minerals

MDPI

Article

Selection of Optimal Parameters for Chemical Well Treatment During In Situ Leaching of Uranium Ores

Kuanysh Togizov, Zhiger Kenzhetaev, Akerke Muzapparova *, Shyngyskhan Bainiyazov, Diar Raushanbek and Yuliya Yaremkiv

Department of Geophysics, Satbayev University, 22 Satbayev Str., Almaty 050013, Kazakhstan; k.togizov@satbayev.university (K.T.); kzhiger@yahoo.com (Z.K.); bayniyazov87@mail.ru (S.B.); draushanbek16@gmail.com (D.R.); yuliyayaremkiv89@gmail.com (Y.Y.)

* Correspondence: a.muzapparova@satbayev.university

Abstract

The aim of this study was to improve the efficiency of in situ uranium leaching by developing a specialized methodology for selecting rational parameters for the chemical treatment of production wells. This approach was designed to enhance the filtration properties of ores and extend the uninterrupted operation period of wells, considering the clay content of the productive horizon, the geological characteristics of the ore-bearing layer, and the composition of precipitation-forming materials. The mineralogical characteristics of ore and precipitate samples formed during the in situ leaching of uranium under various mining and geological conditions at a uranium deposit in the Syrdarya depression were identified using an X-ray diffraction analysis. It was established that ores of the Santonian stage are relatively homogeneous and consist mainly of quartz. During well operation, the precipitates formed are predominantly gypsum, which has little impact on the filtration properties of the ore. Ores of the Maastrichtian stage are less homogeneous and mainly composed of quartz and smectite, with minor amounts of potassium feldspar and kaolinite. The leaching of these ores results in the formation of gypsum with quartz impurities, which gradually reduces the filtration properties of the ore. Ores of the Campanian stage are heterogeneous, consisting mainly of quartz with varying proportions of clay minerals and gypsum. The leaching of these ores generates a variety of precipitates that significantly reduce the filtration properties of the productive horizon. Effective compositions and concentrations of decolmatant (clog removal) solutions were selected under laboratory conditions using a specially developed methodology and a TESCAN MIRA scanning electron microscope. Based on a scanning electron microscope analysis of the samples, the effectiveness of a decolmatizing solution based on hydrochloric and hydrofluoric acids (taking into account the concentration of the acids in the solution) was established for the destruction of precipitate formation during the in situ leaching of uranium. Geological blocks were ranked by their clay content to select rational parameters of decolmatant solutions for the efficient enhancement of ore filtration properties and the prevention of precipitation formation. Pilot-scale testing of the selected decolmatant parameters under various mining and geological conditions allowed the optimal chemical treatment parameters to be determined based on the clay content and the composition of precipitates in the productive horizon. An analysis of pilot well trials using the new approach showed an increase in the uninterrupted operational period of wells by 30%–40% under average mineral acid concentrations and by 25%–45% under maximum concentrations with surfactant additives in complex geological settings. As a result, an effective methodology for ranking geological blocks based on their ore clay content and precipitate composition was developed to determine the rational parameters of decolmatant solutions, enabling a maximized filtration performance

check for updates

Academic Editor: Jean-François Blais

Received: 11 June 2025
Revised: 24 July 2025
Accepted: 25 July 2025
Published: 31 July 2025

Citation: Togizov, K.; Kenzhetaev, Z.; Muzapparova, A.; Bainiyazov, S.; Raushanbek, D.; Yaremkiv, Y. Selection of Optimal Parameters for Chemical Well Treatment During In Situ Leaching of Uranium Ores. *Minerals* **2025**, *15*, 811. https://doi.org/10.3390/min15080811

Copyright: © 2025 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/licenses/by/4.0/).

and an extended well service life. This makes it possible to reduce the operating costs of extraction, control the geotechnological parameters of uranium well mining, and improve the efficiency of the in situ leaching of uranium under complex mining and geological conditions. Additionally, the approach increases the environmental and operational safety during uranium ore leaching intensification.

Keywords: uranium; uranium leaching; precipitation formation; X-ray diffraction analysis; microscopic studies; block ranking; pilot-scale testing; ore clay content

1. Introduction

The consequences of climate change and the trend of increasing electricity demands in developing countries are driving growing interest in the further development of nuclear energy [1,2]. Efforts focused on the design and commissioning of new modular nuclear power plants, both small- and large-scale, featuring compact dimensions and low carbon footprints, can have a significant impact on the reduction in CO_2 emissions worldwide. Uranium is the key element for the sustainable development of nuclear energy across the globe [3,4]. Kazakhstan's uranium mining industry, based on highly efficient in situ recovery (ISR) technology, is capable of making a substantial contribution to meeting the growing demand for uranium resources [5].

The in situ method of uranium mining is the most economically viable and environmentally safe under conditions of deep ore occurrence (below 300 m), where the useful component in the ore mass has a content below 0.1%–0.2%. This technology is the most efficient and does not involve high capital or operating costs during the development of the deposit [6]. The ISR technology involves dissolving the useful component in place using a moving stream of leaching solution, followed by the extraction and lifting of the formed compounds to the surface through production wells. The sulfuric acid used as a leaching reagent reacts with carbonate and clay minerals in the host rocks, clogging the productive horizon [7]. The resulting precipitates increase hydraulic resistance in the formation, reduce the filtration properties of the ores, decrease well productivity, and hinder the uranium in situ leaching processes. As a result, both the productivity and the mean time between failures (MTBFs) of wells are reduced, which lowers the utilization coefficient of the wells and the technological equipment. This increases the operation time of production blocks and, consequently, leads to the increased consumption of sulfuric acid, electricity, and other operating costs for extraction [8]. Wells in such blocks are often shut down for repair and restoration work (RRW), and there arises a need to perform additional work to restore the permeability of the near-wellbore zone (NWBZ) of the formation [9].

Well productivity directly depends on the mineralogical characteristics of the ores and the methods used to improve their filtration properties [10]. The chemical methods applied to restore the permeability of the NWBZ are based on the penetration of the reagent into the NWBZ, the dissolution and removal of the reaction products, the restoration of the original permeability, and the improvement of the well productivity and the mean time between failures [11]. Traditional chemical methods for restoring the permeability of the productive horizon under complex geological conditions at sites with low filtration properties of ores do not yield positive results. During acid treatments of the production well filter zone containing ore with low filtration characteristics, the injected decolmatizing solution penetrates into the near-filter zone of the formation through the upper part of the filter along the most permeable sections. As a result, the main lower part of the near-filter zone remains untreated, containing impermeable ore zones. The decolmatizing solution, moving through

the most permeable areas, reacts with the host rocks and becomes neutralized without interacting with the precipitate formations in the productive horizon. The undissolved precipitates enter the near-filter zone of the well and clog the filter openings, and the well productivity drops sharply. In some cases, comprehensive, multicomponent chemical treatments of wells are required using mineral acids, active additives, and surfactants (SAA). Studying the physicochemical characteristics of ores in the productive horizon will make it possible to develop rational parameters for chemical well treatment under various mining and geological conditions.

The purpose of this study was to improve the efficiency of underground in situ uranium leaching under complex geological conditions of ore occurrence, depending on the filtration properties and clay content of the ores. This involves the development of effective parameters for multifunctional chemical reagent formulations aimed at improving the filtration properties of the ores and preventing precipitation formation during uranium in situ leaching in various mining and geological settings.

