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Iodine–β-Cyclodextrin: An Effective Corrosion Inhibitor for Carbon Steel in Sulfuric Acid Solution - Experimental Design and Investigating Thermodynamic Parameters

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Abstract: Widely used across industries, carbon steel is vulnerable to corrosion in aggressive environments, especially acidic ones. Thus, effective methods to mitigate metal corrosion from acids are crucial. Inhibitors are extensively used to prevent corrosion in industries, with the potential for improved protective performance. The design of experiments was employed to determine the optimal conditions for enhancing the inhibitor efficiency of Iodine–β-Cyclodextrin (Iodine/β-CD) in a sulfuric acid solution at temperatures ranging from 20°C to 50°C. The relationship between the factors and responses was established using response surface methodology (RSM), employing regression statistical analysis and probabilistic analysis. A single response was recorded: inhibitor efficiency was determined by measuring weight loss before and after immersion in the inhibitor solution. Thermodynamic parameters were also computed to determine adsorption and activation processes. The statistical analysis revealed that the quadratic models for inhibition efficiencies (IE) were highly significant with a coefficient of multiple regressions R^2= 0.997. Further validation of the model indicated a good fit (R^2 Adj= 0.994), and the experimentally observed values aligned well with predicted ones, demonstrating a highly significant model with Q^2= 0.978. The theoretical efficiency predicted by the RSM model was 88.41%, whereas the efficiency observed during the experimental test procedure with the best-evaluated variables was 82.45%. In conclusion, this paper aims to identify the optimal conditions for employing Iodine–β-Cyclodextrin as a new corrosion inhibitor for carbon steel, utilizing experimental design methods. The results indicate that iodine/β-CD exhibits remarkable corrosion inhibitory properties for carbon steel under specific conditions.

Keywords: Corrosion inhibition, carbon steel, Iodine–β-Cyclodextrin, Thermodynamic and kinetic parameters, Response surface methodology, Optimization.

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1. INTRODUCTION

Carbon steel is a widely used material in infrastructure, transportation, energy, and other industries due to its cost-effectiveness and mechanical strength. However, its susceptibility to corrosion poses risks such as leaks, pressure losses, and ruptures in piping systems. Consequently, implementing protective measures to counter acid-induced corrosion is vital, employing chemical and other means. Among these methods, corrosion inhibitors offer a cost-effective approach (1), effectively mitigating or preventing corrosion when added in low concentrations (2,3). Researchers have reported organic compounds with hetero-atoms like O, N, and S as efficient inhibitors for various metals (4-8). The inhibitive nature of a molecule and its adsorption onto a metal surface depends on factors such as size, chemical structure, surface charge distribution, specimen surface morphology, corrosive media type, and operating conditions (9-10). Investigating adsorption isotherms provides insights

into interactions between adsorbed molecules and the electrode surface, highlighting physical adsorption and chemical decomposition (11,12).

Cyclodextrins (CDs), important oligosaccharides produced through enzymatic amylose hydrolysis, have been extensively used in supramolecular chemistry due to their affordability and versatility. These molecules possess a central cavity whose size depends on the glucose units in the cycle (13). The central cavity, a functional site of CDs, is involved in molecular transportation and is widely applied in drug delivery (14). Various iodine/β-CD host-guest complexes have been synthesized and investigated for potential pharmacological activities. For instance, Wang et al. demonstrated the bacteriostatic and antifungal activities of iodine/β-CD (15). Additionally, a recent study revealed that sausages fortified with iodine/β-CD complex positively affected the iodine status of volunteers (16).

To efficiently study variables and responses while minimizing experiments, the Design of Experiments (DOE) offers an alternative to the traditional one-factor-at-a-time approach. DOE's utilization empowers researchers to effectively explore relationships among input variables, such as inhibitor concentration, temperature, and immersion time, as well as critical output variables like inhibition efficiency (17-19). This is achieved through statistical techniques, employing a systematic framework for data collection that surpasses traditional methods, thus enabling more efficient and effective analyses. In the domain of experimental design and optimization, the omnipresence of the Response Surface Methodology (RSM) software is evident. Guided by a multivariate methodology central to analytical optimization, the RSM approach enables the precise accommodation of experimental data through the application of a polynomial equation (20,21).

