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SCIENTIFIC JUSTIFICATION FOR INCREASING THE ENVIRONMENTAL SAFETY OF RECEIPT OF IODINE UPON RETURN OF FIELD WATER OF GAS CONDENSATE FIELDS

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Purpose. A focus of this work was to establish the effect of the time of the formation-reservoir water ozonation, molar-ion reagent ratio (*MIR*), acidity (*pH*), mineralization (*M*) of the medium, concentration of iodine ions (*J*) and ferrum (Fe^{2+}), contact surface between the phases on the formation degree (*CO*) of iodine using the rate of the reaction of the transformation from the iodide ions to iodine to improve the redemption of the formation-reservoir water and ecologically safe extraction of iodine. **Methodology.** The research uses model mixtures of field waters, which include, mg per liter: *J* – 10-90, bromide ions 800, sodium chloride 200000, Fe^{2+} – 0-66. To ensure the necessary concentration of *J* – ions, the following is used: KJ of chemically pure grade, bromide ions *KBr* of chemically pure grade., Fe^{2+} – ions $FeSO_4$ of pure grade., sodium chloride of pure grade. To obtain an ozone and mixture, an ozone generator GL with the capacity of $6,63 \cdot 10^{-2} m^3$ /per year with ozone concentration of $6,28 \cdot 10^{-2} g\text{-moll}/m^3$ per cubic meter. Field waters have been ozonized with the use of cylindrical reactor, equipped with a dispersing device. The reactor is loaded with 100–400 ml of field waters and the ozonized mixture is supplied through a dispersant within a specified time. In the reaction mixture the concentration of iodine or iodide ion is determined by titration with sodium hyposulfite according to. The degree of formation of iodine (*DF*) is defined as the ratio of the current iodine concentration in the liquid phase to the theoretically possible concentrations at full conversion of iodide ions to iodine. **Results.** This paper gives the results of the scientific substantiation for increasing the environmental safety in the extraction of iodine from mineralized field water during their processing by ozonation, instead of a number of hazardous reagents, and return to the layer, which concerns the impact of time, acidity, salinity, concentration of iodide ions, molar ion ratio of reactants. Mathematical models have been obtained. In terms of experimentally and theoretically evaluated speed, ozonation reaction of iodide ions classified is classified as instant. **Practical value.** The results of the research are of great practical importance for ecologically safe extraction of iodine the redemption of the formation-reservoir water of gas condensate fields to the reservoir and the improvement of the redemption process. *References 12, tables 2, figures 8.*

Key words: time, concentration, iodine, ozonation, mineralization, water.

НАУКОВЕ ОБГРУНТУВАННЯ ПІДВИЩЕННЯ ЕКОБЕЗПЕКИ ОДЕРЖАННЯ ЙОДУ ПРИ ПОВЕРНЕННІ СУПУТНЬО-ПЛАСТОВИХ ВОД ГАЗОКОНДЕНСАТНИХ РОДОВИЩ

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Приведено результати наукового обґрунтування підвищення екобезпеки при одержанні йоду з мінералізованих супутньо-пластових вод газоконденсатних родовищ під час їх переробки озонуванням, замість застосування ряду шкідливих реагентів, і повернення у пласт, яке стосується впливу часу, кислотності, мінералізації, концентрації йодид-іонів, мольно-іонного відношення реагентів, поверхні контакту фаз, присутності іонів бром, вилучення одержаного йоду з газовою фазою. Показано, що зміни ступеня утворення йоду під впливом часу, кислотності, мінералізації, концентрації іонів йоду характеризуються екстремальними залежностями та лінійною залежністю при мольно-іонних відношеннях реагентів до 2 і зміні концентрації йодид-іонів. Одержано математичні моделі, що відтворюють експериментальні результати з похибкою 3-5%. За експериментально і теоретично оціненими величинами швидкості реакції озонування йодид-іонів віднесено до миттєвих реакцій.