The objectives of this study included determining the physicochemical characteristics of ores in the productive horizon and the composition of precipitates; analyzing characteristics and ranking technological blocks and sites in order to develop multifunctional chemical reagent formulations; conducting microscopic laboratory studies and pilot-scale well tests to select rational parameters of multifunctional chemical reagents; substantiating rational parameters for the application of innovative chemical well treatments aimed at the effective dissolution and prevention of precipitation; and developing an effective methodology for selecting optimal parameters for chemical well treatment depending on the physicochemical characteristics of the ores and precipitates in the productive horizon.

2. Materials and Methods

2.1. X-Ray Diffraction Studies of Ores

To address the issues associated with a sharp decline in well productivity during ore leaching, it is necessary to determine the mineralogical composition of the productive horizon and the structure of the host rocks. The data obtained make it possible to identify the causes of reduced solution filtration, depending on the mineralogical characteristics of ores from productive stratigraphic levels at the Syrdarya depression uranium deposit [12]. To facilitate a comparison of the physicochemical properties of the ores, core samples were collected from the Santonian, Maastrichtian, and Campanian productive horizons of the North Kharasan uranium deposit.

An X-ray diffraction (XRD) analysis was performed using an automated DRON-3 diffractometer (St. Petersburg, Russia) with CuKα radiation and a β-filter. The diffraction patterns were recorded under the following conditions: U = 35 kV, I = 20 mA, the θ–2θ scan mode, and a detector speed of 2°/min. A semi-quantitative phase analysis was conducted on powder samples using the equal-weight method and artificial mixtures. The relative proportions of crystalline phases were determined. The diffraction patterns were interpreted using the ICDD database: the PDF2 (Powder Diffraction File) and reference patterns of pure, impurity-free minerals [13,14]. X-ray diffractograms of the core samples are presented in Figure 1.

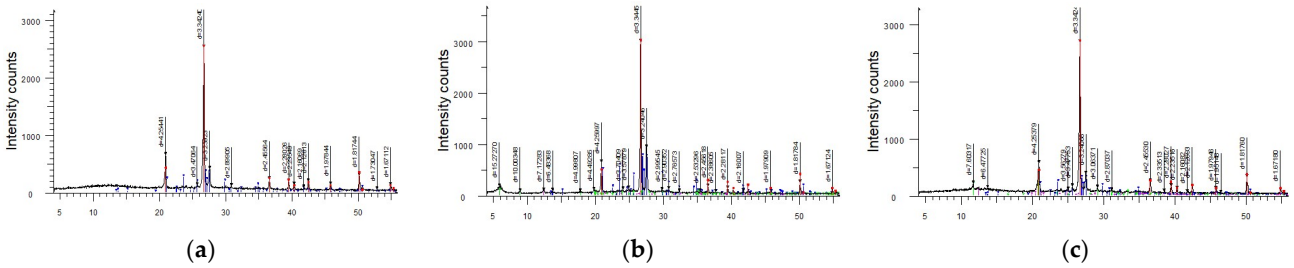


Figure 1. X-ray diffractogram of core samples corresponding to the (**a**) Santonian, (**b**) Maastrichtian, and (**c**) Campanian stages.

2.2. X-Ray Diffraction Studies of Precipitates

The main objective of the XRD studies of precipitate-forming components was to determine and compare the quantitative and qualitative characteristics of precipitates from various ore-hosting horizons of the Santonian, Maastrichtian, and Campanian productive strata under laboratory conditions. Establishing these characteristics will allow for the selection of effective approaches and chemical reagents for their breakdown, dispersion, removal, and long-term prevention of precipitate formation [15]. X-ray diffractograms of the well precipitates are presented in Figure 2.

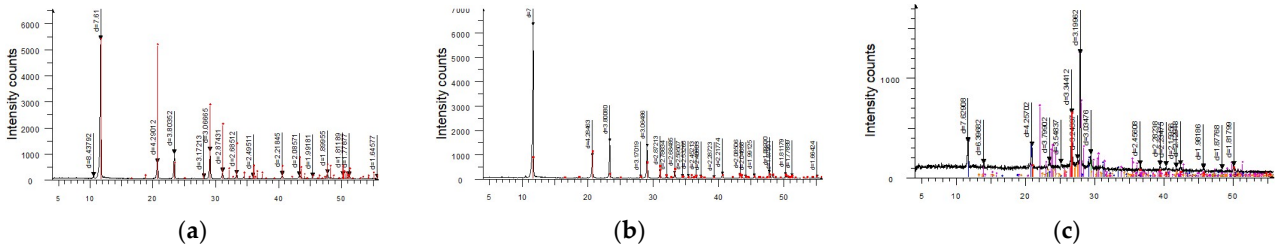


Figure 2. X-ray diffractogram of precipitates from (**a**) the Santonian stage, (**b**) the Maastrichtian stage, and (**c**) the Campanian stage.

2.3. Laboratory Experiments on Precipitate Treatment

The primary objective of the laboratory experiments was to select effective compositions and concentrations of multifunctional chemical reagents for the breakdown and prevention of precipitates during in situ uranium leaching. Precipitate samples were treated using the dropwise application method, which included drying the samples, applying drops of chemical solutions, allowing the treated samples to dry at room temperature for 24 h, and subsequently analyzing the results. Four experimental treatments were conducted: Experiment A1 involved the application of a 5%-by-weight hydrofluoric acid solution (with water as the balance), while Experiment A2 used a 2.5% hydrofluoric acid solution. Similarly, Experiment B1 employed a 5% hydrochloric acid solution, and Experiment B2 used a 2.5% hydrochloric acid solution. The samples used in these experiments were taken from wells in the Campanian horizon, chosen due to their complex structure, multicomponent mineral composition, and mechanical strength.

2.4. Microscopic Studies of Precipitates

The objective of the microscopic studies was to determine and compare the structure of precipitate-forming components before and after the laboratory experiments aimed at identifying effective chemical reagent compositions for the breakdown and prevention of precipitates during sulfuric acid uranium leaching. Surface images of the precipitates, both before and after treatment with various chemical solutions, were obtained using the TESCAN MIRA 3 FEG-SEM—a high-resolution analytical scanning electron microscope

designed for a wide range of research tasks and submicron-level quality control [16]. The SEM TESCAN MIRA is equipped with an electron column featuring a Schottky field emission cathode. The energy range of the electron beam striking the sample extends from 200 eV to 30 keV (down to 50 eV with the beam deceleration technology (BDT) option). An electromagnetic lens is used to adjust the beam current, which can be continuously regulated within a range of 2 pA to 400 nA. The maximum field of view exceeds 8 mm at a working distance (WD) of 10 mm and over 50 mm at the maximum WD. The resolution of the electron column in high vacuum mode is 1.2 nm at 30 keV using the secondary electron (SE) detector, 3.5 nm at 1 keV using the in-beam SE detector, and 1.8 nm at 1 keV with the BDT option.

2.5. Ranking of Technological Blocks by Area

The development of rational parameters for multifunctional chemical reagent formulations used for the treatment of wells with low filtration characteristics involves ranking the sites and blocks based on the clay content coefficient of the ores and the composition of precipitates. This allows for the selection of appropriate reagent concentrations during well repair and restoration (RRW). As an example, Figure 3 presents a schematic overview of the technological blocks from the second area of the North Kharasan deposit, showing the classification according to the clay content, filtration, and carbonate content of the productive horizon.

