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INVESTIGATION OF THE INHIBITORY AND COLLOID-CHEMICAL PROPERTIES OF GXMA (GUANIDINE CHLORIDE METHYLACRYLATE) SYNTHESIZED ON THE BASIS OF GUANIDINE

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Abstract

In this study, guanidine chloride methylacrylate (GXMA), a newly synthesized compound based on guanidine, was investigated as an effective corrosion inhibitor for steel in a 15% hydrochloric acid (HCl) solution. Steel grade St-20 was selected for the experiments, and the inhibitory effect of GXMA at various concentrations (100–300 mg/L) was evaluated using the gravimetric method. The tests were conducted over a period of 300 hours within a temperature range of 343–363 K. The experimental results demonstrated that GXMA significantly reduced corrosion rates. Notably, at a concentration of 300 mg/L, GXMA provided a protection efficiency of 93.3% for St-20 steel. The corrosion rate inhibition coefficient was found to be up to 15 times higher compared to the control. Based on weight loss measurements of steel coupons, the effectiveness of GXMA was found to fully comply with the requirements of GOST (RH)39.0–051:2007. To gain deeper insights into the adsorption mechanisms, graphical analyses were conducted using the Langmuir, Temkin, Frumkin, and Flory-Huggins adsorption isotherms. The obtained R^2 coefficients confirmed that the adsorption of GXMA closely follows the Langmuir model, indicating monomolecular and physico-chemical adsorption on the metal surface. Thermodynamic analyses were carried out based on the Arrhenius equation, and the observed increase in activation energy (Ea), along with a negative entropy change, indicated the predominance of GXMA adsorption processes on the metal surface. The findings revealed that GXMA exhibits maximum inhibitory performance within the concentration range of 250––300 mg/L. This study suggests that GXMA is a promising corrosion inhibitor for protecting metal structures operating in acidic environments, particularly in the oil and gas industry. The results confirm GXMA's potential as a highly efficient, economically viable, and environmentally friendly corrosion inhibitor.

Keywords: *corrosion, inhibitor, guanidine chloride methylacrylate (GXMA), HCl solution, steel coupons, gravimetric method, adsorption isotherm, Langmuir model, activation energy, thermodynamic analysis, physico-chemical adsorption, monomolecular layer, acidic environment, metal protection, oil and gas industry*

1. Introduction

The corrosion of metallic structures operating in acidic environments presents significant technical and economic challenges. Therefore, the use of effective chemical inhibitors is crucial for mitigating corrosion-related damage. In particular, when steel materials used in the oil and gas industry are exposed to strong acidic solutions, there is a pressing need for specialized chemical compounds to extend their service life (Narzullaev A. Kh., Beknazarov Kh. S., Dzhalilov A. T., 2019).

In recent years, guanidine derivatives have gained special attention among organic corrosion inhibitors. This study investigates the corrosion-inhibiting effectiveness of guanidine chloride methylacrylate (GXMA), synthesized from guanidine thiocyanate, formaldehyde, and acrylic acid using locally available raw materials, through the gravimetric method (Beknazarov Kh. S., Dzhalilov A. T., 2015). While guanidine chromate and its derivatives with mineral acids have previously been applied as corrosion inhibitors, the behavior of GXMA in acidic environments remains insufficiently studied. The aim of this research is to evaluate the anticorrosion performance of GXMA and to elucidate its adsorption characteristics (Yaro A. S., Abdul Masih N. Sh., Khadom A. A., 2000; Oguzie E. E., 2006).

During the corrosion process, the adsorption of inhibitors onto the metal surface plays a vital role. To understand these adsorption behaviors, mathematical models are employed. Adsorption isotherms represent mathematical relationships that describe the equilibrium distribution of substances on the surface of the adsorbent. Among these, the Langmuir, Temkin, and Frumkin isotherms are widely used. In particular, the Langmuir isotherm, which assumes monolayer adsorption where each adsorption site binds to only one molecule, is frequently applied (Odiongenyi A. O., Odoemelam S. A. and Eddy N. O., 2009; Olimov B. B., Yoldosheva N. J.). This study explores how these isotherms help explain the interaction of GXMA with the metal surface and assesses their compatibility with experimental results.