Ключові слова: час, концентрація, йод, озонування, мінералізація, вода.

PROBLEM STATEMENT. It shall not be permitted in Ukraine to discharge field waters of gas condensate and oil deposits in the rivers and soils, since the volume reaches $100 km^3$ per year, creating a threatening situation for the environment. The source [1] indicates that the water coming to the surface together with oil and gas may be a source for the production of iodine, bromine and other substances. It is known that iodine content in the waters related to oil and gas fields ranges 10–20 mg per liter, of which 90–95% are iodides [2].

World production of iodine is at the level of 30 tons per year, thus not covering the essential world's needs. Ukraine does not produce iodine. During the development of domestic gas condensate fields, field waters are extracted together with hydrocarbons, which are referred to by-products of the hydrocarbons extraction technology and which should be returned to the Earth without damage to the environment and primary production. In Ukraine, such waters are returned into the deeper horizons. [3] Analysis of the

composition of field waters of the domestic gas condensate fields had found that the concentration of iodide ions generally ranges 10–100 mg per liter. Low concentration of solutions causes the need to develop appropriate technologies for removal of iodine and return to the layer. Under the known technologies, iodine is extracted from concentrated solutions with environmentally and corrosion-dangerous hydrochloric or sulfuric acids, chlorine or sodium hypochlorite, sulfur dioxide, confirming the maximum allowable concentrations of these agents in mg per cubic meter: 0,2; 0,3; 0,03; 0,5, respectively. Known approaches to return field waters in the layer are that after the separation of natural gas, gas condensate or oil, they are collected in a container and after evaluation of component composition for the compatibility with the water of the depleted layer, are delivered to the stations for the return to the layer through injection wells, after elimination of impurities and iron ions, which is achieved by settling [3]. The disadvantage of such approaches is that they do not allow for the use of substances mined from underground horizons such as iodine. The applicable industrial technology to extract iodine from drill waters [4] is the oxidation of iodide in previously acidified water to achieve $\text{pH} \approx 2$ with hydrochloric or sulfuric acid to elemental iodine and treatment with chlorine or chlorinated water. Chlorine causes the reaction $2\text{I}^- + \text{Cl}_2 = \text{I}_2 + 2\text{Cl}^-$ – and molecular iodine emerges in the water, which with a strong flow of air mixed with SO_2 fog is blown out of the water in the gas phase. This gas mixture is fed into a scrubber with the regenerating solution (Na_2SO_3), where iodine is converted into ions I^- – and concentrated to contain $\geq 30 \text{ g/dm}^3$. The consent rate is re-oxidized with chlorine to further release iodine by filtration. Then iodine is cleaned to remove organic impurities. It should be noted that when neutralizing waters acidified with sulfate acid with alkaline, poorly soluble sulfates of calcium and barium fall down and the costs of acids according to the alkalinity can reach up to 1000 kg per 1 kg of iodine. It is known [4] that ozone is used as an oxidant in a weakly acidic medium and for saturation of drilling water with CO_2 . Ozone is added into the water with $\text{pH} 7,8$ to such extent that all iodide ions are oxidized to form iodate ions. Then aggressive corrosive sulfuric acid and 4–5 of such volume of not oxidized initial drilling water are added. Iodide reacts with iodate to form iodine. Disadvantages of these technologies lies in the fact that in their implementation a number of aggressive corrosive substances is used. The use of hydrogen peroxide for the oxidation of iodide followed by the extraction of iodine has also found no development due to the use of expensive reagents [5]. It is known [6] that iodine is extracted from drilling water by processing of previously acidified water air with air with initiated oxidation with ozone in the presence of ferrous iron salts. It is also known that ozone is used to improve water quality for household purposes, indicating the environmental safety of this agent.

