



Research into Uranium Characteristics and Content in a Pregnant Solution During Leaching with Oxygen Saturation

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Abstract

The aim of this study is to intensify the process of in situ leaching of uranium with saturation of the working solution with oxygen using a Venturi tube. One of the ways to increase the efficiency of underground leaching is to saturate the leaching solution with oxygen. However, this oxidizer has not found application due to the complexity and high cost of oxygen saturation in the solution. The results of the study showed that saturation of the leaching solution with oxygen using a Venturi tube leads to a decrease in the concentration of divalent iron and an increase in the concentration of trivalent iron. Thus, this leads to an increase in the average uranium content in the pregnant solution by 21.3% compared with the technology being used. The dependence of changes in the concentration of trivalent iron and the uranium content in the pregnant solution on the leaching time was obtained when the solution was saturated with oxygen. The application of the proposed technology of oxygen saturation in the solution will increase the uranium content in the pregnant solution and thereby shorten the time required to mine uranium reserves in the technological block.

Keywords: Geotechnology; In Situ Leaching; Trivalent Iron; Oxidation; Lead Dioxide; Pregnant Solution.

1. Introduction

Kazatomprom JSC is the national operator of uranium mining in the Republic of Kazakhstan. At the holding's enterprises, metal is mined using the most environmentally friendly and safe method of in situ leaching (ISL) [1]. However, despite the relatively significant reserves and low cost of uranium mining, the constant depletion of profitable deposits necessitates the development of new highly efficient and economically attractive methods for mining metals from poor deposits, as well as improving the completeness of mining [2–4]. The directions for the development and improvement of these methods can be very different. For example, in the geological direction, to improve the quality and completeness of mining, modern geophysical well logging methods of research are used, such as the method of radio-wave geointoscopy, which has shown high efficiency in tracking and controlling technological processes when mining uranium by ISL methods [5–8]. The geotechnological direction is focused on the use of various chemical complexes and reagents for the intensification of mining. Various mining and geological factors affect the process of drillhole in situ leaching, reducing the efficiency of production and increasing the cost of the final product [9–12].

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There is a complete absence or very low concentration of trivalent iron in underground reservoir water and in the ore bed with the transition to mining areas of deposits with complex mining and geological conditions, which is the reason for the decrease in the value of the redox potential of the working solution. Therefore, it is necessary to introduce various types of oxidizing agents into the leaching solution in such areas [13, 14]. A method for intensifying the in-situ leaching of uranium with the addition of trivalent iron salts to a sulfuric acid solution is known. Sulfuric acid solution, along with trivalent iron salts, is a selective solvent that leads to a 20% reduction in sulfuric acid consumption and an increase in the efficiency of uranium leaching by 1.2–1.4 times. This leads to an increase in the value of the redox potential of the solution to 400 mV and above and the uranium extraction coefficient to 0.92–0.98 at a concentration of trivalent iron in a working solution of 1000 mg/l. However, if the amount of leaching solution supplied to the injection well is taken into account, i.e., the amount of trivalent iron salts for dilution, then this technology becomes economically unjustified.

A number of researchers have proposed the use of chemical oxidants by chemolithoautotrophic microorganisms, which can increase the redox potential of the working solution by generating the active form of the oxidizer-trivalent iron and reagent:



This technological solution for the use of chemolithotrophic bacteria for the complex extraction of minerals is used in many countries worldwide [15, 16].

The efficiency of in situ leaching of various metals such as nickel, zinc, gold, copper, and arsenic could be increased when the thionic bacteria *Thiobacillus ferrooxidans* are added to the working solution [17, 18]. The use of bacterial uranium leaching increases the value of the redox potential of the solution up to 20% and the uranium content in the pregnant solution to 12–15%. However, practice shows that bacterial leaching requires the construction and maintenance of a special complex for growing and maintaining the viability of a bacterial colony, which leads to a significant increase in the cost of final products. Another oxidizing agent used, in particular, to improve the efficiency of uranium leaching is nitrous acid or its salts. There are results of studies on the use of hydrogen peroxide, sodium nitrate, and ozone as oxidants. The leaching solution will have a high oxidizing ability at a concentration of hydrogen peroxide in an amount of 100–150 mg/l, and the sodium nitrate concentration of 100 mg/l in the leaching solution leads to a decrease in divalent iron and an increase in the value of the redox potential of the solution [19, 20].

The aforementioned technological schemes have been tested both separately and simultaneously at deposits with different geological and hydrogeological parameters. The obtained experimental data on the technological parameters of the uranium leaching regime have led researchers to develop the main dependences that determine the effectiveness of using this ISL technology. The methods listed for increasing the efficiency of drillhole in situ leaching undoubtedly give good results, but they have some disadvantages, which are the main obstacles to their widespread use. Hydrogen peroxide and sodium nitrate must be stored and transported in compliance with certain safety rules, and the construction of expensive ozonizers must obtain ozone. Furthermore, it is necessary to create special installations that need to be integrated into the leaching solution line to supply these oxidants to the leaching solution [21–24].