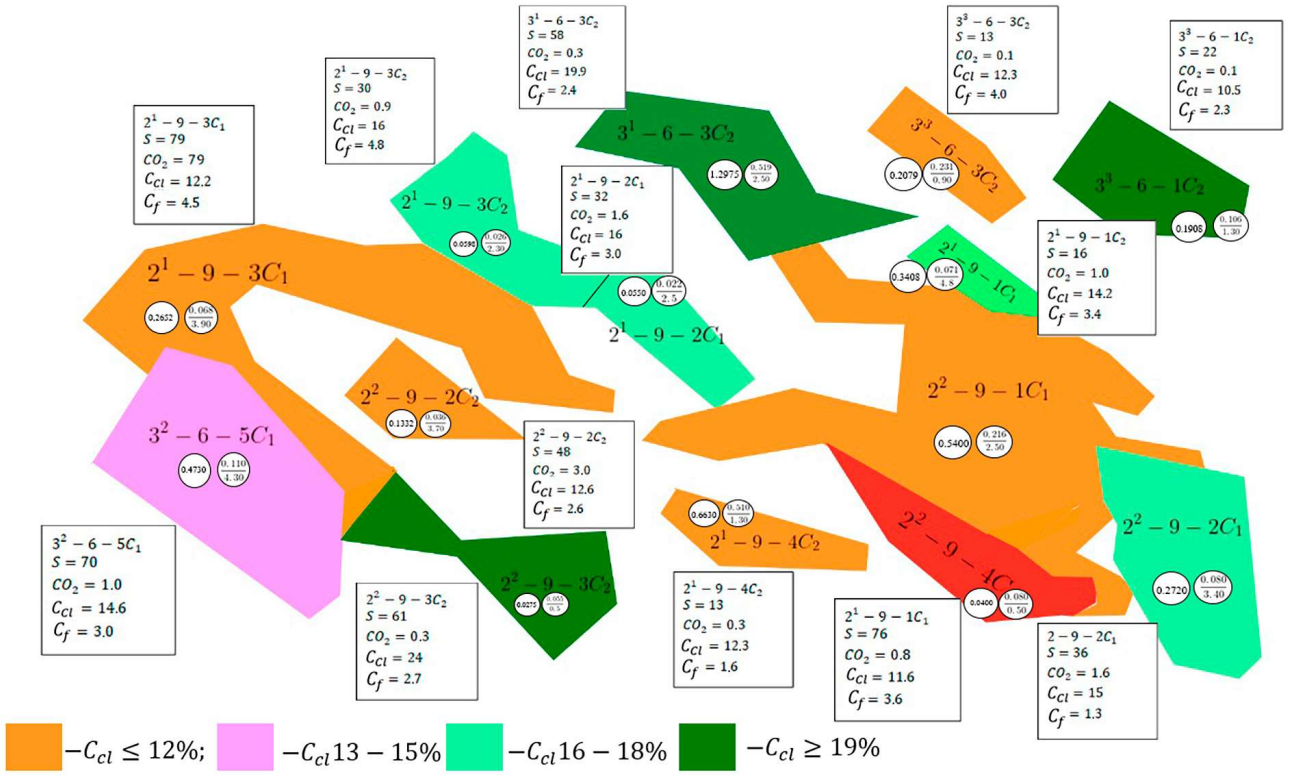


Figure 3. Overview diagram for ranking technological blocks according to the clay content of the productive horizon.

As shown in Figure 3, the technological blocks of the second area consisted of productive horizons from the Santonian, Maastrichtian, and Campanian stages, delineated by red, blue, and green lines, respectively. For the convenience of development and maintenance operations, the authors propose a method for ranking blocks based on the clay content coefficient of the ores in the productive horizon. The blocks are categorized into zones

with minimal, moderate, high, and maximum clay content. Orange indicates blocks with a clay content ranging from 0 to 12% in the productive horizon, red represents blocks with 13%–15%, light green denotes 16%–18%, and dark green indicates 19%–22%.

The rationale behind ranking the blocks by clay content lies in the relatively greater influence of clay minerals on the geotechnological parameters of the block and the difficulty of mitigating their effects. The empirical data from blocks with a low clay content (0%–12% or 13%–15%) show almost no delays in development, while blocks with a high clay content (16%–18% or above 19%) experience performance lags relative to design targets. The primary reason for the reduced performance in ores with a higher clay mineral content is the significant decrease in filtration properties, which disrupts the technological regimes of block development. The interaction of sulfuric acid solutions with the host rocks in the productive horizon leads to the swelling of smectite and kaolinite clays, resulting in reduced filtration properties. This causes a decline in the production well yield and the injection well acceptability due to the clogging of the productive horizon, ultimately lowering well utilization rates because of the increased frequency of repair and restoration work (RRW).

The zoning of blocks based on these characteristics allows for the selection of effective concentrations and volumes of multifunctional chemical reagent complexes to dissolve and prevent precipitate formation during uranium in situ leaching.

2.6. Development of a Well Chemical Treatment Method

The development of a new chemical treatment method for wells enables the direct dissolution of precipitates and prevents dilution due to the preferential flow of reagents through permeable rock layers. The use of multifunctional chemical reagent solutions must be carried out according to a specially designed method using dedicated technological equipment, while strictly adhering to occupational safety regulations [17]. The innovative method involves treating the filter zone of the well with specially developed decolmatant solutions, which ensure the maximum breakdown and long-term prevention of precipitate formation [18]. In addition, this approach reduces the specific consumption of sulfuric acid, electricity, labor, and other operational costs during well-based uranium extraction from diverse geological blocks [19].

Figure 4 presents the developed workflow diagram for intensifying uranium recovery via well mining.

As shown in Figure 4, the main accumulation of precipitates (3) occurs within the productive horizon (1), directly in the discharge zone of the leaching solutions, where the flow velocity increases between injection wells (5) and production wells (4). Chemical treatment using a complex of chemical reagents involves the preparation of solutions using specialized equipment (6), followed by delivery through a pressurized hose (7) to the filter zone of the production wells (4). The specially formulated solution is supplied from a storage tank (8) and enters the near-filter zone of the well, where it spreads through capillaries and pores, interacts with the precipitates, and dissolves them, thereby enhancing the filtration properties of the ores. The reaction products are pumped out of the well, allowed to settle, filtered, and returned to the circulation cycle of the leaching solution.

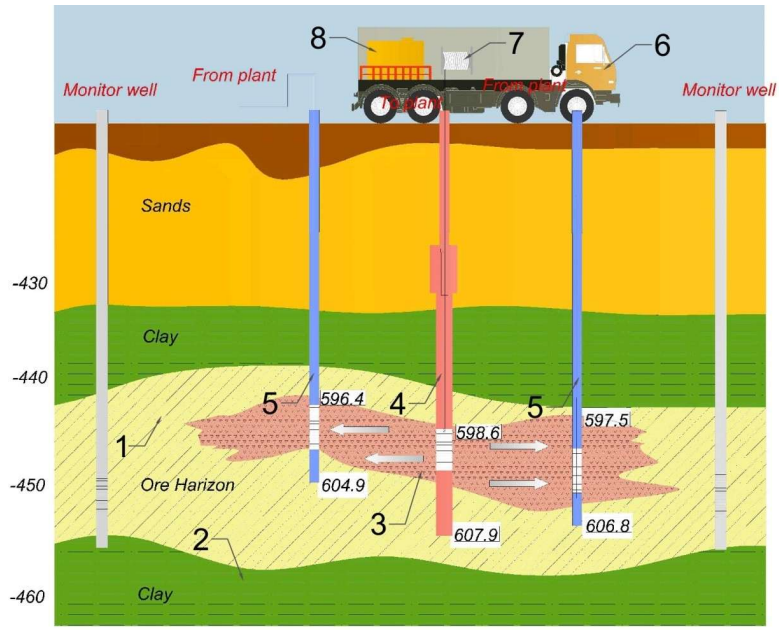


Figure 4. Scheme for the intensification of downhole uranium production: 1—productive horizon; 2—impenetrable rocks; 3—sedimentation in the NZF; 4—pumping wells; 5—injection well; 6—equipment for chemical treatments; 7—pressure hose; and 8—tank.