EXPERIMENTAL PART AND RESULTS OBTAINED. The research uses model mixtures of field waters, which include, mg per liter: I^- – 10–90, bromide ions 800, sodium chloride 200000, Fe^{2+} – 0–66. To

ensure the necessary concentration of I^- ions, the following is used: KJ of chemically pure grade, bromide ions KBr of chemically pure grade., Fe^{2+} - ions FeSO_4 of pure grade., sodium chloride of pure grade. To obtain an ozone and mixture, an ozone generator GL with the capacity of $6,63 \cdot 10^{-2} \text{ m}^3/\text{per year}$ with ozone concentration of $6,28 \cdot 10^{-2} \text{ g-moll/m}^3$ per cubic meter. Field waters have been ozonized with the use of cylindrical reactor, equipped with a dispersing device. The reactor is loaded with 100–400 ml of field waters and the ozonized mixture is supplied through a dispersant within a specified time. In the reaction mixture the concentration of iodine or iodide ion is determined by titration with sodium hyposulfite according to [7]. The degree of formation of iodine (DF) is defined as the ratio of the current iodine concentration in the liquid phase to the theoretically possible concentrations at full conversion of iodide ions to iodine.

The results of research of the impact of technological parameters on the ozonation of field waters

Due to the fact that almost all domestic deposits of natural gas contain carbon dioxide, field waters are saturated with this component. Therefore, the acidity of water can vary in terms of pH from $\approx 3,5$ pH to $6,5$ – $7,0$. Accordingly, the effect of the time of treatment of field waters with ozone PCI has been studied (Fig. 1) at different values of initial pH_0 (C_n , g-ion/dm^3 , AI), mg KOH/dm^3 , where C_n – concentration of hydrogen ions, AI – acid index): 3,78 ($1,659 \cdot 10^{-4}$; 9,31), 5,86 ($1,38 \cdot 10^{-6}$; 0,077) і 6,26 ($5,495 \cdot 10^{-7}$; 0,03). For comparison, the results of ozonation in the absence of acidity are given. Fig. 1 shows that the increase in the processing time leads to an increase in the degree of formation of iodine and it reaches its maximum at the lowest pH in the range of 150–200 s and eventually begins to decline. Reduction of the acidity to 5,86 and 6,26 causes a shift in the maximum formation of iodine in the range of 200–250 s (Fig. 1). It should be noted that with increasing acidity of the reaction after 200 s there is a marked decrease in iodine and therefore the degree of formation of iodine is reduced. At the same time, the dependences are described by regression equations with the correlation coefficient between 0,985 and 0,992, which can be used for prediction and adjustment of the ozonation process.

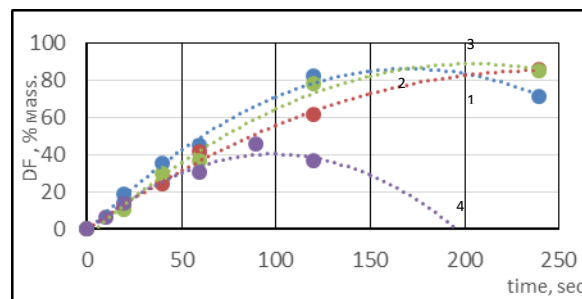


Figure 1 – Dependence of the degree of formation (DF) of iodine on the time of ozonation and acidity where: 1 – $\text{pH}_0 = 3,78$; 2 – $\text{pH}_0 = 5,86$; 3 – $\text{pH}_0 = 6,26$; 4 – $\text{pH}_0 = 7,3$

In the weak alkaline environment, accumulation of iodine is also originally observed, like in acidic environments, and there is a tendency towards its reduction over time.

