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NEW MULTIFUNCTIONAL CORROSION INHIBITOR OF STEEL IN FORMATION WATER WITH OIL CONTAINING HYDROGEN SULFIDE AND CARBON DIOXIDE

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Gravimetric methods were initially employed to examine the influence of inhibitor IB-1 on the corrosion rate of steel in formation water with oil, which contained hydrogen sulfide, carbon dioxide, and a combination of both. In order to assess the effectiveness of inhibitor IB-1, laboratory tests were conducted using samples of steel of grade St3. Corrosion experiments were conducted within sealed containers with a volume of 0.5 liters, using samples sized 30×20×1 mm. The effectiveness of the IB-1 inhibitor in formation water with oil, containing hydrogen sulfide, carbon dioxide, and a simultaneous presence of hydrogen sulfide and carbon dioxide, exhibited a variation within the ranges of 88.3% to 98.0%, 72.4% to 92.7%, and 60.22% to 94.83%, respectively. The laboratory investigations allowed for the determination of the optimal concentration of inhibitor IB-1 to inhibit the growth of sulfate-reducing bacteria and protect steel of grade St3 from corrosion induced by hydrogen sulfide, carbon dioxide, and the concurrent presence of hydrogen sulfide and carbon dioxide.

Keywords: protection, corrosion, hydrogen sulfide, carbon dioxide, inhibitor, bacteria, sulfate reduction, bactericide, effectiveness.

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Introductэon

Corrosion of metal equipment leads to huge economic and environmental damage in many industries. Significant losses are observed in oil-gas producing industry due to the presence of aggressive technological environments containing acid gases (H_2S, CO_2). The appearance of hydrogen sulfide and the growth of its concentration are associated mainly with the vital activity of sulfate-reducing bacteria (SRB), which enter the productive formations with surface waters of formation pressure maintenance systems. The corrosion rate is 1.5 mm/year or more, and the life cycle of field oil and gas pipelines does not exceed 2–3 years, while a standard period is 10 years. The most dangerous ones are local corrosion lesions in the form of pits and ulcers, which in some cases lead to ruptures 6–8 months after the exploitation of a new pipeline [1–5].

The use of corrosion inhibitors is one of the most common techniques to reduce the level of corrosion losses during the exploitation of field equipment and oil and gas pipelines. To date, considerable practical experience has been accumulated in this field. However, the difference in corrosive aggressiveness of working environment and changes in the exploitation conditions of equipment and facilities at different stages of development put forward new requirements for the selection of inhibitors and the improvement of inhibitor protection technology [6–10].

Despite the wide range of reagents available, there is a constant search for new inhibitors and inhibitory compositions being able to provide a comprehensive protective effect: not only slowing down the corrosion process, but also effectively protecting equipment from corrosion-mechanical destruction and reducing the growth of SRB and other bacterial cultures [11–13].

This research was aimed at investigating the anticorrosive and bactericidal properties of an organic compound, inhibitor IB-1, as a new multifunctional corrosion inhibitor of steel in formation water with oil containing hydrogen sulfide and carbon dioxide.

Experimental

The investigations were carried out on samples of steel St3 in formation water with oil, containing hydrogen sulfide, carbon dioxide and a simultaneous presence of hydrogen sulfide and carbon dioxide. For corrosion tests, steel samples were used in the form of rectangular plates 30×20×1 mm in size.

Prior to the investigation, the surface of the samples was grinded (using a grinding machine) on abrasive paper with successively decreasing grain sizes. Final grinding was carried out on paper with a grain size of 10−50 μm («zero»). After grinding on paper of each grain type, the metal surface was washed with cold tap water and dried with filter paper. After final grinding, the processed surface was washed with water, dried with filter paper, and degreased with isopropyl alcohol. Inhibitor IB-1 was tested as a corrosion inhibitor.

Corrosion tests were carried out in a special instrument recommended by the state standard SS 9.506-87. The tests were carried out at a fluid flow rate of 1 m/s, temperature of 25 ± 2^0C; and testing time of 6 hours. After corrosion testing, the samples were removed from the container, washed with running water, corrosion products were removed, washed with water, dried with acetone, kept over molten $CaCl_2$ in a desiccator, and weighed on an analytical balance with an accuracy of 0.0001 g [14,15].