2. Materials and Methods

An experimental investigation was conducted to evaluate the corrosion-inhibiting properties of the synthesized GXMA compound. Various concentrations of guanidine chloride methylacrylate were prepared using hydrochloric acid (HCl) solution. Steel samples of grade St-20 were used in the experiments. The steel coupons were polished using SiC abrasive papers, degreased in 96.6% ethanol, and then dried with acetone (Panoev E. R., Dustov H. B., Akhmedov V. N., 2021; Bobir O., Mashhura S., Islom B., 2022).

The cleaned steel coupons were tied with thread and immersed in 100 mL of test solutions maintained at temperatures ranging from 343 to 363 K. A total of six metal coupons were prepared for each test series. One coupon was immersed in a 15% HCl solution without the inhibitor, serving as the control, while the remaining five were placed in 15% HCl solutions containing GXMA at concentrations of 100, 150, 200, 250, and 300 mg/L, respectively.

Table 1. *Dependence of corrosion-inhibiting characteristics of GXMA on its concentration. The experiment was conducted on St-20 steel*

Sample	S, 10^{-4} m^2	τ, hour	mass m_0, g	mass m, g	Δm	Consentration of inhibitor, mg/ml	Rate of corrosion, g/hour·m^2	Z, %	γ
1.	21	300	23,52	22,7	0,813	–	1,29	–	–
2.	21	300	23,68	23,48	0,193	100	0,306	76,2	4,21
3.	21	300	23,76	23,6	0,158	150	0,25	80,6	5,16
4.	21	300	23,61	23,51	0,096	200	0,153	88,1	8,43
5.	21	300	23,65	23,57	0,082	250	0,131	89,8	9,84
6.	21	300	23,77	23,72	0,054	300	0,086	93,3	15

To analyze weight loss due to corrosion, the coupons were kept in the solutions for a duration of 300 hours. At the end of the exposure period, the following post-treatment steps were performed:

— Coupons were cleaned with a soft brush;
— Rinsed with distilled water;
— Washed with acetone;
— Dried until a constant mass was achieved.

The difference between the initial and final mass of each coupon was calculated using the gravimetric method. The results of the gravimetric analysis are presented in Table 1.

3. Results and Discussion (Part)

The effects on the surface of steel samples (coupons) subjected to corrosion were examined over a 300-hour period in both a 15% HCl solution containing guanidine chloride methylacrylate (GXMA) and in an uninhibited 15% HCl solution. In the inhibitor-containing solution, almost no visible changes were observed on the metal surface. In contrast, clear signs of corrosion were evident on the surface of the sample exposed to the uninhibited acidic solution.

According to the results presented in Table 1 and in compliance with clause 5.3 of document NGH (RH)39.0–051:2007, the protective efficiency determined by the gravimetric method on steel samples should not be less than 90% at concentrations not exceeding 500 mg/L for general corrosion resistance.

The determination of the corrosion inhibitor's mechanism of action requires analyzing the energy state of the thermodynamic system. Accordingly, it is necessary to evaluate the system's thermodynamic energy during corrosion in both inhibited and uninhibited conditions at different concentrations. To analyze energy changes, calculations were conducted based on the Arrhenius equation:

$$CR = A \cdot \exp\left(\frac{-E_a}{RT}\right)$$

Where:

E – is the activation energy (kJ/mol),

R – is the universal gas constant, 8.314 J/mol·K,

T – is the temperature in Kelvin,

$A \cdot \exp$ – is the pre-exponential factor.

The application of a corrosion inhibitor in the solution leads to observable changes in activation energy (E_a). When the inhibitor is added, the activation energy of the corrosion process increases, and this increase becomes more pronounced with higher inhibitor concentrations. A higher value of activation energy indicates the predominance of physical adsorption, whereas unchanged or lower values of E suggest chemical adsorption. A significant increase in the activation energy compared to the uninhibited system confirms that the inhibitor is adsorbing onto the steel surface. In the absence of an inhibitor, the activation energy of the corrosion process is determined based on the concentration of iron ions released into the solution in their active state.

