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Inhibition of Mild Steel Corrosion in 1M Hydrochloric Medium by the *cherimoya seeds*

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Abstract

In the present study, the inhibition performance of mild steel by cherimoya seeds extract was investigated using weight loss measurements, electrochemical techniques, and scanning electron microscope (SEM) coupled with energy-dispersive X-ray spectroscopy (EDX). The experimental data suggested that the cherimoya seeds exhibited a high inhibition performance, which increases with increasing their concentrations. Cherimoya seeds extract present maximum inhibition efficiency of 94% at an optimal concentration of 1g/L. The principal observations that resulted from electrochemical studies are that cherimoya seeds affected both anodic and cathodic reactions (mixed inhibitors). Their adsorption, which is a combination of chemisorption and physisorption, obeyed the Langmuir isotherm model. Furthermore, the temperature effect was carried out at various temperatures ranging from 303 to 328 K to verify the corrosion inhibition performance of cherimoya seeds at higher temperatures. Moreover, SEM-EDX analysis confirmed that cherimoya seeds can ensure remarkable prevention against corrosion through the adsorption onto the metal surface.

Keywords: *cherimoya seeds extract; Mild steel; hydrochloric acid; Corrosion. Electrochemical*

1. Introduction

In industry, many acidic solvents are extensively used, particularly for removing different types of locally occurring deposits such as rust or scale. Among them, chloride acid is very often used [1,2]. In addition, the most extensively used metal in these industrial sectors is mild steel on account of its outstanding mechanical performance, extreme thermal stability, high tensile strength, its manufacturability, and economical price [3–9]. Despite such importance, mild steel is highly susceptible to corrosion in acidic media [10]. Industrial installations and equipment that are likely to be damaged can be designed and built, taking into account the available anti-corrosion treatments. Instance, hydrochloric acid solutions are commonly used for pickling, industrial acid cleaning, acid de-scaling, and oil well-acidifying processes [11–13]. Because of the aggressiveness of acid solutions, mild steel corrodes severely during these processes, particularly with the use of hydrochloric acid, which results in a terrible waste of both resources and money [14, 15]. Therefore, the use of inhibitors is one of the usual methods of protecting metallic materials against corrosion in acidic environments, i.e., inhibitors compound is often added in the acid. Moreover, in the corrosion studies, the selection of a good corrosion inhibitor is controlled by its economic availability, its efficiency to inhibit the substrate material and its environmental side effects. The majority of synthetic compounds have good anticorrosion action, but most of them are highly toxic to humans and the environment [16]. Therefore, due to environmental concerns, different inhibitors extracted from natural plants (vegetable oils or plant tannins) have allowed researchers to achieve high inhibitory efficiency values. In recent years, plant extracts as corrosion inhibitors attracted great attention due to their properties like low cost, environment-suitability, renewability and also due to the cost effectiveness and simplicity of the methods utilized during the extraction of these plants [17]. In this context, the literature In this context, the literature review reveals that exclusive and extensive works have been done in the research areas related to plant leaves, bark, and stem as corrosion inhibitors for steel in HCl medium like Grapefruit [18], Apricot [19], Thymus leptobotrys [20], Lavandula stoechas [21], Thymus broussonnetii [22], Phyllanthus amarus [23], Tabernaemontana divaricata [23], Pimenta dioica [24], Bryophyllum Pinnatum [25]. Furthermore, the existing literature discovered that adsorption of corrosion inhibitors takes place through adsorptive interactions between concerned compounds and metal surface. Generally, organic compounds containing nitrogen, sulfur and/or oxygen atoms and polar functional groups are considered to be good corrosion inhibitors in wet corrosion environments [26,27]. In the present work, we chose cherimoya seeds extract as a corrosion inhibitor. The aim of this study is to evaluate the inhibitory action of the extract of cherimoya seeds as well as its mode of action on mild steel in a solution of 1.0M HCl using several methods including weight loss measurements, adsorption isotherms, potentiodynamic polarization (PDP), and electrochemical impedance spectroscopy (EIS). In addition, further experimental investigations were performed to evaluate the effect of immersion time and temperature on the inhibition efficiency. Moreover, the inhibition effect of the target inhibitors was also investigated through the morphological characterization of the mild steel substrates by scanning electron microscope/energy-dispersive X-ray spectroscopy (SEM/EDX) analysis.

2. Material and Methods

Preparation of the Extract, the seeds of cherimoya are milled in a blender and kept until use.

Preparation of the work electrode, the material used as working electrode in this study is mild steel whose chemical and mass composition is given in Table 1.