The study of changes in the degree of formation of iodine over time when changing the mineralization from 10 g/dm³ to 200 g/dm³ (Fig. 2) indicates that over time the degree of formation of iodine increases, reaching at different mineralization a certain maximum, which is observed in ≈ 200 – 250 s. The greatest degree of formation of iodine is obtained at the most surveyed mineralization. The comparison of the degree of formation of iodine with changing mineralization suggests that during one and the same time of ozonation, increased mineralization causes almost linear changes in the degree of formation in the range of 20 – 60 s (Fig. 2). Increased ozonation up to 120 – 240 s determines an unclear linear relationship between the degree of formation of iodine and changes of mineralization, which is characterized by the minimum level with mineralization at 100 g/dm³ (Fig. 2). Signs of such minimum are observed with less time of ozonation. In all cases, a greater degree of formation of iodine occurs within 240 s of ozonation.

In the change of concentration of iodide ions (Fig. 3) the degree of formation depending on time of ozonation is characterized by maxima which are shifted with increased concentration of ions J⁻ – towards an increased in the processing time. That is, increased concentrations of iodide ions require more time to reach the maximum value of the degree of formation.

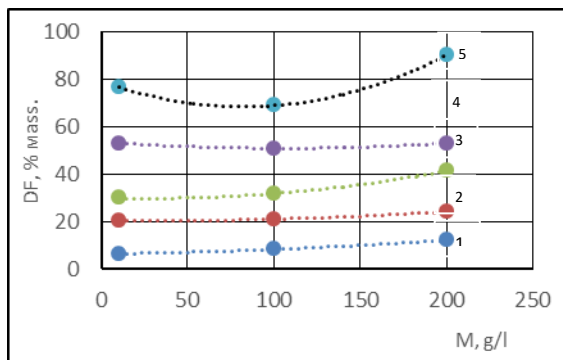


Figure 2 – Changes in the degree of formation (DF) of iodine with mineralization (M) and ozonation time where 1– 20 s; 2 – 40 s; 3 – 60 s; 4 – 120 s; 5 – 240 s.

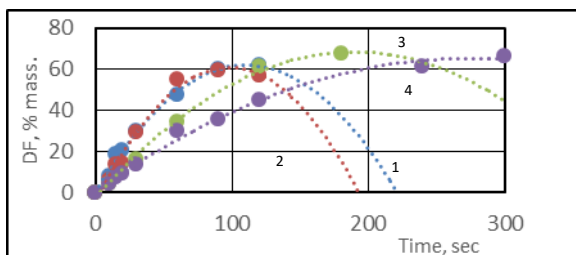


Figure 3 – Changes in the degree of formation (DF) of iodine over time by changing the concentration of J⁻: 1– 10 mg/dm³; 2 – 30 mg/dm³; 3 – 50 mg/dm³; 4 – 70 mg/dm³

Analysis of changes of the degree of formation depending on the ion and molar ratio of reactants (Fig. 4) shows that at concentrations J⁻ ion 10–70 mg/dm³ at the start of ozonation linear dependence is observed. In this case, at a concentration of 10 mg/dm³ of iodine the linear and nonlinear dependences are almost the same, because approximation coefficients are equal to 0,9612 and 0,9879, respectively. Increased concentration up to 30–70 mg/dm³ causes deviation from linear patterns. However, at the concentration of iodide ion 30 – 70 mg/dm³ the IMR value is maintained at ≈ 2, there is a linear relationship between IMR and degree of formation (Fig. 4b). Taking into account that the maximum dependence (Fig. 4a) begins with IMR >2, then such simplified dependence can be used for predicting the degree of iodine formation at the cost of ozone providing for ionic molar ratio (IMR) ≤ 2.

Comparison and analysis of research.

Based on the study of the impact of time of ozonation and mineralization on the degree of formation of iodine (Figure 2), a mathematical statistical model has been created

$$DF = (a_1 - a_2)M^2 - (a_3 - a_4)M + (a_5t + a_6);$$

where t is time of ozonation, s; M is mineralization, g/dm³; $a_1 - 7E-6$, $a_2 - 0,0002$; $a_3 - 0,0013$; $a_4 - 0,0614$; $a_5 - 0,2869$; $a_6 - 12,726$.

