Enhancing Corrosion Resistance of Carbon Steel in Sulfuric Acid Using βcyclodextrin as an Inhibitor

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The corrosion process leads to substantial economic losses, particularly in the industrial sector, emphasizing the importance of preventive measures. This study aimed to investigate the impact of inhibitor concentration, immersion duration, and temperature on the corrosion of carbon steel in a sulfuric acid environment, assessed through mass loss measurements. Inhibition efficiency displayed a positive correlation with increasing inhibitor concentration, while it declined as temperatures rose. In a 0.5 M sulfuric acid solution, β -cyclodextrin (β -CD) exhibited an inhibition efficiency of 62.26% at a concentration of 1 mM. The addition of potassium iodide (KI) significantly enhanced the inhibition efficiency to 97.98%, indicating a synergistic effect. The study encompassed various calculations, including the determination of activation energy (E_a), activation enthalpy (ΔH_a), activation entropy (ΔS_a), adsorption-free energy (ΔG_{ads}), adsorption enthalpy (ΔH_{ads}), and adsorption entropy (ΔS_{ads}). The adsorption of the inhibitor onto the carbon steel surface conformed to the Langmuir adsorption isotherm. Additionally, Scanning Electron Microscopy (SEM) investigations provided further verification of β -cyclodextrin's adsorption on the carbon steel surface.

Introduction

Carbon steel is commonly used as a construction material and is susceptible to corrosion, particularly in acidic environments. Strong acids are widely employed in various industrial applications such as cleaning, scaling, pickling, acidification of oil wells, and other isothermal processes [1]. Consequently, protective measures need to be implemented to reduce the rate of acid corrosion through chemical and other means. Among the available methods, the use of corrosion inhibitors is the most cost-effective approach [2]. A corrosion inhibitor is a chemical substance that, when added in low concentrations to the environment, can mitigate or prevent corrosion [3-4]. The effectiveness of corrosion inhibitors depends on multiple factors, including the corrosive

environment, temperature, pH, immersion time, metal composition, as well as the concentration and chemical properties of the inhibitor [5-6]. The adsorption isotherm provides valuable insights into the interaction between adsorbed molecules and the electrode surface. Two main types of interactions can be described by the adsorption isotherm: physical adsorption and chemical adsorption [7-8]. Physical adsorption involves the electrostatic forces between the ionic charges or dipoles on the adsorbed substance and the charges at the metal/solution interface. The heat absorbed during physical adsorption is relatively low, resulting in stability only at relatively low temperatures. On the other chemical adsorption occurs hand, when molecules inhibit charge transfer or share electrons with the metal surface, forming bonds with similar coordination. Compared to other types of adsorption, chemical adsorption typically exhibits higher adsorption energy, thus providing stability greater at higher temperatures. The use of β -cyclodextrin and its derivatives as corrosion inhibitors has been explored in numerous studies [9,10]. The study demonstrated that β -CD exhibited remarkable inhibition efficiency against corrosion, attributed to its adsorption on the metal surface and formation of a protective barrier. Furthermore, β-CD was found to have low toxicity and good water solubility, making it a promising choice for corrosion protection. Cyclodextrin is extensively employed in the pharmaceutical industry to

enhance stability, eliminate unpleasant odor and flavor, and improve the solubility and bioavailability of drugs [11].

The objective of this study is to investigate the inhibitory effect of β -cyclodextrin on the corrosion of carbon steel in sulfuric acid and evaluate the thermodynamic parameters associated with the corrosion process. Additionally, the adsorption of the inhibitor on the surface of the carbon steel will be examined.

Experimental

Materials and methods

The test solution of 0.5 M sulfuric acid was prepared by diluting analytical grade 98% H₂SO₄ with double-distilled water in a volume of 50 mL. The composition of the carbon steel sample (by weight) is as follows: 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, and the remaining element is Fe. Different types of emery paper (ranging from 400 to 1200) were used for polishing and cleaning the electrode. The samples were weighed using an analytical balance before and after exposure, with weight measurements recorded to the nearest five decimal places. The weight loss experiment was conducted two hours after exposure. βcyclodextrin solutions with concentrations ranging from 0.01 to 1 mM were prepared in 0.5 M sulfuric acid.