Table 1: chemical and mass composition of mild steel

Elements	Fe	C	Si	Mn	Cu	S	Cr	Co	Ti	Ni
Composition (w%)	98.307	0.38	0.23	0.68	0.16	0.016	0.077	0.09	0.011	0.059

To have a good reproducibility of measurements, it is necessary to have a clean surface state. The surface of the samples undergoes mechanical polishing on abrasive papers (carbon, silica) of increasing particle size ranging from 80 to 1200 mm followed by rinsing with distilled water and drying. *Preparation of the Corrosive Solution*, The corrosive solution consists of a molar solution of 1M hydrochloric acid prepared from a commercial solution of hydrochloric acid (37%) using bidistilled water. *Electrochemical Studies*, Potentiodynamic polarization (PDP) and electrochemical impedance spectroscopy (EIS) tests are carried out by a Volta lab potentiostat (Tacussel Radiometer PGZ 100) controlled by the Voltamaster 4 software. This assembly has three electrodes: mild steel as working electrode (ET), platinum as auxiliary electrode (CE) and Ag/ AgCl electrode as reference electrode. The working electrode is immersed in the test solution for 30 min until a steady-state open circuit potential (E_{ocp}) is established. The intensity-potential curves or polarization curves of the metal/solution interface are obtained in potentiodynamic mode. The potential applied to the sample varies continuously from −800 to −200mV vs. ECS, with a sweep rate of 1 mVs^{-1}. The intensity of the current is measured between the working electrode and the platinum counter-electrode. Before drawing these curves, the working electrode is maintained at its abandonment potential for 30 min. For impedance measurements, the amplitude of the sinusoidal disturbance applied to the dropout potential is chosen to satisfy the linearity conditions (10mV peakto-peak). The frequencies scanned during these impedance measurements go from 100 kHz to 10 mHz at the rate of 5 points per decade. The direct current (DC) voltage was taken from the Ecorr to the reference electrode obtained from the OCP. *Gravimetric Measurements*, the principle of this method is based on the measurement of the weight loss experienced by a surface sample S, during the time t of immersion in the corrosive solution, in the absence and in the presence of the tested inhibitor, maintained at a constant temperature. The gravimetric tests are carried out in a double walled cell equipped with a condenser and a thermometer. However, a circulating water thermostat keeps the electrolyte at the desired temperature. As well as the electrolyte volume is 100 ml, and the samples are in rectangular form of dimensions 2 cm × 2 cm × 0.2 cm. Before any measurement, the samples are polished with sandpaper of decreasing grain size up to 1200 followed by washing with distilled water and acetone and drying in air. After weighing accurately, the samples are immersed in beakers containing 100 ml of acid solutions without and with different concentrations of the extract at a temperature of 303 K for 6 hours immersion time. At last, each value of the gravimetric tests is the average of three trials.

3. Results and Discussion

3.1. Influence of Inhibitors Concentrations

3.1.1. Polarization Curve.

The inhibition effectiveness of cherimoya seeds extract is evaluated using potentiodynamic polarization experiments at different concentrations at 298K. The important electrochemical corrosion parameters including corrosion potential (E_{corr}), corrosion current density (I_{corr}), and cathodic Tafel slope were determined with the help of polarization curves as shown in Fig. 1, and they are listed in Table 2. Looking at Fig. 1, it is apparent that the addition of cherimoya seeds inhibitor to corrosive solution decreases considerably the current densities and a change in the anodic and cathodic curves is observed. This means that the presence of this inhibitor affects both cathodic and anodic reactions of mild steel substrate. It is also clear that the cathodic parcels are parallel and the inhibition effect of cherimoya seeds extract is more remarkable on cathode than on the anode part indicating that the inhibitor retarded the hydrogen evolution reaction without modify the mechanism of cathodic reaction [28]. Moreover, it can be seen from the data in Table 2 that the I_{corr} decreases in function of cherimoya seeds concentration, while the inhibition efficiency is enhanced with the increase of

the variable dose of cherimoya seeds extract. Also, in this study, the displacement in E_{corr} is less than 85 mV compared to the blank solution, which means that that cherimoya seeds acts as mixed type inhibitor with predominant cathodic effectiveness [29].

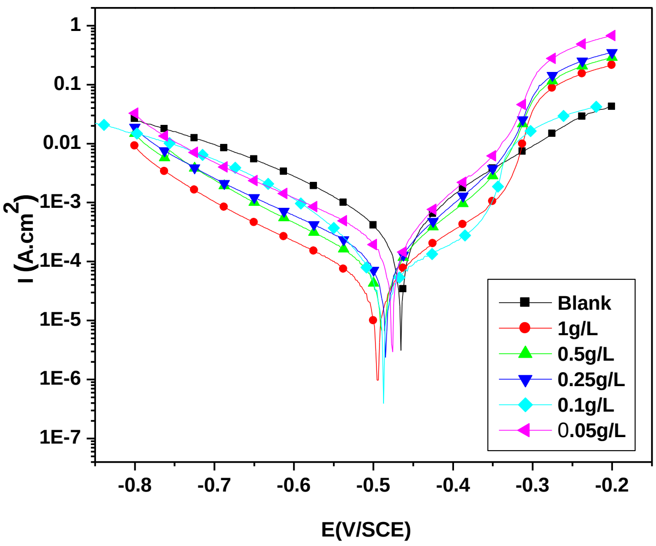

Fig.1: PDP curves for mild steel in 1M HCl at various concentrations of cherimoya seeds extract at 298K.