The corrosion rate was calculated by the formula:

$$K = \frac{\Delta m}{S \cdot \tau},$$

where Δm is the difference in the sample weights before or after exposure; S is the surface area of the sample; and τ is the exposure time.

Braking ratio was calculated as:

$$\gamma = \frac{K_0}{K_{inh}},$$

where K_0 is the corrosion rate of the sample in the absence of inhibitor; and K_{inh} is the corrosion rate in the presence of inhibitor ($g/m^2 \cdot h$).

The protective effectiveness of inhibitor was calculated by the following formula:

$$Z = \frac{K_0 - K}{K_0} \cdot 100\%,$$

where K_0 and K are the corrosion rates in non-inhibited and inhibited solutions, respectively.

Corrosion penetration was calculated by the formula:

$$\Pi_K = 1.12 \cdot K.$$

The bio-corrosive aggressiveness of formation water (pure and with additives of inhibitors) was determined by the method of limiting dilutions in Postgate environment [17]. This procedure is based on the standard microbiological method. Sodium lactate was used as a source of nutrition to detect sulfate-reducing bacteria (SBR); lactic acid and sodium acetate were used as a source of carbon nutrition to identify bacteria with other nutritional needs; metallic powder was used to identify bacteria that do not need a mandatory supply of a large amount of organic matter, but use molecular hydrogen.

The development of sulfate reduction was recorded based on the increase in biogenic hydrogen sulfide in seed flasks, which was determined by a qualitative-visual method, quantitative-iodometric titration, as well as by the amount of SRB in 1 ml of the investigated water [16].

Visually, the determination of SRB and formation of hydrogen sulfide was observed by the formation of black precipitate-iron sulfide, formed as a result of the reaction of hydrogen sulfide with ferrous iron ions contained in the environment. The presence of living SRB cells was determined using a microscope after incubation in a thermostat, for 15 days, of seed penicillin sample bottles with Postgate nutrient environment [17], and taken after the formation of black precipitate in them. Bactericidal properties of the combined inhibitor were tested according to RD 39-3-973-83. In order to study the bactericidal properties under laboratory conditions, enrichment culture of sulfate-reducing bacteria isolated from formation water of oil fields was used according to the method [18].

Quantitative determination of biogenic hydrogen sulfide was carried out after 15 days of incubation, the contents of the seed bottles were transferred into a flask, and chemical analysis was performed using the iodometric method. The amount of hydrogen sulfide was calculated based on the analysis results.

When calculating the amount of SRB in the grown crop, where the maximum dilution is used, it is considered that one bacterial cell is present.

Taking into account the dilutions, the content of bacteria in 1 ml of inoculum was calculated and their number was expressed as an order of units, tens, hundreds, thousands, etc.

During the period of incubation of samples, the emergence of black precipitate-iron sulfide, formed in the interaction of biogenic hydrogen sulfide (the result of the vital activity of SRB) with iron salts contained in the nutrient environment, characterizes the rate of development of SRB. This rate was estimated over time in terms of the activity index.

The number of cells in 1 ml of the initial suspension was calculated according to the formula:

$$M = \frac{a \cdot 1000}{h \cdot S} \cdot n,$$

where M is the number of cells in 1 ml of suspension; a is the average number of cells in the grid square; h is the chamber depth (mm); S is the square of the grid (mm^2); and n is the dilution of the initial suspension.

The suppression coefficient of the number of SRB cells of the studied compositions was calculated by the ratio:

$$N = \frac{n_0 - n_{ina}}{n_0} \cdot 100\%,$$

where n_0 and n_{ina} are the number of microorganisms in the absence and presence of inhibitors, respectively, other conditions being equal.

The effectiveness of the inhibitor was determined from the magnitude of the suppression degree of microorganisms' vital activity:

$$S = \frac{C_0 - C_i}{C_0} \cdot 100\%,$$

where C_0 and C_i are the concentrations of biogenic hydrogen sulfide in the absence and presence of inhibitors, respectively.

