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Application of Expired Amoxicillin as a Green Corrosion Inhibitor for Carbon Steel in Acidic Conditions

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Abstract

The aim of this study is to investigate the potential of expired amoxicillin as an environmentally friendly corrosion inhibitor for carbon steel (CS) in a 1.0 M hydrochloric acid (HCl) solution. Electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP) techniques were employed to evaluate the inhibition efficiency, along with weight loss measurements over a concentration range of 50-300 ppm and a temperatures range of 303-333 K. SEM and AFM were used to analyze the surface morphology. Discoveries indicate that the expired amoxicillin is a mixed-type inhibitor, reaching the peak efficacy of 91.2 % at 300 ppm and 303 K. Adsorption is by the Langmuir Isotherm as an unstirred physical adsorption. The surface examination established the growth of a protective coating that minimized the corrosive losses. This research indicates the utility of the expired pharmaceuticals as a more affordable and cost-effective substitute corrosion inhibitor that is environmentally friendly.

Keywords: Corrosion inhibition, Expired amoxicillin, and Electrochemical impedance spectroscopy.

1. Introduction

Corrosion of metal is a major concern in many industries, such as petrochemicals, oil and gas, and industrial manufacturing [1, 2]. Carbon steel is widely used due to its favorable mechanical properties and low cost.

However, it is highly susceptible to corrosion, especially during processes such as acid pickling, descaling, and oil-well acidification, where aggressive acidic solutions such as HCl, are employed [3]. Even though there are always the practices of applying corrosion inhibitors to reduce this

problem. The application of conventional inorganic compounds such as chromates and nitrites are not only harmful and risky to the environment but also to human health since they are toxic [4].

This is leading to an increasing trend towards the production of non-toxic, biodegradable and economically viable green inhibitors [5]. Recent reports have identified expired pharmaceutical compounds as effective green corrosion inhibitors [5, 6]. Such materials are frequently polar in nature and are often heteroatomic (including N, O and S). Which enables them to adsorb onto metal surfaces [7].

A suitable candidate is expired amoxicillin, a commonly used β-lactam antibiotic containing carboxyl (–COOH) and amine ($–NH_2$) groups. For better understanding the inhibition, the study uses weight loss, electrochemical measurement, and surface characterization to evaluate the suppression of carbon steel corrosion in a 1.0 M HCl solution with an expired amoxicillin solution [8].

2. Experimental Methodology

2. 1 Materials and Test Solutions

Metal Substrate included carbon steel coupons that were used with the following composition (wt.%) C 0.17, Si 0.38, Mn 0.65, P 0.02, S 0.02 and the balance Fe. For gravimetric tests, specimens with dimensions of 2.5 cm × 0.2 cm × 2.0 cm were prepared. Electrochemical measurements, a carbon steel sample with an exposed area 1.0 cm^2 was cemented in the epoxy resin [9]. Amoxicillin capsules (500 mg) were acquired in a local pharmacy.

The content of the capsule was taken out and modified into powder and then dissolved in the test solution to reach the concentrations of 50, 100, 200 and 300 ppm. Later, analytical grade 37% HCl was diluted with distilled water to create a 1.0 M HCl solution. Every experiment was carried out in naturally aerated, unstirred circumstances. [10].

2. 2 Weight Loss Measurements

Carbon steel coupons were pre-weighed before being submerged in 250 mL of test solution, with and without the inhibitor, for 24 hours at 303 K. Coupons were withdrawn from the water, cleaned, dried, and weighed again. Using conventional formulae, computed the corrosion rate (CR) and inhibition efficiency (ηw%).

For guaranteed repeatability, each experiment was performed three times [11].

2. 3 Electrochemical Measurements

A standard three-electrode cell with carbon steel as the working electrode, platinum mesh as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode was utilized to conduct electrochemical studies using a Gamry Interface 1000 potentiostat [12].