Table 2: PDP parameters obtained for the mild steel at various concentrations of the cherimoya seeds extract in 1.0 M HCl and the corresponding inhibition efficiency at 298 K.

Inhibitor	Concentrations	$-E_{corr}$ (mV/SCE)	I_{corr} (µA/cm^2)	$-b_c$ (mV/dec)	E (%)
Blank	**1M**	463	636	168	-
cherimoya seeds extract	**1.00 g/L**	494	41	132	93.55
	0.50 g/L	484	72	148	88.68
	0.25 g/L	494	102	161	83.97
	0.10 g/L	473	146	214	77.04
	0.05 g/L	464	218	152	65.72

3.1.2. Electrochemical impedance spectroscopy (EIS) study

In Figure 2, Nyquist and Bode diagrams for the steel electrode are shown with a range of concentrations of the tested compounds in 1.0 M HCl at 298 K. In the case of the Nyquist impedance plots, they represent the imaginary component of the plotted impedance as a function of the real component. In contrast, the Bode plots show the logarithm of the impedance modulus |Z| and phase angle as a function of the logarithm of the frequency f. Based on the following equation, the double- layer electrical capacity (C_{dl}) for each inhibitor concentration is calculated [30]:

$$C_{dl} = \frac{\sqrt[n]{Q.R_{ct}}}{R_{ct}} \quad (1)$$

With n indicating as always the phase shift, which allows calculating the surface heterogeneity of mild steel [31,32]. Based on the EIS technique, for the determination of inhibitory efficacy, the following formula is used:

$$E_{Rct}(\%) = (\frac{R'_{ct} - R_{ct}}{R'_{ct}}).100 \qquad (2)$$

Where R'_{ct} and R_{ct} signify the present the charge transfer resistance without and with inhibitor, Electrochemical parameter values estimated according to EIS curves are given in Table 3. The obtained plots of impedance are showing a capacitive loop that increases in size with increasing inhibitor concentrations, that is attributable to the charge transfer process. Therefore, R_{ct} values with corrosion resistance significance have been achieved by fitting the EIS diagrams with the equivalent circuit shown in the insert Fig. 3.