For quantitative analysis of the change in the concentration of hydrogen sulfide, a coefficient γ_e was determined from the following ratio:

$$\gamma_e = \frac{C_{(H_2S)_0}}{C_{(H_2S)}},$$

where $C_{(H_2S)_0}$ is the concentration of hydrogen sulfide in non-inhibited environments; and $C_{(H_2S)}$ is the concentration of hydrogen sulfide in inhibited environments.

2,4-Dichloro-1-{2-iodo-1[(prop-2-in-1-il)oxy]-ethyl}benzene was used in work [19] as a bactericide-inhibitor (chemical formula of organic compound is $C_{11}H_9Cl_2IO$, and its designation is IB-1). Organic substances of IB-1 are more soluble in water and in various other solvents, so they have a stronger bactericidal-inhibitory effect.

Table 1

Results of laboratory tests to determine the effectiveness of the IB-1 inhibitor*

C_{inh}	S, m^2	M_1, g	M_2, g	M_1–M_2, g	V_1, g/m^2·h	V_2, g/m^2·h	γ	K_p, mm/year	Z, %
H_2S environment									
0	0.0013	8.6697	8.6663	0.0034	0.4326	–	–	–	–
10	0.0013	8.6697	8.6693	0.0004	0.4326	0.0506	8.54	0.0566	88.3
15	0.0013	8.6697	8.6694	0.0003	0.4326	0.0328	13.18	0.0367	92.4
20	0.0013	8.6697	8.6696	0.0001	0.4326	0.0190	22.76	0.0212	95.6
25	0.0013	8.6697	8.66963	0.00007	0.4326	0.0086	50.30	0.0096	98.0
CO_2 environment									
0	0.0013	8.6697	8.6680	0.0017	0.2234	–	–	–	–
10	0.0013	8.6697	8.6692	0.0005	0.2234	0.0616	3.62	0.0689	72.4
15	0.0013	8.6697	8.6694	0.0003	0.2234	0.0408	5.47	0.0456	81.7
20	0.0013	8.6697	8.6695	0.0002	0.2234	0.0232	9.62	0.0259	89.6
25	0.0013	8.6697	8.6696	0.0001	0.2234	0.0163	13.7	0.0182	92.7
H_2S+CO_2 environment									
0	0.0013	8.6697	8.6692	0.0005	0.06231	–	–	–	–
10	0.0013	8.6697	8.6695	0.0002	0.06231	0.0247	2.52	0.0276	60.22
15	0.0013	8.6697	8.6696	0.0001	0.06231	0.0187	3.33	0.0209	69.86
20	0.0013	8.6697	8.6692	0.0005	0.06231	0.0080	7.78	0.0089	87.15
25	0.0013	8.6697	8.66968	0.00002	0.06231	0.0032	19.47	0.0035	94.83

Note: * – «0» stands for the case without the use of inhibitor; S is the sample area; M_1 and M_2 are the weights of the sample before and after the test, respectively; M_1–M_2 is the weight loss of the sample; V_1 and V_2 are the corrosion rates in non-inhibited and inhibited environments, respectively; K_p is the penetration coefficient; and Z is the degree of corrosion inhibitor protection.

Results and dəscussion

The protective properties of IB-1 inhibitor were investigated in formation water with oil containing hydrogen sulfide, carbon dioxide, and a combination of both. The results of laboratory tests on the definite effectiveness of corrosion inhibitors and studies of the inhibitor IB-1 effect are shown in Table 1. In all cases, the average value of the sample weight was taken from two parallel tests on samples in the amount of at least three for each test.

As is seen from Table 1, an increase in the concentration of IB-1 reagent in the range of 10–25 mg/l in all three aggressive corrosive environments leads to a decrease in the corrosion rate during 6 hours tests.

In both cases, the greatest effect was observed at a reagent amount of 25 mg/l. As can be seen from Table 1, this effect was 98%, 92.7%, and 94.8% in the hydrogen sulfide environment, in the carbon dioxide environment, and in the mixed environment (H_2S+CO_2), respectively. Thus, based on the analysis results of laboratory experiments, it was determined that the optimal concentration of the investigated inhibitor IB-1 for corrosion protection is 25 mg/l.

Figures 1, 2 and 3 provide information on the effectiveness of the inhibitor percentage. The highest efficiency was observed at a concentration of 25 mg/l.