Figure 3 shows the inflow profile distribution along the filter column, depending on the filtration properties of the ores in the near-wellbore zone of the formation. These data were obtained using a geophysical method known as well flowmetry. This approach enables the assessment of filtration properties along the entire length of the filter. As illustrated, the main volume of the solution flows through the upper section of the filter, while the injectivity of the lower section is significantly lower due to the swelling of clay minerals, gravitational effects, the clogging of the near-wellbore zone, and other factors. The use of a multifunctional reagent composition for chemical well treatment helps reduce the permeability in already-washed areas and redirects the decolmatant solution toward the lower part of the filter column. As a result, the solution penetrates low-permeability zones, reacts with precipitates, dissolves them, and creates or enlarges pore channels. The multifunctional reagent composition also interacts with insoluble fine and ultra-fine particles in the near-wellbore zone, binding them together to form larger, permeable aggregates. This combined effect dissolves precipitates and converts insoluble fines into larger particles, thereby increasing the permeability of the formation under complex geological conditions.

2.7. Experimental Field Trials on Uranium ISR Wells

Chemical methods used in RRW aim to dissolve and remove precipitates formed as a result of the interactions between leaching solutions and the host rocks of the productive horizon. The use of hydrofluoric acid solutions with the addition of polymeric and binding agents during RRW is conducted in accordance with a specially developed methodology using dedicated technological equipment, and in compliance with occupational safety standards.

The experimental field trials for the chemical treatment of wells involve injecting an innovative multifunctional reagent composition into the filter section of the well, where it reacts with the precipitates. The resulting products are then removed from the well via airlift pumping. The monitoring of the well productivity and the mean time between repair cycles is carried out to assess the effectiveness of the selected treatment parameters.

The concentration and volume of hydrofluoric acid, as well as the type and proportion of polymeric and binding reagents, are selected based on the well productivity, previous treatment experience, and the clay content of the technological block. Sulfuric acid is added to the formulation to strengthen the solution, reduce the pH, and prevent the premature neutralization of the primary solvent in the productive horizon (Table 1).

Table 1. The compositions and parameters of the multifunctional decolmatant solutions.

Decolmatant Solution Composition	HF, Kg	H_2SO_4, Kg	Surfactant, Kg	Ore Clay Content Coefficient	MTBF Before, Days (HCl)	MTBF After, Days (HF)
HF + H_2SO_4	150 (2.0%)	120		0–12	31	60
HF + H_2SO_4	200 (2.5%)	120		13–15	38	55
HF + H_2SO_4 + SAA 0.5%	250 (3.2%)	120	15	16–18	34	47
HF + H_2SO_4 + SAA 1.0%	300 (4.0%)	120	30	19–22	31	58

The decolmatant solution was prepared using technical-grade hydrofluoric acid with an initial concentration of 37%, in quantities of 150, 200, 250, and 300 kg per total solution volume of 3.0 m^3. This resulted in concentrations of 2.0%, 2.5%, 3.2%, and 4.0% by total mass, respectively. Additionally, 120 kg of sulfuric acid is added, representing 4.0% of the total mass. To ensure the effective interaction and complete dissolution of the precipitates along the filter column, a surfactant (0.5%–1.0% by weight) was added to highly concentrated solutions containing hydrofluoric acid.

3. Discussion of Results

An X-ray diffraction analysis was carried out using the equipment described in the methodology section (Chapter: Methods, Section 2.1). The content of the major crystalline phases was calculated. Table 2 presents the results of the semi-quantitative X-ray diffraction analysis of core samples. Table 3 provides the results of the semi-quantitative X-ray diffraction analysis of precipitate samples.

Table 2. Results of semi-quantitative X-ray diffraction analysis of core material samples.

Mineral	Formula	Santonian Stage, %	Maastrichtian Stage, %	Campanian Stage, %
Quartz	SiO_2	90.8	54.7	66.3
Smectite	$KAl_2(AlSi_3O_{10})$ $(OH)_2$	-	27.0	-
K-Feldspar	$KAlSi_3O_8$	9.2	10.1	5.7
Kaolinite	$Al_2(Si_2O_5)$ $(OH)_4$	-	6.7	11.6
Gypsum	$CaSO_4$ 2 (H_2O)	-	-	16.4

Table 3. Results of semi-quantitative X-ray diffraction analysis of crystalline phases in precipitate samples.

Mineral	Formula	Santonian Stage, %	Maastrichtian Stage, %	Campanian Stage, %
Quartz	SiO_2	-	5.1	35.6
Gypsum	$CaSO_4$ 2 (H_2O)	100	88	16.7
Calcite	$Ca(CO_3)$	-	-	8.9
Albite	(Na, Ca) (Al, Si)$4O_8$	-	6.9	33.9
K-Feldspar	$KAlSi_3O_8$	-	-	4.9

The results of the X-ray diffraction analysis of core samples from different ore-bearing stratigraphic stages revealed a general similarity in the mineralogical composition of the productive horizons at the North Kharasan deposit. However, the ores exhibited variations in the content of certain minerals across a broader range: quartz varied from 54.7% to 90.8%, potassium feldspar (K-feldspar) varied from 5.7% to 10.1%, and kaolinite varied from 6.7% to 11.6%. Additionally, gypsum (up to 16.4%) in the samples from the Campanian and Maastrichtian stages indicated the formation of ion-exchangeable, mechanical, and chemical precipitates, which hinder the in situ mining process. The quantity of kaolinite governs the scale of ion-exchange colmatation, as it swells upon interaction with sulfuric acid, whereas the presence of gypsum contributes to the development of chemical colmatation. In contrast, ores from the Santonian stage are more homogeneous and show minimal precipitation during in situ leaching.

The swelling of clay minerals and gypsum deposition in solution discharge zones lead to the formation of impermeable areas within the productive horizon. This results in a change in the flow direction of leaching solutions toward barren zones, a reduction in the uranium concentration in the pregnant solution (PLS), and a consequent delay in uranium recovery from the subsurface.

The results of the X-ray diffraction analysis of precipitate samples from wells in the Santonian horizon of the Syrdarya depression indicate that the deposits are single-component and consist entirely (100%) of gypsum, a chemically derived product.

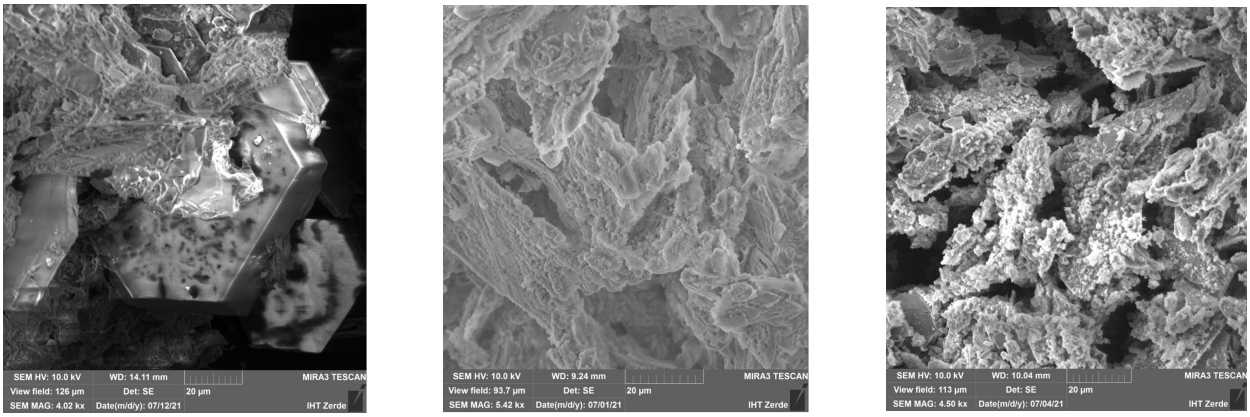
The precipitates from wells in the Maastrichtian stage were similar in composition but differed slightly, containing 88% gypsum, 5.1% quartz, and 6.9% albite. The dominance of gypsum confirmed the chemical nature of the deposits, while the presence of quartz (5.1%) and albite points to the existence of minor mechanical impurities.