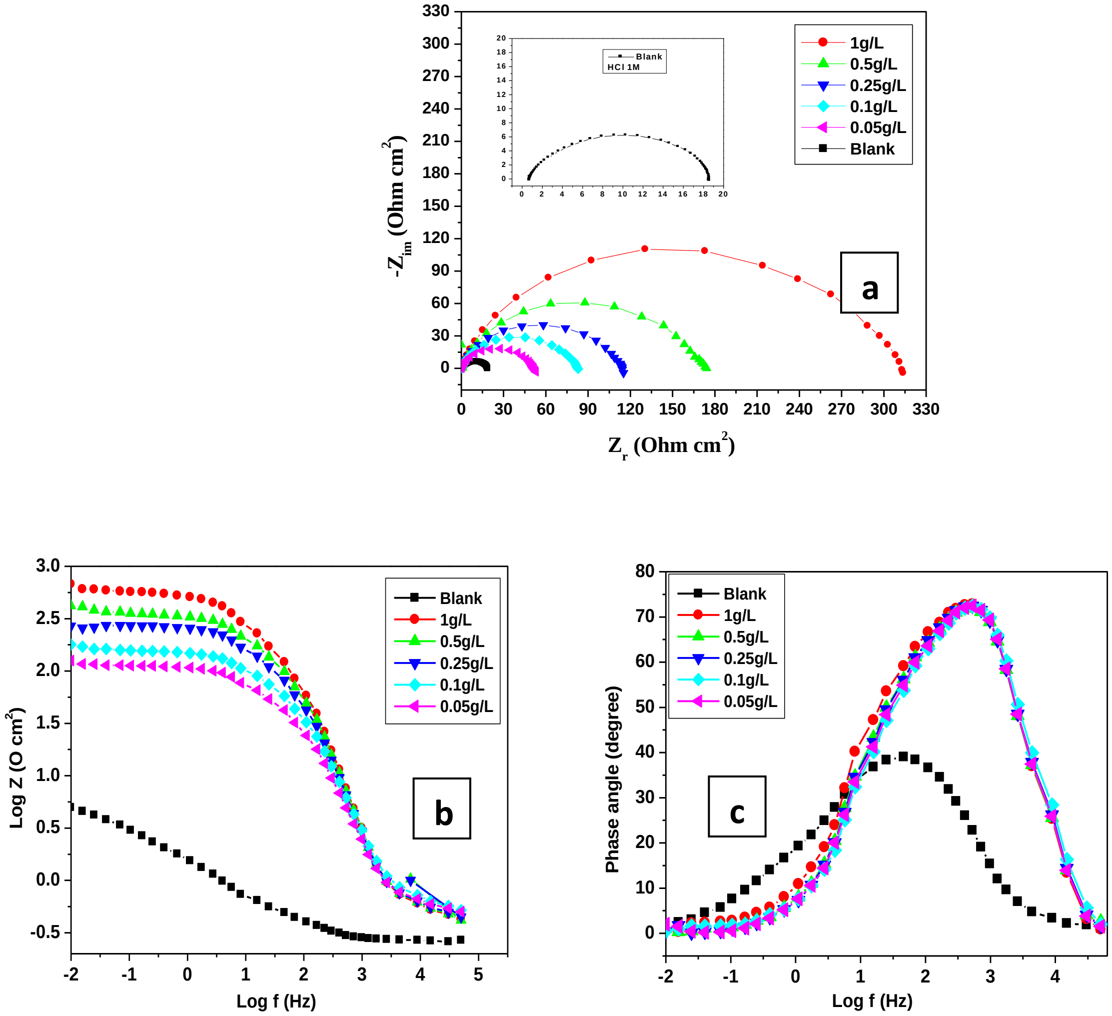

Fig 2. Nyquist (a), Bode (b), and phase angle (c) plots of mild steel in 1.0 M HCl with and without various concentrations of cherimoya seeds inhibitor.

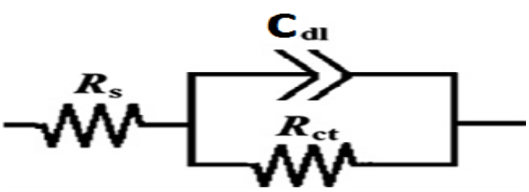

Fig 3. Equivalent circuit model applied to fit and simulate the impedance data

Table 3. Electrochemical parameter values estimated according to EIS curves of mild steel in HCl solution in the presence and absence of several concentrations of cherimoya seeds at 298 K..

Inhibitors	Concentration	R_{ct} ($\Omega\times cm^2$)	n	$Q\times10^{-4}$ ($s^n/\Omega\times cm^2$)	C_{dl} ($\mu F/cm^2$)	E_{Rct} (%)	Θ
Blank	1M	18	0.79	2.092	49	-	-
cherimoya seeds	**1.00 g/L**	312	0.82	0.3624	13	94.23	0.94
	0.50 g/L	174	0.81	0.4857	15	89.65	0.89
	0.25 g/L	116	0.80	0.7427	22	84.48	0.84
	0.10 g/L	82	0.81	0.8457	26	78.04	0.78
	0.05 g/L	53	0.80	1.0453	28	66.03	0.66

3.1.3. Gravimetric Study

The concentration effect is determined by the immersion of the substrates in the corrosive solution, without and with the addition of the extract of cherimoya seeds at different concentrations (0.05, 0.1, 0.025, 0.5, and 1g/L). The inhibitory efficacy is determined after 6 h at 298 K. The corrosion rate (W) and the inhibition efficiency are determined using Equations (3) and (4) given below:

$$W_{corr} = \frac{m_i - m_f}{S.t} \quad (3)$$

Where m_i and m_f are the weights of the samples before and after immersion in the tested solution, On the surface of the mild steel specimen (cm^2) and t is the exposure time (*h*). W_{corr} and W'_{corr} respectively, represent the corrosion rates of the steel after immersion in the absence and in the presence of the inhibitor

$$E_w(\%) = \frac{W_{corr} - W'_{corr}}{W_{corr}} \times 100 \quad (4)$$

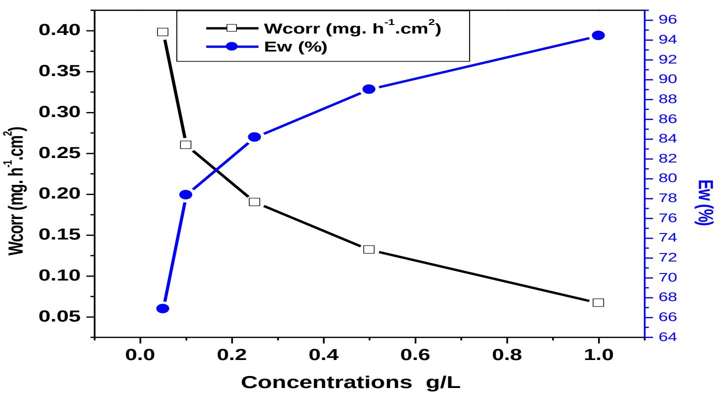

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Fig 4. The variation of corrosion rate and protection efficiency with the extract of cherimoya seeds concentrations for mild steel in 1.0 M HCl at 298 K.