After 30 minutes of immersion stabilizing the open-circuit potential (OCP), potentiodynamic polarization (PDP) curves were taken between -250mV and +250mV vs OCP at 1 mV/s scan rate [13]. Electrochemical impedance (EIS) was recorded at the OCP with frequencies ranging between 100 KHz to 10 MHz and amplitude AC of 10 m V [14].

2. 4 Surface Morphology Analysis

The surface morphology of carbon steel coupons after 24 hours of immersion in blank (1.0 M HCl) and inhibited (300 ppm amoxicillin) solutions were analysed using scanning electron microscopy (SEM) and atomic force microscopy (AFM) [15].

3. Results and Discussion

3. 1 Weight Loss Measurements

Gravimetric analysis provides a direct measure of inhibitor performance. Table one summarizes the corrosion rates and corresponding inhibition efficiencies at 303 K. It can be observed that the corrosion rate decreases significantly with increasing inhibitor concentration. The maximum inhibition efficiency reached 91.2% at 300 ppm compared to 71.4% at 50 ppm. This concentration effect has been attributed to a slow absorption of the molecules of an inhibitor to a carbon steel surface.

As the amount of the inhibitor increases, the size of the protective layer developed gets smaller and denser, acting as a physical shield that separates the metal and the corrosive medium and therefore decreases the anodic dissolution and cathodic hydrogen release. Surface coverage (θ) is also similar and is approaching full surface coverage at the highest concentration, which suggests coverage of surfaces [16].

Table 1: Corrosion parameters of CS obtained based on weight loss at 303 K, in 1.0 M HCl.

Inhibitor Conc. (ppm)	Corrosion Rate ($mg \cdot cm^{-2} \cdot h^{-1}$)	Inhibition Efficiency η_w (%)	Surface Coverage (θ)
(Blank)	1.82	-	-
50	0.52	71.4	0.714
100	0.31	83.0	0.830
200	0.19	89.6	0.896
300	0.16	91.2	0.912

3. 2 Electrochemical Analysis

3. 2. 1 Potentiodynamic Polarization (PDP)

PDP is an effective technique for determining kinetic parameters and elucidating the corrosion inhibition mechanism. The polarization curve of carbon steel in 1.0 M HCl in the presence and absence of expired amoxicillin is shown in figure 1. The addition of the inhibitor leads to a significant decrease in both anodic and cathodic current densities. This combined inhibition of dissolution of metals (anodic) and the evolution of hydroxyl ions (cathodic) without any noticeable change of corrosion potential (Ecorr) is the property of a mixed-type inhibitor.

The corrosion potential values vary slightly (between -476 and -460 mV vs. SCE), with the largest difference being less than 20 mV, it is confirmed again that mixed-type behavior is observed in table two [17]. The density of the corrosion current (Icorr) changes drastically between 650 μA/cm^2 at blank and 55 μA/cm^2 at 300 ppm. That is, PDP inhibition effectiveness is 91.5 %, which is quite consistent with the weight loss rates. The Icorr decrease implies that an inhibitor can raise the energy barrier to the corrosion process by blocking active sites on the metal surface. The anodic (βa) and cathodic (βc) Tafel slope variations indicate that the inhibitor does not simply alter the kinetics of one of the two half-reactions but rather probably by creating some physical barrier retards the ion and electron movements across the metal-electrolyte interface [18].

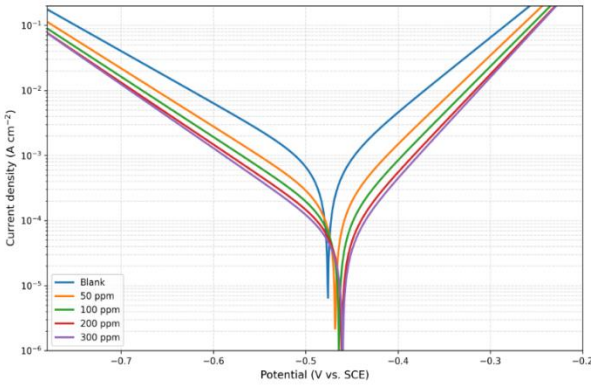

Figure 1: Potentiodynamic Polarization Curves of CS at 303 K with different concentrations of expired amoxicillin in 1.0 M HCl.

