



Eco-Friendly *Pancreaticum Foetidum Pom* Extracts as Corrosion inhibitors for Mild Steel in 1M HCl Media

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Abstract

Currently, corrosion has become the concern of many industries, for this reason and to prevent chemical electrochemical phenomenon, many studies especially in acid were carried out in recent years. In this work, we sought to understand the influence of methanolic and dichlorometanic extracts on corrosion of mild steel in HCl medium by the electrochemical measurements: polarization curve, electrochemical impedance spectroscopy and for weight loss measures. The results showed that the efficiency of inhibition is proportional to the concentration of the inhibitor and can reach 93% in the presence of dichloromethane extract of *Pancreaticum foetidum Pom* at 1 g / l to 308K. The temperature effect on the corrosion behavior showed that the inhibition efficiency of the two extracts decreases significantly with temperature. The adsorption of different extracts on the steel surface follows the Langmuir adsorption isotherm.

Keywords: *Pancreaticum foetidum Pom*, Green Corrosion, EIS, Langmuir Adsorption, Methanol and Dichloromethane extracts.

1. Introduction :

Corrosion can be defined as the interaction (electrochemical reaction) of a metal with the surrounding environment, causing a slow, steady, and irreversible deterioration in the metal, to both its physical and chemical properties. These phenomena have major impacts in various fields, particularly in industry. The work stoppage and change of corroded material for the industry has huge financial cost. The use of inhibitors is one of the best techniques that can prevent corrosion of materials and alloy with its environment. A corrosion inhibitor is a chemical substance which, when added in small concentrations to an environment reduce the corrosion rate of the material [1]. The selection of the inhibitors is not only based on their inhibition efficiency but also their environmental impact. Artificial and heterocyclic chemicals are not suitable for the corrosion inhibition process, in spite of their high efficiency, due to their hazardous impact on the environment. Natural compounds and green inhibitors are the most suitable concerning their safe impact towards the environment. The pure plants extracts had efficient performance in preventing the corrosion of different metal in acidic media [15, 17].

Green inhibitors like natural products from plant extracts and substances from other renewable sources are of interest to researchers who are interested in “green chemistry” or “eco-friendly” technologies. [2-3]

Pancreatium foetidum Pom belongs to Amaryllidaceae family which gathers 75 types and 1100 species, it's a plant endemic to north west of morocco and east of Alger according to Jahandiez and Maire[4]. *Pancreatium foetidum Pom* is known by several biologic effect such as, antimicrobial*, antifungal*, analgesic*, anticholinesterase*, due the presence of alkaloid compounds used in the study of cytotoxic* and antimitotic* effects.

However, the amaryllidaceae family - which *Pancreatium foetidum Pom* belong to it – is the inspiration of the alkaloids studies [5-6]. Furthermore, several investigation concern the isolation and the identification of the alkaloids of the amaryllidaceae are published, those compounds showed many pharmacologic properties (antiviral, antibacterial ...etc.), also they presented a great interest in the treatment of Alzheimer [7-8]. The inhibition of corrosion by the alkaloids was the aim of many studies [9-11].

The aim of our study is to investigate the influence of both extracts methanol and dichloromethane of *Pancreatium foetidum Pom* on the corrosion of mild steel in HCl acid medium by electrochemical measurements: weight loss, temperature effect, polarization curve and electrochemical impedance spectroscopy.

1. Materials and methods

2.1. Preparation of Extracts

Pancreatium foetidum Pom plant obtained from Saïdia-Oujda, Fig. 1, was washed with distilled water, dried, powdered into small pieces, according to the following procedure to obtain the two extracts.



Figure 1: *Pancreatium foetidum Pom*

Methanolic extract EMe:

The dried bulbs were cut into small pieces and ground in methanol using a mixer. After 48 hours of maceration in methanol, and the filtrate obtained is evaporated to dryness. The crude methanol extract is obtained; a first CCM analysis shows that this extract is rich in alkaloids.

Dichloromethane extract EDi:

The methanol extract is resumed in ether (250ml). This phase has eliminated the maximum neutral substances. 250ml of distilled H₂O were added. The mixture thus obtained is decanted to separate two immiscible phases: an aqueous phase and an ether phase. This operation was repeated at least three times. The rich aqueous phase alkaloids is separated from the ether phase containing natural substances (lipids, sterols ...). The rotary evaporator temperature was 20-25 ° C as the ether is very volatile. We got in late the ether extract and separate the second aqueous phase. After this phase, the acid phase comes (hydrochloric acid). It is necessary that the pH has a value between 3 and 4. Then the extraction is done with dichloromethane. This operation causes a passage of alkaloids in the aqueous phase, however, the organic compounds such as lipids, resins ...etc., are eliminated in dichloromethane. The aqueous phase is then made alkaline with ammonia and then with dichloromethane, water-soluble compounds (sugar, inorganic and organic salts) are removed in the aqueous phase. After recovery of the dichloromethane phase decantation, the dichloromethane is completely removed by evaporation in vacuum and dichloromethane extract was obtained [36].