3.1.4. Influence of the Temperature on Inhibitor Performances

In parallel to the concentration effect, a temperature effect study was performed to further examine and give more insight into stability of inhibitor under higher temperatures taking into consideration the wide range of applications of mild steel. This part discusses the findings obtained from weight loss measurements carried out at various temperatures ranging from 298 K to 328 K. The effect of temperature on steel electrode dissolution in terms of corrosion rate in 1.0 M HCl was evaluated in the absence and presence of various concentrations of cherimoya seeds extract. The temperature impact on the corrosion rate and effectiveness of

cherimoya seeds is inserted in Table 4, while Table 5 provides thermodynamic parameters related to the temperature effect computed from analyzing the Arrhenius plots.

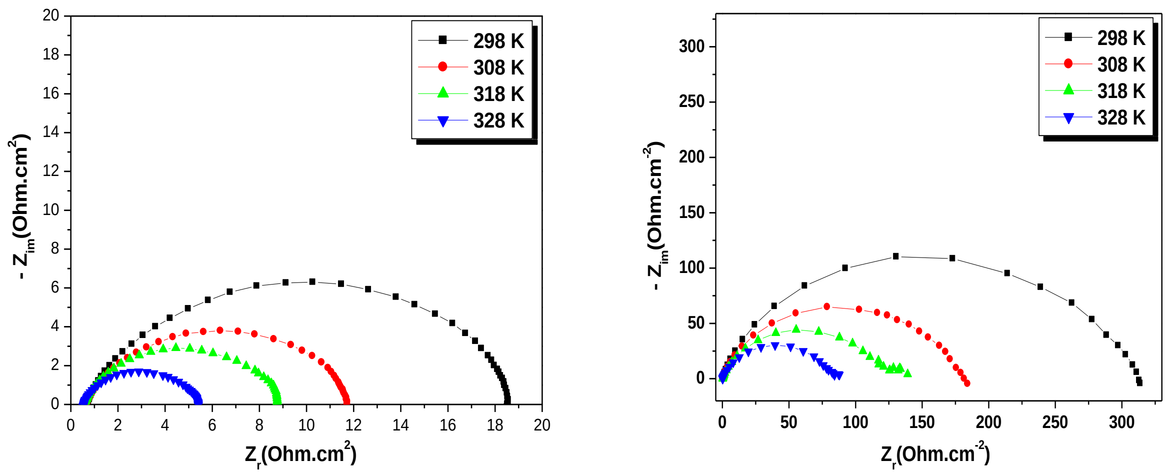

Fig. 5. Nyquist diagrams for carbon steel in 1 M HCl + 1 g/L of cherimoya seeds at different Temperatures.

Table 4. Influence of temperature toward the corrosion and inhibition efficiency for mild steel in 1.0 M HCl with and without the cherimoya seeds compound.

Inhibitor	Temp (K)	R_s (Ω.cm^2)	R_{ct} (Ω.cm^2)	*n*	Q×10^{-4} (s^n/Ω×cm^2)	C_{dl} (μF /cm^2)	E_{Rct} (%)
Blank	298	0.42	18	0.79	2.092	48	-
	308	0.42	11	0.77	2.69	7	-
	318	0.48	8	0.80	2.37	5	-
	328	0.44	5	0.81	2.17	4	-
cherimoya seeds 1 g/L	298	0.45	312	0.82	0.36	13	94.23
	308	0.19	180	0.81	0.47	16	93.88
	318	0.22	124	0.82	0.66	19	93.54
	328	0.16	84	0.81	0.74	22	94.04

Arrhenius curves were fitted to determine the thermodynamic parameters such as enthalpy change of activation (ΔH*), entropy change (ΔS*), and activation energy (Ea) for the mild steel corrosion in 1.0 M HCl without and with different concentrations of tested compounds using the following equations:

$$I_{corr} = A\exp(-\frac{E_a}{R.T}) \quad (5)$$

$$I_{corr} = \frac{R.T}{Nh}.\exp(\frac{\Delta S^*}{R}).\exp(-\frac{\Delta H^*}{RT}) \quad (6)$$

where A denotes the pre-exponential factor, h signifies the Planck constant, N denotes the number of Avogadro, R is the universal gas constant, and T represents the temperature. The kinetic activation parameters derived from Arrhenius and transition state plots, shown in Figure 6a,b, are listed in Table 5.