The model adequately with an average deviation of 3% abs. describes the experimental data (Table 1).

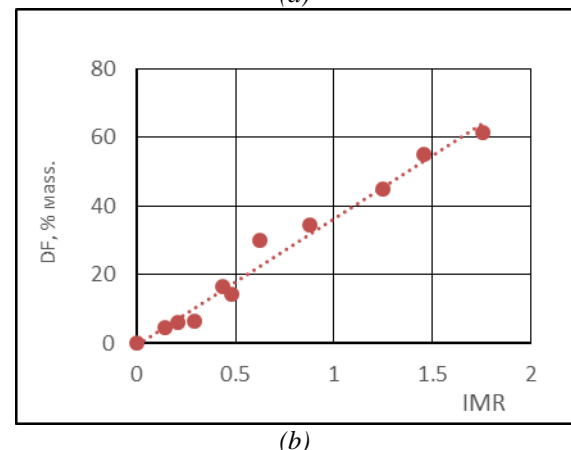
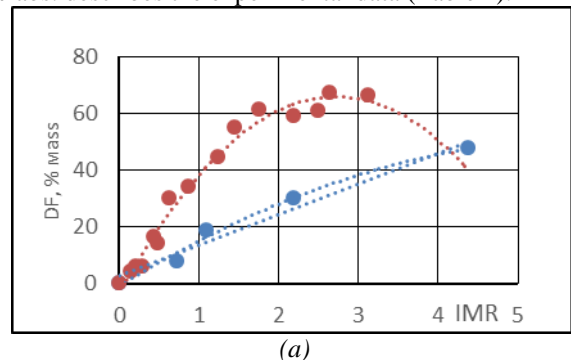


Figure 4 – Changes in the degree of formation of ion molar ratio (IMR) at a concentration J⁻ 10 mg/dm³ (1) and 30–50–70 mg/dm³ (2) – (a) and only at concentrations 30–50–70 mg/dm³ (b)

Based on changes in the degree of formation of iodine in the liquid phase on IMR and iodine ion

concentration of 30–70 mg per cubic dm (Fig. 4) the following equation is obtained

$$DF = a \cdot IMR^2 + b \cdot IMR + c$$

where $a = -9403$; $b = 51.1$; $c = -3.473$.

Calculations of iodine formation degree under this equation are consistent with experimental results with deviations within 0.1-5.1% at an average of 2.6% (Table. 2).

Table 1 – Comparison of experimental (e) and calculated (c) values of iodine formation degree with changing the time of ozonation of water salinity

Time, s	The degree of formation of iodine wt%. with mineralization, g/dm ³								
	10 g/dm ³			100 g/dm ³			200 g/dm ³		
	e	c	Δ	e	c	Δ	e	c	Δ
20	6.3	17	10.7	8.2	14.3	6.1	12.0	9.0	3.0
40	20.3	21.9	1.6	20.9	25.9	5.0	24.1	25.8	1.4
60	29.8	29.7	0.1	31.7	30.5	1.2	42.1	41.2	0.9
120	68.8	71.3	2.5	50.8	44.1	6.7	53.0	53.8	0.8
240	76.6	79.1	2.5	68.8	71.3	2.5	90.1	90.6	0.5

Note: Δ - deviation defined as [e-c].

Evaluation of simultaneous impact of the molar ion ratio of reactants and reaction time (Fig. 5) indicates that the increase in these parameters leads to increase in the degree of formation of iodine to the maximum possible level with a significant decrease in the concentration of ozone in the ozone and air mixture, which then can be completely used for ozonation of ions of iron Fe²⁺ with their conversion into ions of Fe³⁺ [3].

