**Experimental and Theoretical Study of Guanidine-Based Corrosion Inhibitors for St20 Steel**

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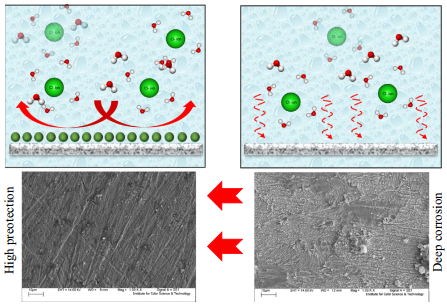
**Abstract:** Currently, various new types of corrosion inhibitors are being studied, which is why guanidine derivatives are being applied. In this work, new composite organic corrosion inhibitors FKDIGK-3 and FKG2T-4 have been synthesized based on guanidine nitrate, orthophosphoric acid, glycerol, and thiourea. These organic FKDIGK-3 and FKG2T-4 inhibitors were applied for the first time as corrosion inhibitors for St20 grade steel in working solutions of 1 M HCl and produced water (pH=4.5). The study of the corrosion process on the metal surface was fully characterized using weight loss (gravimetric) analysis. The obtained results indicate that the FKG2T-4 inhibitor exhibits a corrosion inhibition efficiency of 94.36% at a concentration of 100 mg/L within the temperature range of 293–323 K. Gravimetric analysis confirmed that FKG2T-4 acts as a mixed-type inhibitor.

**Keywords:** Dicyandiamide, Urea, Ammonium nitrate, Silicon (IV) oxide, Hydrochloric acid, Guanidine nitrate, Orthophosphoric acid, Glycerol, Thiourea, FKG2T-4 organic inhibitor, Produced water, St20 grade steel.

**INTRODUCTION**

The term "corrosion" originates from the Latin word "corroder," meaning "to gnaw away" or "to decay." Corrosion of metals and alloys is the process of their spontaneous deterioration due to chemical or electrochemical reactions with the external environment, and sometimes under the influence of mechanical factors. In such environments, metals release free electrons in accordance with kinetic laws, leading to a decrease in their energy. As a result of corrosion, metal structures of plants and factories suffer significant material damage. Developing methods to prevent and protect against corrosion is crucial for ensuring the long-term and reliable operation of metal components. One of the most important methods for protecting metals from corrosion is the use of inhibitors. In recent decades, numerous organic compounds have been studied and utilized as corrosion inhibitors for metals [1-3].

Several methods exist to protect metal structures from corrosion. Among the protection methods, one of the most effective and economical is the inhibition technique, which involves adding a small amount of inhibitor (10-6 to 10-3 mol/L) to the corrosive environment. Inhibitors are substances active in corrosive media; when introduced into the corrosion process, they cause the corrosion to slow down or cease completely [4].



**FIGURE 1.**High protection efficiency of the inhibitor against pitting corrosion on the metal surface.

In the chemical industry, solutions of strong acids are frequently used in various cleaning and pickling processes. St20 steel is susceptible to corrosion under the influence of these acidic solutions, leading to economic and environmental risks. Currently, compounds such as imidazolines, amines, amino acids, surfactants, polymers, plant extracts, ionic liquids, and natural polymer composites are widely used to protect carbon steel from acid corrosion [5-8]. The reason these aforementioned compounds are employed as corrosion inhibitors is that they contain numerous electron-rich heteroatoms (S, N, O, and P), π-electrons, aromatic rings, coordinating functional groups, and aliphatic amines. They dissolve and act as adsorbents on the steel surface in acidic solutions [9-12].

Metal Sample and Corrosive Solution

For experimental analysis, St20 grade steel was selected as it is a widely used metallic material in the chemical industry. Corrosion of St20 steel is a major concern in the oil, gas, and metallurgy industries. Protecting St20 steel from corrosion in aggressive environments is one of the critical tasks in engineering.

St20 steel,according to GOST 1050-88, is a carbon steel grade 20 with a composition of Fe 97.215-97.775%, C 0.17-0.24%, Si 0.17-0.37%, Mn 0.35-0.65%, Cu and Ni up to 0.3%, Cr up to 0.25%, As up to 0.08%, S up to 0.04%, and P up to 0.035% [13].

Prior to experiments, the St20 steel samples were cleaned by abrading with fine-grit sandpaper (grades 150-1000). Subsequently, the steel was rinsed sequentially four times with distilled water and ethanol, degreased with acetone, and then dried. The first working solution used as the corrosive medium was 1M hydrochloric acid (pH=4.56) (Solution-1), and the second was a solution of produced water (graderniy water) with a pH of 4.5 (Solution-2). In this study, the organic inhibitor FKG2T-4 was applied within a concentration range of 25, 50, 75, and 100 mg/L. The pH levels of the pure solutions were constantly monitored and adjusted to approximate values typical for industrial water systems.