4. Results and Discussion

The newly synthesized inhibitor demonstrated a protection efficiency of 93.3%, as determined by the gravimetric method, which meets the requirements of established standards. In this study, the corrosion inhibitor was synthesized using locally available raw materials. The inhibitory properties of the synthesized compound were tested under laboratory conditions in an artificially created aggressive environment. Gravimetric analysis revealed that at a concentration of 300 mg/L, the GXMA inhibitor provided a 93.3% protection efficiency for St-20 steel, with a 15-fold reduction in the corrosion rate compared to the uninhibited solution. The investigation was carried out across a range of inhibitor concentrations from 100 mg/L to 500 mg/L. However, it was found that the optimal inhibitory effect was achieved at 300 mg/L. The Langmuir adsorption isotherm is a specific mathematical model related to adsorption phenomena. It is primarily used to describe the equilibrium state of adsorption and helps in understanding the nature of the bonding between the adsorbate and the surface. According to Equation (1), the model assumes that adsorption occurs on a homogeneous surface with a fixed number of identical sites, where each site can hold only one adsorbate molecule, and there is no interaction between adsorbed molecules. This equation describes monolayer adsorption, implying that the adsorption process occurs in a single molecular layer (monomolecular adsorption). One of the key assumptions of the Langmuir model is that each adsorption site binds to only one molecule, the adsorbate molecules do not interact with one another, and they are uniformly distributed over the adsorbent surface.

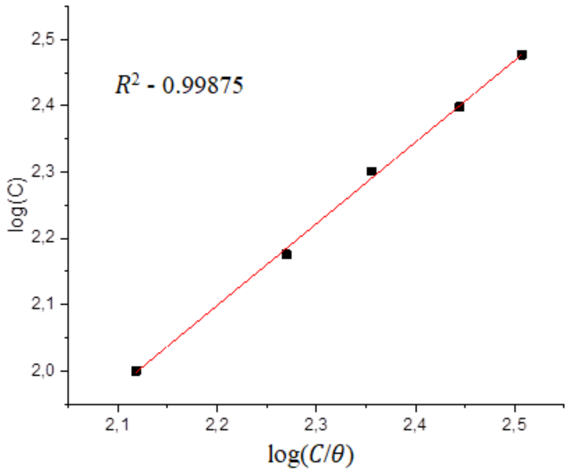
$$\frac{C}{\theta} = \frac{1}{K} + C \qquad (1)$$

$$long\left(\frac{C}{\theta}\right) = long\,C - long\,K \qquad (2)$$

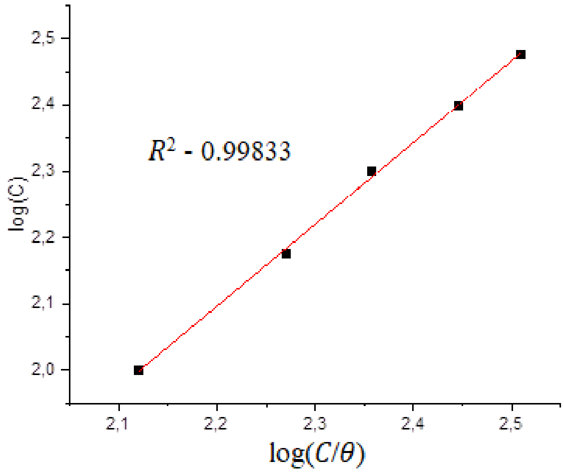
To evaluate the compatibility of the experimental results with theoretical models, the data were tested using various adsorption isotherm equations. At temperatures of 343 K, 353 K, and 363 K, the relationship between log(C/θ) and log(C) was analyzed. As shown in Figure 1, the resulting plots exhibited a linear correlation, indicating that the adsorption process conforms well to the assumptions of the isotherm models at the given temperatures.

