4%), polyethyleneimine (0.65%), and 1% nano-silica (particle size 28.6 nm) has been developed. Core waterflooding experiments (SWG field, permeability 130 mD) showed a 58% increase in the residual resistance factor (RRF) compared with a control sample without nanoparticles. DSC analysis confirmed a reduction in free-water content and increased gel stability. [8]

3. Methods for the following processes:

- The effect of silicate gels on rock permeability was studied for the first time. Laboratory experiments demonstrated that a 1% NaOH solution effectively destroys the silicate gel, reducing the residual resistance factor from 300 to 15.7. Waterflooding experiments on a sand-pack formation model (permeability 1,200 mD, temperature 60 °C) showed that NaOH removes up to 95% of gel plugging after a 12-hour holding period. [4]

- An innovative method for water isolation has been developed using a temporary plugging agent together with a gel-forming composition. Laboratory experiments used a suspension of cellulose fibres (3%) in a xanthan-polymer solution (0.5%) to create a filter cake preventing gel penetration into oil-bearing zones. After injecting the polymer gel (based on PHPA) followed by enzymatic breakdown of the filter cake, the restored core permeability was 92% of the original value. [2]

- As a result of implementing the developed compositions for consolidating the bottomhole formation zone and isolating water shut-off, the following was achieved:

- Field trials on wells No. 1004, 1007 and 1009 of the Verkhne-Shapshinskoye field confirmed the effectiveness of the developed composition for stabilizing sand reservoirs. Injecting 15–20 m³ of polymer solution followed by displacement with 8–10 m³ of buffer fluid reduced solid-particle production by 61% and increased the mechanical strength of the bottomhole zone by 39%. After treatment, a 54% reduction in mechanical-impurity content in production was observed, which extended the service life of pumping equipment and reduced the frequency of repair work. As a result, additional oil production over the observation period amounted to 125 tonnes, and the duration of the treatment effect without the need for repeat operations reached 7 months.

- Selective water shut-off isolation operations using the developed gel-forming composition were carried out on wells No. 1325 and 1350 of the Urengoy OGCF. Injecting 15–20 m³ of solution provided effective sealing of water-bearing zones and reduced produced-fluid water cut. Monitoring results showed that the volume of produced water decreased by 3,659 m³, while hydrocarbon production increased by 460 tons over the observation period. An additional increase in the hydrocarbon recovery factor and a reduction in operating costs — due to reduced requirements for produced-water separation and disposal — were also noted.

The main content and results of the dissertation have been published in the following works:

1. Shovgenov, A. D. Gel composition for water shut-off isolation / A. D. Shovgenov // SOCAR Proceedings, – 2020. No. 3, – pp. 59–71.

2. Veliyev, E. F., Shovgenov, A. D. Novel water shut-off method based on temporary plugging agent and gel composition shut-off / E. F. Veliyev, A. D. Shovgenov // SOCAR Proceedings, – 2023. No. SI1, – pp. 96–101.

3. Veliyev, E. F., Shovgenov, A. D., Mehdiyev, B. R. Assessing silica-based gel system for high-temperature water shut-off applications / E. F. Veliyev, A. D. Shovgenov, B. R. Mehdiyev // SOCAR Proceedings, – 2023. No. SI2, – pp. 26–32.

4. Veliyev, E. F., Shovgenov, A. D., Mehdiyev, B. R. A study on the mitigation of formation damage caused by excessive silica-based gel application in water shut-off treatments / E. F. Veliyev, A. D. Shovgenov, B. R. Mehdiyev // SOCAR Proceedings, – 2023. No. SI2, – pp. 33–38.

5. Veliyev, E. F., Shovgenov, A. D. Enhancing sand control in loose reservoirs: nanofluid application / E. F. Veliyev, A. D. Shovgenov // SOCAR Proceedings, – 2024. No. SI1, – pp. 6–11.

6. Shovgenov, A. D. Gel composition for water shut-off isolation / A. D. Shovgenov // Ashirov Readings, – 2020. No. 1(12), – pp. 124–131.

7. Shovgenov, A. Application of a Chemical Composition with Nanoparticle Additives for Sand Control // V International Scientific Conference of Young Researchers. – Baku, – April 29–30, 2020, – pp. 268–271.

8. Suleymanov, A. B., Shovgenov, A. D. Nanocomposite polymer composition for water shut-off treatment at high formation temperature // SPE Annual Caspian Technical Conference, – Virtual: October, – 2021. SPE-207066-MS.

9. Shovgenov, A. D. Application of a gel-forming composition for combating sand production // Bulatov Readings: Proceedings of the V International Scientific-Practical Conference, – Krasnodar: –31 March, 2021, – pp. 277–280.

Personal contribution of the applicant

Works [1, 6, 7, 9] were carried out independently;
in works [2–5, 8], the applicant participated in problem
formulation, conducting the research, and summarizing the results.

A handwritten signature in blue ink, appearing to read 'Aliper', is centered on the page.

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