The aggressive solutions of 1 M HCl were prepared by dilution of an analytical grade 37% HCl with double distilled water.

Different concentrations (0.125, 0.25, 0.5 and 1 g/l) were prepared from the *Pancreatium foetidum* powder by dissolving the required amount of *Pancreatium Foetidum Pom* in 100 ml of 1 M HCl with stirring at room temperature. 100 ml of 1 M HCl without inhibitor was used as blank test solution.

2.2. Preparation of Specimens

The mild steel sheets were cut into $2 \times 2 \times 0.25$ cm³ dimensions with the following composition:

Composition	P	Al	Si	Mn	C	S	Fe
	0.09%	0.01%	0.38%	0.05%	0.21%	0.05%	Balance

Prior to all measurements, the sheets surfaces were abraded with 180, 400, 800, 1000, 1200 grades of emery papers. The specimens are washed thoroughly with bidistilled water degreased and dried with ethanol.

2.3. Weight Loss Method

Gravimetric measurements of square steel specimens of size 2 x 2 x 0.25 cm in 1 M HCl with and without addition of different concentrations of both **EDi** and **EMe** Extracts. The corrosion rate was determined after 6 h of immersion period, at a temperature of 308K in by the following equation:

$$\vartheta = \frac{\Delta m}{S \times t} \quad (1)$$

Where: Δm is the average weight loss, S the total area, and t is immersion time. With the corrosion rate calculated, the inhibition efficiency (E %) is determined as follows:

$$E\% = \frac{\vartheta^{\circ} - \vartheta}{\vartheta^{\circ}} \times 100 \quad (2)$$

Where ϑ° and ϑ are the corrosion rates in the absence and presence of the inhibitor, respectively.

2.4. Electrochemical Measurements:

Electrochemical experiments were monitored using potentiostat PGZ 100 piloted by Voltmaster software, the electrochemical measurements were carried out in conventional three electrode electrolysis cylindrical Pyrex glass cell. The working electrode (WE) has the form of a disc cut from the steel sheet, the area that is exposed to the corrosive solution was 1 cm². A saturated calomel electrode (SCE) and a platinum electrode were used respectively as reference and auxiliary electrode. The temperature of the cell was controlled by a thermostatically at 308 K. Before start any test, it is necessary that the potential must stabilized for 30 min.

The inhibition efficiency IE_{Rt} % was defined as:

$$E_p \% = \frac{I_{corr}^{\circ} - I_{corr}}{I_{corr}^{\circ}} \times 100 \quad (3)$$

Where I_{corr}° and I_{corr} represent corrosion current density values in the absence and presence of the inhibitor, respectively.

The electrochemical impedance spectroscopy (EIS) measurements are carried out with the electrochemical system, which included a digital potentiostat model Voltalab PGZ100 computer at E_{corr} after immersion in solution without bubbling. After the determination of steady-state current at a corrosion potential, sine wave voltage (10 mV) peak to peak, at frequencies between 100 kHz and 10 mHz are superimposed on the rest potential. Computer programs automatically controlled the measurements performed at rest potentials after 0.5 hour of exposure at 308 K. The impedance diagrams are given in the Nyquist representation. [12-19]

Each experiment was duplicated to check the reproducibility.

The inhibition efficiency IE_{Rt} % was defined as:

$$IE\% = \frac{R_{ct}^{\circ} - R_{ct}}{R_{ct}^{\circ}} \times 100 \quad (4)$$

where, R_{ct}° and R_{ct} are the charge transfer resistance values in the absence and presence of inhibitor, respectively:

2. Results and Discussion

2.1. Weight Loss Measurements

3.1.1 Inhibitor concentration effect

The corrosion rates and inhibition efficiency values were calculated using weight loss data, for different concentrations (0.125, 0.25, 0.5 and 1 g/l) at 308K of the two extracts (**EDi** and **EMe**) for the corrosion of carbon steel in 1 M HCl solution are listed in Table 1.

Table 1. Effect of **EDi** and **EMe** on the corrosion rate of mild steel in 1 M HCl at 308K.