Table 2: Electrochemical Parameters from PDP Measurements.

Conc. (ppm)	-E_{corr} (mV vs. SCE)	I_{corr} (μA/cm²)	β_a (mV/dec)	-β_c (mV/dec)	η_{pdp} (%)
Blank	476	650	88	125	-
50	468	185	74	112	71.5
100	464	106	70	108	83.7
200	461	70	67	105	89.2
300	460	55	65	102	91.5

3. 2. 2 Electrochemical Impedance Spectroscopy (EIS)

EIS provides detailed information on the interfacial properties of the corroding system. The Nyquist plots in figure two show

that all tested conditions exhibit a single depressed semicircle. The diameter of this semicircle, corresponding to the charge transfer resistance (Rct), increases significantly with increasing inhibitor concentration. Values of Rct increase with 38 $\Omega \cdot cm^2$ at blank to 435 $\Omega \cdot cm^2$ at 300 ppm as listed in table 3.

This significant growth indicates that the adsorbed layer of inhibitor is demonstrated to be a good covering over the metal surface and that this surface presents a considerable obstacle to an emphasis of transfer between the metal and the corrosive species (H, Cl-) [19]. The constant phase element (CPEdl) values, which indicate a value of the doubled layer capacitance, decrease in value with the addition of the inhibitor. The CPEdl value decreases between 410 $\mu F \cdot s^{n-1} \cdot cm^{-2}$ at blank and 55 $\mu F \cdot s^{n-1} \cdot cm^{-2}$ at 300 ppm. This reduction is explained by the substitution of water molecules and ions on the interface between metal and electrolyte by larger and less polar molecules of the inhibitor [19].

The protective layer results make the electrical double layer thicker, and hence the capacitance ($C \propto \varepsilon A/d$) of the electrical double layer is less. These are credited through the Bode plots in figure 3. An upward trend of the impedance modulus at lower frequencies and a drop in the change in the phase angle peak at higher frequencies which is indicative of a more capacitive and protective surface film. The imprecision of the results is confirmed by the fact that the inhibition efficiencies obtained using EIS (ηeis) are like those obtained by using PDP and weight loss [20].

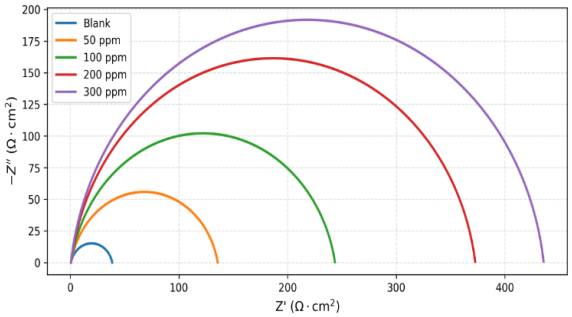

Figure 2: Nyquist plots of CS at a temperature of 303 K at varying concentrations of expired amoxicillin in 1.0 M HCl.

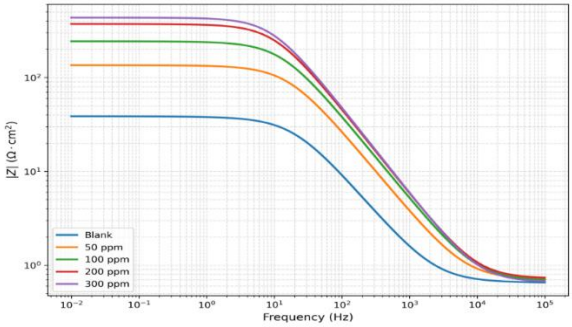

Figure 3: Bode graphs of CS in 1.0 M HCl at different concentrations of expired amoxicillin at 303 K.

Table 3: EIS Measures of electrochemical parameters.