Ozonation requires significant energy costs to generate high ozone concentrations. From this, it can be concluded that various methods of intensifying uranium mining by leaching require large capital and operating costs for equipment, as well as for the biological protection of personnel. This requires the development of new effective methods for activating oxidizing processes with low energy and material costs, which do not require complex technological equipment and are low labor intensive. It is therefore proposed to use air oxygen as an oxidizing agent to intensify leaching processes. Oxygen is a gas that is poorly soluble in water. Its solubility is directly proportional to the partial pressure of oxygen over the solution and, to a significant extent, depends on the temperature and aqueous phase composition.

Equally important is the development of technologies for saturating the leaching solution with air oxygen. At first glance, the obvious way to saturate the solution with oxygen is to supply gas through an air-falling hose from a compressor connected to the main pipe. However, the main problem is that at enterprises, through the main pipe itself, from 60m³/h to 130m³/h of solution passes. Alternatively, it is possible to supply air directly into the mother liquor, which is located under the open sky. However, since there is a relatively low pressure at the air supply point, almost all the oxygen will instantly evaporate, negating all attempts to intensify the solution.

Given these factors, it is proposed to use the Venturi tube as a way for oxidizing the solution. The Venturi tube is a short, specially designed tube with a narrowing or reduction in diameter in the middle and an air suction hole. When the solution passes through the narrowing of the Venturi tube, the flow rate increases with simultaneous air suction [25]. According to D. Bernoulli's principle, the volume of fluid or leaching solution that passes through a pipe of a larger diameter will be the same volume of fluid that passes through a narrow section per unit of time. To provide the same

volume of fluid conditionally at two points, the fluid flow rate in the narrow section of the pipe is increased. The higher the flow rate, the lower the static pressure. The ratio of fluid velocity and static pressure is directly proportional; that is, by increasing the fluid flow rate through the reduced pipe diameter, the static pressure can be reduced to such a value that air can be sucked from the hole in the narrowed section of the pipe.

The novelty of the proposed technology lies in the use of a Venturi tube for suction of air, transfer of oxygen from the gaseous state to the liquid phase, and saturation of the solution during in situ leaching of uranium. The essence of the technology is that there is an active mixing of air with the solution, which contributes to the efficient transfer of oxygen from air into the solution. The leaching solution first passes through the large diameter of the pipe, then in the section of the pipe narrowing and from the laminar flow changes into a turbulent flow, where chaotic, highly irregular movement of fluid occurs. It is accompanied by active transverse mixing and velocity and pressure pulsations, which in turn leads to the fragmentation of the gas into small parts directly in the moving fluid, after which the solution saturated with gas passes back into the section with a large diameter. In this section, the flow rate is reduced, where the solution changes back to the laminar regime of the fluid flow and the static pressure increases proportionally, which contributes to the dissolution of gases in water. With increasing pressure, the air oxygen solubility increases. In the study of Aben et al. [26], it has been proven that the saturation of the leaching solution with oxygen can be doubled using a Venturi tube.

2. Material and Methods

Laboratory research on core material from the Central Mynkuduk uranium deposit was performed to determine the influence of an oxygen-saturated solution on divalent-trivalent iron concentration, the oxidation–reduction potential value of the solution, and the uranium content in the pregnant solution. According to the mineralogical composition, the ores of the Mynkuduk horizon of the Mynkuduk deposit and the Central site, in particular, are coffinite-nasturan (pitchblende). In the total balance of uranium minerals in the site, calculated from the results of X-ray diffraction analysis and electron microscopic studies, coffinite is 15%, and nasturan is 85% (with a total number of tests – 127).

The core material, as shown by the field study, is gray sand. The research was conducted in two stages. The core material was selected, and a model installation was made during the first stage. The core material from a uranium deposit was sampled from different depths of wells at intervals of 20 cm, and each core is described. A total of 6 m core was selected. To determine the average uranium content in the core material, all the cores were crushed, and the uranium content in the core materials was 0.035% on average. The model laboratory setup (Figure 1) consists of a container for the leaching solution, a vessel with the core material, and a container for the pregnant solution.