Figure 1. *Langmuir adsorption isotherm plots of GXMA inhibitor on steel surface at: (a) 343 K, (b) 353 K, and (c) 363 K*

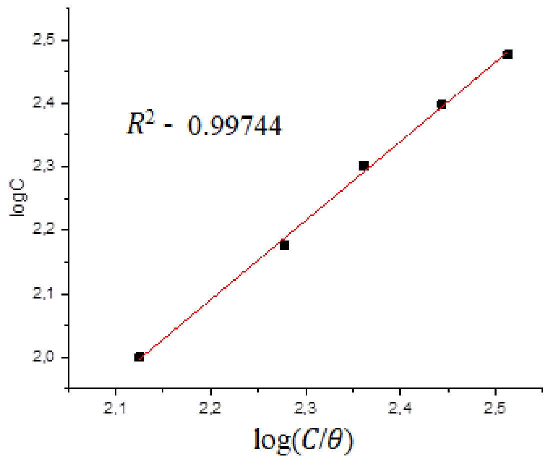
a)



b)



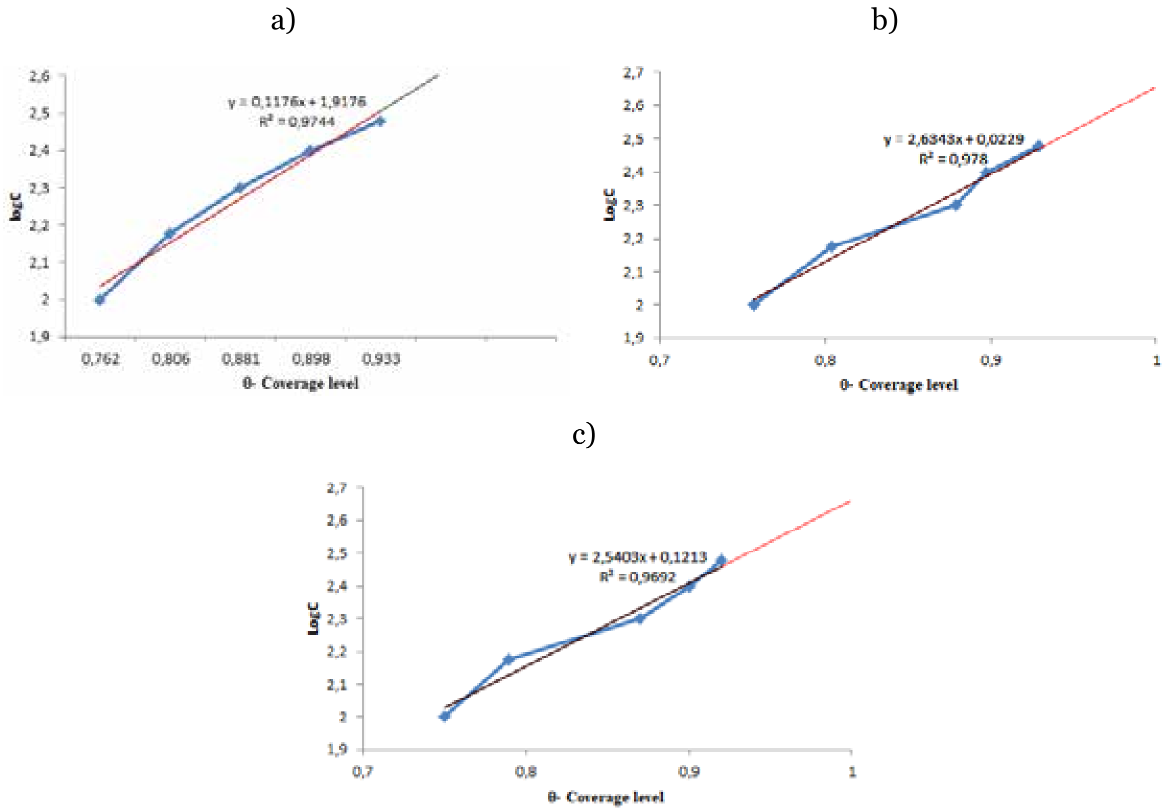
c)



Based on the data obtained at these temperatures, the calculated R^2 values were 0.99875, 0.9833, and 0.99744, respectively. These results indicate a strong correlation with the Langmuir adsorption isotherm, confirming the applicability of the model to the adsorption behavior of GXMA on the steel surface. The high degree of fit suggests that the process follows a monomolecular physico-chemical adsorption mechanism, characterized by interactions between the donor atoms of the inhibitor molecules and the iron ions on the steel surface. This conclusion is supported by the assumptions of the Langmuir equation, which describes monolayer adsorption without interaction between adsorbed species.