The bactericidal properties of IB-1 inhibitor in relation to sulfate-reducing bacteria were evaluated in Postgate nutrient environment, composition, g/l: NH_4Cl 1.0; K_2HPO_4 0.5; $MgSO_4{\cdot}7H_2O$ 2.0; Na_2SO_4 0.5; $CaCl_2$ 0.1, calcium lactate 2.6.

The investigated microorganisms were obtained in the laboratory and identified as *Desulfovibriodesulfuricans* and *Desulfomicrobium*.

The investigation of the bactericidal properties of IB-1 inhibitor showed that it effectively suppresses the increase in the number of SRB in Postgate nutrient environment, i.e. in the most comfortable conditions for their development and vitality.

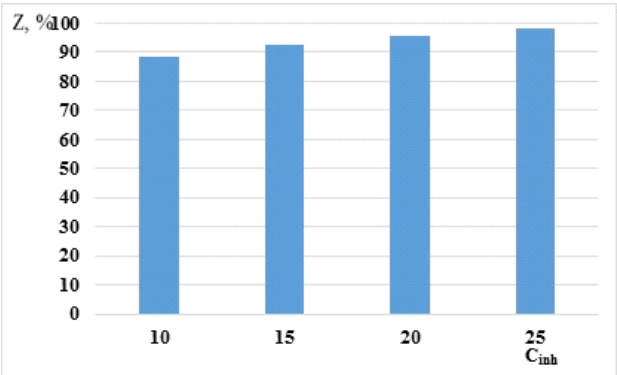

Fig. 1. Diagram of inhibitor protection degree in H_2S environment

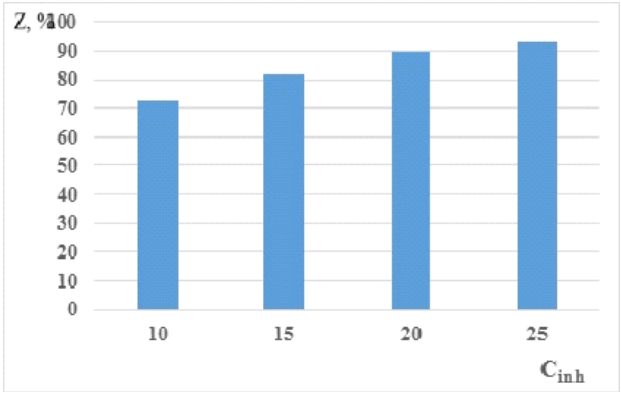

Fig. 2. Diagram of inhibitor protection degree in CO_2 environment

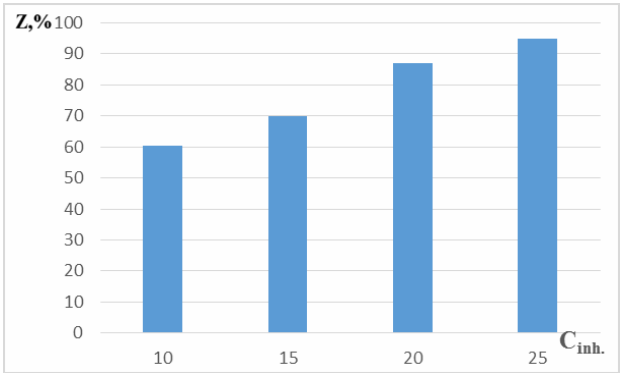

Fig. 3. Diagram of inhibitor protection degree in H_2S+CO_2 environment

The introduction of IB-1 inhibitor and an increase in its concentration in Postgate environment reduces the number of microorganisms of both types (Figs. 4 and 5).

When the concentration of the inhibitor changes from 10 to 25 mg/l, already on the first day, the suppression coefficient of the number of SRB cells (N) of *Desulfovibriodesulfuricans* increases from 15% to 35% (Fig. 4), whereas it does not exceed