Conc. (ppm)	R_s ($\Omega \cdot cm^2$)	R_{ct} ($\Omega \cdot cm^2$)	CPE_{dl} ($\mu F \cdot s^{n-1} \cdot cm^{-2}$)	n	η_{eis} (%)
Blank	0.65	38	410	0.86	-
50	0.70	135	125	0.88	71.9
100	0.68	243	82	0.89	84.4
200	0.72	372	61	0.91	89.8
300	0.66	435	55	0.92	91.3

3. 3 Thermodynamic Parameters and Adsorption Isotherm

Inhibitor's adsorption process on the metal surface is crucial for explaining the inhibition mechanism. Surface coverage (θ) values obtained from weight loss measurements were fitted to different adsorption isotherms. The Langmuir isotherm is expressed as:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C$$

With a high correlation coefficient (R2 = - 1) the linear plot of C/ θ versus C. According to the Langmuir model, the adsorption of the monolayer is assumed to be on a smooth surface in the absence of interaction between the adsorbed molecules as shown in figure 4 [21].

Using the following formula, the standard free energy of adsorption (-ΔGads) was determined by multiplying the equilibrium constant of the adsorption process (Kads) by the intercept of the Langmuir plot.

$$K_{ads} = \frac{1}{55.5} \exp\left(\frac{-\Delta Gads^{\circ}}{RT}\right)$$

Computed value of ΔGads was -27.4 kJ /mol. As a rule, the ΔGads were less than -20 kJ /mol, physisorption (electrostatic interactions) whereas -40 kJ/mol or above indicated chemisorption (electrostatic interaction). The value, which was yielded as -27.4 kJ/mol indicates that adsorption of expired amoxicillin on the surface involved on the carbon steel is a spontaneous process that is being driven by a mixed mechanism in which the primary interaction involves physisorption.

This is likely to happen due to electrostatic attraction between the molecules of the protonated inhibitors and the negatively charged metallic surface in the acidic media, the vacant d-orbitals in iron and the free pair of electrons in the inhibitor have some donor-acceptor interactions [22]. The mode of adsorption is the key factor affecting the inhibition performance and stability of the protective film.

The calculated value of the standard free energy of adsorption (ΔG°ads = -27.4 $kJ \cdot mol^{-1}$) indicates that the adsorption of expired amoxicillin on carbon steel. Surface is mainly physisorption but can be accompanied by weak chemisorption. Typically, ΔG°ads values less negative than

−20 kJ·mol^{-1} are related to electrostatic interactions between the charged metal surface and the charged inhibitor molecules, while more negative values (more negative than −40 kJ·mol^{-1}) are related to chemisorption with charge sharing or transfer [23]. In the present study, the intermediate value suggests mixed types of adsorptions, with the initial adsorption process dominated by electrostatic attraction between positively charged amoxicillin molecules and the negatively charged steel surface in acidic solution.

This is consistent with the presence of heteroatoms (N and O) and π-electrons in the structure of the molecules, which can lead to donor-acceptor interactions with the empty d-orbitals of iron atoms [19, 21]. This kind of physisorption-dominated adsorption is usually reversible and temperature-dependent, which may result in a drop in corrosion efficiency with increasing temperature. But it also allows for quick adsorption and the formation of a protective film, as indicated by the high inhibition efficiency and surface analysis.

Thus, the adsorption process of expired amoxicillin can be considered successful for corrosion inhibition in the present study, especially at room temperatures where physisorption is stable [23].

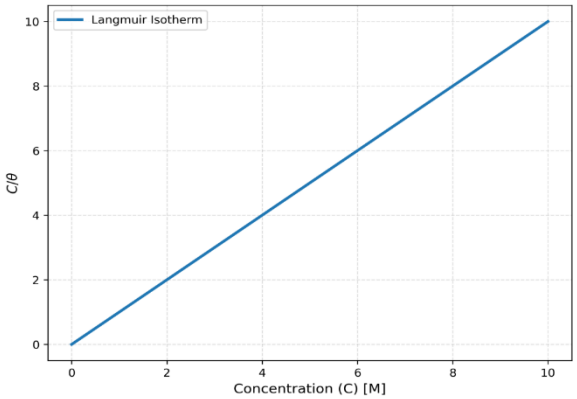

Figure 4: Langmuir adsorption isotherm of expired amoxicillin on CS surface in 1.0 M HCl at 303 K.