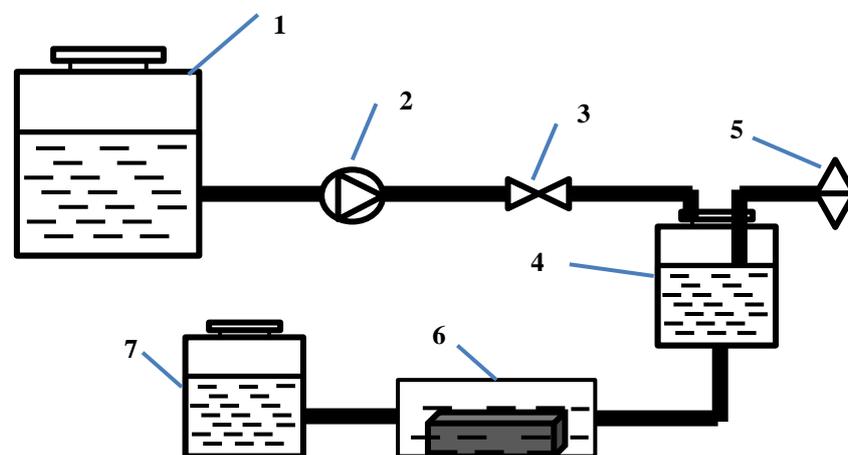


Figure 1. Model laboratory setup: 1 – container for a leaching solution; 2 – pump; 3 – venturi tube; 4 – dissolved oxygen measuring vessel; 5 – oxygen meter; 6 – sealed container with core; 7 - a container for a pregnant solution

A flexible hose is connected to the 50 l plastic container, and a horizontal pump is then installed to provide the required fluid flow rate. The fluid, at high velocity, provided by the pump, passes through the Venturi tube. The oxygen-saturated solution is drained into a container to measure the dissolved oxygen concentrations in the fluid. The vessel for measuring oxygen in a fluid consists of a sealed lid on one side and an oxygen meter on the other. The vessel is designed in such a way that a hole for filling the container with water and air is drilled in a sealed lid, and an oxygen meter for measuring dissolved oxygen is arranged on the bottom of the vessel. The uranium leaching time is 9 h for each core material, with sampling done every hour. A total of 10 samples were taken, including one sample of the leaching solution (initial solution) and 9 samples of the pregnant solution. Oxygen saturation is measured using an oxygen meter, pH value and oxidation–reduction potential (ORP) are measured using an IT-1101 device, and uranium content and divalent-trivalent iron concentration are determined in the mine laboratory.

Laboratory studies were carried out with a solution without oxygen saturation (traditional leaching technology) and then with the proposed technology with saturated oxygen leaching solution to obtain comparative data during the second stage. The course of laboratory work is shown in Figure 2.

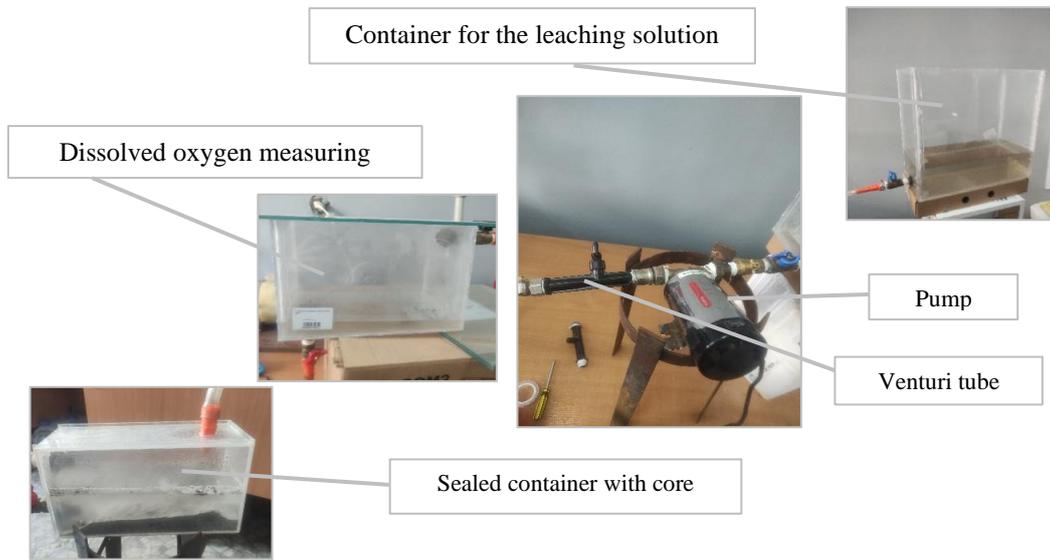


Figure 2. Course of laboratory work

The results of laboratory studies were processed using MatLab software. The concentration of divalent iron, ORP, uranium content in the pregnant solution, and the amount of extracted uranium were analyzed both during leaching with a solution without oxygen saturation and with oxygen saturation of the solution over time.

A flowchart of the oxygen saturation and leaching processes using the proposed technology is shown in Figure 3.