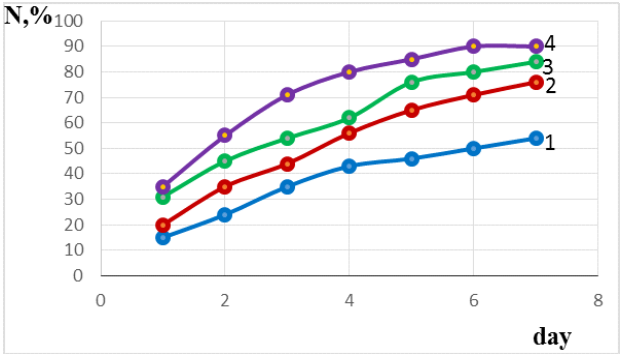

Fig. 4. Dependence of the suppression degree of the number of *Desulfovibriodesulfuricans* cells on the concentration of inhibitor IB-1 mg/l: 1 – 10; 2 – 15; 3 – 20; and 4 – 25

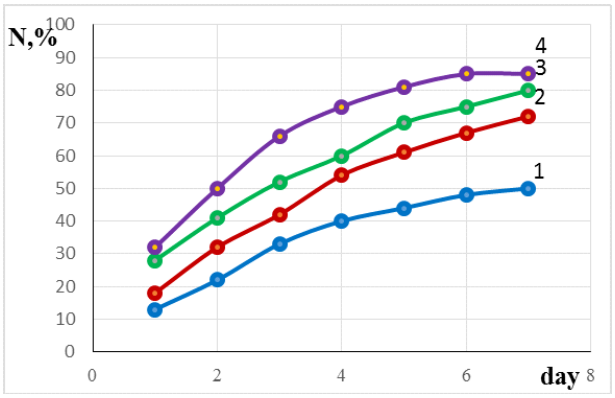

Fig. 5. Dependence of the suppression degree of the number of *Desulfomicrobium* cells on the concentration of inhibitor IB-1 mg/l: 1 – 10; 2 – 15; 3 – 20; and 4 – 25

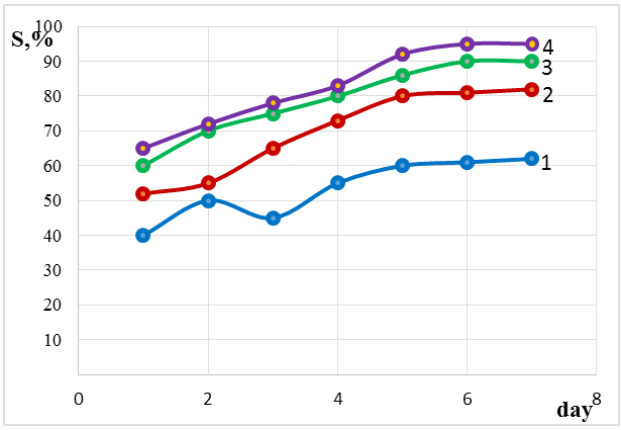

Fig. 6. Dependence of the suppression degree of hydrogen sulfide production by *Desulfovibriodesulfuricans* bacteria on the concentration of inhibitor IB-1 mg/l: 1 – 10; 2 – 15; 3 – 20; and 4 – 25

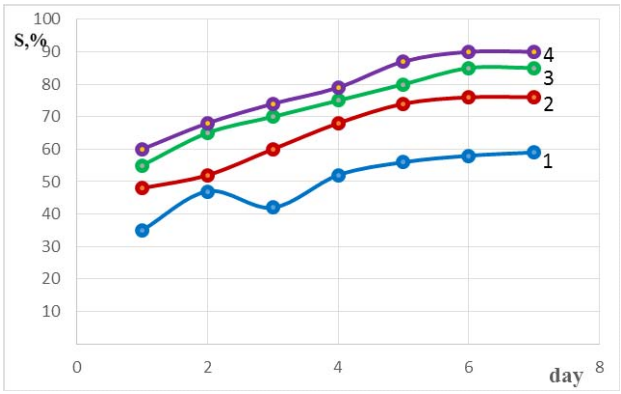

Fig. 7. Dependence of the suppression degree of hydrogen sulfide production by *Desulfomicrobium* bacteria on the inhibitor concentration IB-1mg/l: 1 – 10; 2 – 15; 3 – 20; and 4 – 25