Table 5. Thermodynamic activation parameters for the mild steel electrode in 1.0 M HCl in the presence and absence of cherimoya seeds.

Inhibitor	Ea (kJ/mole)	ΔH* (kJ/mole)	ΔS* (J/mole K)	Ea-ΔH
Blank	36.38	33.79	-191.53	2.60
cherimoya seeds	37.66	35.06	-210.75	2.60

As shown in Table 5, the values of activation energy (Ea) are generally seen as a factor strongly related to the concentration of inhibitor, and the Ea value calculated for inhibited systems are greater than those of the blank acid solution. The higher value of Ea in the presence of cherimoya seeds when compared to the uninhibited system, suggests that the dissolution of mild steel becomes difficult in the presence of the cherimoya seeds inhibitor because the energy barrier was higher. Consequently, a protective film on the surface of mild steel could be formed [33,36]. Moreover, it is apparent from Table 5 that the enthalpy values increase compared to the uninhibited case and are positive, which indicate the endothermic nature of mild steel dissolution during the corrosion process [37,38]. In terms of the disordering of the molecule during the inhibition process, the high negative values of ΔS* in comparison to the blank solution indicate that there is a decrease in the disorder during the switch from reagents to the activated complex. Furthermore, these findings might be a result of the adsorption of the activated compound which prefers the association rather than dissociation on the mild steel surface. This is a quasi-substitution phenomenon between inhibitor molecules in the acidic solution and H_2O molecules present at the mild steel /electrolyte interface [39]. In addition, the obtained results (Table 5) indicate that there is a perfect relationship between Ea and ΔH* values, which further confirm and validate all these observations.

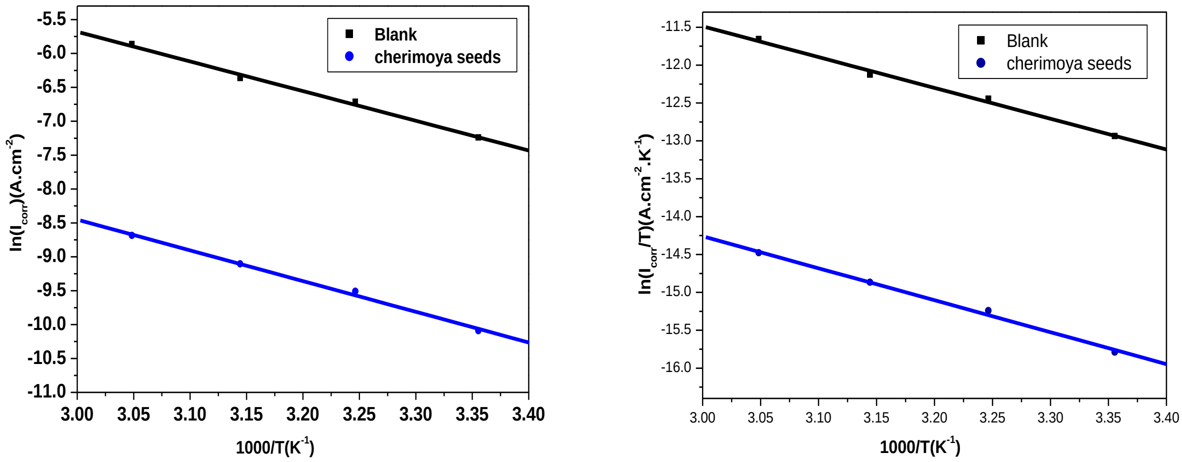

Fig 6. Arrhenius (a) and transition state (b) plots of the corrosion rate for mild steel dissolution in 1.0 M HCl solution in the absence and presence of different concentrations of cherimoya seeds.

3.1.5.Adsorption Isotherm

One of the most typical processes for inhibitory molecules is adsorption. Due to this, it is possible to confirm the adsorption behavior of the molecule under examination by adapting the obtained results to many known adsorption isotherms [40]. Along with the Langmuir adsorption isotherm model, three other different types of adsorption isotherms, including Freundlich and Frumkin were evaluated. In this study, of the previously mentioned model of isotherms, it should be noted that the best fit was accomplished from the Langmuir model. According to the Langmuir isotherm, **Θ** is related to the inhibitor concentration $\mathbf{C_{inh}}$ by the following equation (7):

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \qquad (7)$$

In which C_{inh} signifies the inhibitor concentration, K_{ads} means the constant of adsorption equilibrium, while θ denotes the coverage area. The plot of Langmuir adsorption isotherm of the cherimoya seeds using the data of gravimetric studies is shown in Fig. 7, which present a linear regression with a slope approximately equal to 1 and correlation coefficient value near to unity ($R^2 > 0.999$).