In this study, the primary investigation centers on the inhibitory effect of Iodine–β-Cyclodextrin on carbon steel corrosion in sulfuric acid using the weight loss method. Additionally, the study aims to evaluate thermodynamic parameters linked to the corrosion process and to examine the collaborative effects of inhibitor concentration, temperature, and immersion time using response surface methodology (RSM).

2. EXPERIMENTAL

2.1. Specimen Preparation

The carbon steel specimen utilized in the current study has the following chemical compositions (in wt%): 0.370% C, 0.230% Si, 0.680% Mn, 0.016% S, 0.077% Cr, 0.011% Ti, 0.059% Ni, 0.009% Co, 0.160% Cu, with the remaining content being iron. The specimens underwent thorough mechanical polishing using SiC (300–1200) emery papers, followed by degreasing with acetone, washing with doubly distilled water, and final drying. All solvents and chemicals employed were of AR grade. Additionally, doubly distilled water was used to prepare the various concentrations of test solutions.

2.2. Weight Loss Method

The weight-loss method assesses the corrosion rate of a metal sample by quantifying the reduction in mass over a designated timeframe of exposure to a corrosive milieu. It yields outcomes that better reflect uniform corrosion compared to electrochemical methods, owing to its alignment with realistic experimental conditions (22). The weight loss measurements followed the procedures detailed in our prior work by Attar et al (23-25). Each measurement was conducted in triplicate, and the resulting weight loss means were both noted and documented.

The corrosion rate, designated as 'CR,' and the inhibitor's efficacy were computed using the subsequent equations:

$$\mathrm{CR} = \Delta w/(S \times t) \quad (1)$$

where Δw refers to the weight loss in milligrams (mg), S stands for the sample area in square centimeters (cm²), and t symbolizes the immersion time in hours (h).

The corrosion inhibition efficiency (IE %) and surface coverage (θ) were calculated from the values of CR:

$$\mathrm{IE}(\%) = 100 \times (\mathrm{CR} - \mathrm{CR_{Inh}})/\mathrm{CR} \quad (2)$$

where CR represents the corrosion rate acquired in the absence of the inhibitor, while CR_{Inh} corresponds to the corrosion rate observed in the presence of the inhibitor.

2.3. Experimental Design

The investigation employed MODDE Software Version 9.1 for experimental design and statistical analysis. The primary aim was to develop a precise analytical model, discerning variable effects on response function and optimal conditions. The study employed RSM for experiment design, modeling, and corrosion inhibition process optimization. Parameters included temperature, inhibitor concentration, and exposure time at three levels. Seventeen experiments were conducted using RSM. Independent parameters and their levels are detailed in Table 1.

Table 1: Optimization of inhibition efficiency of an iodine–β-cyclodextrin on carbon steel in acid medium: weight loss and RSM approach.

Sl. no	Parameter	Code	Unit	Level		
				− 1	0	+ 1
1	Inhibitor Concentration	Con	mol/L	1×10^{-5}	5.05×10^{-4}	1×10^{-3}
2	Temperature	Temp	°C	20	30	40
3	Immersion Time	Tim	hrs	1	2	3

2.4. Adsorption Considerations and Activation Energy Calculations

Adsorption and activation play crucial roles in the inhibition of corrosion for metals. Adsorption refers to the phenomenon where inhibitor molecules attach to the metal surface, creating a protective layer that effectively reduces the corrosion rate. To elucidate the adsorption mechanism of iodine/β-CD on the surface of the studied samples, a thorough analysis of various models of adsorption isotherms was conducted. The findings reveal that the Langmuir equation provides a more accurate description of this phenomenon for iodine/β-CD within a 0.5 M H_2SO_4 solution. The mathematical representation of the Langmuir adsorption isotherm takes the form:

$$C_{inh}/\theta = 1/K_{ads} + C_{inh} \quad (3)$$

Where C_{inh} represents the molar concentration of iodine/β-CD in units of mol/L, while θ signifies the surface coverage (expressed as a percentage of IE/100). The parameter K_{ads} corresponds to the adsorption constant, quantified in units of L/mol. By linear fitting of C_{inh}/θ versus C_{inh}, K_{ads} can be determined.

The determination of adsorption enthalpy (ΔH_{ads}) was accomplished employing the Van't Hoff equation:

$$\ln(K_{ads}) = (-\Delta H_{ads}/RT) + \text{Const.} \quad (4)$$

Through the execution of a regression analysis between the natural logarithm of K_{ads} and the reciprocal of temperature (1/T), a linear relationship manifested itself, revealing a slope equivalent to ($-\Delta H_{ads}/R$).