3. 4 Surface Morphology Analysis

The protective effect of the inhibitor was visually confirmed using SEM micrographs as shown in figures 5-8. The carbon steel surface exposed to blank 1.0 M HCl solution exhibited severe corrosion damage, characterized by a rough and uneven morphology with deep pits, cracks, and a loosely adherent porous corrosion product layer [22]. This morphology is typical of aggressive acid attack resulting in which there is excessive dissolution of the metal surface [23].

On the contrary, the surface of the specimen in the solution with 300 ppm expired amoxicillin is much smoother and even [24]. There is a clear and tight coating

covering the metal underneath and this layer is protective in nature. This coating is a protective layer that keeps the metal out of contact with the corrosive electrolyte [24]. The lack of pitting or cracking also proves the fact that the inhibitor is also capable of preventing uniform and localized corrosion. This would be quantitatively confirmed by AFM analysis as shown in figure 9-12. The average carbon surface roughness (Ra) is significantly lowered with an inhibited sample, which is in accordance with the growth of a coherent protective layer [24].

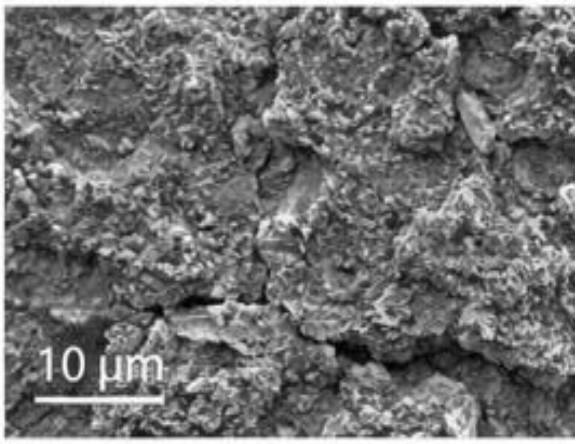

Figure 5: SEM images micrographs (x1000) of carbon steel surfaces following a 24-hour immersion in 1.0 M HCl without inhibitor.

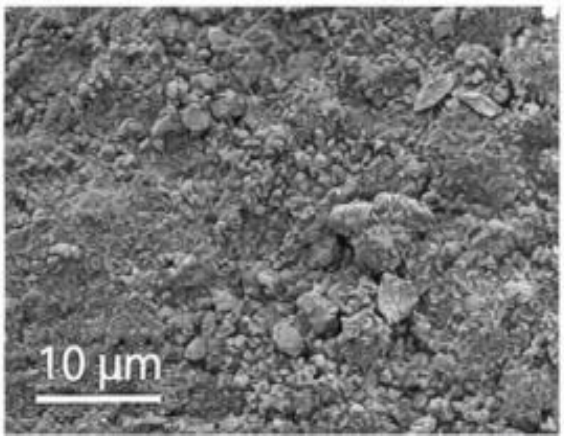

Figure 6: SEM images micrographs (x1000) of carbon steel surfaces following a 24-hour immersion in 1.0 M HCl with 50 ppm inhibitor.

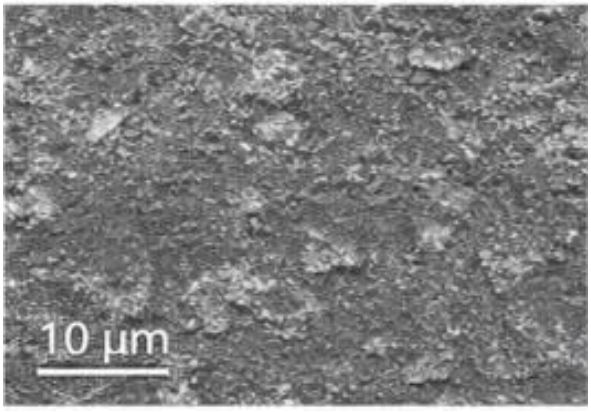

Figure 7: SEM images micrographs (x1000) of carbon steel surfaces following a 24-hour immersion in 1.0 M HCl with 200 ppm inhibitor.