Inhibitor	Concentration g/l	Corrosion rate mg/cm ² .h	E (%)
Blank	-	9.07	-
EMe	1	1.41	84
	0.5	1.60	82
	0.25	2.90	68
	0.125	4.11	54
EDi	1	0.65	93
	0.5	2.03	78
	0.25	2.41	73
	0.125	2.48	72

The Table 1 of results showed clearly that the inhibition efficiency increased by increasing the concentration due to the adsorption of the compounds on the steel surface. The methanol extract is full of organic families such as alkaloids, polyphenols, flavonoids, etc.... The dichloromethane extract **EDi** which contains a great amount of alkaloids decreased the corrosion rate and led to a high efficiency reach until 93% for the concentration of 1g/l. On the other side, the methanol extract **EMe** has the lowest efficiency; it reaches until 84% at the same concentration. This could be explained by the antagonist effect of the other compounds and the alkaloid family in methanol extract. Comparing the two extracts, we conclude that **EDi** is a better than **EMe** as a green inhibitor. Generally, the inhibitor suppresses the metal dissolution by forming a protective film adsorbed to the metal surface and separating it from the corrosion medium.

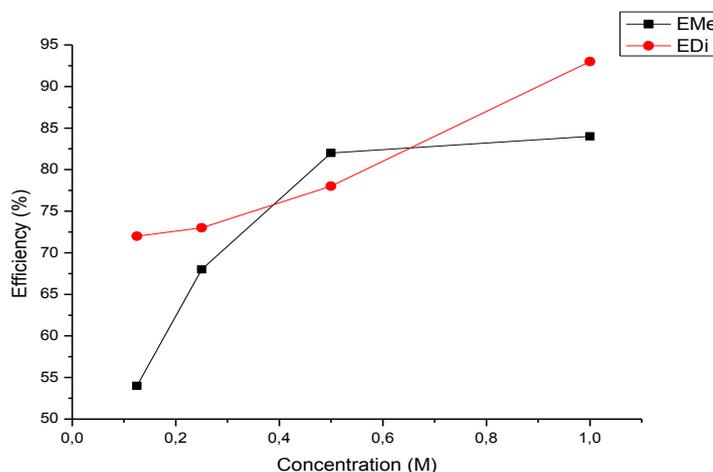


Fig. 1: Variation of the inhibition efficiency of different concentrations of extracts in HCl medium

3.1.2 Temperature effect:

The temperature effect study was carried out by weight loss method in the temperature range 313-343 K, in 1 M HCl by the absence and presence of 1 g/l of inhibitor after 1 h of immersion time. The results obtained are listed in Table 2.

Table 2. Effect of temperature on the corrosion rate of mild steel in 1 M HCl at 1g/l of extracts concentration for 1h.

Inhibitor	Temperature (K)	Corrosion rate (g/m ² .h)	E (%)
Blank	313	11.56	-
	323	22.03	-
	333	37.95	-
	343	69.15	-
EDi	313	1.64	86
	323	4.68	79
	333	11.25	70
	343	23.21	66
EMe	313	3.39	71
	323	7.47	66
	333	17.66	53
	343	52.30	24

The Table 2 and Figure 2 showed clearly that the corrosion rate increases by the rise of temperature in the HCl medium in the absence and the presence of both EDi and EMe extracts of *Pancreaticum foetidum Pom*, the corrosion rate of mild steel decreased at any given temperature due to the increasing of the degree of surface coverage. The results showed clearly in the Fig. 2, that the inhibition efficiency decreased with increasing of the temperature

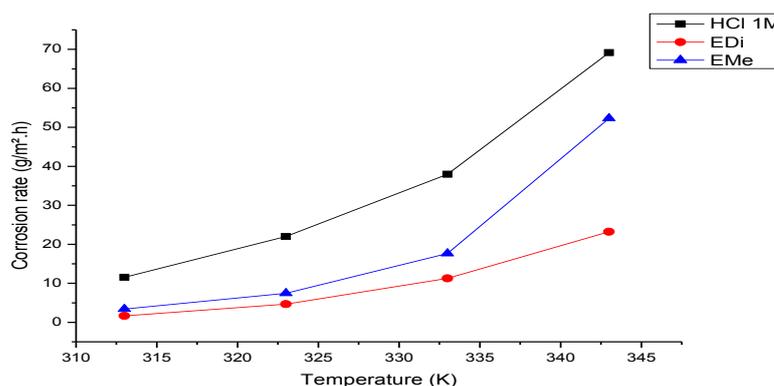


Fig. 2: Variation of the corrosion rate of extracts on the mild steel in HCl medium versus several temperatures

To calculate the activation parameters for the corrosion process based on Arrhenius Eq. (5) and transition state Eq. (6) were used [20]:

$$V = A \exp\left(-\frac{E_a}{RT}\right) \quad (5)$$

$$V = \frac{RT}{Nh} \exp\left(\frac{\Delta S_{ads}^\circ}{R}\right) \exp\left(-\frac{\Delta H_{ads}^\circ}{RT}\right) \quad (6)$$

Where E_a is the apparent activation corrosion energy; R is the universal gas constant; k is the Arrhenius pre-exponential factor. h is Plank's constant. N is Avogrado's number. ΔS_a is the entropy of activation and ΔH_a is the enthalpy of activation.