At the "Muborak Gas Processing Plant" JSC, activated carbon sorbent brand AU-60 was used to purify the closed-cycle cooling (graderniy) water from acids and salts. The results of laboratory-scale adsorption purification tests for industrial recirculating cooling water using reactivated AU-60 carbon sorbent are presented (Table 1).

**TABLE 1.** Results of ion content removal from produced water using the adsorption purification method with AU-60 activated carbon sorbent.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Adsorbents** | **Indicators of cooling water, mg/l** | | | | | | |
| **pH** | **Na+** | **Ca2+** | **Mg2+** | **Cl-** | **SO42-** | **HCO3-** |
| **The amount of ions in the cooling water** | **4,5** | **21** | **92** | **61,2** | **640** | **903** | **11** |

Gravimetric method. Equipment and materials: St-20 steel plates 10x18x1x 2-4 mm, laboratory beakers of various sizes, glass rods, hydrochloric acid, table salt and Graderni water, distilled water, capron thread, etc. determination of corrosion stability of St-20 steel sample.

Corrosion rate W (g/m2 h) was calculated based on the following formula.

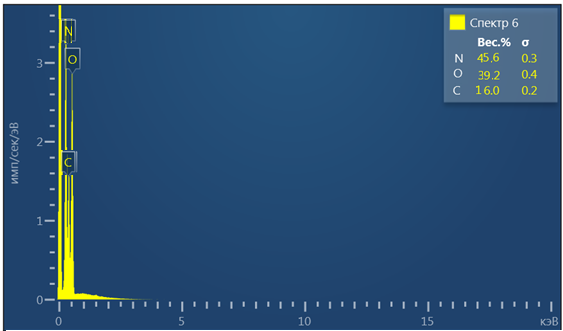
 (1)

where: **W** - is the corrosion rate (g/cm2·h)  
 **m1** = the metal plate's mass in grams before to the experiment; (g)  
 **m2** = the metal plate's mass (in grams) following the experiment (g)  
 **S** = the surface of the metal plate, (cm²)  
 **τ** = time of the experiment (hours)

Based on the corrosion rate values with inhibitor (**W**\_**ing**) and without inhibitor (**W₀**), the inhibition coefficient **γ** (2), protection efficiency **Z%** (3), and surface coverage degree **θ** (4) are calculated [14].

(2) (3) (4) 

Elemental analysis of the synthesized guanidine nitrate was performed, and the results are presented in Figure 2 [15].



**FIGURE 2.** Elemental analysis of guanidine nitrate

Urea (chemical formula (NH2)2CO; Mr = 60.07) is a white crystalline substance that is soluble in polar solvents such as water, ethanol, and liquid ammonia (Table 2).

**TABLE 2.** Physicochemical properties of urea.

|  |  |  |
| --- | --- | --- |
| **№** | Parameter Name | Standart per **SST 2081-2010** |
| 1 | State | White crystals |
| 2 | Melting point, °C | 132.7 |
| 3 | Boiling point, °C | 174 (decomposes) |
| 4 | Solubility in water (20 °C), g/100 g | 51.8 |
| 5 | Density, g/cm3 | 1.32 |

Orthophosphoric acid (phosphoric acid) –with the empirical formula H₃PO₄, is a hygroscopic crystalline substance under standard conditions and a medium-strength weak inorganic acid. Typically, the term orthophosphate (or phosphate) acid refers to its 85% aqueous solution (a colorless, odorless, syrupy liquid). It is soluble in water, ethanol, and other solvents. Its molecular weight is 98 g/mol, density is 1.685 g/ml, and boiling point is 158 °C (Table 3).

**TABLE 3.** Quality indicators of pure orthophosphoric acid

|  |  |  |
| --- | --- | --- |
| **№** | Parameter Name | Standard per **SST 6552-80** |
| 1 | Mass fraction of orthophosphoric acid, %, not less than | 85 |
| 2 | Density, g/cm3 | 1.60 |

Steel grade St-20,according to SST 1050-88, is a structural carbon steel alloy of grade 20 and consists of the elements listed in Table 4 [16].