In this research paper, the Gibbs free energy of adsorption (ΔG_{ads}) was calculated at different temperature levels to explore the adsorption behavior of iodine/β-CD in a 0.5 M sulfuric acid solution. Gibbs free energy of adsorption, which is useful for determining inhibitor adsorption, can be calculated using the following equation:

$$\Delta G_{ads} = -RT\ln(55.5\ K_{ads}) \quad (5)$$

where ΔG_{ads} is Gibbs free energy of adsorption, J/mol; R is 8.314 J/(mol/K); T is the absolute temperature, K.

Additionally, the enthalpy of activation (ΔH_a) and entropy of activation (ΔS_a) were calculated using the alternative Arrhenius equation:

$$\ln(CR/T) = [\ln(R/Nh) + (\Delta S_a/R)] - \Delta H_a/RT \quad (6)$$

In this equation, h denotes Planck's constant, and N represents Avogadro's number. Plotting ln(CR/T) against 1/T resulted in a straight line with a slope of ($-\Delta H_a/R$) and an intercept of [ln(R/Nh) + (ΔS_a/R)].

By applying the thermodynamic relation (Eq.7), one can calculate the variation in free energy of adsorption and activation for the corrosion process at different temperatures.

$$\Delta G = \Delta H - T\,\Delta S \quad (7)$$

3. RESULTS AND DISCUSSION

For the study, three independent variables, namely inhibitor concentration (Con), temperature (Temp), and immersion time (Tim) were selected across three distinct levels (equation 8). This equation delineates how the experimental variables interact to influence the Inhibition Efficiency (IE%).

$$\mathrm{IE}(\%) = 73.606 + 2.636(Con) - 7.589(Temp) + 2.345(Tim) + 1.795(Con)^2 + 2.141(Temp)^2 - 9.769(Tim)^2 - 0.651(Con \times Temp) - 1.366(Con \times Tim) - 2.583\ (Temp \times Tim) \quad (8)$$

It was evident that the variables with the most substantial impact on IE were inhibitor concentration (Con) and immersion time (Tim), while the influence of temperature (Temp) was comparatively smaller. Coefficients in the model characterized by positive values denote a synergistic effect, whereas negative values indicate an antagonistic effect (26). Among the model factors, Con^2 and $Temp^2$ positively contribute to the formulation, whereas Tim^2, Con×Temp, Con×Tim, and Temp×Tim exert a negative influence on the developed model.

3.1. Statistical Test and Analysis of Models

The quadratic model generated was fitted to the data, and the response is depicted in the summary of the fit plot (Figure 1), which provides information about the model's strength and robustness, including Model Validity, Reproducibility, R^2, and Q^2. The values of these parameters are displayed in the following table.