The precipitates from wells in the Campanian horizon of the Syrdarya depression were multicomponent and exhibited a complex structure. The presence of quartz (35.6%), albite (33.9%), and potassium feldspar (4.9%) confirmed the predominance of a mechanical clogging mechanism. At the same time, the presence of gypsum (16.7%) and calcite (8.9%) indicated the formation of chemically derived precipitates.

In reference [20], data on the mineralogical composition of well precipitates relative to the productive horizon are presented. Practical experience in well regeneration confirms that homogeneous precipitates are effectively removed using conventional chemical methods involving hydrochloric acid. However, multicomponent precipitates tend to be denser, are less soluble, and exhibit a reduced responsiveness to standard chemical treatments. Quartz-, gypsum-, and feldspar-based precipitates tend to accumulate in the filter section of wells, reducing the filtration properties of the productive horizon and obstructing the flow paths of the leaching solution. As a result, the solution is diverted to barren zones, reducing the uranium recovery efficiency.

Laboratory experiments enabled the selection of effective chemical reagent compositions and concentrations for the breakdown and prevention of precipitates, depending on their mineralogical characteristics. Microscopic studies allowed for a detailed examination of the surface structure of precipitate samples before and after treatment and facilitated a comparative analysis of the dissolution effectiveness of different chemical reagent formulations.

Figure 5 shows scanning electron microscope (SEM) images of precipitates from a well in the Campanian horizon of the Syrdarya depression: (a)—untreated sample; (b)—after treatment in Experiment A1; and (c)—after treatment in Experiment A2.



(**a**) (**b**) (**c**)

Figure 5. Surface images of Campanian horizon samples: (**a**) original sample; (**b**) sample after Experiment A1; and (**c**) sample after Experiment A2.

The image of the untreated sample shown in Figure 5a reveals a solid structure composed of intergrown hexagonal prisms of various sizes, with overgrowths of small, irregularly shaped crystals bonded together. The hexagonal prisms varied in size, and the smaller elongated crystals were tightly packed, forming a continuous structure with no visible fractures or gaps.

Figure 5b shows that, after treatment with the decolmatant solution from Experiment A1, structural damage and fissures appeared. The hexagonal prism crystals were dissolved, and the material lost its original mechanical strength, taking on a loose and porous texture. Fine crystals were no longer visible, and larger crystals were significantly thinned.

In Figure 5c, after treatment in Experiment A2, the structural changes were similar to those observed in Experiment A1. The hexagonal crystals were dissolved, with the appearance of cracks and erosional features. However, the remaining crystals were relatively larger and less thinned, and small crystals were still present. The overall structure appeared denser and showed fewer ruptures compared to that in Experiment A1.

The more pronounced dissolution observed after Experiment A1 and the comparatively milder effect in Experiment A2 were attributed to the action of hydrofluoric acid on siliceous, gypsum, aluminosilicate, and carbonate minerals in the precipitate composition. The breakdown of gypsum, aluminosilicate, siliceous, and carbonate deposits in the productive horizon occurs under the influence of hydrofluoric acid (HF), according to Reactions (1)–(4).

$$CaSO_4 \cdot 2\,HF + 2\,H_2O \rightarrow CaF_2 + H_2\,SO_4 + 2\,H_2O \quad (1)$$

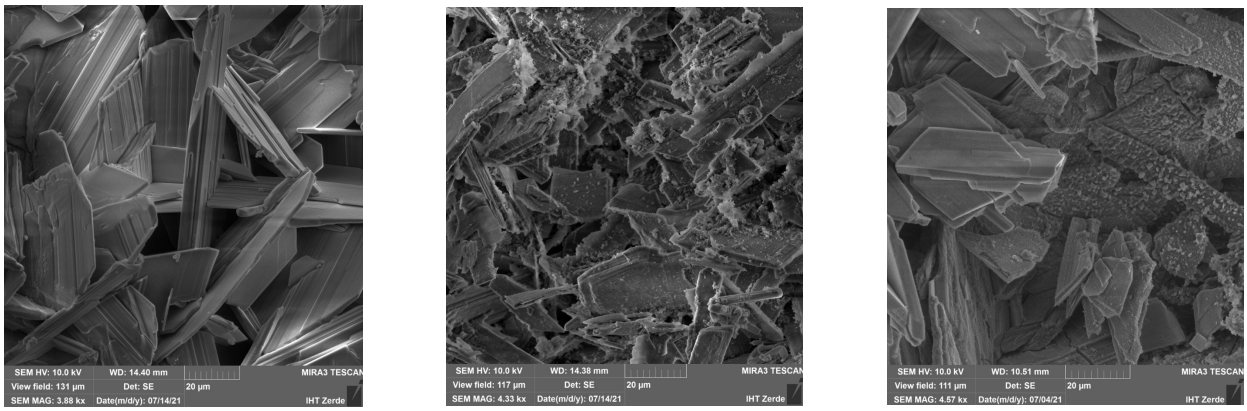
$$CaAl_2SiO_8 + 16\,HF \rightarrow 2\,AlF_3 + 2\,SiF_4 + 8\,H_2O + CaF_2 \quad (2)$$

$$6\,HF + SiO_2 \rightarrow SiF_4 + 2\,HF + 2\,H_2O \quad (3)$$

$$CaCO_3 + 2\,HF \rightarrow H_2CO_3 + CaF_2 \quad (4)$$

As demonstrated by Interaction Equations (1)–(4), the complete dissolution of gypsum, aluminosilicate, siliceous, and carbonate precipitates in the near-filter zone of the wells was achieved through the application of a 2.5%–5% hydrofluoric acid solution in volumes sufficient to penetrate 0.5 to 1.0 m along the entire length of the well filter, thereby creating an acid bath. This approach enables the restoration of filtration properties in the near-wellbore zone of the formation and enhances the productivity of geotechnological wells affected by complex, multicomponent precipitate compositions.

Figure 6 presents scanning electron microscope (SEM) images of a precipitate sample from a well in the Campanian horizon of the Syrdarya depression: (a)—original sample; (b)—after treatment in Experiment B1; and (c)—after treatment in Experiment B2.



(**a**) (**b**) (**c**)

Figure 6. Surface images of Campanian horizon samples: (**a**) original sample; (**b**) sample after Experiment B1; and (**c**) sample after Experiment B2.

As shown in Figure 6a, the original precipitate sample consisted of large, elongated crystals arranged in a chaotic pattern. The crystal surfaces were smooth and uniformly structured, with no visible fractures or discontinuities, displaying a characteristic skeletal (framework) morphology.

Figure 6b shows a scanning electron microscope (SEM) image of the sample after treatment with the decolmatant solution from Experiment B1. Significant structural degradation was observed: the crystals decreased in size, became thinner, and showed signs of erosion. The surfaces were deformed, and fine flakes formed from dissolved crystals were clearly visible.