Gravimetric method

Weight loss methods provide a more realistic assessment of uniform corrosion compared to electrochemical methods, as they closely resemble real-world conditions. For the weight loss measurements, we followed the same procedures as described in our previous work by Attar [12-13].

The measurements were conducted in triplicate, and the mean value of the weight loss was recorded and reported. To determine the corrosion rate 'CR' and evaluate the inhibitor's effectiveness, the following equations were employed [14]:

$$CR = \Delta w / (S \times t)$$
 (1)

Where, the weight loss (Δw) is expressed in milligrams (mg), the sample area (S) is represented in square centimeters (cm²), and the immersion time (t) is denoted in hours (h).

The corrosion inhibition efficiency (IE %) and surface coverage (θ) were determined based on the values of CR, as per the following equation [14]:

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

$$\theta = IE/100 \tag{3}$$

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

Results and discussion

Effect of immersion time

In order to evaluate the effect of time on the corrosion rate of the steel coupons in the acid medium, the corrosion rate of carbon steel in 0.5 M of H_2SO_4 solutions was separately investigated at 1 h, 2 h, 3 h, 4 h, 6h and 24 h using 1 mM of β -cyclodextrin at 303 K.

Figure 1 demonstrates a significant change in the weight loss per unit area of the carbon steel sample during prolonged immersion in the corrosive solution with and without the inhibitor. This indicates an accelerated corrosion rate accompanied by a deceleration in inhibition efficiency. Throughout the experiment, the carbon steel sample immersed in the acid solution exhibited significantly higher weight loss compared to the corresponding sample in the presence of the inhibitor, across all immersion durations. The disparity in weight loss, resulting from the presence of the inhibitor, can be attributed to the formation of adsorptive layers. These layers act as a protective barrier, shielding the reactive metal ions from the corrosive media. As a result, the weight loss from the substrate is reduced, leading to an increase in inhibition efficiency. Nevertheless, as the immersion time progressed, a notable decline in both weight loss and inhibition efficiency became apparent. This phenomenon can likely be attributed to two potential factors. Firstly, the inhibitor molecules might undergo simultaneous desorption from the sample surface, leading to subsequent dissolution in the exposed area. Secondly, the size, orientation, and interaction of the inhibitor molecules could give rise to specific defects within the inhibitor layer.



Figure 1. Correlation between Immersion Time and Weight Loss of carbon steel in 0.5 M H_2SO_4 with and without 1 mM of β -cyclodextrin at 303 K.

Effect of concentration and temperature

The weight loss measurements were conducted in 0.5 M H₂SO₄ solution in the absence and presence of various concentrations of βcyclodextrin (0.01 to 1 mM) at temperatures ranging from 293 K to 303 K, with an immersion time of 2 hours. The results were analyzed and presented in Figure 2, showing the inhibition efficiency of β -cyclodextrin. It was observed that the inhibition efficiency increased up to 62.26% at a higher concentration of 1 mM. This indicates that the inhibition efficiency is directly proportional to the concentration of the inhibitor, suggesting the formation of a stable adsorbed layer on the surface of the carbon steel. According to the results presented in Figure 2, it is evident that at higher temperatures, there is a significant decrease in the adsorption of the inhibitors on the metal surface, which subsequently results in an increase in the corrosion rate.



Figure 2. Variation in inhibition efficiency with different concentrations of β -Cyclodextrin for carbon steel in 0.5 M H₂SO₄ at various temperatures.

Adsorption Considerations

The adsorption process is significantly influenced by factors such as the structure, charge, and electrolyte composition of the metal surface [15]. In an acidic environment, the inhibitor primarily acts through chemisorption and/or physical adsorption at the metal surface [16]. Analyzing adsorption isotherms allows for a deeper understanding of the physicochemical interactions occurring at the interface between the adsorbed corrosion inhibitor and the metal substrate. By examining the adsorption equilibrium constant (Kads) and Gibbs free energy of adsorption (ΔG_{ads}), and by correlating the surface coverage (θ) with the concentration of the adsorbed species, valuable insights into the adsorption behavior can be obtained [17, 18].