In the Temkin isotherm, the relationship between log C and θ was analyzed at three different temperatures. The calculated R^2 values were 0.9744 at 343 K, 0.9780 at 353 K, and 0.9692 at 363 K. These values indicate a reasonable correlation with the Temkin isotherm model, suggesting that the adsorption process is moderately consistent with the assumptions of this model at the investigated temperatures (Figure 2).

Figure 2. *Temkin adsorption isotherm plots of the GXMA inhibitor on the steel surface at: (a) 343 K, (b) 353 K, and (c) 363 K.*



For the Frumkin isotherm, the relationship between log $[C\cdot(\theta/(1-\theta))]$ and θ was examined. The obtained R^2 values were 0.9891 at 343 K, 0.9892 at 353 K, and 0.9691 at 363 K. These values reflect a strong correlation with the Frumkin isotherm, indicating a good fit between the experimental data and the model. This suggests that intermolecular interactions between the adsorbed species may play a role in the adsorption mechanism of GXMA on the steel surface.

The adsorption process is characterized by the adsorption of inhibitor molecules onto the metal surface and the simultaneous desorption of water molecules. This can also be considered as an exchange process, in which the inhibitor molecules displace water molecules and adhere to the metal surface, forming a protective layer. The surface coverage (θ) increases proportionally with the inhibitor concentration, leading to enhanced inhibition efficiency. The parameter θ represents the percentage of the metal surface covered by the inhibitor and is expressed as a value from 0 to 1 (or 0% to 100%). The increase in θ indicates greater surface protection. The following graph illustrates the change in corrosion rate at various temperatures and inhibitor concentrations. The results obtained during the study show that at inhibitor concentrations of 250 mg/L and 300 mg/L, the calculated R^2 values were 0.9989 and 0.9976, respectively. These high values confirm the accuracy and reliability of the experimental procedures and the validity of the observed trends (Figure 4).

Figure 3. *Frumkin adsorption isotherm plots of the GXMA inhibitor on the steel surface at: (a) 343 K, (b) 353 K, and (c) 363 K.*