Arrhenius plots for the corrosion rate of mild steel are given in Fig. 3 and Fig. 4. Values of apparent activation energy of corrosion (E_a) for mild steel in 1 M HCl with the absence and the presence of various concentrations of **EDi** and **EMe** were determined from the slope of $\ln(v)$ versus $1/T$ plots of *Pancreaticum Foetidum Pom* extracts and shown in Table. 4.

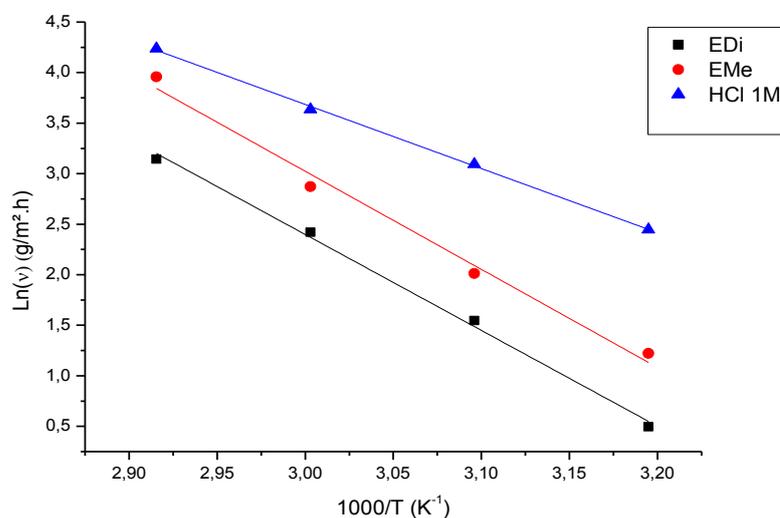


Fig. 3: Arrhenius Plots in the absence and presence of extracts in HCl medium

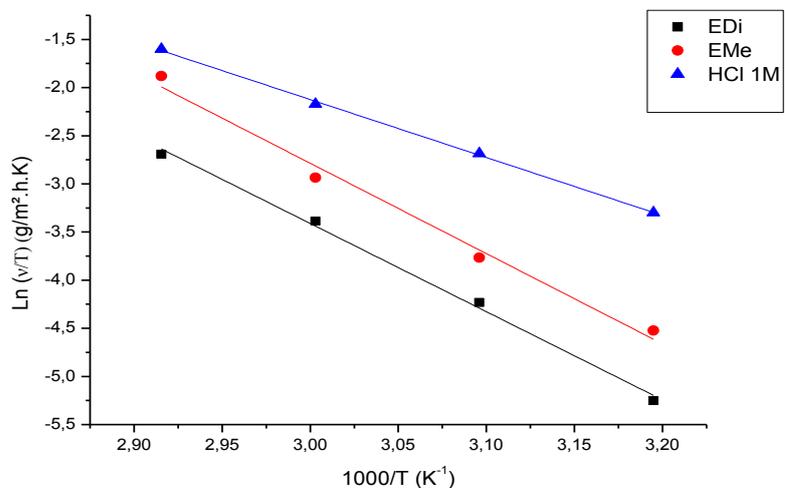


Fig. 4: Variation of $\ln(v/T)$ versus $1000/T$ with different extract in HCl medium

The relationship between the temperature and the corrosion rate does not only show the corrosion behavior of the mild steel against changing temperature, it also provides us with a lot of information including the activation of thermodynamic parameters such as the activation energy E_a activated entropy ΔS_a and enthalpy ΔH_a by using Arrhenius equation :

$$\ln(v) = \ln(A) - \frac{E_a}{RT} \quad (7)$$

$$\ln\left(\frac{v}{T}\right) = \left(\ln\left(\frac{R}{Nh}\right) + \frac{\Delta S_a}{R}\right) - \frac{\Delta H_a}{RT} \quad (8)$$

Where V is the corrosion rate, A is a constant depends on metal type and electrolyte, E_a is the apparent activation energy, h is the Planck's constant (6.626176×10^{-34} Js), N is the Avogadro number (6.02252×10^{23} mol⁻¹). R is the universal gas constant and T is the absolute temperature, ΔH_a and ΔS_a is the enthalpy and the entropy of activation, respectively. All the parameters are listed in Table 3.