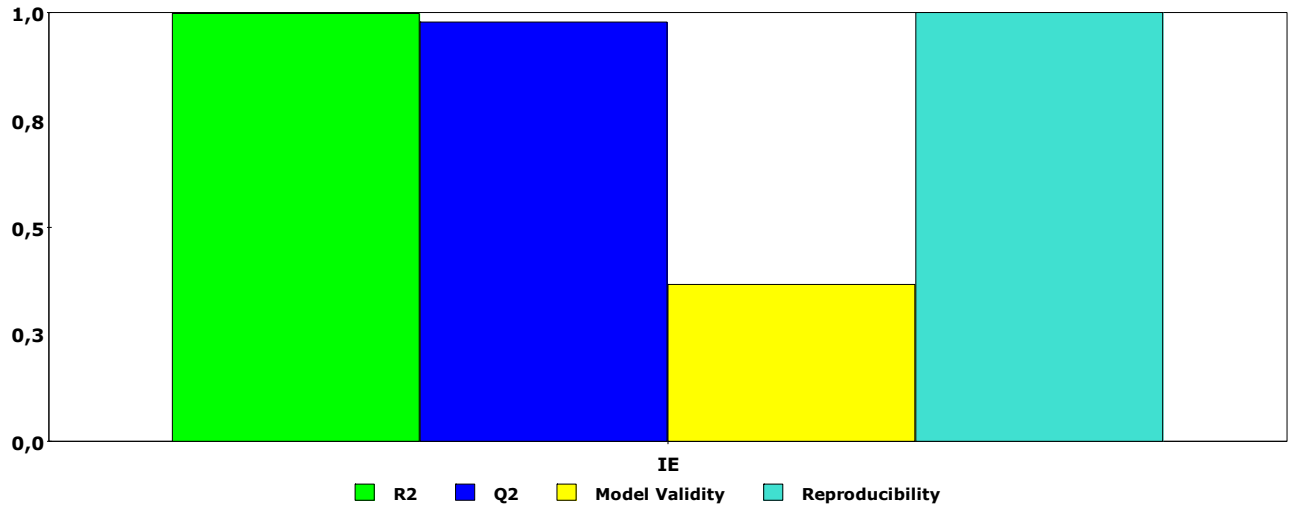

Figure 1: Summary of fit plot showing model fit (R^2), predictability (Q^2), model validity, and reproducibility.

Table 2 displays the statistical parameters acquired through ANOVA. The coefficient of determination (R^2) denotes the model's fit quality, with R^2 (0.997) suggesting that only 0.3% of the overall variations remain unaccounted for by the model. Correspondingly, the value of Adj. R^2 (0.994) affirms the model's strong significance, signifying excellent concurrence between the predicted and experimental efficiencies of Iodine/β-CD (27).

Table 2: Displays the coefficients of factors, interactions, and probability values of approximate polynomials for the response variables in the experimental design.

R^2	R^2Adj.	Q^2	RSD	Conf. lev.	Model Validity	Reproducibility
0.997	0.994	0.978	0.623	0.95	0.366	0.999

The Q^2 value serves as an indicator of the model's predictive performance for future outcomes (28). As shown in Table 2, the obtained Q^2 value exceeds 0.9, indicating an excellent model for this study. A Q^2 value should surpass 0.1 for a significant model and exceed 0.5 for a good model. Furthermore, when Q^2 is above 0.9, the model is considered excellent (19, 29). Additionally, the difference between R^2 and Q^2 should be smaller than 0.3 for a good model. The model's residual standard deviation (RSD) was calculated to be 0.623. A small RSD value suggests a strong model that closely aligns predicted values with actual responses (19).

The analysis maintains a 95 percent confidence level. When the Model Validity (0.366) bar exceeds 0.25, the model does not exhibit a Lack of Fit. Reproducibility refers to the variability among replicates about the overall variability. In this study, a value of 0.999 is nearly equivalent to unity. A reproducibility value of 1 signifies perfect reproducibility.

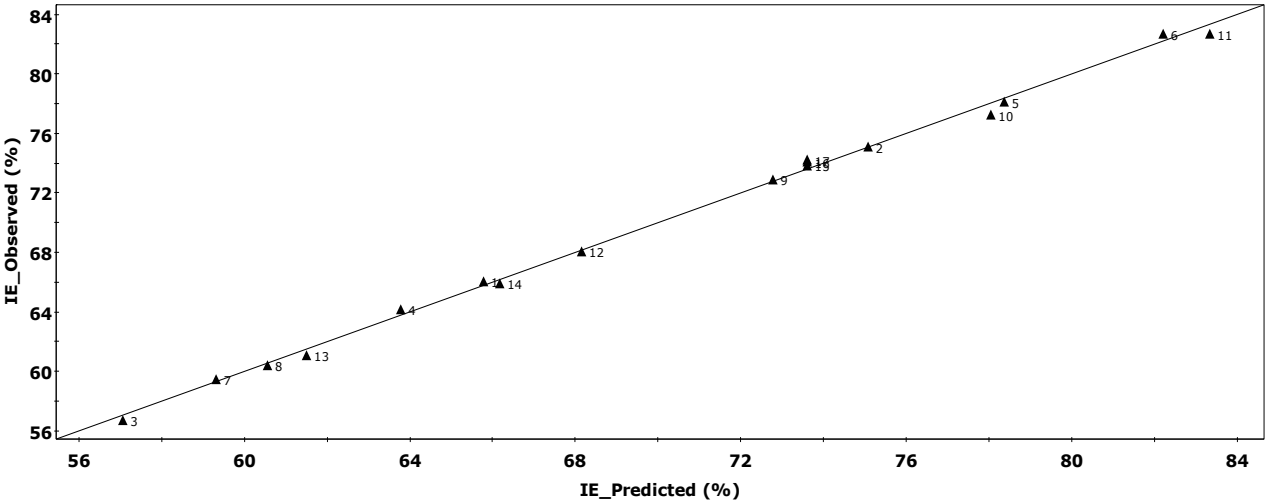

Figure 2: Illustrates the model fit graph, depicting the relationship between the experimental and predicted values.