Assessment of the impact of surface (S) of contact between the phases (Fig. 6) shows that when increasing it the degree of formation of iodine grows and taking into account the low concentration of ozone in the air that the limiting stage of the process of ozonation may be mass transfer, including diffusion of ozone in the liquid phase

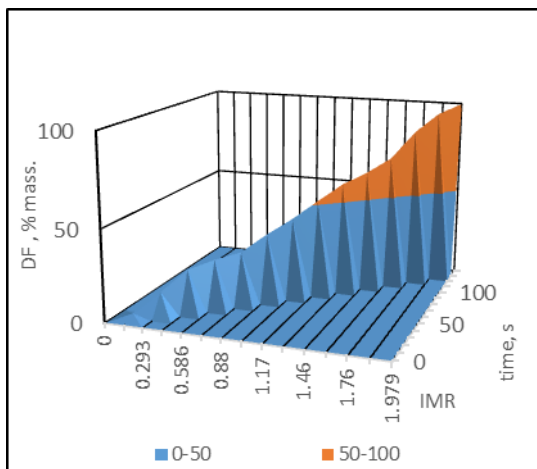


Figure 5 – Changes in the iodine formation degree depending on the time of ozonation and the molar & ion ratio (IMR)

With the assumption that the absorbed ozone

completely reacts with J – ion, the ozone absorption rate is equal to the rate of spending J – ions, the reaction rate constant can be estimated by J – ions [8], as done in [9]. Then the rate (V) of the reaction:

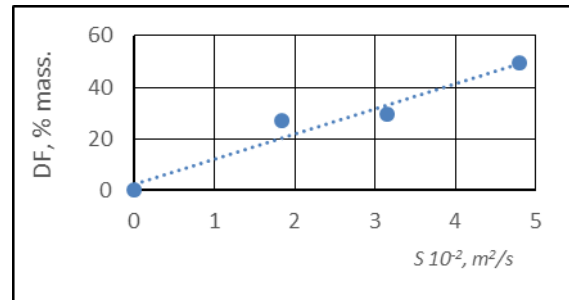


Figure 6 – The dependence of the degree of formation of iodine on the contact surface of phases

$nO_3 + 2J + H_2O \rightarrow J_2 + O_2 + OH$
 can be written as $V = n(-dJ/dt) = [O_3]a(D_{O_3}kJo)^{0.5}$, which after integration gains a kind of linear equation (Fig. 6).

$$(J_0)^{0.5} - (J)^{0.5} = \{[O_3]a(D_{O_3}k)^{0.5}\}t = Kt,$$

where J_0 , J, primary and current concentration of J – ions, $[O_3]$ - the concentration of ozone, a - contact surface, D_{O_3} - ozone diffusion ratio, k – reaction rate constant of second order.

The processing of experimental results by this expression indicates that the resulting dependence is linear (Fig. 7). This shows that the reaction of ozone may be subject to the laws of the second order reactions. The actor K found allows to evaluate the rate constant of this reaction by the expression $k^{0.5} = K/([O_3]aD_{O_3}^{0.5})$. The reaction rate constant found is estimated with the value of $7 \cdot 10^7$ g-mol/m³ sec. This confirms that the reaction of J – ion and ozone interaction is very fast, and the ozonation process itself is determined by diffusion of ozone, since the diffusion coefficient in water is 1,74 square meter per second and in the air it is 0,178 square meter per second at a ratio of mass transfer of ozone into water $2,8 \cdot 10^{-4}$ meter per second [9].