According to the results summarized in the table 3, we conclude that the activation energy E_a in the absence of the extract is lower than those in presence of **EDi** and **EMe** meaning that the adsorption process is physical (electrostatic) [21-23], Furthermore, the positive values of ΔH_a showed that the corrosion process is endothermic.

The negative values of entropies ΔS_a imply that the activated complex in the rate determining step represents an association rather than a dissociation step, meaning that a decrease in disordering takes place ongoing from reactants to the activated complex and suggesting that the dissolution reaction will be more blocked sites from the metal surface [24].

Inhibitor	Concentration g/l	E_a (kJ mol ⁻¹)	ΔH_a (kJ mol ⁻¹)	ΔS_a (kJ mol ⁻¹)	$E_a - \Delta H_a$
Blank	-	52.744	50.017	-218.306	2.72
EDi	1	78.886	76.164	-435.696	2.72
EMe	1	80.687	77.968	-450.696	2.72

Table 3: Activation parameters for the steel dissolution in 1 M HCl in the absence and the presence of *Pancreatium Foetidum Pom* extracts at 1 g/l of concentration

2.2. Adsorption isotherm and standard adsorption free energy

The adsorption of the organic compounds can be described by two main types of interaction: physical adsorption and chemisorption that are influenced by the nature and charge of the metal, the chemical structure of the inhibitor and the type of electrolyte. If one supposes that, the adsorption of this inhibitor follows the Langmuir adsorption isotherm. Correlation between surface coverage (θ) defined by $E\%/100$ and the Langmuir adsorption isotherm, the isotherm is given by [25,26] can represent the concentration of inhibitor (C):

$$\frac{C}{\theta} = \frac{1}{k} + C(5)$$

Where K is the adsorption constant, C is the concentration of the inhibitor and surface coverage values (θ) are obtained from the weight loss measurements for various concentrations.

The adsorption isotherm parameters and correlation coefficients found from the slopes and intercepts are summarized in Table 4 from Fig.5.

The Langmuir adsorption isotherm is the best fit was made according to the correlation coefficient since $R^2 > 0.98$ [27,28]. Langmuir adsorption isotherm assumes that the adsorption of organic molecules on the adsorbent is monolayer.

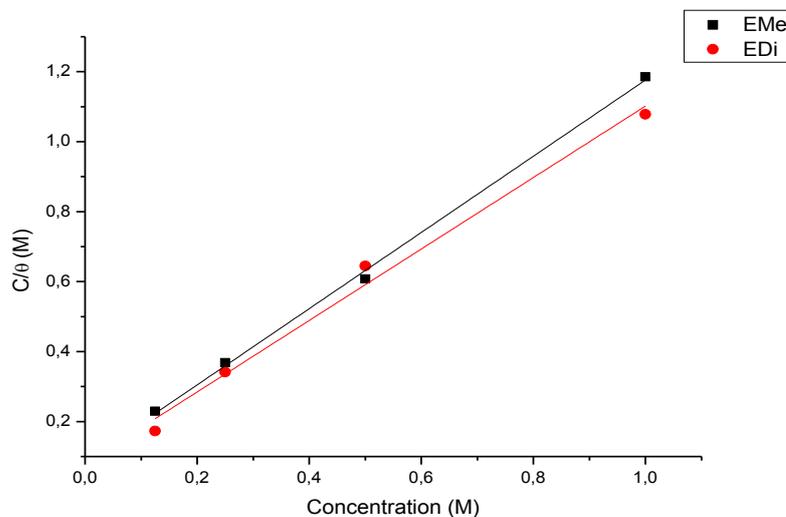


Fig. 5. Langmuir adsorption of **EDi** and **EMe** on the mild steel in HCl medium

Table 4: Parameters of the linear regression between C/θ and C and thermodynamic values of adsorption extracts on the steel mild in HCl media.

Inhibitor	Linear correlation	Slope	K
EMe	0.99783	1.0894	11.49
EDi	0.98431	1.0216	12.45

3. Potentiodynamic polarization curves:

The electrochemical measurement was carried out to understand the kinetic process of the anodic and cathodic reactions. The mild steel electrode maintained at corrosion potential for 30 min and thereafter pre-polarized at 800 mV for 10 min. The potential start from -800 mV to -200 mV with a scan rate of 1 mV/s.

Fig. 6 illustrates the cathodic and anodic polarization curves recorded for the mild steel in 1 M HCl solutions without and with various concentrations of both **EDi** and **EMe**. Electrochemical corrosion parameters including corrosion current density (I_{corr}), corrosion potential (E_{corr}), cathodic Tafel slope (β_c), anodic Tafel Slope (β_a) and inhibition efficiency ($E_p(\%)$) obtained by Tafel extrapolation method are given in Table 5. The $E_p(\%)$ was calculated from polarization measurements according to the relation given below.