**TABLE 4.** Chemical composition of St-20 steel, %

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Fe** | **C** | **Si** | **Mn** | **Ni (max)** | **Cu (max)** | **Cr (max)** | **As (max)** | **S (max)** | **P (max)** |
| 97.215-97.775 | 0.17-0.24 | 0.17-0.37 | 0.35-0.65 | 0.3 | 0.3 | 0.25 | 0.08 | 0.04 | 0.035 |

The strength of steel is largely dependent on the carbon concentration, as an increase in its concentration enhances the strength and brittleness of the steel. Considering these properties, grade 20 steel is defined as a "structural quality carbon steel." Such steel is commonly used to manufacture pipes and turbines of various diameters, various reactor parts, bearings, and many other components utilized across different industrial sectors.

Dicyandiamide – a colorless crystalline substance, soluble in water (3.3% by weight at 20°C) and in methanol (3.8%), with a density of 1.405 g/cm3. It is insoluble in liquid NH3, DMF, and benzene. In terms of chemical properties, dicyandiamide resembles cyanamide (Table 5).

**TABLE 5.** Chemical properties, dicyandiamide resembles cyanamide

|  |  |  |
| --- | --- | --- |
| **№** | Substance Property | Standard per **SST 6988-73** |
| 1 | Appearance | Fine white crystals |
| 2 | Mass fraction of dicyandiamide (dry basis), % | 93 |
| 3 | Mass fraction of water-insoluble residue, % | 0.3 |
| 4 | Mass fraction of water, % | 0.7 |
| 5 | Melting point, °C | 201-203 |

Synthesis of the Organic Corrosion Inhibitor FKDIGK-3 from Phosphoric Acid, Dicyandiamide, Glycerol, and Urea**.**





**FIGURE 3.** Synthetic pathway of the organic corrosion inhibitor FKDIGK-3.

A white-colored mass of 38.2 g with a melting point of 160–180 °C was obtained as the organic corrosion inhibitor FKDIGK-3 (decomposition occurs above 160 °C).

Synthesis of the organic corrosion inhibitor FKG2T-4 based on phosphoric acid, glycerol, guanidine nitrate, and thiourea.







**FIGURE 4.** Synthetic pathway of the organic corrosion inhibitor FKG2T-4.

A white-yellowish substance of 43.2 g with a melting point of 130–150 °C was obtained as the organic corrosion inhibitor FKG2T-4 (decomposition occurs above 160 °C).

This FKG2T-4 organic corrosion inhibitor molecule is water-soluble and contains multiple electron-rich functional groups. Figure 4 illustrates the molecular structure of FKG2T-4, which incorporates heteroatoms (N, O, P, and S) derived from components such as glycerol, phosphoric acid, guanidine nitrate, and thiourea [17].

Based on investigations conducted in Solution-1 (1M hydrochloric acid, pH=4.56) and Solution-2 (produced water, pH=4.5), experiments were performed at inhibitor concentrations of 25, 50, 75, 100, and 125 mg/L to determine the optimal inhibitor concentration. According to the results of the performed experiments and studies, an inhibitor concentration of 100 mg/L was identified as the optimal concentration. Throughout the experiments, it was established that as the concentration increased, the protection efficiency initially rose significantly and subsequently remained almost unchanged.

**TABLE 6.** Dependence of corrosion protection efficiency on concentration for inhibitors FKDIGK-3 and FKG2T-4 in Solution-1 and Solution-2 at 298 K.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **C, mg/l** | **Inhibitor efficiency, %** | | | | |
| **FKDIGK-3** | **FKG2T-4** | **FKDIGK-3** | **FKG2T-4** | |
| **Solution-1** | | **Solution-2** | | |
| 25 | 70,52 | 76,51 | 73,25 | | 79,65 |
| 50 | 78,48 | 82,51 | 79,86 | | 85,38 |
| 75 | 83,64 | 88,32 | 89,82 | | 93,64 |
| 100 | 86,78 | 90,68 | 94,63 | | 96,76 |
| 125 | 89,88 | 92,88 | 94,89 | | 96,78 |

**TABLE 7.** Protection efficiency values of the FKG2T-4 inhibitor in working solutions at 298 K.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Inhibitor, Name | Inhibitor  amount mg/l | time  (hours) | Solution-1 | | Solution-2 | |
|  | **Ζ%** |  | **Ζ%** |
| FKDIGK-3 | 25  50  75  100 | 4  6  8  10 | 0,68  0,84  0,88  0,96 | 88,45  91,75  93,32  94,68 | 0,85  0,87  0,84  0,82 | 92,38  93,66  94,79  95,87 |
| FKG2T-4 | 25  50  75  100 | 4  6  8  10 | 0,79  0,87  0,89  0,98 | 89,56  92,68  94,79  96,89 | 0,84  0,86  0,89  0,85 | 93,56  94,78  95,86  96,78 |

As evident from the data in Table 7, it was established that the corrosion rate of the metal decreases with an increase in inhibitor concentration.