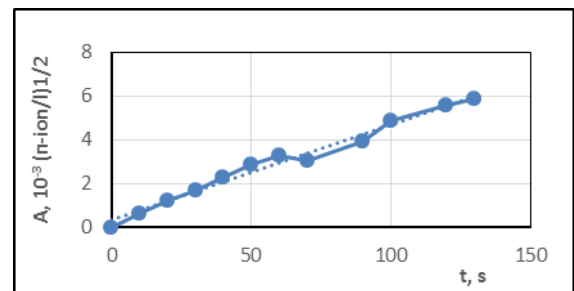


Figure 7 – Dependence $[Jo]^{0.5} - [J]^{0.5} (A)$ on the ozonation time (t)

It is also known [10] that the constant rate of iodine formation by the interaction of ozone with iodide ion with the formation of the NJO acid reaches $2,4 \cdot 10^9$ m⁻¹s⁻¹, and its conversion into iodine – with a constant rate of $4,4 \cdot 10^{12}$ m⁻¹s⁻¹. Relevant reactions involving bromine [11] may occur with the rate constants of 160 m⁻¹s⁻¹ and

330 m⁻¹s⁻¹, respectively. Therefore, we can expect the formation of minor impurities in the extraction of bromine reaction:

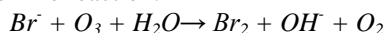


Table 2 – Comparison of experimental (e) and calculation (c) values of the iodine formation degree with changes of the ion and molar ratio (IMR). Ozone: J - ion concentrations and J – 30, 50, 70 mg/dm³.

IMR	The degree of formation of iodine, % wt., At concentrations of J · mg/dm ³								
	30			50			70		
	e	c	Δ	e	c	Δ	e	c	Δ
0.146				4.4	3.9	0.5			
0.208							5.9	6.7	0.8
0.293	6.3	10.7	4.4						
0.44				16.5	17.3	0.8			
0.486	14.3	19.1	4.8						
0.625							29.9	24.9	5.1
0.88				34.3	34.2	0.1			
1.25							44.8	45.7	0.9
1.459	55.0	51.1	3.9						
1.76				61.2	57.4	3.8			
2.19	59.3	63.4	4.1						
2.5							61.1	65.5	4.4
2.64				67.2	65.9	1.3			
3.12							66.5	64.5	2.0

Note: Δ - deviation defined as [e-c].

Simultaneous determination of the bromine concentration in the reaction mixture indicates that this response in the studied conditions is practically not taking place. This is confirmed by changes of bromine ion concentration over the time of ozonation:

t,s	0	10	30	60	90	120	180	300
Br ⁻ , mg/dm ³	40	40	39	39	39	400	390.4	400
	0	0	5.	5.	5.			
			5	5	5			

The maximum deviation of the concentration of bromide ions from the initial concentration does not exceed 2.4%, and the average is 1%. These research results are consistent with the data [11], according to which the reaction rate of conversion of bromide ions into bromine by ozonation is significantly lower than the reaction rate of ozonation of iodide ions [10].

Based on the fact that the rate of reactions of iodine formation is characterized by values 10⁹ - 10¹² located at the level of rates of vibrations between atoms, it can be assumed that the ozonation reaction occurs without energy activation (ΔE = 0) or with the value close to 0. In this case, the temperature has almost no effect on the reaction and the reaction rate equation: $V = kT/h(\Delta S/R)(-\Delta E/RT)$ is simplified to $V = k T/h (\Delta S/R)$, where k, h - Boltzmann and Planck constants, respectively, and ΔS is an entropy. Given that in the course of chemical reactions such entropy mainly varies from - 100t₀ + 100 J/mol K and put these values with the values of the constants in a simplified expression for the rate of reaction, we get the value of 10⁹ - 10¹⁴ s⁻¹, confirming the high rate of the ozonation reaction. Based on the theoretical calculations and experimental results obtained, it can be referred to instant reactions. The high rate of the reaction requires an appropriate design of the process with the use of maximum surface contact between heterogeneous phases and removal of iodine flow of gas phase - its capture technology.

Comparison of changes in the concentrations of iodide ions with changes in the concentration of iodine in the liquid phase (Fig. 8) during the same reaction time indicates that the experimental dependence deviates from the theoretical dependence (y = x) towards the reduction of iodine concentration with increased conversion of iodide ions. It can be assumed that during the ozonation an iodine is extracted from the reaction zone the gas flow. This is confirmed by the test analysis [12] of the gas phase. This discrepancy between the experimental and theoretical dependences with increased degree of conversion of iodide ions and consequently the ozone reaction time increases.