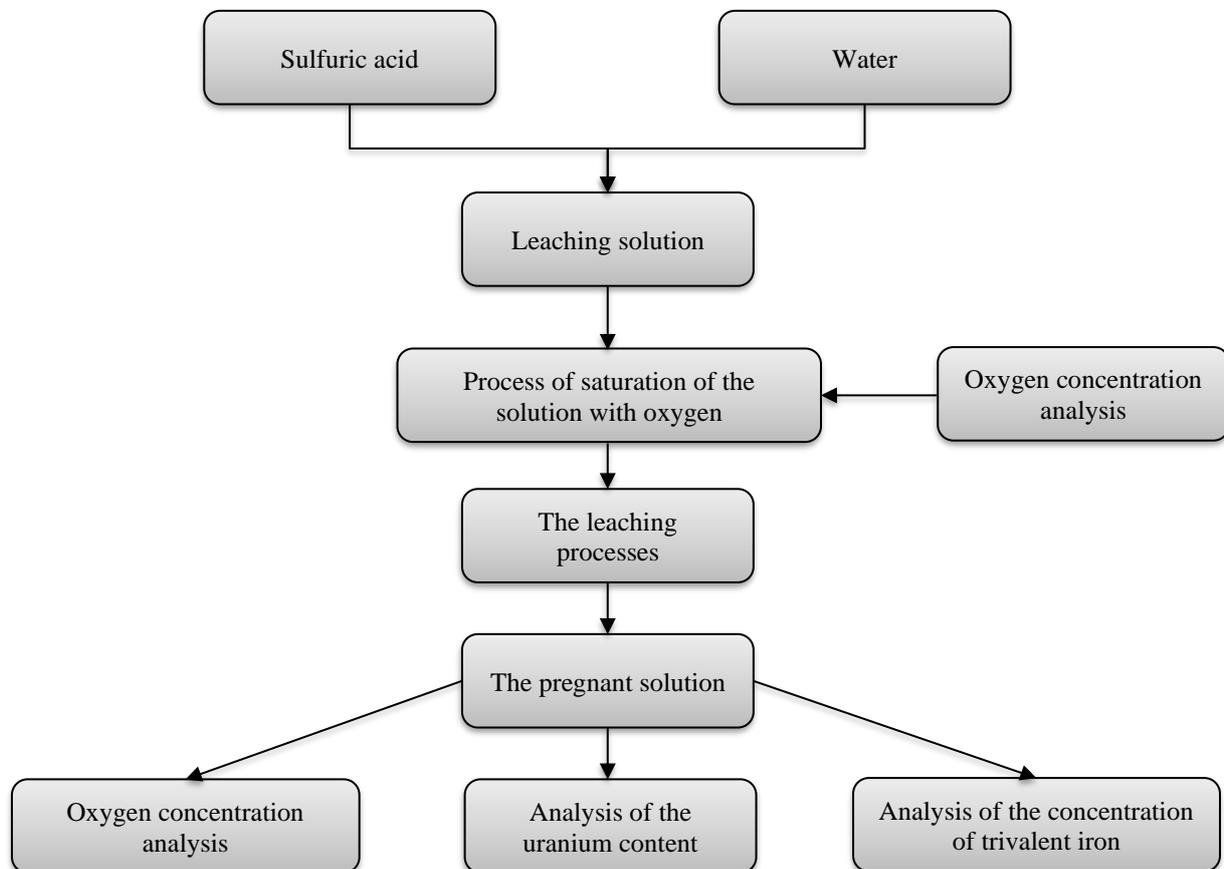


Figure 3. Flowchart of oxygen saturation and leaching processes using the proposed technology

Studies were first conducted without saturation of the solution with oxygen (traditional leaching technology) and then with the proposed technology with saturated oxygen leaching solution to obtain comparative research data.

3. Results

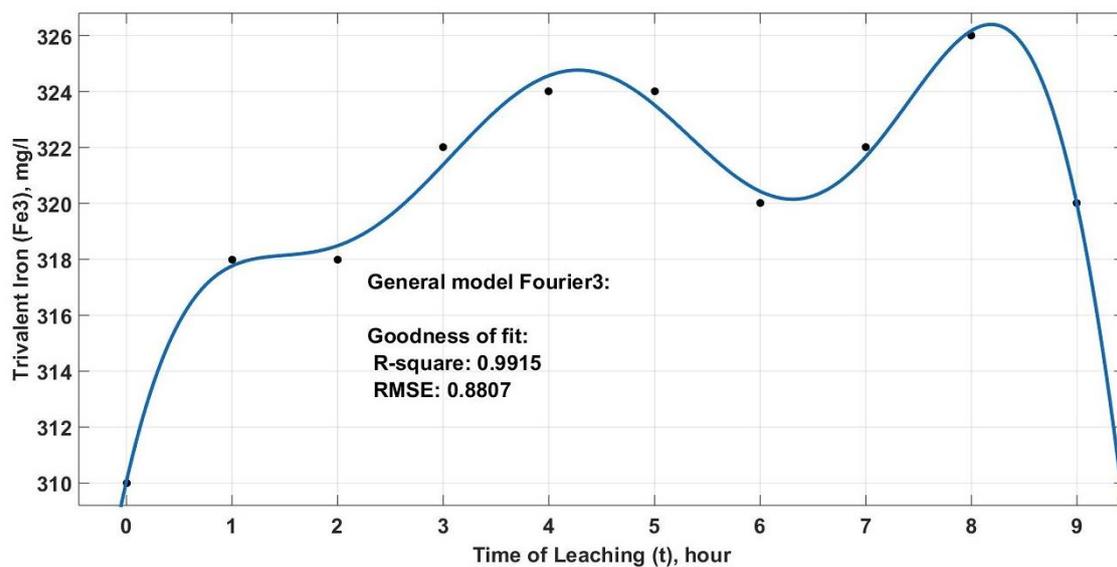
The results of the analysis of the uranium content, redox potential values, and divalent and trivalent iron without saturation of the solution with oxygen are shown in Table 1.