As illustrated in Figure 2, a clear linear relationship emerged in the diagnostic plots comparing predicted inhibition efficiency to actual inhibition efficiency. These findings affirm the appropriateness of the design model for predicting the inhibition efficiency of the Iodine/β-CD. Furthermore, it demonstrates the model's suitability for predicting the response variables based on the experimental data (17, 27).

3.2. Main Effects

Figure 3 illustrates the impact of each examined parameter. Notably, the values attributed to each point correspond to the average corrosion IE achieved at that specific level, regardless of other parameter variations. The comprehensive mean is depicted across each panel. The observed correlation between the escalation of corrosion IE and temperature elevation is evident within this study (Figure 3). As the concentration of iodine/β-CD increased, the inhibitor efficiency (IE%) also increased. The increase in surface coverage enhances accessibility and facilitates the adsorption of the active inhibitor components onto the surface of the corroding metal (30). The optimal efficiencies (IEs) are achieved when carbon steel is immersed in a solution with a concentration of 1×10^{-3} mol/L iodine/β-CD for a duration of 2 hours, at a temperature of 20°C. Conversely, the lowest IE was observed when carbon steel was subjected to a solution containing 1×10^{-5} mol/L of iodine/β-CD, immersed for 1 hour, and maintained at a temperature of 40°C.

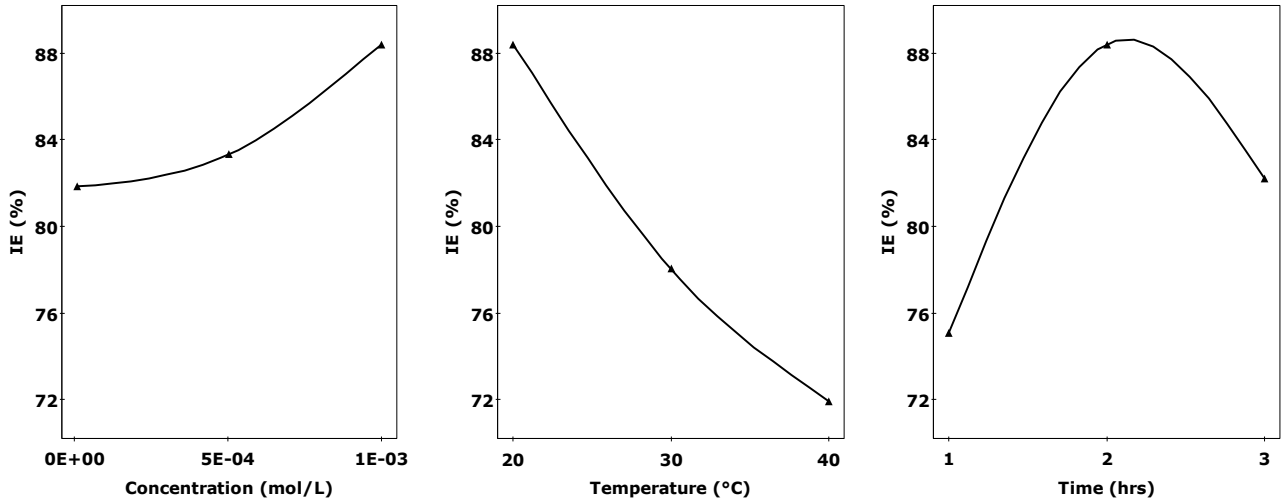

Figure 3: Optimizing the inhibition efficiency of Iodine/β-CD on carbon steel in sulfuric acid medium – medium-weight loss and RSM approach.

3.3. Contour Plots

Contour plots serve as graphical tools for illustrating the interplay among two or more variables using a three-dimensional space (31). Within corrosion inhibition research, these plots find frequent application in displaying how various variables collaboratively influence the effectiveness of corrosion inhibitors.