The estimated relative number of extracted iodine in the gas phase according to the results (Fig. 8) over the ozonation time changes as follows:

t,s	0	10	30	60	90	120
J ₂ , % weight	0	10	16.5	12.4	10.5	22.6

So, during ozonation not only iodine forms from iodide ions, but also iodine particles are extracted in the gas phase, indicating a possible way to simplify the process compared to the air adsorption technology [4].

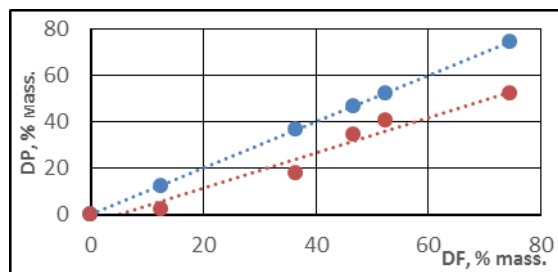


Figure 8 – The dependence of the degree of conversion of J - ions (DP) on the degree of formation (DF) of iodine in the liquid phase (mineralization of 200 g/dm³, the value of pH₀ = 5.84, the concentration of iodide ions is 50 mg/dm³, bromid-ion = 400 mg/dm³, ions Fe²⁺ = 44 mg per cubic dm).

The obtained results taken together suggest that through the use of ozone-and air mixtures with low

concentration of ozone instead of a number of hazardous reagents iodine can be obtained and thus environmental safety in the processing and return of filed waters in layers is increased.

CONCLUSIONS.

1 It is scientifically proved that the use of ozone instead of a number of environmentally harmful reagents used for extracting iodine from drilling water allows to receive iodine and improve environmental safety in the return of field waters into the layer.

2 The research has found that most changes in the degree of formation of iodine under the influence of time, acidity, salinity, concentration of iodine ions are characterized by extreme dependencies.

3 The mathematical statistical models have been found which under certain conditions adequately describe the degree of iodine formation on the mineralization, ozonation time, molar and ion ratio of reactants, with a tolerance of changes.

4 It has been determined that the degree of formation of iodine increases with an increased surface of contact between heterogeneous phases, and in terms of the value of the experimentally and theoretically estimated rate constant, the ozonation reaction of iodide ions can be attributed to the instant reactions.

5 It has been shown s that during the ozonation a part of iodine formed is extracted from the reaction zone by the gas flow.

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НАУЧНОЕ ОБОСНОВАНИЕ ПОВЫШЕНИЯ ЭКОБЕЗОПАСНОСТИ ПРИ ВОЗВРАТЕ ПОПУТНО-ПЛАСТОВИХ ВОД ГАЗОКОНДЕНСАТНЫХ МЕСТОРОЖДЕНИЙ

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Приведено результаты научного обоснования повышения экобезопасности при получении йода с минерализованных попутно-пластовых вод газоконденсатных месторождений при их переработке озонированием, вместо применения ряда вредных реагентов, и возвращении в пласт, которое относится к влиянию времени, кислотности, минерализации, концентрации йодид-ионов, мольно-ионного отношения реагентов, поверхности контакта фаз, присутствия ионов брома, извлечения йода с газовой фазой. Показано, что изменения степени образования йода под влиянием времени, кислотности, минерализации, концентрации ионов йода характеризуются экстремальными зависимостями и линейной зависимостью при мольно-ионных отношениях реагентов до 2 с изменением концентрации йодид-ионов. Получены математические модели, которые с погрешностью 3-5 % совпадают с экспериментальными результатами. За экспериментально и теоретически оцененными величинами скорости реакции озонирование йодид-ионов отнесено к мгновенным реакциям.

Ключевые слова: время, концентрация, йод, озонирование, минерализация, вода.