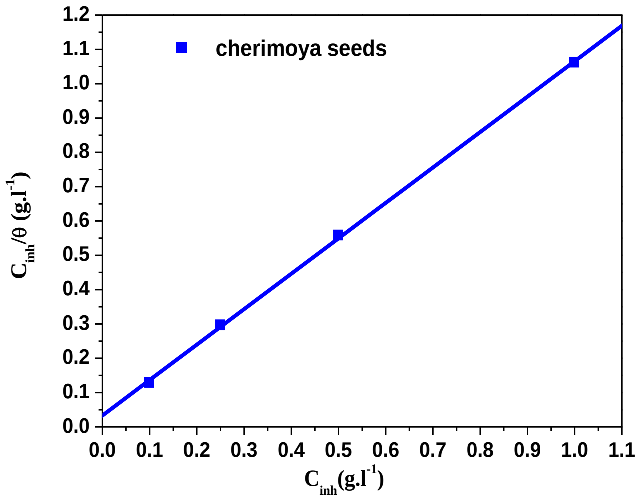

Fig.7. Plots of the Langmuir adsorption isotherm of the cherimoya seeds on the mild steel surface at 298 K.

Table 6: The adsorption parameters for the corrosion of mild steel in 1.0 M HCl at 298K.

Inhibitor	**Slope**	**K_{ads} (M^{-1})**	**R^2**
cherimoya seeds	1.03	30.06	0.99982

3.1.6.FT-IR analysis

FT-IR spectrum analysis is a powerful tool that can be used to identify the type of bonding predominantly functional groups present in the inhibitor. The spectra of cherimoya seeds shown in Figure 8 the absorption band observed at 1466.42 cm^{-1} is associated with hydroxyl or/and N–H groups. The band at 1661.07cm^{-1} indicates the presence of C = O groups. The band around at 1160 cm^{-1} can be assigned to the stretching mode of the C–N group. The shift in the absorption bands of the inhibitor on the mild steel surface strongly supports the interaction between the phytochemical compounds of the inhibitor and mild steel surface [41].

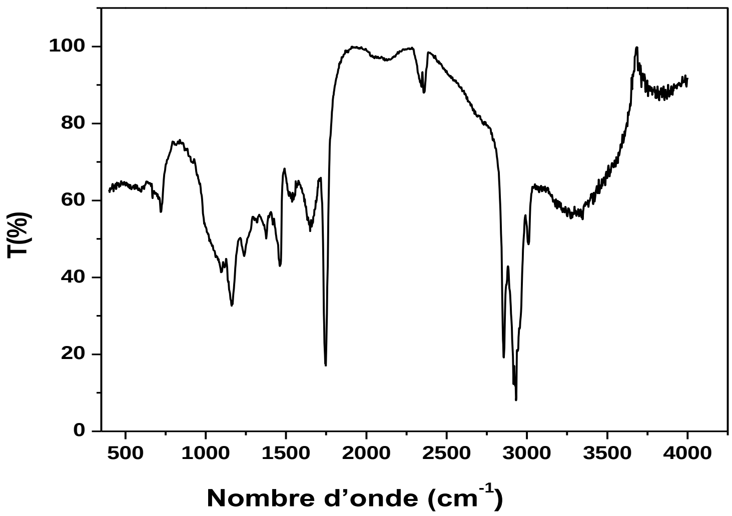

Fig.8.FT-IR spectra of cherimoya seeds

3.1.7.Surface Morphological Study

SEM analysis is an effective method for studying the morphology of mild steel surface after being immersed in various mediums. This technique is particularly useful to characterize the development of a protective organic layer on the surface that allows a high degree of inhibition resistance. Figure 9a,b illustrates the surface morphology of processed mild steel surfaces immersed for 24 h in two different solutions; non-inhibited solution and that with 1g/L of cherimoya seeds inhibitor. As Figure 9 shows, there is a significant difference between the two SEM micrographs. From Figure 9a,.