32% for *Desulfomicrobium* (Fig. 5). On the third day, in the first case, N reaches 71% when C_{inh}= =25 mg/l, and it reaches up to 90% on the seventh day. In the second case, N reaches 66% on the third day at the same inhibitor concentration and increases to 85% on the sixth and seventh days. If in the first case, N=76–84% with C_{inh}=20 mg/l on days 5–7, and N=85–90% at a concentration of 25 mg/l, then N=85% in the second case on seven days. Thus, the investigated inhibitor in the first three days more effectively suppresses the number of *Desulfovibriodesulfuricans* cells than *Desulfomicrobium*, and by the end of the life cycle of bacteria, N value is 90% in the first case, and it is 85% in the second case when C_{inh}=25 mg/l. when C_{inh}=20 mg/l, N=84% and N=80% for *Desulfovibriodesulfuricans* and *Desulfomicrobium*, respectively (Figs. 4 and 5).

The suppression degree of hydrogen sulfide production (Figs. 6 and 7) by microorganisms *Desulfovibriodesulfuricans* is 92–95% at an inhibitor concentration of 20 and 25 mg/l on 7 days, while it is S=87–90% for *Desulfomicrobium*. Thus, the inhibitor more effectively suppresses the production of H_2S on the seventh day in the first case than in the second one.

In both cases, IB-1 inhibitor reduces the amount of biogenic hydrogen sulfide, but does not completely stop the sulfate reduction process. Obviously, the inhibitor, preventing the reproduction of SRB in a nutrient environment, cannot completely stop the processes of their metabolism. In this case, the inhibitor-bactericide, easily dissolving the plasma membrane of the bacterial cell wall and penetrating into the cell, prevents the growth of bacteria.

The number of bacteria germinating in the environment was determined under a microscope. It was found that the number of bacteria was $n=10^7$ in the non-inhibited control environment.

As is seen from Table 2, IB-1 demonstrates bactericidal effect of 70%, 78%, 92%, and 98% at a concentration of 10 mg/l, 15 mg/l, 20 mg/l, and 25 mg/l. Thus, the IB-1 inhibitor shows high bactericidal properties at a concentration of 25 mg/l.

As can be seen from Fig. 8, the number of bacteria is $n=1\cdot10^7$ in non-inhibited environment, whereas in inhibited environment, the number of bacteria decreases from 10^7 to 10^5, 10^3, 10^2 and 10^1 at a concentration of 10 mg/l, 15 mg/l, 20 mg/l and 25 mg/l, respectively.

Conclusions

1. By means of gravimetric testing, the inhibitory properties of inhibitor IB-1 were investigated for the corrosion of steel in formation water with oil containing hydrogen sulfide, carbon

Table 2

Results of the bactericidal effect of the IB-1 inhibitor

Concentration of IB-1, C, mg/l	Number of bacteria (number of cells/ml)	H_2S content, mg/l	Bactericidal effect, %
0	10^7	270	–
10	10^5	80	70
15	10^3	60	78
20	10^2	22	92
25	10^1	5.4	98

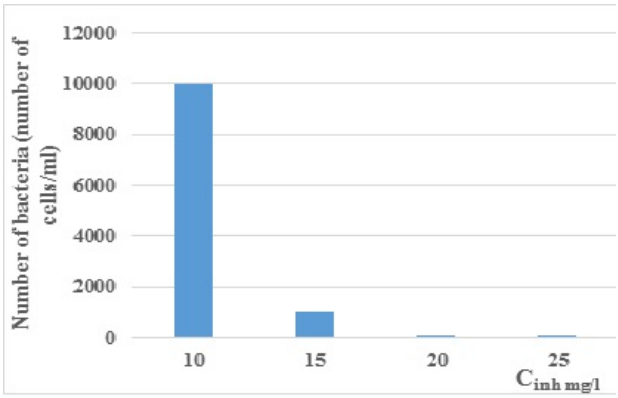

Fig. 8. Diagram of the impact of IB-1 on the amount of SRB

dioxide and a combination of both. The protective effect of inhibitor IB-1 was investigated in aggressive environment of H_2S, CO_2 and H_2S+CO_2 for 6 hours. Experiments were carried out at a temperature of 25^{0}C for IB-1 inhibitor concentrations of 10, 15, 20 and 25 mg/l to determine the corrosion rate of steel sheets St3 by weight loss. The highest effect of IB-1 inhibitor was found in the amount of 98% at 25 mg/l in hydrogen sulfide environment, 92.7% in carbon dioxide environment and 94.83% in mixed environment (H_2S+CO_2).