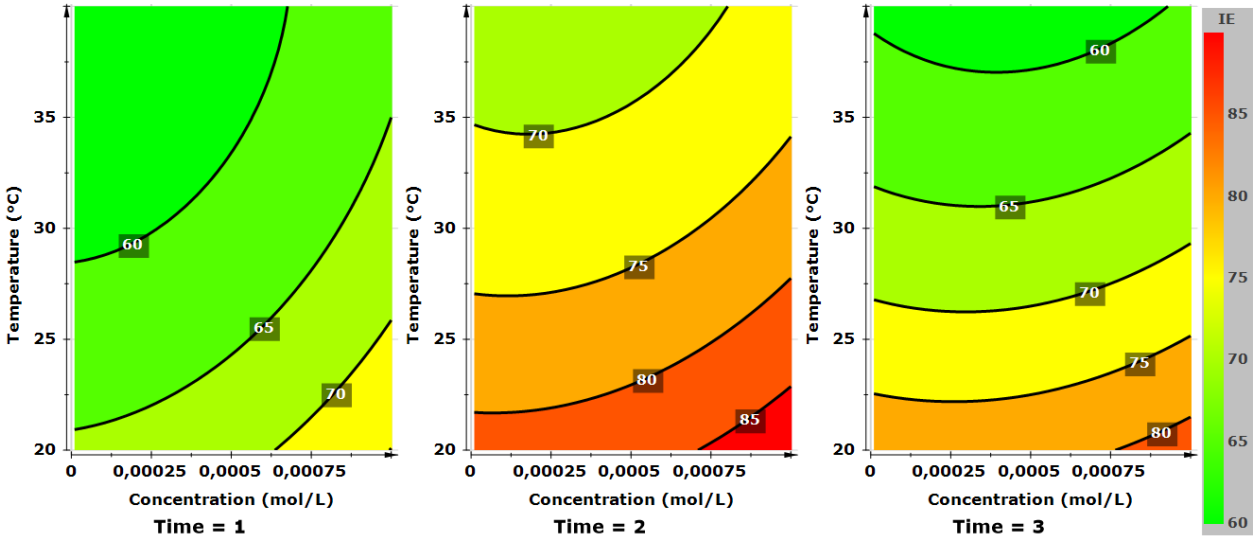

Figure 4: Contour plots illustrating the interactive effects of concentration, temperature, and immersion time on the corrosion inhibition efficiency of Iodine/β-CD on carbon steel in sulfuric acid, obtained through weight loss and RSM approach.

Figure 4 demonstrates the interaction effect through a contour plot involving temperature, inhibitor concentration, and exposure time on corrosion inhibition efficiency (IE). It confirms that corrosion IE moderately increases as the inhibitor concentration rises from the minimum to the maximum level. For any amount of Iodine–β-Cyclodextrin in the system, IE increases with an immersion time of up to 2 hours. The IE reaches its peak at the lowest temperature of 20°C and the highest concentration of Iodine/β-CD, as depicted in Figure 4. The results obtained align with those reported in the literature (32).

3.4. Adsorption Considerations and Activation Energy Calculations

3.4.1. Adsorption considerations

Table 3 provides a comprehensive summary of the equilibrium constants, free energy, enthalpy, and entropy associated with the adsorption process. The positive adsorption equilibrium constants demonstrate the feasibility of the inhibitor's adsorption onto the metal surface (33). This finding aligns with the trend observed, indicating that the inhibitor with better inhibition efficiency has a positive effect on the adsorption process.

Table 3: Thermodynamic parameters of Iodine/β-CD adsorption on carbon steel surface in sulfuric acid at various temperatures.

T (K)	R^2	K_{ads} (L/m^3)	ΔH_{ads} (kJ/mol)	ΔS_{ads} (J/mol K)	ΔG_{ads} (kJ/mol)
293	0.999	229.12	-35.61	-14.47	-39.85
303	0.999	177.43		-16.33	-40.56
313	0.998	92.49		-14.69	-40.21
323	0.998	62.72		-14.98	-40.45

The thermodynamic parameters for inhibitor adsorption provide valuable insights into the corrosion inhibition mechanism. An exothermic adsorption process ($\Delta H_{ads}<0$) may imply either chemisorption, physisorption, or a mixture of both processes. Conversely, an endothermic adsorption process ($\Delta H_{ads}>0$) is unequivocally assigned to chemisorption (34). A negative adsorption entropy value signifies a reduction in metal surface degradation, whereas a positive value indicates an enhancement in system disorder. The negative values of the free energy adsorption indicate the spontaneity of the process. Typically, if ΔG_{ads} values exceed -20 kJ/mol, the physisorption mechanism is favored, while values of -40 kJ/mol or lower suggest chemisorption. In this study, the obtained ΔG_{ads} values fall within the range of -39.85 to -40.45 kJ/mol, which lies between −20 and −40 kJ/mol (35,36). Based on the study's findings, the calculated enthalpy and free energy of adsorption values suggest a mechanism that extends beyond pure chemical or physical processes. Instead, they indicate a coexistence of physisorption and chemisorption between the inhibitor and the metal surface (37).