Figure 6c presents the SEM image of the sample after treatment with the decolmatant solution from Experiment B2. The surface exhibited partial deformation—some crystals were partially dissolved and reduced in size, while others remained unaffected. A minor presence of flakes from partially dissolved crystals was noted, but the overall structure and density of the sample remained largely unchanged.

The limited dissolution observed after Experiment B1 and the minimal effect seen in Experiment B2 were attributed to the action of hydrochloric acid, which reacted primarily with the gypsum and carbonate minerals, but not with the siliceous or aluminosilicate components present in the precipitate samples. The dissolution of gypsum and carbonate precipitates in the near-filter zone of the formation occurred under the influence of hydrochloric acid according to Reactions (5) and (6).

$$CaSO_4 + 2\,HCl = CaCl_2 + H_2SO_4 \quad (5)$$

$$Ca(CO_3) + 2\,HCl = CaCl_2 + CO_2\uparrow + H_2O \quad (6)$$

As shown in Equations (5) and (6), the complete dissolution of gypsum and carbonate deposits in the near-filter zone of the formation was achieved by applying a 2.5%–5.0% hydrochloric acid solution in a volume sufficient to penetrate 0.5–1.0 m from the well filter, thereby forming an acid bath. The restoration of filtration characteristics in wells is ensured only when gypsum and carbonate minerals dominate the precipitate composition.

Determining the ore structure and the composition of precipitate-forming components within the productive horizons of uranium deposits in the Syrdarya depression enables

the development of universal methods for intensifying wellfield extraction. These new approaches involve the application of specially formulated solutions tailored to the mineralogical composition of the ores and precipitates in the productive horizon. The developed techniques allow for the effective dissolution and prevention of mineral deposits in the productive zone and significantly increase the solvent capacity of the working reagent (WR) used in well-based uranium leaching operations [21,22].

This technology offers several key advantages: it enhances the uranium concentration in the pregnant solution (PLS), restores filtration properties in the productive horizon at deposits in the Syrdarya depression, and reduces the labor intensity, power consumption, and other operating costs associated with uranium extraction.

The selection of the concentration and composition for the multifunctional chemical reagent complex depends on the well productivity, the clay content, and the filtration characteristics of the ore in the productive horizon of the technological block or the specific well being treated. Well productivity is influenced by the type of filter and the lithology of the productive formation and is calculated according to Equation (7).

$$Q = \frac{l\ d}{\alpha} \tag{7}$$

where Q—well productivity, m^3/h; l—filter length, m; and α—empirical coefficient, dependent on the filtration characteristics of the formation. For fine-grained sand with a filtration coefficient ranging from 2 to 5, $\alpha = 90$.

The rational volume of the decolmatant solution is determined based on the composition of the precipitate-forming materials, the productivity of the well, and the effectiveness of previous chemical treatments. The volume is calculated by taking into account the porosity coefficient of the productive horizon rocks and the radial dispersion range of the decolmatant solution from the well filter, which is determined according to Equation (8).

$$Q_d = \pi r^2\ H_e\ \ k_\rho \tag{8}$$

where $k_\rho = 0.22$; H_e—effective thickness of the productive horizon and r—radial dispersion distance of the decolmatant solutions.

The effectiveness of selecting rational parameters for the multifunctional chemical reagent complex also depends on the filtration characteristics of the rock within the block or well, and is calculated according to Equation (9).

$$K_f = \frac{\mathrm{Q}(\lg R - lg\, r)}{1366(H^2\ - h^2)} \tag{9}$$

where R—radius of the depression cone, m; r—radius of the well, m; H—thickness of the aquifer, m; and h—water column in the well after pumping, m.

Experimental studies on the dissolution of precipitates made it possible to determine the optimal concentrations and volumes of multifunctional chemical reagents to enhance the productivity of production wells. To ensure experimental consistency, a single treatment of the filter section was carried out on each of 30 pre-selected wells using a prepared decolmatizing solution with a volume of 3 m^3, containing hydrofluoric acid at concentrations ranging from 2.0%, 2.5%, 3.2% and 4.0%, along with the addition of complexing agents, 5–10 wells for each method. The subsequent monitoring and analysis of the well performance and the operational duration before and after treatment allowed for an assessment of the effectiveness of the innovative chemical (RRW) using a multifunctional reagent composition based on hydrofluoric acid and surfactants (SAA). The effectiveness was evaluated by measuring the mean time between failures (MTBF) of the wells.

Figure 7 shows a diagram comparing the uninterrupted operation periods of uranium ISR wells before and after the experimental treatments conducted on the wells.

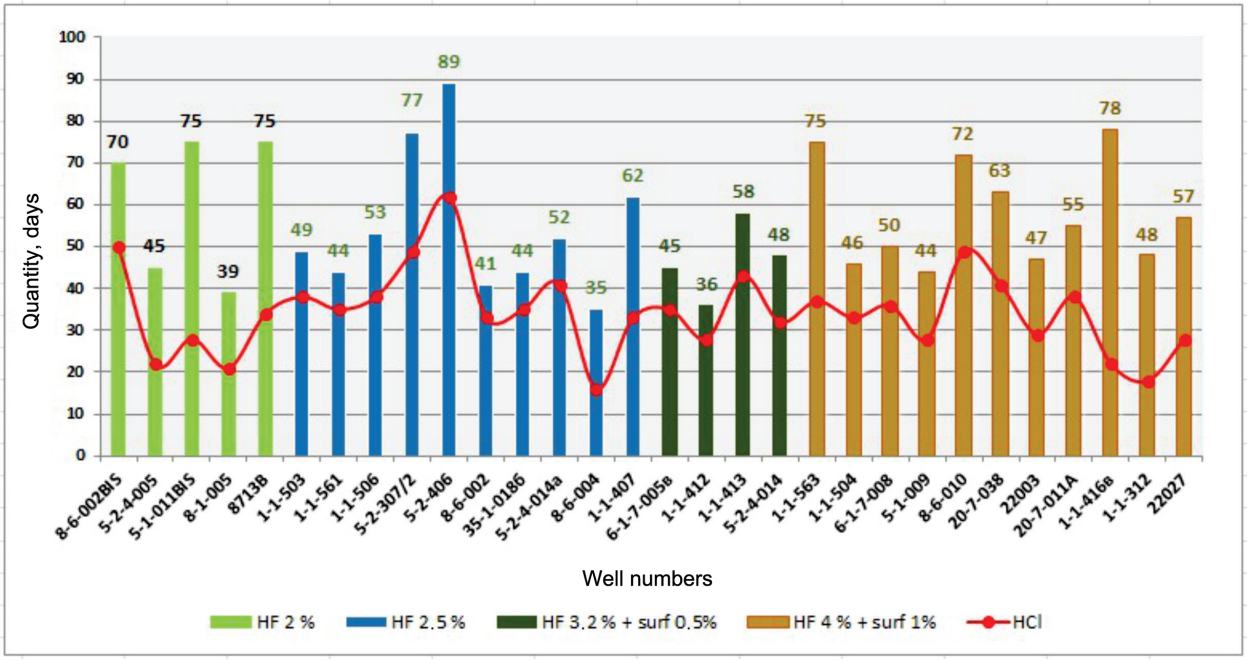


Figure 7. Results of comparative analysis of well performance before and after chemical treatment.

A comparative analysis of the performance of geotechnological wells before and after chemical treatment revealed that the average mean time between failures (MTBF) prior to treatment was 34 days when hydrochloric acid (HCl) was used in quantities of 150, 200, 250, or 300 kg.