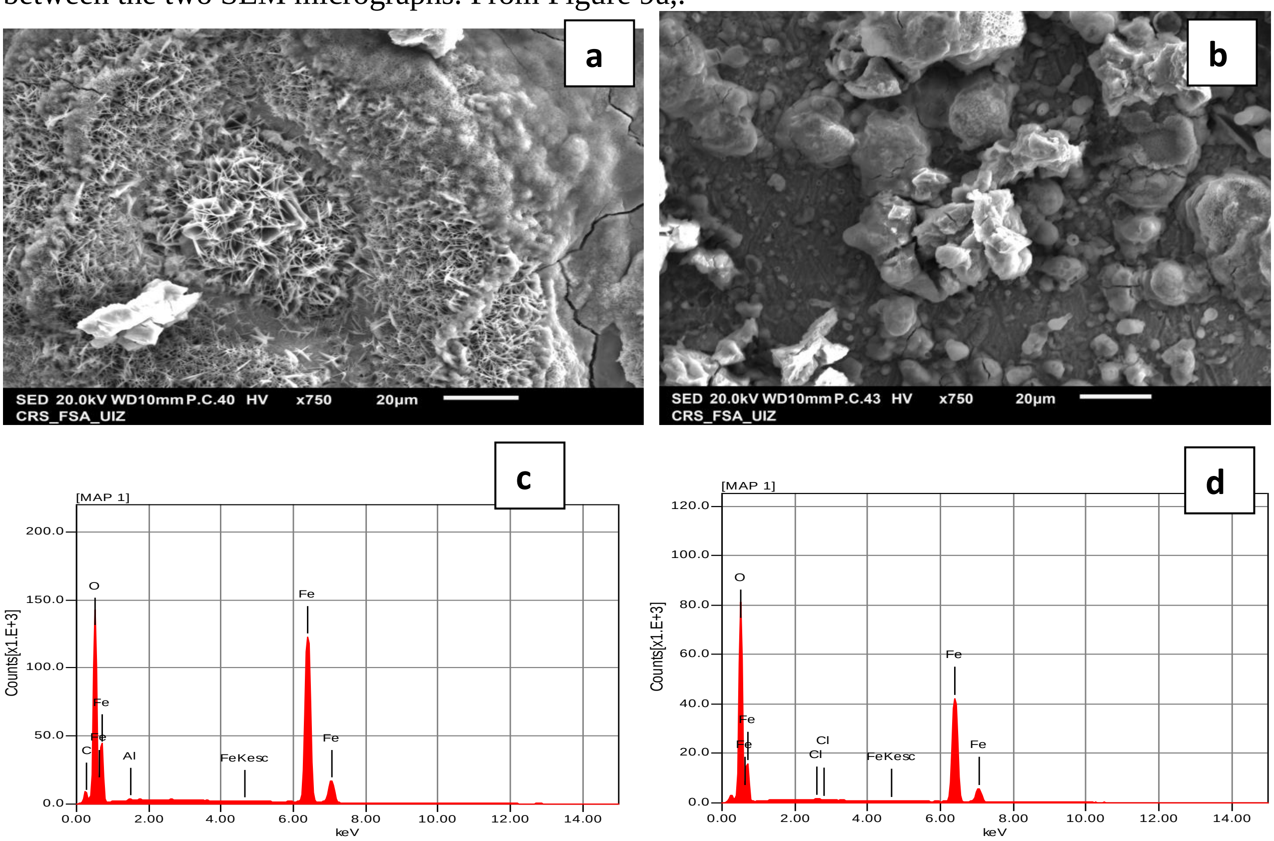

Fig.9. SEM and EDX photographs of mild steel specimens after 24 h of immersion in 1.0 M HCl solution in the absence (a,c) and the presence of 1g/L cherimoya seeds (b, d).

we can see that without inhibitor, the electrode surface is highly corroded and contains internal corrosion damage, which is due to rapid corrosion attack in the acid solution; hence, the steel surface becomes inhomogeneous and rough. However, Figure 9b shows that with the presence of the cherimoya seeds inhibitor, the damage of the mild steel surface is significantly reduced, meaning that the surface morphology is more protected due to the cherimoya seeds adsorption onto the steel electrode surface. In order to examine the protection mechanism, EDX analyses of the steel surface were performed after 24 h of immersion in HCl. In this latter medium, the presence of peaks corresponding to oxygen, carbon, and iron were detected by EDX analysis on the steel substrate before the addition of the inhibitor (Figure 9c). By comparing the EDX spectrum of the sample in the presence of tested extract with that of the blank, it is clear that the peak of chlorine and oxygen declines in the EDX spectra when cherimoya seeds are present. Such remarks approve that the cherimoya seeds prevents metal corrosion through the formation of an electrolyte-limiting layer to the metal [41,43]

4- Conclusions

The corrosion inhibition effect of cherimoya seeds for mild steel in a solution of 1.0 M HCl were investigated using chemical, electrochemical, and surface characterization techniques. The outcomes clearly show that the increase in the concentration of inhibitor towards an optimal value of 1g/L considerably impacted the inhibitory efficacy of the cherimoya seeds. Nevertheless, the favorable inhibitory effect of cherimoya seeds was studied under different temperatures ranging from 298 to 328 K. However, corrosion inhibition was almost unchanged with increasing temperatures, which implies the long-term stability of the inhibiting effect. It was also concluded from the cherimoya seeds results that acted as mixed-type inhibitor with a distinctly cathodic character, and that the adsorption followed the Langmuir isotherm model. The results from SEM analysis confirmed that the cherimoya seeds stopped the corrosion of steel by forming a layer that limits the access of the electrolyte to the surface.

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