The values of corrosion rate (W), inhibition coefficient (γ), and protection efficiency (Z) determined in our studies via the gravimetric method for the organic corrosion inhibitor FKG2T-4 in two different working solutions over 120 and 240 hours at T=298 K are presented in Table 8.

**TABLE 8.** Gravimetric method for the organic corrosion inhibitor FKG2T-4

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | **W,** gr/(cm2·hour) | **γ** | **Z, %** | **W,** gr/(cm2·hour) | **γ** | **Z, %** |
|  | **120 hour** | | | **240 hour** | | |
| Solution-1 | **1,242** | - | - | **1,231** | - | - |
| FKDIGK-3 | 0,136 | 8,65 | 88,78 | 0,128 | 10,32 | 90,12 |
| FKG2T-4 | 0,124 | 11,29 | 92,18 | 0,087 | 13,53 | 94,53 |
| Solution-2 | **1,106** | - | - | **1,102** | - | - |
| FKDIGK-3 | 0,105 | 10,67 | 91,86 | 0,069 | 10,48 | 93,26 |
| FKG2T-4 | 0,042 | 17,63 | 94,65 | 0,043 | 19,23 | 96,67 |

Based on the results from tests conducted in working solutions Sof-1 and Sof-2 at a concentration of 100 mg/L, our organic inhibitor demonstrated a protection efficiency ranging from 91.86% to 94.65% over 120 hours, and from 93.26% to 96.67% over 240 hours [18].

Given that the protection efficiency of the FKDIGK-3 organic inhibitor was observed to be slightly lower than that of the FKG2T-4 inhibitor, we considered it appropriate to proceed with further analyses using the FKG2T-4 inhibitor. To gain more comprehensive information about its surface coverage and protective mechanisms, studies were conducted via the gravimetric method. These investigations involved varying inhibitor concentrations across a temperature range from 298 K to 323 K (Table 9).

**TABLE 9.** Protection Efficiency of the FKG2T-4 Inhibitor in Sof-2 Working Solution at Various Temperature and Concentration Ranges

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Inhibitor | **T,** (K) | **C,**  (mg/l) | **W,** gr/(cm2·hour) | **γ** | **Z, %** | **θ** |
| **FKG2T-4** | **298** | - | **1,32** | - | - | - |
| 25 | 0,292 | 4,12 | 76,01 | 0,7601 |
| 50 | 0,228 | 5,28 | 81,23 | 0,8123 |
| 75 | 0,139 | 8,35 | 88,45 | 0,8845 |
| 100 | 0,108 | 11,86 | 91,67 | 0,9167 |
| **303** | - | **1,46** | - | - | - |
| 25 | 0,351 | 4,25 | 78,61 | 0,7861 |
| 50 | 0,262 | 5,73 | 83,45 | 0,8345 |
| 75 | 0,158 | 9,42 | 88,76 | 0,8876 |
| 100 | 0,115 | 13,32 | 92,45 | 0,9245 |
| **313** | - | **1,75** | **-** | **-** | **-** |
| 25 | 0,389 | 4,34 | 76,26 | 0,7626 |
| 50 | 0,286 | 5,89 | 82,45 | 0,8245 |
| 75 | 0,172 | 9,72 | 90,72 | 0,9072 |
| 100 | 0,117 | 14,21 | 93,67 | 0,9367 |
| **323** | **-** | **1,95** | **-** | **-** | **-** |
| 25 | 0,429 | 4,41 | 76,34 | 0,7634 |
| 50 | 0,312 | 6,06 | 84,63 | 0,8463 |
| 75 | 0,186 | 9,83 | 88,86 | 0,8886 |
| 100 | 0,125 | 14,61 | 94,36 | 0,9436 |

**CONCLUSION**

The change in protection efficiency with increasing temperature indicates the occurrence of chemical adsorption of the inhibitor on the steel surface. A relatively smaller increase in protection efficiency with corresponding temperature rise suggests that, alongside chemical adsorption, physical adsorption also takes place during the inhibition process. The simultaneous observation of both chemical and physical adsorption phenomena demonstrates that the organic inhibitor exhibits a mixed-type mechanism of action. From our experiments, the corrosion inhibitor FKG2T-4 displayed a maximum protection efficiency of 94.36% at a concentration of 100 mg/L and a temperature of 323 K.

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