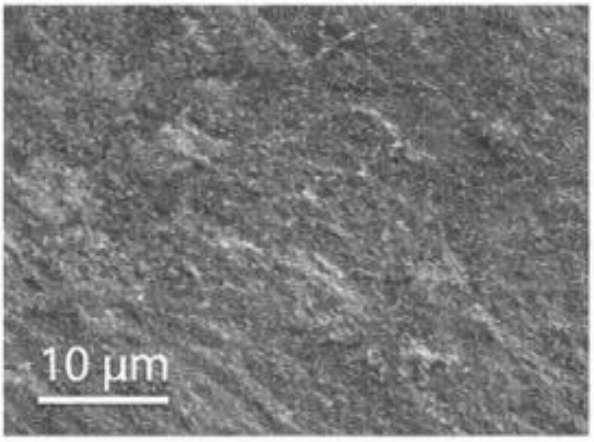

Figure 8: SEM images micrographs (x1000) of carbon steel surfaces following a 24-hour immersion in 1.0 M HCl with 300 ppm inhibitor.

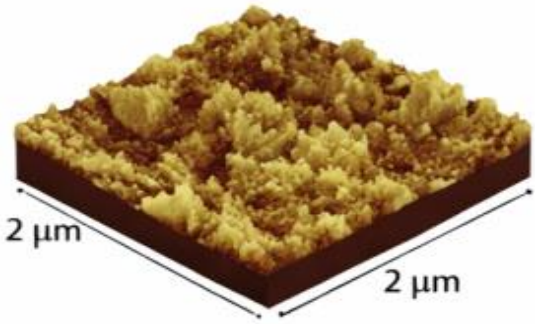

Figure 9: AFM images micrographs (x1000) of carbon steel surfaces following a 24-hour immersion in 1.0 M HCl without inhibitor.

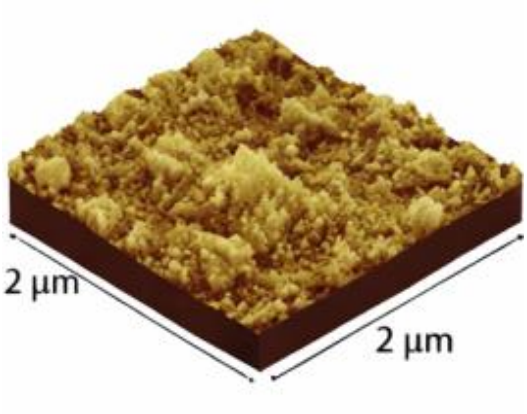

Figure 10: AFM images micrographs (x1000) of carbon steel surfaces following a 24-hour immersion in 1.0 M HCl with 50 ppm inhibitor.

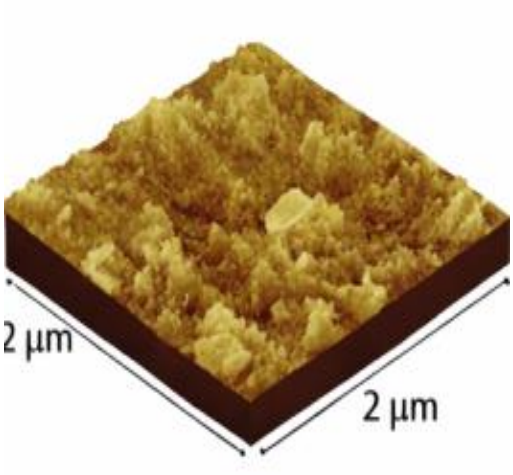

Figure 11: AFM images micrographs (x1000) of carbon steel surfaces following a 24-hour immersion in 1.0 M HCl with 200 ppm inhibitor.