As shown in Figure 7, the application of a multifunctional chemical reagent complex containing hydrofluoric acid (HF at 2.0%) increased the MTBF for wells with a clay content coefficient (*kc*) $\leq$ 12 from 21 to 50 to 45 to 75 days. For wells with *kc* $\leq$ 15, the use of a 2.5% HF solution increased the MTBF from 35 to 62 to 44 to 89 days. In wells with a high clay content (*kc* $\leq$ 18), a multifunctional solution containing 3.2% HF and 0.5% surfactant (SAA) was applied, resulting in an increase in the MTBF from 28 to 43 to 45 to 58 days. For wells with a very high clay content (*kc* $\geq$ 19), a multifunctional reagent solution with 4.0% HF and 1.0% surfactant was used, leading to an MTBF increase from 18 to 36 to 50 to 78 days. To reproduce the research results at sites with similar lithological properties, it is necessary to apply a declogging solution with the described concentrations of hydrofluoric acid, adjusted according to the carbonate content, clay content, or grain size of the productive horizon. The results will help improve the efficiency of restoring filtration properties and ISR well performance, while reducing reagent consumption and labor costs at wells with sufficient or high effectiveness.

The increase in the MTBF was attributed to the reactive capability of hydrofluoric acid, which effectively dissolves precipitates in the near-filter zone of the wells. The presence of surfactants in high-clay wells helps prevent the solution from bypassing through previously flushed areas of the filter, allowing the reagent to reach and dissolve deposits in the lower part of the filter column. Moreover, the surfactant does not negatively affect the filtration properties of the wells, as it is water-soluble and is fully removed during the post-treatment pumping of the calculated solution volume prior to putting the well back into operation.

The lower efficiency observed in areas with a very high clay content can be explained by the even-lower performance of conventional chemical methods under similar conditions.

Hydrofluoric acid effectively destroys the primary compounds in precipitate formations, while the surfactant, especially in complex geological areas, reduces the permeability in the upper washed zones of the filter. This prevents the uncontrolled spreading and dilution of the decolmatant solution within the productive horizon. Consequently, the reagent is distributed more uniformly along the filter column, promoting declogging and activating the lower sections of the well screen.

As confirmed by pilot-industrial trials, the application of the multifunctional chemical reagent composition resulted in more stable well operation and longer MTBF intervals. The wells required fewer repair and restoration interventions, and the effectiveness of subsequent operations increased by approximately 15% compared to previous treatments. However, the use of hydrofluoric acid requires additional safety measures for monitoring and ensuring the quality of specialized clothing, footwear, and protective equipment to safeguard the skin and respiratory organs from accidental splashes.

4. Conclusions

These comparative quantitative and qualitative mineralogical studies demonstrated a predominance of quartz in the ore intervals of the Syrdarya depression: 90.8% in the Santonian stage, 54.7% in the Maastrichtian stage, and 66.3% in the Campanian stage. The second most common mineral across these intervals was potassium feldspar (K-feldspar): 9.2% in the Santonian, 10.1% in the Maastrichtian, and 5.7% in the Campanian horizon. The presence of kaolinite (6.7% in Maastrichtian and 11.6% in Campanian ores), along with gypsum (16.4% in the Campanian ores), indicates the complex and heterogeneous structure of the Maastrichtian and Campanian productive horizons. According to operational data, the well productivity in these blocks remains unstable and requires additional measures to improve the ore filtration characteristics.

An X-ray diffraction analysis of the precipitate samples revealed that, in wells of the Santonian horizon, the dominant precipitate type is chemical gypsum (100%). In the Maastrichtian wells, the precipitates are relatively homogeneous, composed mainly of chemically deposited gypsum (88%) with minor amounts of quartz (5.1%) and albite (6.9%). In contrast, precipitates in the Campanian horizon are multicomponent and consist of quartz (35.6%), albite (33.9%), K-feldspar (4.9%), calcite (8.9%)—formed from mechanical particle transport—and gypsum (16.7%), resulting from chemical reactions. Homogeneous and slightly contaminated precipitates are effectively removed without an additional operational impact, while multicomponent deposits are more difficult to eliminate and require increased effort and resources. In such cases, prolonged well operation and increased maintenance efforts are often necessary.

Microscopic studies of the samples before and after laboratory testing made it possible to select effective chemical compositions and concentrations for well treatment, depending on the geological conditions and precipitate composition. A 2.5% hydrofluoric acid solution effectively destroyed homogeneous and moderately complex precipitates, with noticeable structural degradation and crystal deformation. A 5.0% HF solution showed even stronger effects on multicomponent precipitates, significantly reducing the crystal sizes. A 2.5% hydrochloric acid solution caused only minor changes, with slight deformation and no reduction in crystal size, whereas 5.0% HCl induced moderate damage and changes in structure and size. These findings confirm that selecting the appropriate concentration and composition of chemical reagents can enhance the uranium in situ leaching efficiency and reduce the operational costs and labor.

A preliminary analysis of the geological parameters and block performance enabled the classification of sites and wells based on the filtration coefficient, clay content, and MTBF, supporting the selection of optimal chemical treatment parameters. Zones with

$kc \leq 12$ or $kc \leq 15$ demonstrated stable well operation, requiring only low-concentration HF (2.0%–2.5%) or high-concentration HCl (4.0%). Blocks with $kc \leq 18$ are considered moderately challenging and can be treated using 3.2% HF with spot application of surfactants, while HCl-based treatments above 4.0% are ineffective. For areas with $kc \geq 19$, complex treatments involving 4.0% HF with surfactants are required, based on previous performance data.

As a result of pilot-industrial tests conducted on geotechnological wells used for in situ uranium leaching, optimal parameters for chemical well treatment under various mining and geological conditions were developed. Monitoring and comparative analysis of well performance before and after experimental treatments with multifunctional chemical reagent solutions demonstrated the high efficiency of the innovative method compared to traditional chemical treatment approaches.

At wells with low clay content (kc $\leq$ 12), the use of a hydrofluoric acid solution (HF—2.0%) increased the average inter-repair cycle from 31 to 60 days. In areas with a clay coefficient of kc $\leq$ 15, a declogging solution containing hydrofluoric acid (HF—2.5%) was applied, resulting in an average increase in the inter-repair cycle from 38 to 54 days. For wells with an ore clay coefficient of kc $\leq$ 18, treatment with a multifunctional solution containing hydrofluoric acid at a concentration of 3.2% and a surfactant (0.5%) increased the average inter-repair cycle from 34 to 46 days. In geotechnological blocks with high clay content in the productive horizon (kc $\geq$ 19%), the application of multifunctional solutions with a hydrofluoric acid concentration of 4.0% and a surfactant concentration of 1.0% increased the average inter-repair cycle from 32 to 57 days. The increase in the inter-repair cycle of wells enabled a greater extraction of productive solution from the subsurface and the greater injection of leaching solution, due to the reduced downtime for maintenance and restoration activities.

Based on the collected data, the effectiveness of applying multifunctional chemical reagent complexes for well treatment in challenging geological conditions was validated through field trials. The results substantiate the use of tailored chemical treatment parameters based on the ore and precipitate composition. Block and well classification, coupled with parameter selection, improves productivity, extends uninterrupted operation, reduces equipment stress, and enhances environmental and occupational safety. The rational parameters for well treatment, based on the ore clay content, filtration rate, carbonate content, and precipitate composition, increase the ISR efficiency and reduce the chemical consumption and operating costs by 10%–15%.

These studies have generated a large body of data that will guide the future development of improved and novel methods to enhance uranium leaching in low-permeability ores.