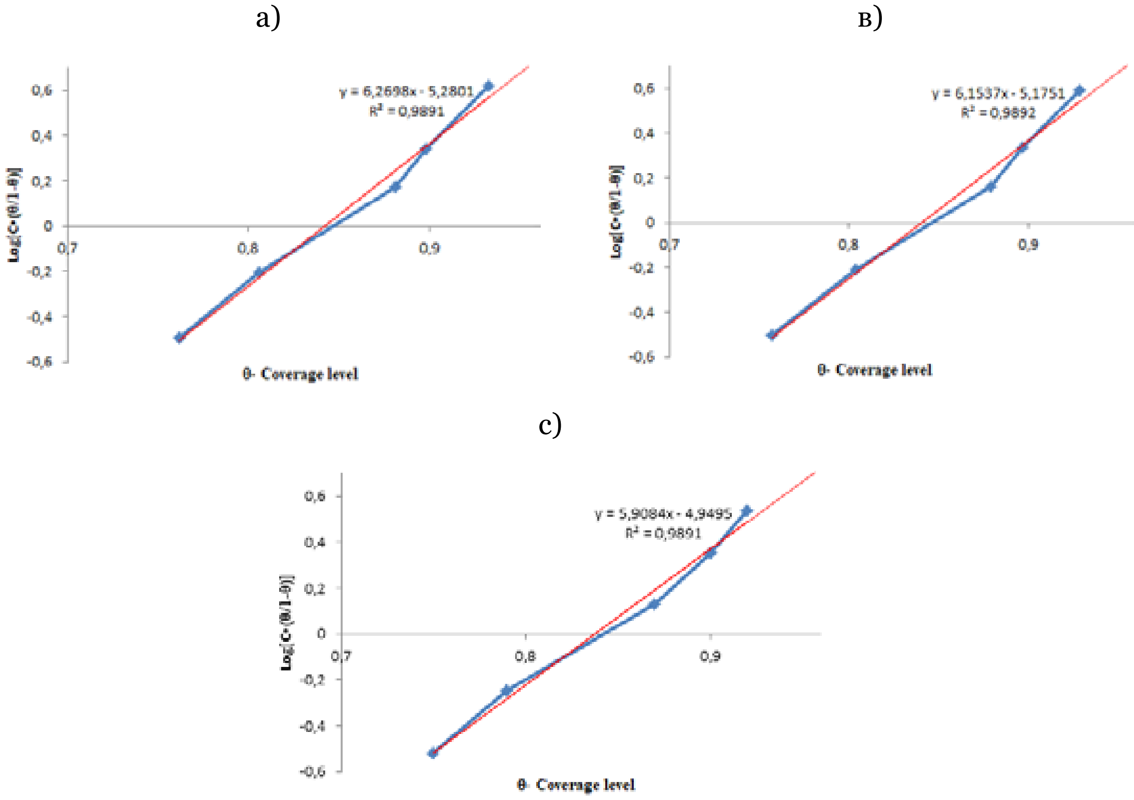
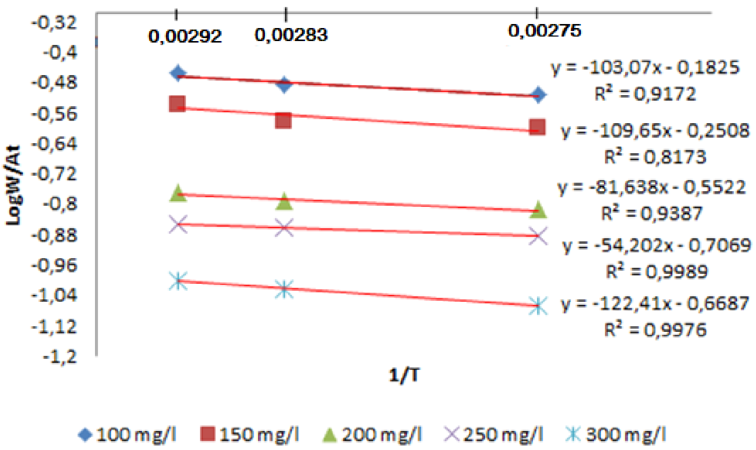


Figure 4. *Arrhenius plot for the activation energy of the GXMA corrosion inhibitor in an aggressive environment*



This analysis emphasizes the importance of thermodynamic and kinetic parameters in gaining a deeper understanding of the corrosion inhibition mechanisms. The conclusions derived from these calculations, along with the results obtained from the previously discussed adsorption isotherms, confirm that the adsorption of GXMA on the steel surface follows a monomolecular pattern and is of a physico-chemical nature.

4. Conclusion

In this study, the corrosion inhibition performance of guanidine chloride methylacrylate (GXMA), synthesized from locally available raw materials, was thoroughly investigated using the gravimetric method in a 15% HCl acidic environment on steel surfaces. At a concentration of 300 mg/L, GXMA provided up to 93.3% protection, which fully complies with the GOST standard (RH 39.0–

051:2007). After 300 hours of exposure in hydrochloric acid, a significant reduction in corrosion rate was observed based on mass loss measurements of the steel coupons.

The GXMA compound was found to form an effective monomolecular adsorption layer on the metal surface, acting as a strong physical barrier in an acidic medium. Detailed analysis of the adsorption process was carried out using Langmuir, Temkin, and Frumkin isotherms. The high correlation coefficients, particularly for the Langmuir model ($R^2 \approx 0.99$), confirmed that GXMA undergoes monomolecular adsorption on the metal surface. This suggests that each inhibitor molecule occupies a distinct active site on the metal, displacing water molecules in the process.

Thermodynamic evaluations, including calculations based on the Arrhenius equation, indicated an increase in activation energy (Ea) and a negative entropy change, both of which support a physico-chemical nature of adsorption. These results imply that the interaction between GXMA and the metal surface is not only superficial but also involves stable bonding at the ionic level.

It was also established that the optimal inhibitory efficiency of GXMA was achieved within the 250–300 mg/L concentration range, highlighting the compound's potential as an economically viable and highly effective corrosion inhibitor.

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