Table 1. Analysis results of the uranium content, ORP values, divalent-trivalent iron content using the basic technology

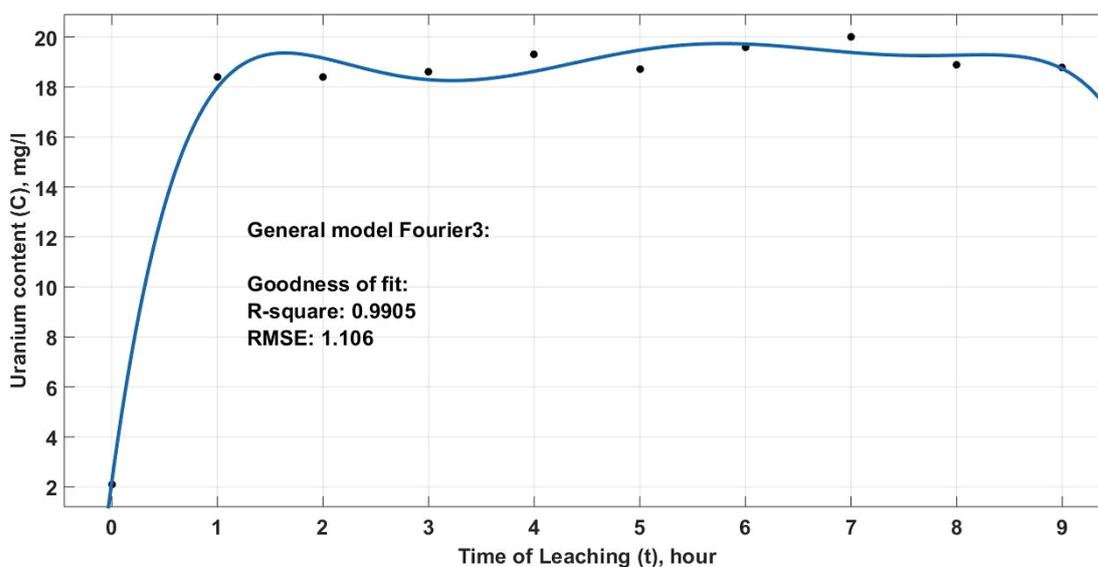
Sample No.	Divalent iron, mg/l	Trivalent iron, mg/l	Uranium content, mg/l	Oxidation-reduction potential, mV	Sampling time, h
Leaching solution sample	270	310	2.10	340	0
Pregnant solution sample 1	300	318	18.4	340	1
Pregnant solution sample 2	290	318	18.4	344	2
Pregnant solution sample 3	362	322	18.6	345	3
Pregnant solution sample 4	365	324	19.3	346	4
Pregnant solution sample 5	368	324	18.7	345	5
Pregnant solution sample 6	362	320	19.6	346	6
Pregnant solution sample 7	360	322	20.0	346	7
Pregnant solution sample 8	356	326	18.9	348	8
Pregnant solution sample 9	354	320	18.8	346	9

As shown in Table 1, as the leaching time increases, the ORP value changes slightly from 340 to 348 mV. At the same time, the trivalent iron concentration in the leaching solution is 310 mg/l, and with increasing leaching time it increases to 324 mg/l, but then decreases to 320 mg/l. The divalent iron concentration increases from 270 mg/l to 368 mg/l, but then slightly decreases to 354 mg/l. The uranium content in the pregnant solution does not change significantly within the range of 18.4–20.0 mg/l during the leaching period. In general, the average uranium content in the pregnant solution is 18.63 mg/l, and the divalent iron concentration increases by 27%. Additionally, 170.7 mg of uranium was extracted after 9 h of leaching.

Graphs of changes in the concentration of trivalent iron (a) and uranium content (b) from the time of leaching without saturation of the solution with oxygen were obtained by processing the data in Table 1 (Figure 4).