2. The bactericidal action of the investigated inhibitory compositions was studied using two cultures of SRB (*Desulfomicrobium* and *Desulfovibriodesulforicans*). It was shown that the investigated inhibitor in the first 3 days more effectively suppresses the number of *Desulfovibriodesulfuricans* cells than *Desulfomicrobium*, and by the end of the life cycle of bacteria, N value is 90% in the first case, and in the second case it is 85% when C_{inh}=25 mg/l. When C_{inh}=20 mg/l, it is N=84% for *Desulfovibriodesulfuricans*, while it is N=80% for *Desulfomicrobium*.

3. The suppression degree of hydrogen sulfide production by microorganisms *Desulfovibriodesulfuricans* is 92–95% at an inhibitor concentration of 20 and 25 mg/l on 7 days, respectively, and it is 87–90% for *Desulfomicrobium*. Thus, the inhibitor more effectively suppresses the production of H_2S on the seventh day in the first case than in the second one.

The obtained experimental data can be used by specialists from oil and gas companies to arrange inhibitor protection of oil and gas complex equipment from hydrogen sulfide, carbon dioxide and microbiological corrosion.

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НОВИЙ БАГАТОФУНКЦІОНАЛЬНИЙ ІНГІБІТОР КОРОЗІЇ СТАЛІ У ПЛАСТОВІЙ ВОДІ З НАФТОЮ, ЩО МІСТИТЬ СІРКОВОДЕНЬ ТА ДІОКСИД ВУГЛЕЦЮ

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У роботі застосовувалися гравіметричні методи для вивчення впливу інгібітора ІВ-1 на швидкість корозії сталі в утворюваній воді з нафтою, яка містила сірководень, діоксид вуглецю та обидва одночасно. Для визначення ефективності інгібітору ІВ-1 були здійснені лабораторні випробування на зразках сталі марки St3. Корозійні випробування проводилися в герметичних контейнерах об'ємом 0,5 л на зразках розміром 30×20×1 мм. Ефективність інгібітора ІВ-1 в утворюваній воді з нафтою, яка містила сірководень, діоксид вуглецю та одночасно сірководень та діоксид вуглецю коливалася у межах 88,3−98,0%, 72,4−92,7% та 60,22−94,83%, відповідно. Лабораторні дослідження дозволили визначити оптимальну концентрацію інгібітору ІВ-1 для пригнічення росту сульфат-відновлюючих бактерій та захисту сталі марки St3 від корозії, спричиненої сірководнем, діоксидом вуглецю та одночасною присутністю сірководню та діоксиду вуглецю.

Ключові слова: захист, корозія, сірководень, діоксид вуглецю, інгібітор, бактерії, зниження сульфатів, бактерицид, ефективність.

NEW MULTIFUNCTIONAL CORROSION INHIBITOR OF STEEL IN FORMATION WATER WITH OIL CONTAINING HYDROGEN SULFIDE AND CARBON DIOXIDE

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Gravimetric methods were initially employed to examine the influence of inhibitor IB-1 on the corrosion rate of steel in formation water with oil, which contained hydrogen sulfide, carbon dioxide, and a combination of both. In order to assess the effectiveness of inhibitor IB-1, laboratory tests were conducted using samples of steel of grade St3. Corrosion experiments were conducted within sealed containers with a volume of 0.5 liters, using samples sized 30×20×1 mm. The effectiveness of the IB-1 inhibitor in formation water with oil, containing hydrogen sulfide, carbon dioxide, and a simultaneous presence of hydrogen sulfide and carbon dioxide, exhibited a variation within the ranges of 88.3% to 98.0%, 72.4% to 92.7%, and 60.22% to 94.83%, respectively. The laboratory investigations allowed for the determination of the optimal concentration of inhibitor IB-1 to inhibit the growth of sulfate-reducing bacteria and protect steel of grade St3 from corrosion induced by hydrogen sulfide, carbon dioxide, and the concurrent presence of hydrogen sulfide and carbon dioxide.

Keywords: protection; corrosion; hydrogen sulfide; carbon dioxide; inhibitor; bacteria; sulfate reduction; bactericide; effectiveness.

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