3.4.2. Activation energy calculations

The activation energy values for the corrosion reaction under different conditions are as follows: E_a = 35.96 kJ/mol for the blank, and E_a = 53.87 to 55.29 kJ/mol for iodine/β-CD, respectively (Table 4). When an inhibitor is present, the activation energy can either be lower or remain unchanged compared to the blank test, indicating a chemisorption mechanism. Conversely, higher values of E_a in the presence of the inhibitor suggest a physical adsorption mechanism (38).

The presence of positive ΔH_a values (Table 4), both in the absence and presence of the inhibitor, indicates the endothermic nature of the metal dissolution process, suggesting a gradual dissolution of carbon steel (39,40).

Table 4: Activation parameters for carbon steel corrosion in sulfuric acid with varying Iodine/β-CD concentrations.

C (mol/L)	R^2	E_a (kJ/mol)	ΔH_a (kJ/mol)	ΔS_a (J/mol K)	ΔG_a (kJ/mol)			
					293	303	313	323
-	0.971	35.96	33.41	-226.53	99.78	102.05	104.31	106.58
1×10^{-5}	0.994	53.87	51.32	-275.90	132.16	134.92	137.67	140.43
5×10^{-5}	0.993	54.46	51.91	-277.12	133.11	135.87	138.64	141.42
5×10^{-4}	0.988	55.29	52.73	-279.13	134.51	137.30	140.09	142.88
1×10^{-3}	0.991	54.92	52.36	-277.05	133.53	136.31	139.07	141.84

Upon comparing the ΔS_a values, it becomes evident that the entropy of activation reduces in the presence of the studied inhibitor compared to the free acid. The lower ΔS_a value further corroborates the decelerated metal dissolution in the presence of iodine/β-CD (41).

The ΔG_a values exhibited a positive trend, intensifying as the temperature elevated. This temperature rise corresponded to an increased spontaneity of the corrosion process, indicating enhanced solubility of the activated complex at higher temperatures (34). Moreover, the augmentation in concentration resulted in higher free energy of activation, attributed to the formation of

an unstable activated complex in the rate-determining transition state (42).

4. CONCLUSION

In this study, Iodine–β-Cyclodextrin was employed to investigate the efficiency of corrosion inhibition in a 0.5 mol/L sulfuric acid solution concerning carbon steel. The investigation considered various parameters, including inhibitor concentration, exposure time, and solution temperature. The results indicate that inhibition efficiency increases with higher inhibitor concentrations and decreases with rising temperatures. In the inhibited solution, the activation energy for the corrosion process exceeded that in the uninhibited solution. The enthalpy of activation signifies the endothermic character of metal dissolution, while the entropy of activation signifies a reduction in the rate of metal dissolution. Among the assortment of adsorption isotherm models, the Langmuir equation emerged as a more suitable descriptor for the phenomenon involving Iodine–β-Cyclodextrin within a sulfuric acid solution. Furthermore, the observed values of the Gibbs adsorption free energy, ranging from -39.85 to -40.45 kJ/mol, indicate the coexistence of both chemical and physical adsorption mechanisms for Iodine–β-CD.

The RSM optimization utilized in this study meticulously predicted process parameters to achieve the highest corrosion inhibition efficiency. This technique also offers crucial insights applicable across various scales, from laboratory experimentation to industrial processes. The statistical model developed for corrosion inhibition efficiency using RSM was found to be effective, as this equation successfully elucidated the experimental data with a confidence level of 95%. Numerical optimization of the corrosion inhibition process for Iodine–β-CD revealed that the optimal conditions for achieving maximum IE (88.41%) were a concentration of 1×10^{-3} mol/L, a temperature of 20 °C, and an exposure time of 2 hours. Employing response surface methodology (RSM) in conjunction with analysis of variance (ANOVA), substantial RSM-based models were constructed to predict IE. Additionally, R^2 statistics, adequate precision, and diagnostic plots were employed as primary metrics to validate the accuracy and sufficiency of the IE models.

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