(a)



(b)

Figure 4. Changes in the concentration of trivalent iron and uranium content in the pregnant solution from the time of leaching without saturation of the solution with oxygen

As can be seen from Figure 4, the convergence of experimental data on changes in the concentration of trivalent iron gives at a Fourier to the term of 3, and the uranium content at the same fit, which allows concluding that the level of convergence is fairly high. The results of laboratory work using the proposed technology with oxygen saturation in the solution are shown in Table 2.

Table 2. Analysis results of the uranium content, ORP and divalent-trivalent iron values when the solution is saturated with oxygen

Sample No.	Divalent iron, mg/l	Trivalent iron, mg/l	Uranium content, mg/l	Oxidation-reduction potential, mV	Sampling time, h
Leaching solution sample	300	320	1.80	360.0	0
Pregnant solution sample 1	296	331	20.91	360.8	1
Pregnant solution sample 2	298	320	21.65	370.2	2
Pregnant solution sample 3	298	330	24.28	373.9	3
Pregnant solution sample 4	294	335	23.84	375.8	4
Pregnant solution sample 5	294	338	23.46	380.6	5
Pregnant solution sample 6	292	345	23.0	382.8	6
Pregnant solution sample 7	294	341	23.15	387.6	7
Pregnant solution sample 8	296	347	24.0	380.5	8
Pregnant solution sample 9	294	347	23.20	381.6	9

As can be seen from Table 2, the concentration of divalent and trivalent iron in the leaching solution were 300 and 320 mg/l, respectively, before saturation of the solution with oxygen. After saturation of the solution with oxygen, the concentration of trivalent iron increased to 347 mg/l, and the concentration of divalent iron decreased to 292 mg/l. It can be concluded that when the solution is saturated with oxygen, divalent iron is oxidized to trivalent iron. There was also an increase in the ORP value after saturation of the solution with oxygen with an increase in the leaching time from 360 mV to 387 mV. The uranium content in the pregnant solution varied within the range of 20.9 – 24.2 mg/l during leaching. The average uranium content in the pregnant solution was 23.05 mg/l, the concentration of trivalent iron increased by 5%, and the ORP value increased by 4.7%. Moreover, 207.49 mg of uranium was extracted after 9 h of leaching when the leaching solution was saturated with oxygen.

Then, regression equations can be formed that show the relationship between the change in the concentration of trivalent iron and the uranium content from the leaching time using the data from Table 2, as well as using the R-square and RMSE coefficients to make an estimate of own method.

It can be seen from Table 3 that Fourier with the number of terms 3 is better suited for trivalent iron according to R-square, and Exponential with the number of terms 2 is best suited for uranium content according to R-square and RMSE.

Therefore, Fourier better predicts the behavior of trivalent iron from the leaching time, and Exponential better predicts the uranium content.

Table 3. Equations of regression for changes in the concentration of trivalent iron and uranium content from the leaching time according to the proposed technology

Uranium Characteristics	Fit	Sum of Sine			Exponential		Fourier	Gaussian
		Number of terms	1	2	3	1	2	3
Trivalent iron	R-square	0.85	0.86	0.94	0.85	0.85	0.95	0.85
	RMSE	4.43	5.59	7.48	4.2	4.8	4.98	4.4
Uranium content	R-square	0.65	0.938	0.985	0.298	0.99	0.99	0.579
	RMSE	4.6	2.55	2.5	6.05	0.78	1.32	5.005

Graphs of the dependences of the concentration of trivalent iron (a) and the content of uranium (b) in the pregnant solution on the leaching time with the proposed technology were obtained by processing the data in Table 2 (Figure 5).