The corrosion inhibition of carbon steel in a 0.5 M H₂SO₄ medium can be attributed to the adsorption phenomenon of β -cyclodextrin on the metal surface. By introducing varying concentrations of β -CD, the inhibition of corrosion is observed and can be attributed to the adsorption of β -cyclodextrin onto the metal surface. Various adsorption isotherms, including Langmuir, Temkin, Freundlich, Frumkin, and El-Awady, were examined in this study. The analysis revealed that the Langmuir adsorption isotherm provided the best fit for the experimental data, indicating that it accurately describes the adsorption behavior observed.

The standard adsorption free energy (ΔG_{ads}) can be determined using the equation [19]:

$$\Delta G_{ads} = -RT \times \ln(55.5 \times K_{ads})$$
(4)

where R represents the universal gas constant, T denotes the thermodynamic temperature, and the value 55.5 corresponds to the molar concentration of water in the solution.

The adsorption heat (ΔH_{ads}) was determined by performing a regression analysis between ln (K_{ads}) and 1/T, (**Figure 3**) resulting in a straight line with a slope equal to $(-\Delta H_{ads}/R)$.

The Van't Hoff equation [21] can be utilized to calculate the adsorption heat using the equation [20, 21]:

$$\ln(K_{ads}) = (-\Delta H_{ads}/RT) + Const.$$
 (5)

The temperature increase leads to a decrease in the equilibrium constants for the adsorption process, as shown in **Table 1**. It is well-known that K_{ads} represents the adsorption capacity of the inhibitor on the metal surface. Notably, β -cyclodextrin exhibits higher K_{ads} values at lower temperatures, indicating a strong adsorption onto the iron surface [3, 13].

Consequently, as the temperature rises, the inhibition efficiency slightly decreases due to enhanced desorption of β -cyclodextrin from the metal surface.



Figure 3. Relationship between $Ln(K_{ads})$ and 1/T for carbon steel corrosion in 0.5 M H₂SO₄ solution with β -cyclodextrine.

The obtained enthalpy value for the adsorption process of β -CD on the carbon steel surface in the sulfuric acid solution is -39.76 kJ/mol, signifying an exothermic nature. The negative enthalpy value confirms the release of energy during the adsorption process of β -CD onto the carbon steel surface in the presence of sulfuric acid solution. Large negative values of ΔG_{ads} , along with their magnitude, are typically indicative of a strong interaction and highly efficient adsorption. When considering ΔG_{ads} values, those up to -20 kJ/mol generally correspond to physisorption, while values around -40 kJ/mol or higher are associated with chemisorption. Chemisorption occurs due to the sharing or transfer of electrons from organic

molecules to the metal surface, resulting in the formation of coordinate bonds [22].

Table 1. Thermodynamic parameters for the adsorption of β -cyclodextrin in H₂SO₄ solution on carbon steel electrodes at different temperatures.

Τ,	\mathbf{R}^2	K _{ads,}	$\Delta H_{ads,}$	$\Delta S_{ads,}$	$\Delta G_{ads,}$
K	к	m ³ /mol	kJ/mo)	J/mol K	kJ/mol
293	0.997	44.10		-13.34	-35.85
303	0.996	22.84	•• • •	-14.39	-35.40
313	0.995	15.13	-39.76	-13.62	-35.49
323	0.987	9.48		-13.57	-35.37

In the present study, the calculated ΔG_{ads} values for β -cyclodextrin range from -35.37 to -35.85 kJ/mol (**Table 1**). These values indicate that the adsorption mechanism of β -cyclodextrin on metal surface in acid solution at the investigated temperatures likely involves a combination of physisorption and chemisorption [23]. The negative adsorption entropy value indicates a decrease in the destruction of the metal surface, while the positive value suggests an increase in system disorder.

Activation energy calculations

The inhibitory mechanism of the studied inhibitor can be comprehended by considering the thermodynamic and activation parameters. The influence of temperature on the inhibitory effect can be effectively illustrated using the Arrhenius equation. This equation is represented as follows [24]:

$$CR = A \times \exp(-E_{act} / RT) \tag{6}$$

In this equation, CR denotes the corrosion rate of carbon steel, A represents the Arrhenius preexponential factor, E_a (kJ/mol) represents the activation energy, R signifies the gas constant (8.314 J/mol K), and T represents the temperature (K).