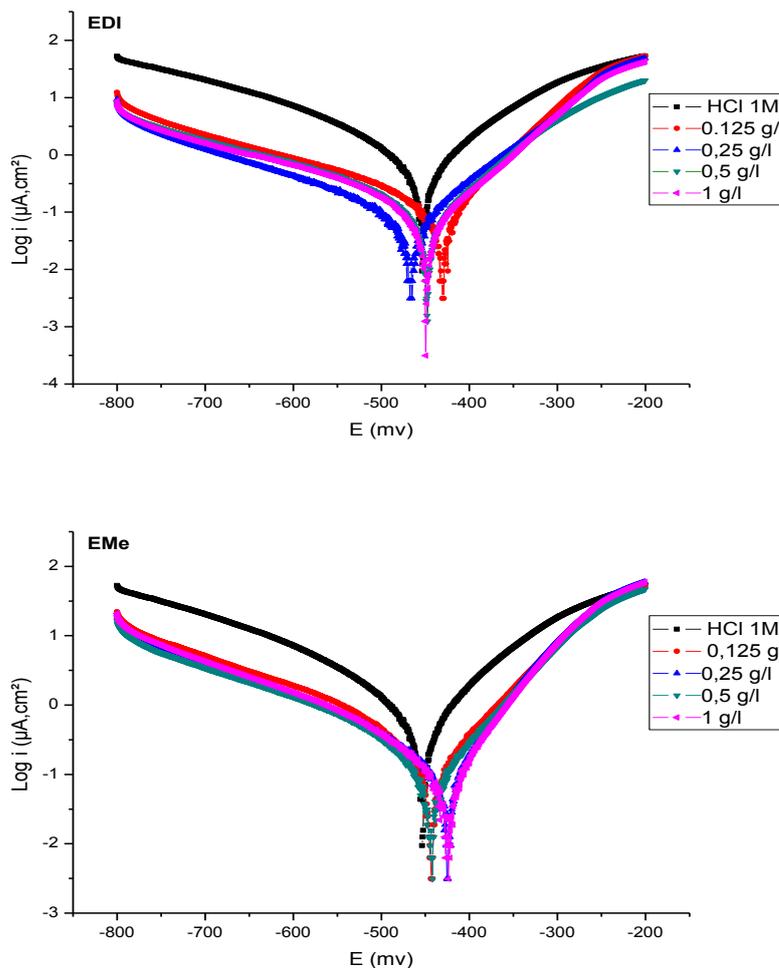
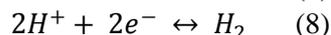
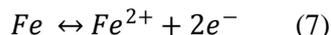


Fig. 6: Polarization curves in presence and absence of extracts at different concentrations in medium 1M HCl

As it can be seen from Fig. 6, the inhibitor, indicating that both **EDi** and **EMe** acts as mixed type inhibitors, affects the anodic and cathodic reactions. The addition of **Edi** and **EMe** to HCl solution, therefore reduces the anodic dissolution of iron and retards the cathodic hydrogen evolution reaction, see Eqs. (7) and (8).



Potentiodynamic polarization curves of carbon steel in 1 M HCl containing *Pancreatium Foetidum Pom* extracts at 308 K are shown in **Fig.6**. The electrochemical corrosion parameters

Inspection of the **fig 6** shows that the addition of **EDi** and **EMe** extracts has an inhibitive effect in the both anodic and cathodic parts of the polarization curves, it shifted the E_{corr} value towards the positive direction compared to the uninhibited mild steel. Thus, addition of this inhibitor reduces the mild steel dissolution as well as retards the hydrogen evolution reaction. The presence of *Pancreatium Foetidum Pom* extracts does not prominently shift the corrosion potential, which indicates the studied *Pancreatium foetidum Pom* extracts inhibitor act as mixed-type inhibitor [13-16]. Furthermore, in the presence of this inhibitor, the slight change of β_c indicates that the cathodic corrosion mechanism of steel does not change.

The **table5** represent different electrochemical parameters including corrosion potential E_{corr} , corrosion current density I_{corr} , cathodic Tafel slope β_c , anodic Tafel slope β_a and the inhibition efficiency $E\%$. The results revealed that **EDi** is the best inhibitor with an efficiency reach until 95, contrary, the **EMe** showed a low efficiency reach until 93%.