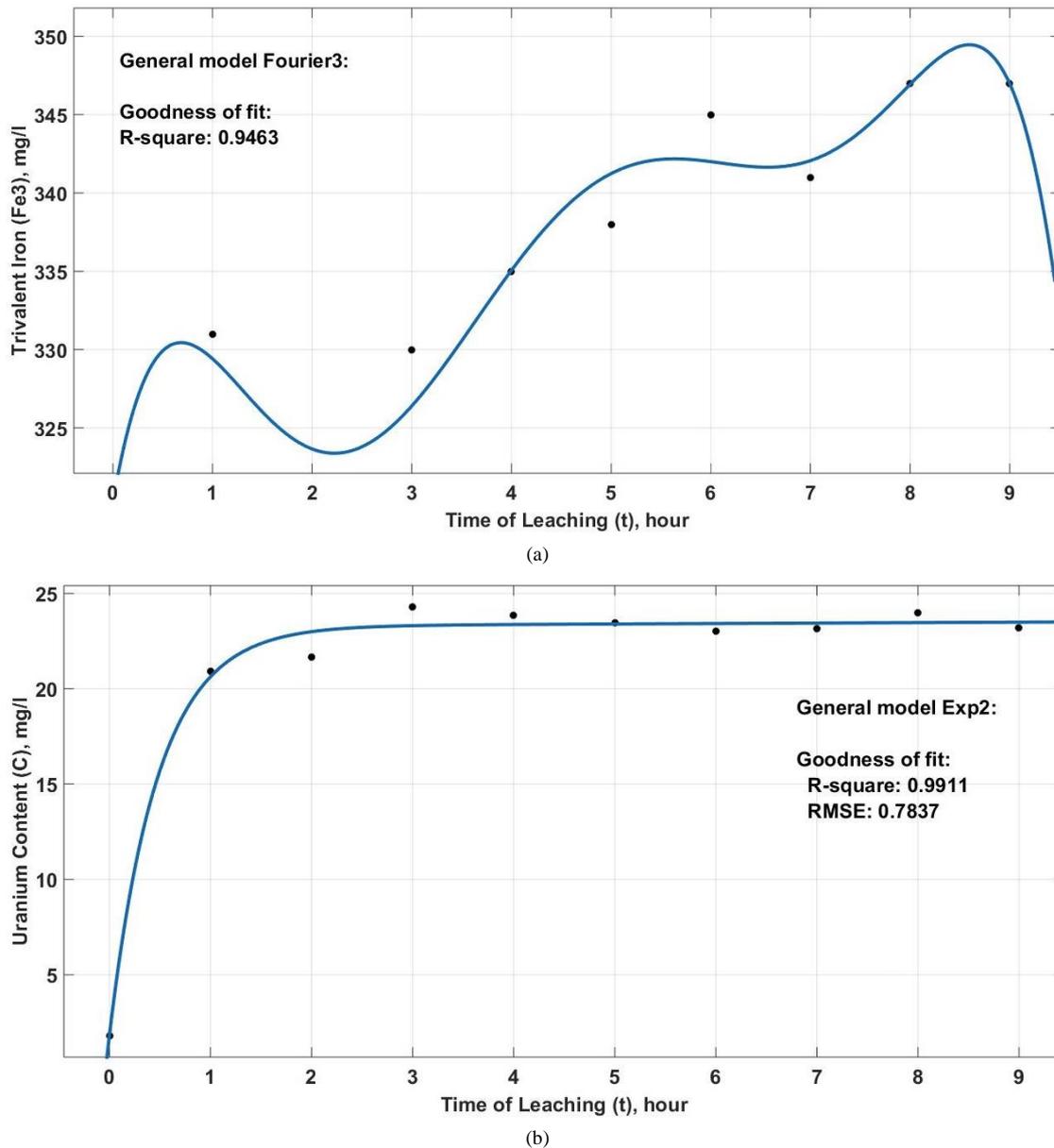


Figure 5. Change in the concentration of trivalent iron and the uranium content in the pregnant solution from the time of leaching using the proposed oxygen saturation technology

As can be seen from Figure 4, the correlation coefficient between the experimental points and the obtained dependencies is higher than that in studies without oxygen saturation of the solution. The general trend of decreasing uranium content over time is explained by a decrease in the uranium content in the core material during leaching.

4. Discussion

Table 4 summarizes the comparative results of the studies on uranium leaching with solutions without and with oxygen saturation.

Table 4. Analysis results of the uranium content, ORP and divalent-trivalent iron values when the solution is saturated with oxygen

Solution without oxygen saturation			Solution with oxygen saturation		
Divalent iron, mg/l	Trivalent iron, mg/l	Uranium content, mg/l	Divalent iron, mg/l	Trivalent iron, mg/l	Uranium content, mg/l
346.3	321.6	19.0	295.1	337.1	23.05

A comparative column chart of the concentration of divalent and trivalent iron and the uranium content in the pregnant solution was obtained by processing the data in Table 4 (Figure 6).