The values of apparent activation energy for carbon steel in sulfuric acid, both in the presence and absence of different concentrations of β -CD, were determined by examining the slope of ln(CR) versus 1/T (**Figure 4**). The activation enthalpy (Δ H_a) and activation entropy (Δ S_a) can be calculated using the slope (- Δ H_a/R) and intercept (ln(R/Nh) + Δ S_a/R) of ln(CR/T) versus 1/T (**Figure 5**), employing the following transition state equation [25]:

 $\ln(CR/T) = \left[(\Delta S_a/R) + \ln(R/Nh) \right] - \Delta H_a/RT \quad (7)$

Where h represents Planck's constant, N denotes Avogadro's number, ΔS_a corresponds to the entropy of activation, and ΔH_a represents the enthalpy of activation.

The calculated values for activation energy, enthalpy change, entropy change, and Gibbs free energy change are provided in **Table 2**. The presence of an inhibitor can lead to lower or unchanged values of activation energy compared to the blank test, suggesting a chemisorption mechanism. Conversely, higher values of E_a in the presence of the inhibitor indicate a physical adsorption mechanism.

The activation energy values for the corrosion reaction in the absence and presence of β -CD and the blank are as follows: $E_a = 35.96$ kJ/mol for the blank, and $E_a = 48.10$ to 52.09 kJ/mol for β -CD, respectively. This increase in

activation energy implies a significant decrease in the adsorption of the inhibitor on the carbon steel surface with increasing temperature. As the desorption of inhibitor molecules increases at higher temperatures, a larger surface area of the carbon steel is exposed to the aggressive environment, leading to an increase in corrosion rates with rising temperature.



Figure 4. Arrhenius plot for the dissolution of carbon steel in $0.5 \text{ M H}_2\text{SO}_4$ with and without inhibitor at various temperatures.



Figure 5. Transition Arrhenius plots of carbon steel in 0.5 M H_2SO_4 with and without inhibitor at various temperatures

The entropy values (ΔS_a) suggest that the activated complex in the rate-determining step involves an association rather than a dissociation process. This indicates a decrease in disorder as the system transitions from reactants to the activated complex. The positive values of the free Gibbs energy indicate a nonspontaneous corrosion reaction, and these values tend to increase with both the concentration of the inhibitor and temperature. Comparing the ΔG_a values of the process with the presence of the inhibitor to those in its absence, higher values are observed, indicating physisorption as the dominant mechanism. Conversely, lower ΔG_a values are associated with chemisorption [26].

Table 2. Activation parameters for the dissolution of carbon steel in 0.5 M H_2SO_4 in the absence and presence of various concentrations of β -CD

C _{Inh,} mM	R ²	E _{a,} kJ/mol	ΔH _{a,} kJ/mol	-∆S _{a,} J/mol K	ΔG _{a,} kJ/mol
0.00	0.971	35.96	33.41	168.09	84.34
0.01	0.971	48.10	45.55	131.61	85.42
0.05	0.966	49.40	46.84	128.32	85.72
0.50	0.970	50.60	48.05	125.37	86.03
1.00	0.994	52.09	49.54	122.25	86.58

Synergy effect

Synergistic inhibition is achieved through the combination of β -cyclodextrin and iodide ions, effectively preventing corrosion in carbon steel immersed in a 0.5 M H₂SO₄ solution. Halide ions, due to their affinity for organic cations, exhibit a greater tendency to be adsorbed on the metal surface. To evaluate the synergistic effect between inhibitors and iodide ions, the calculation of the synergistic parameter (S_{θ}) was performed based on the method proposed in [27]:

$$S_{\theta} = \frac{1 - \theta_{1+2}}{1 - \theta_{1+2}}$$
(8)

and

$$\theta_{1+2} = (\theta_1 + \theta_2) \tag{9}$$

Where θ_1 inhibition efficiency of iodide ions; θ_2 inhibition efficiency of β -CD and θ'_{1+2} :inhibition efficiency measured from β -CD in combination with iodide ions.