Table 5: Corrosion parameters in the presence and absence of inhibitor obtained from polarization measurements

Inhibitor	Concentration (mol l ⁻¹)	E_{corr} (mV/SCE)	I_{corr} (mA/cm ²)	β_c (mV/dec)	E %
Blank	0	-454	1.9477	-182	-
	1	-440	0.0665	-151	97
EDi	0.5	-444	0.1377	-222	92
	0.25	-449	0.1705	-216	91
	0.125	-432	0.1753	-213	90
	1	-465	0.0759	-108	96
EMe	0.5	-426	0.2253	-212	88
	0.25	-444	0.2414	-212	87
	0.125	-444	0.3105	-195	84

The E_{corr} values shifted slightly to the cathodic potential, according to literature If the displacement in E_{corr} is more than ± 85 mV relating to the corrosion potential of the blank, the inhibitor can be considered as a cathodic or anodic type, If the change in E_{corr} is less than ± 85 mV, the corrosion inhibitor may be regarded as a mixed type. [17-18] in our case maximum displacement is 30 mV lower than ± 85 mV indicating that our inhibitors act as mixed-type.

4. Electrochemical impedance spectroscopy :

The electrochemical behavior of *Pancreatium foetidum Pom* was recorded by the electrochemical impedance spectroscopy in the studied acid solutions without and with presence of different concentration of both **EDi** and **EMe** extracts. The experiments has been carried out using a Volta lab (Tacussel- Radiometer PGZ 100) potentiostat and controlled by Tacussel corrosion analysis software model (Voltmaster 4) at under static condition. The corrosion cell used had three electrodes. The reference electrode was a saturated calomel electrode (SCE). A platinum electrode was used as auxiliary electrode of surface area of 1cm². The working electrode was carbon steel. All potentials given in this study were referred to this reference electrode. The working electrode was immersed in test solution for 30 minutes to a establish steady state open circuit potential (E_{ocp}). After measuring the E_{ocp} , the electrochemical measurements were performed. All electrochemical tests have been performed in aerated solutions at 308 K. The EIS experiments were conducted in the frequency range with high limit of 100 kHz and different low limit 10 Hz at open circuit potential, with 10 points per decade, at

the rest potential, after 30 min of acid immersion, by applying 10 mV ac voltage peak-to-peaks. Nyquist plots were made from these experiments. The best semicircle can be fit through the data points in the Nyquist plot using a non-linear least square fit so as to give the intersections with the x -axis [19].

In all cases, the charge transfer resistances are increased with increasing the concentration. Nyquist plots obtained at different concentration (1, 0.5, 0.25, 0.125 g/l) in 1 M HCl solution without and with *Panocratium foetidum Pom* extracts are shown in Fig. 7. These spectra reveal that although values the charge transfer resistances are increased with increasing the concentration, these values are quite similar to those obtained by the weight loss and electrochemical curves measurement. Inspection of data in Table 6 shows clearly that charge transfer resistance R_{ct} and double layer capacitance C_{dl} values have opposite trend at the whole concentration range (R_{ct} increases and C_{dl} decreases with extract concentration).