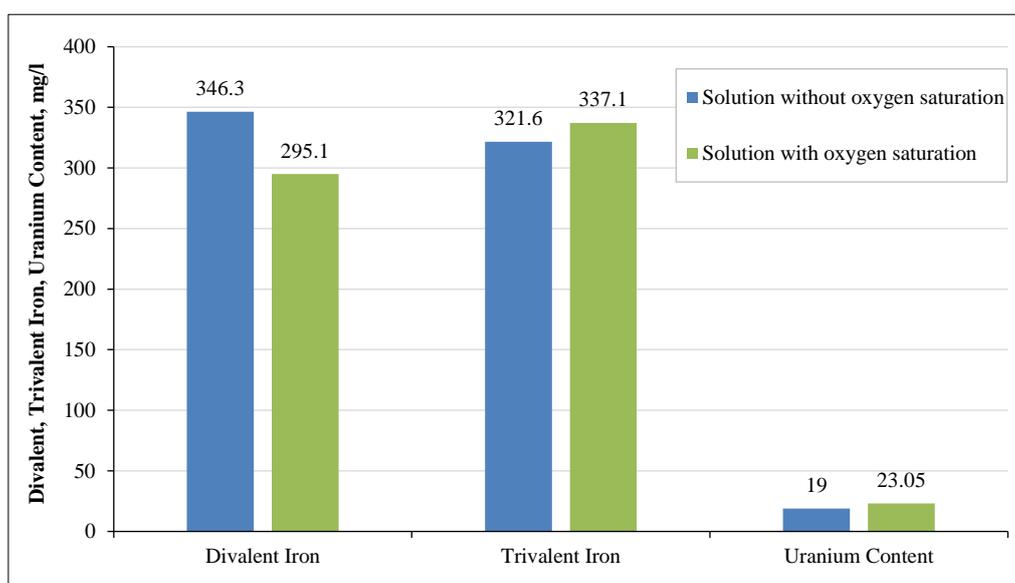


Figure 6. Comparative histogram of the concentration of divalent and trivalent iron and uranium content in the pregnant solution

As can be seen from Figure 6, the concentration of divalent iron in the solution decreases from 346.3 mg/l to 295.1 mg/l, and the concentration of trivalent iron increases from 321.6 mg/l to 337.1 mg/l when leaching with an oxygen-saturated solution. This increases the uranium content in the pregnant solution from 19 mg/l to 23.05 mg/l.

The comparative results of previous studies using oxidants in the form of hydrogen peroxide [15] and sodium nitrate [27] with the proposed technology are shown in Table 5.

Table 5. Comparative results of previous studies using oxidants and the proposed technology

	Increase of trivalent iron, %	Reduction of divalent iron, %	Increase of the uranium content, %	Average price of the oxidant, \$/kg
Solution using hydrogen peroxide	11.7	8	28	1.5
Solution using sodium nitrate	13.3	17	23	0.85
Proposed technology	8.5	14.7	21.3	0

A comparison with the results of previous studies shows that adding hydrogen peroxide as an oxidizer to the leaching solution in an amount of 0.3 l per 1000 l (an average of 2,800 liters per hour is supplied to only one technological well) would increase the uranium content in the pregnant solution by an average of 28%.

The results of previous studies show that the optimal amount of sodium nitrate, which gives good results, is 100 mg/l of the solution. This leads to an increase in the ORP of the solution from 374 mV to 395 mV, a decrease in the concentration of divalent iron by 17%, and an increase in the uranium content in the pregnant solution by 23%. However, these oxidizers have not found application and are not competitive in comparison with the proposed technology due to the significant cost of hydrogen peroxide (1.5 USD/kg) and sodium nitrate (-0.85 USD/kg (prices of the Republic of

Kazakhstan) and the consumption of hydrogen peroxide per a technological unit of 6000 kg/day and sodium nitrate 600 kg/day, as well as special requirements and additional costs for storage, transportation, and the creation of special devices for supplying them to the leaching solution line.

5. Conclusion

The use of a Venturi tube will saturate the leaching solution with oxygen because of the transition of the laminar flow of the solution into a turbulent flow and vice versa, which increases the transfer of oxygen from a gaseous state to a liquid state. There is no need to create special conditions for the storage and transportation of the oxidizer with the proposed technology, which is environmentally safe and can be easily integrated into the leaching solution line. The cost of the purchase and installation of a Venturi tube is about \$10 per injection well. The dependence of changes in the concentration of trivalent iron and the uranium content in the pregnant solution on the leaching time was obtained both during leaching with and without oxygen saturation. A decrease in the concentration of divalent iron and an increase in the concentration of trivalent iron prove the intensification of the oxidation of divalent iron in solution to trivalent iron when uranium is leached with an oxygen-saturated solution. Thus, this leads to an increase in the uranium content in the pregnant solution, a reduction in the uranium mining time by an average of 21.3%, and a reduction in the cost of the final metal. It should be noted that 170.7 mg of uranium was extracted from the core material without saturation of the solution with oxygen during 9 h of uranium leaching, and 207.49 mg of uranium was extracted when the solution was saturated with oxygen, which is 25.5% more.

The obtained laboratory results can serve as a basis for conducting semi-industrial tests under production conditions and further expand the application of uranium mining through drillhole in situ leaching.

6. Declarations

6.1. Author Contributions

Conceptualization, E.A.; methodology, E.A. and S.Y.; software, N.S.; validation, D.A. and E.Y.; formal analysis, S.Y.; investigation, D.A.; resources, N.S.; data curation, E.A.; writing—original draft preparation, S.Y. and E.A.; writing—review and editing, D.A., E.Y., and N.S.; visualization, D.A. and N.S.; supervision, E.A.; project administration, E.A.; funding acquisition, E.A. All authors have read and agreed to the published version of the manuscript.

6.2. Data Availability Statement

The data presented in this study are available on request from the corresponding author.

6.3. Funding

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6.4. Conflicts of Interest

The authors declare no conflict of interest.

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