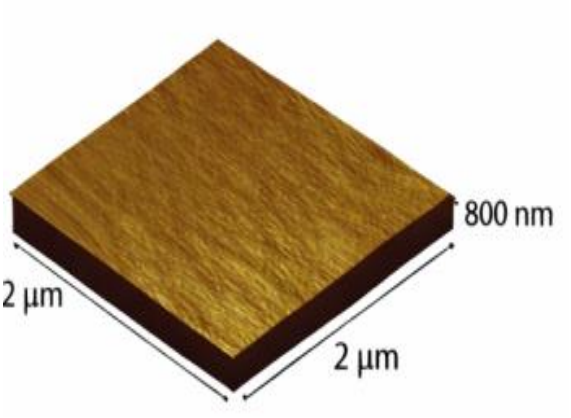

Figure 12: AFM images micrographs (x1000) of carbon steel surfaces following a 24-hour immersion in 1.0 M HCl with 300 ppm inhibitor.

Comparative evaluation of the corrosion inhibition performance of expired amoxicillin, used as the inhibitor in this study, alongside several reported green corrosion inhibitors for carbon steel in acidic media as listed in table 4. An inhibition efficiency of 91.2% was achieved at a concentration of 300 ppm in 1.0 M HCl. This level of performance is considered comparable to levels that are reported by many green inhibitors in literature. Efficiencies in the range of 85–94% and 85-92% have been reported for quinazoline [2] and pyrazine derivatives [10] respectively.

A slightly higher efficiency range (90-95%) has been observed for fifth-generation α-amino phosphonates under similar concentration conditions [5]. In contrast, lower efficiencies (70-85%) have been documented for Luffa cylindrica leaf extract [11]. Furthermore, expired pharmaceutical compounds have been reported to exhibit inhibition efficiencies reaching approximately 90%, placing amoxicillin at the upper end of this range. Overall, expired amoxicillin functions as an effective, environmentally friendly, and economically viable corrosion inhibitor, with the additional advantage of facilitating the reuse of pharmaceutical waste as listed in table four.

Table 4: Comparative performance of expired amoxicillin and selected green corrosion inhibitors for carbon steel in acidic media.

Inhibitor Type	System / Medium	Concentration (ppm)	Inhibition Efficiency (%)	Reference
Expired Amoxicillin	CS in 1.0 M HCl	300	91.2	Present study
Luffa cylindrica extract	CS in HCl	200-500	70-85	11
Quinazoline derivatives	Mild steel in 1 M HCl	100-300	88-94	2
α-Aminophos-phonate	CS in HCl/H_2SO_4	150-300	90-95	5
Pyrazine derivatives	Steel in HCl	100-500	85-92	10
Expired drugs (general class)	Steel in acid	Variable	Variable (up to~90%)	7

Expired drugs (general class) Steel in acid Variable Variable (up to ~90%) Gecé, 2011. Note: CS = Carbon Steel; HCl = Hydrochloric acid. Values are reported from literature under comparable experimental conditions.

4. Conclusion

This study reveals that expired amoxicillin is a safe and effective corrosion inhibitor for carbon steel in 1.0 M HCl. It attained its maximum effectiveness of 91.2% at 300 ppm. It is a mixed-type inhibitor that inhibits both anodic and cathodic processes, as evidenced by its potentiodynamic polarization. Scanning electron microscopy (SEM) and electrochemical impedance spectroscopy (EIS) were used to assess the creation of the protective coating that can be linked to the decrease in double-layer capacitance and the rise in resistance to charge transfer. The adsorption was like the monolayer covering Langmuir Isotherm.

Free energy of adsorption (-27.4 kJ/mol) is evidence of spontaneous adsorption which is predominantly physical. The study offers the opportunity of implementing the use of expired pharmaceuticals like amoxicillin as the sustainable and economical alternative to toxic inhibitors that offer the possibility of waste valorization and corrosion prevention.

5. References

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