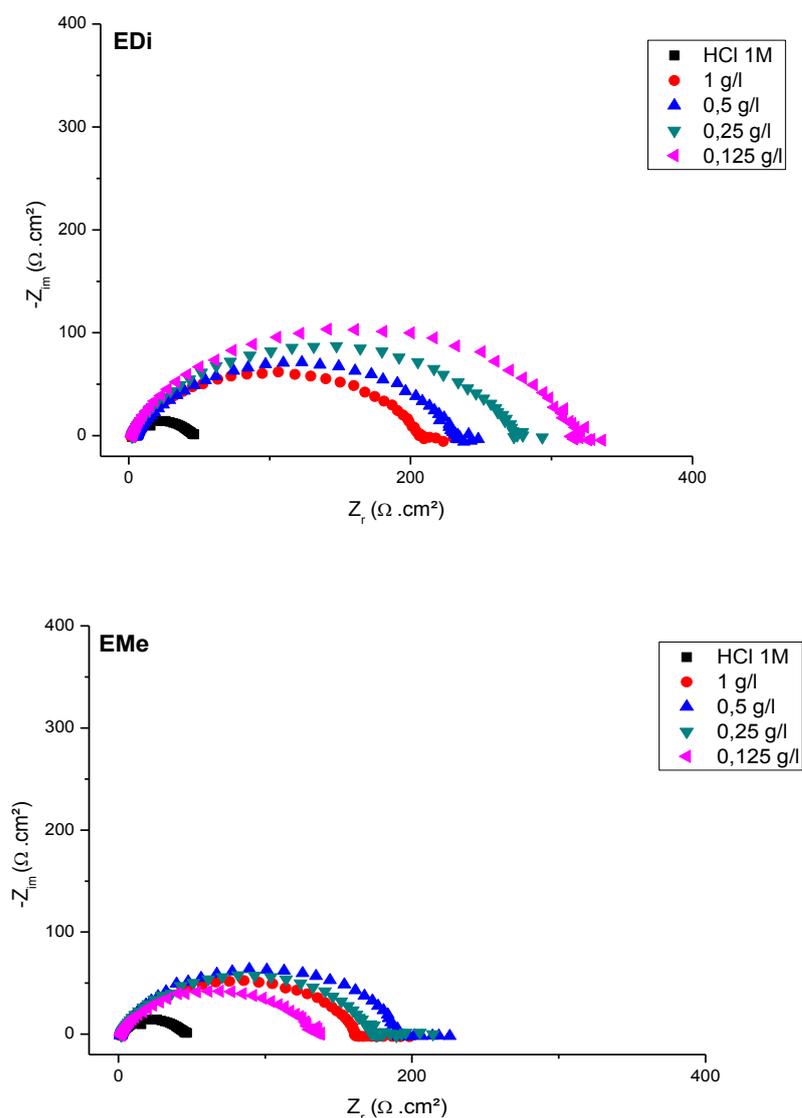


Fig.7: Nyquist plots of **EDi** and **EMe** in absence and presence of different concentrations in

The decrease in this capacity with increase in plant extract concentrations may be attributed to the formation of a protective layer on the electrode surface [30].

The Nyquist plots for carbon steel obtained by adding **EDi** and **EMe** in at 308 K are represented in **fig.7**, the existence of a single semicircle indicating the presence of a single transfer process, also the Nyquist plots characterized by one capacitive loops either in presence or in absence of the inhibitors. The capacitive loops are

not perfect semicircles because of the non-homogeneity and roughness of the carbon steel surface [31]. The increase in the concentration of the inhibitors implies to an increase in the inhibition efficiency values. The double layer capacitance C_{dl} given as in the Helmutz model by:

$$C_{dl} = \frac{\varepsilon^0 \times \varepsilon}{\delta} S \quad (9)$$

Where δ is the thickness of the deposit, S is the surface of the electrode, ε^0 is the permittivity of the air and ε is the medium dielectric constant. The decrease in C_{dl} values could be interpreted either by a decrease of local dielectric constant ε [33] or by the thickness of the adsorbate layer of inhibitor at the metal surface [32].

Table 6. Corrosion parameters obtained by impedance measurements for mild steel in 1M HCl at various concentrations of extracts.

Compounds	Concentrations (g/l)	R_{ct} (ohm.cm ²)	f_{max} (Hz)	C_{dl} (μF/cm ²)	E (%)
Blank	0	43.94	25	$1.48 \cdot 10^{-4}$	-
	1	190.2	20	$4.18 \cdot 10^{-5}$	77
EMe	0.5	172.1	20	$4.62 \cdot 10^{-5}$	73
	0.25	160.7	20	$4.95 \cdot 10^{-5}$	75
	0.125	134	20	$5.94 \cdot 10^{-5}$	67
	1	319.3	15.82	$3.15 \cdot 10^{-5}$	86
EDi	0.5	275.7	15.82	$3.65 \cdot 10^{-5}$	84
	0.25	227.3	15.82	$4.42 \cdot 10^{-5}$	80
	0.125	206.1	15.82	$4.88 \cdot 10^{-5}$	78

The decrease in C_{dl} values with the addition of inhibitor is due to the adsorption of the inhibitor molecules replacing water at the metal–solution interface that led to the decrease in local dielectric constant and/or an increase in the thickness of the electrical double layer [34,35].

Conclusion

We can summarize our study by the following points:

- *Pancretium foetidum Pom* Extracts had a considerable inhibition effect on carbon steel corrosion in 1 M HCl solution. **EDi** full of alkaloids is better than **EMe** as corrosion inhibitors
- The polarization plots indicated that the inhibitor inhibits both anodic metal dissolution and cathodic hydrogen evolution reaction and act as mixed type inhibitors. Impedance measurements indicate that with increasing inhibitor concentration,
- The transfer charge resistance (R_t) and the double layer capacitance (C_{dl}) values have opposite trend at the whole concentration range (R_{ct} increases and C_{dl} decreases with extracts concentration)
- The adsorption by the Langmuir isotherm is the best fitting between the coverage surface and concentration we conclude that the carbon steel surface obeys the Langmuir adsorption isotherm. The thermodynamic activation parameters were calculated and discussed.
- Using the three famous method weight loss – Electrochemical curves – Electrochemical impedance showed a good agreement.

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