When S_{θ} is less than unity, it suggests the presence of an antagonistic effect, indicating potential competitive adsorption.

Table 3. Synergism parameter (S_{θ}) for 1 mM of β -CD both in the absence and presence of 10⁻³ mM of KI, determined through weight loss measurements at various temperature.

	Inhibition	efficiency (%)	Synergic parameter
Т, К	β-CD	β-CD +KI	S_{θ}
293	64.63	95.32	0.26
303	62.26	97.98	0.30
313	51.93	98.19	0.401
323	35.07	98.51	0.59
333	21.85	99.03	0.84

As S_{θ} approaches unity, the interaction between the inhibitor compounds diminishes. Conversely, when S_{θ} exceeds unity, it signifies a synergistic effect between the chosen inhibitors [28].

The relationship between the efficiency and concentration of each inhibitor, combined with potassium iodide (KI), at different temperatures is presented in **Table 3**.

The results obtained in this study clearly demonstrate a significant enhancement in

inhibition efficiency upon the addition of KI. It is worth noting that the maximum efficiency achieved through the synergistic effect reaches 97.98%, whereas the efficiency without KI present is 62.26%. (at 303 K). An increase in the inhibition efficiency is observed upon the introduction of potassium iodide ions, which can be attributed to their positive effect. Furthermore, the inhibition efficiency shows an upward trend with rising temperatures. In this study, the observed synergism parameter suggests the presence of an antagonistic interaction attributed to competitive adsorption between the inhibitor and the halide ions.

The adsorption of the inhibitor occurs through charge attraction with the metallic surface [29], while iodide ions undergo chemisorption. This interaction between the inhibitor and halide ions leads to enhanced surface coverage, resulting in an increased efficiency [30]. The study concludes that action leads to the formation of more effective barrier films composed of inhibitor molecules on the steel surface, effectively blocking the active sites and protecting the carbon steel from corrosion [31].

Scanning electron microscope

To evaluate the protective ability of β cyclodextrin against carbon steel corrosion in a 0.5 M of sulphuric acid environment, SEM images were captured. Initially, a smooth surface was observed on the carbon steel sample (**Figure 6.a**). In contrast, **Figure 6.b** reveals the presence of pitting corrosion when exposed to a 0.5 M H₂SO₄ solution. The introduction of β -CD resulted in a significant decrease in the corrosion rate of carbon steel in H₂SO₄ solution, as depicted in **Figure 6.c**. The visual analysis reveals a reduction in the occurrence of cracks and pitting compared to **Figure 6.b**. This outcome can be attributed directly to the adsorption of β -cyclodextrin compounds onto the metal surface. It is worth mentioning that the exposed material surface exhibited uniformity when exposed to the acidic medium [32,33].



(c)

Figure 6. SEM images of carbon steel specimens subjected to corrosion in a 0.5 M sulfuric acid solution: (a) prior to immersion (polished), (b) in the absence of β -cyclodextrin, and (c) in the presence of β -cyclodextrin.

Conclusions

The results obtained underscore the remarkable and highly effective inhibitory

properties of β -cyclodextrin in mitigating the corrosion of carbon steel within a 0.5 M H₂SO₄ medium, as assessed through the weight loss method and scanning electron microscopy. The findings highlight a positive correlation between inhibition efficiency and higher β -CD concentrations, while also indicating a decrease in efficiency with rising temperatures. Notably, the activation energy of the corrosion process in the inhibited solution exceeds that of the uninhibited solution.

Furthermore, our investigation demonstrates that the adsorption behavior of the studied inhibitor adheres Langmuir's to adsorption isotherm range across а of temperatures, characterized by an exothermic and spontaneous adsorption process.

Additionally, the observed values of the Gibbs adsorption free energy, averaging -35 kJ/mol, suggest the coexistence of both physical and chemical adsorption mechanisms for β -cyclodextrin. The scanning electron microscopy (SEM) images visually confirm the formation of a protective layer on the carbon steel's surface, further supporting the inhibitor's effectiveness.

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