Author Contributions: Conceptualization, K.T. and Z.K.; methodology, K.T. and Z.K.; software, A.M. and Y.Y.; validation, K.T., Z.K. and A.M.; formal analysis, S.B. and D.R.; investigation, K.T., Z.K. and D.R.; resources, S.B., Y.Y. and A.M.; data curation, K.T. and Z.K.; writing—original draft preparation, K.T., Z.K., S.B. and A.M.; writing—review and editing, K.T., Z.K. and D.R.; visualization, A.M.; supervision, K.T.; project administration, K.T.; funding acquisition, K.T. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (grant No. AP19575556—The innovative uranium underground leaching intensification technology using polymer and binder components under downhole uranium mining conditions).

Data Availability Statement: The data are contained within the article.

Acknowledgments: The authors express their gratitude to the Limited Liability Partnership "Institute of High Technologies" for providing the computer program and for the possibility of using the laboratory facilities.

Conflicts of Interest: The authors declare no conflicts of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

References

1. Khawassek, Y.M.; Taha, M.H.; Eliwa, A.A. Kinetics of Leaching Process Using Sulfuric Acid for Sella Uranium Ore Material, South Eastern Desert, Egypt. *Int. J. Nucl. Energy Sci. Eng.* **2016**, *6*, 62–73. [CrossRef]
2. Rashad, M.M.; Mohamed, S.A.; EL sheikha, E.M.; Miraa, H.E.; Abd el Wahaba, G.M.; Zakia, S.A. Kinetics of uranium leaching process using sulfuric acid for Wadi Nasib ore, South western Sinai, Egypt. *Aswan Univ. J. Environ. Stud.* **2020**, *2*, 171–182. [CrossRef]
3. Bahig, M.A.; Mohamed, A.G.; Mohamed, F.C. Kinetics of uranium and iron dissolution by sulfuric acid from Abu Zeneima ferruginous siltstone, Southwestern Sinai, Egypt. *Euro-Mediterr. J. Environ. Integr.* **2018**, *3*, 39.
4. Chen, J.; Zhao, Y.; Song, Q.; Zhou, Z.; Yang, S. Exploration and mining evaluation system and price prediction of uranium resources. *Min. Miner. Depos.* **2018**, *12*, 85–94. [CrossRef]
5. Togizov, K.; Kenzhetaev, Z.; Temirkhanova, R.; Muzapparova, A.; Omirgali, A.; Altaibayev, B. The Inflence of the Physicochemical Characteristics of Ores on the Effiiency of Underground Well Leaching of Uranium Deposits in Kazakhstan. *Minerals* **2024**, *14*, 381. [CrossRef]
6. Rakishev, B.R.; Mataev, M.M.; Kenzhetaev, Z.S. Analysis of mineralogical composition of sediments in in-situ leach mining of uranium. *Min. Inf. Anal. Bull.* **2019**, *7*, 123–131. [CrossRef]
7. Kenzhetaev, Z.; Nurbekova, M.; Togizov, K.; Abdraimova, M.; Toktaruly, B. Methods for intensification of borehole uranium mining at the fields with low filtration characteristics of ores. *Min. Miner. Depos.* **2021**, *15*, 95–101. [CrossRef]
8. Nikitina, Y.G.; Poyezzhayev, I.P.; Myrzabek, G.A. Improvement of opening schemes of wellfilds to optimize the cost of mining uranium. *Gorn. Vestn. Uzb.* **2019**, *1*, 6–11.
9. Rakishev, B.R.; Bondarenko, V.I.; Matev, M.M.; Kenzhetaev, Z.S. Influence of chemical reagent complex on intensification of uranium well extraction. *Nauk. Visnyk Natl. Hirnychoho Univ.* **2019**, *6*, 25–30. [CrossRef]
10. Rakishev, B.; Mataev, M.M.; Kenzhetayev, Z.; Altaybayev, B.; Shampikova, A. Research into leaching of uranium from core samples in tubes using surfactants. *Min. Miner. Depos.* **2020**, *14*, 97–104. [CrossRef]
11. Yusupov, H.A.; Aliev, S.B.; Dzhakupov, D.A.; El'zhanov, E.A. Primenenie biftorida ammoniya dlya himicheskoj obrabotki skvazhin pri podzemnom vyshchelachivanii urana. *Gorn. Z.* **2017**, *4*, 57–60. [CrossRef]
12. Mataev, M.M. The impact of ammonium bifluoride complex on colmataging formations during the process ofin situ uranium leaching. *Int. J. Adv. Res.* **2017**, *5*, 147–154. [CrossRef] [PubMed]
13. Rakishev, B.; Kenzhetayev, Z.; Shampikova, A.; Toktaruly, B. Increasing of filtration characteristics of ore bodies in borehole uranium mining. In Proceedings of the 2nd International Conference Essays of Mining Science and Practice, RMGET 2020, Dnipro, Ukraine, 22–24 April 2020.
14. Yusupov, H.A.; Dzhakupov, D.A.; Bashilova, E.S. Povyshenie effektivnosti otrabotki slozhnyh gidrogennyh mestorozhdenij urana s primeneniem peroksida vodoroda. *Gorn. Z. Kaz.* **2018**, *2*, 18–21.
15. Kuandykov, T.; Nauryzbayeva, D.; Yelemessov, K.; Karmanov, T.; Kakimov, U.; Kolga, A. Development and justification of a hydro-impulse method for increasing ore permeability in conditions of uranium borehole production. *News Natl. Acad. Sci. Repub. Kaz. Ser. Geol. Tech. Sci.* **2020**, *6*, 126–133. [CrossRef]
16. Gorbatenko, O.A. Remontno-vosstanovitel'nye raboty na geotekhnologicheskikh skvazhinakh predpriyatiy PSV urana. In *Almaty, Kazakhstan: NAK Kazatomprom*; KazNRTU: Almaty, Kazakhstan, 2017; 194p.
17. Zammit, C.M.; Brugger, J.; Southam, G.; Reith, F. In situ recovery of uranium—The microbial influence. *Hydrometallurgy* **2014**, *150*, 236–244. [CrossRef]
18. Kuandykov, T.A.; Karmanov, T.D.; Kuldeyev, E.I.; Yelemessov, K.K.; Kaliev, B.Z. New technology of uncover the ore horizon by the method of in-situ leaching for uranium mining. *News Natl. Acad. Sci. Repub. Kaz. Ser. Geol. Tech. Sci.* **2022**, *3*, 142–154. [CrossRef]
19. Kosunov, A.O.; Shavanda, V.V.; Kopbaeva, M.P.; Myrzabek, K.A.; Mataev, M.M.; Kenzhetaev, Z.S.; Bishimov, K.E.; Lipinsky, V.K.; Tretyakov, S.Y. Mobile Chemical Treatment Unit Technological Wells. Innovation Patent of the Republic of Kazakhstan No. 2153, 9 September 2016.
20. Kenzhetaev, Z.S.; Togizov, K.S.; Abdraimova, M.R.; Nurbekova, M.A. Selecting the rational parameters for restoring filtration characteristics of ores during borehole mining of uranium deposits. *Min. Miner. Depos.* **2022**, *16*, 1–10. [CrossRef]

21. Panfilov, M.; Uralbekov, B.; Burkitbayev, M. Reactive transport in the underground leaching of uranium: Asymptotic analytical solution for multi-reaction model. *Hydrometallurgy* **2016**, *160*, 60–72. [CrossRef]
22. Polynovsky, K.D. Approach complex to solve the problem of intensification of in situ leaching of uranium. *Min. Inf. Anal. Bull.* **2012**, *7*, 64–73.

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.