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RESEARCH

A comparative study on the inhibitive effect of *Crataegus oxyacantha* and *Prunus avium* plant leaf extracts on the corrosion of mild steel in hydrochloric acid solution

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Abstract

The inhibitive action of plant leaf extracts, *Crataegus oxyacantha* (*Hawthorn*) and *Prunus Avium* (*Sweet Cherry*) *on* the corrosion of mild steel in 0.5 M HCl solution was investigated using open circuit potential-time measurements (OCP), potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques. Functional groups of these plants' leaf extracts and their absorption bands were identified by Fourier transform infrared spectroscopy (FTIR) and Ultra-Violet Spectrophotometer (UV), respectively. The leaf extracts showed good inhibition efficiency in hydrochloric acid solution. Potentiodynamic polarization curves revealed that *Crataegus oxyacantha* and *Purnus Avium* plants leaves extracts acted as mixed type inhibitors. Theoretical fitting of different isotherms, Langmuir, Florry–Huggins and the kinetic–thermodynamic models was tested to describe the mode of inhibitors' adsorption on mild steel surface. UV spectra proved that the inhibiting action takes place through simple physical adsorption of the extracts molecules on mild steel surface.

Keywords Mild steel · Electrochemical techniques · Leaf extracts · Hydrochloric acid · Corrosion inhibition

Introduction

Mild steel has been extensively used in several applications including constructions of tanks, petroleum refineries equipment and flow lines as well as transmission pipelines due to its ease of fabrication and low cost [1–6]. However, it is vastly exposed to corrosion and deterioration especially in acidic media. In metallurgical industry, hydrochloric acid is widely applied in various processes such as pickling and descaling of metals, chemical or electrochemical processes in oil refinery as well as deactivation of equipment in atomic power establishments'. Various corrosion controlling methods were used to protect metals such as protective coatings, cathodic protection and the use of corrosion inhibitors [7]. Among these methods, the latter is one of the most practical methods especially in acidic media. Such inhibitors can significantly decrease the corrosion rate when added to a corrosive environment in small concentrations [8–10].

Most of the organic compounds containing nitrogen, oxygen and phosphorus atoms are expected to act as effective corrosion inhibitors of different metals and alloys [11]. Unfortunately, nowadays the use of such compounds is restricted due to their high cost and toxicity for both human and environment. Recently, efforts are directed towards the use of plant extracts as corrosion inhibitors. Such extracts consist of diverse natural ingredients that are eco-friendly, easily available and of low cost [2].

Numerous researchers reported the successful use of natural plant extracts on the corrosion inhibition of mild steel in different media [12–23]. Most of these investigated plant extracts exhibit a moderate to high inhibition efficiency in the range 55–90% in acidic media.

Crataegus oxyacantha (Hawthorn) also known as maybush, or whitehorn, is part of a genus of spiny shrubs and trees native to temperate regions in the Northern Hemisphere in Europe, Asia, and North America. It belongs to the Rosaceae family and consists of bright green leaves, white flowers, and bright red berries. Flavonoids such as vitexin, hyperoside, rutin, or vitexin-2″-*O*-α-L-rhamnoside, and catechin/

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epicatechin derived oligomeric procyanidins are the most important constituent of Hawthorn extract [24].

Prunus Avium (sweet cherry) is geographically distributed around the world, with greater prevalence in areas with a temperate climate, which encompasses much of Europe (Mediterranean and Central), north Africa, Near and Far East, South Australia and New Zealand, and temperate zones of the American continent (USA and Canada, Argentina and Chile) [25]. Sweet cherries have been reported to contain various phenolics and anthocyanins which contribute to total antioxidant activity [26].

This work aims to explore the influence of *Crataegus oxyacantha* and *Prunus Avium*, leaf extracts on the corrosion of mild steel in hydrochloric acid using electrochemical techniques.

Experimental studies

Solution preparation

0.5 M HCl solutions were prepared by dilution of 37% concentrated grade acid, from Scharlau chemical industries using distilled water.

Extraction procedure

Crataegus oxyacantha and *Prunus Avium*s stock solutions were obtained by drying the plant leaves for 2 h in an oven at 80 °C and grinding to powdery form. A 10 g sample of the powder was refluxed in 100 mL distilled water for 1 h. The refluxed solutions were filtered to remove any contamination. The concentrations of the stock solutions were determined by evaporating 10 mL of the filtrates and weighing the residues. Prior to each experiment, an appropriate volume of 4 M HCl is added to an appropriate volume of the stock solution of plant leaf extract and double distilled water to obtain a solution of 0.5 M HCl solutions and the required concentration of the extract.

Electrochemical studies

Electrochemical impedance (EIS) and polarization measurements were done using frequency response analyzer (FRA)/potentiostat supplied from ACM instruments (UK). The frequency range for electrochemical impedance spectroscopy (EIS) measurements was 0.1 to 96 × 10^3 Hz with applied potential signal amplitude of 10 mV around the rest potential. The data were obtained in an electrochemical cell of three-electrode mode; platinum wire and saturated calomel electrodes (SCE) were used as counter and reference electrodes. The mild steel used for constructing the working electrode was of the following chemical composition (wt%) (C: 0.164, Mn: 0.710, Si: 0.260, S: 0.001, P: 0.005 and Fe: 96.2). The steel plate of cylindrical shape was encapsulated in Teflon in such a way that only one surface was left uncovered. The exposed area (0.7853 cm^2) was mechanically abraded with a series of emery papers of variable grades, starting with a coarse one and proceeding in steps to the finest (800) grade. Before polarization and EIS measurements, the working electrode was left for 20 min to attain the open circuit potential in the used solution. Linear polarization measurements (LPR) were carried out at a sweep rate of 10 mV min^{-1} within a potential range of ± 10 mV from the rest potential. Polarization curve measurements were obtained at a scan rate of 30 mV/min starting from cathodic potential (E_{corr} = − 250 mV) going to anodic direction (E_{corr} = + 250 mV). All the measurements were done at 30 ± 0.1 °C using WiseCircu water bath (Germany) in solutions open to the atmosphere under unstirred conditions.

To obtain the activation parameters, the measurements were carried out at 30–60 °C. To test the reliability and reproducibility of the measurements, duplicate experiments were performed, under the same conditions, in each case and found to be within 2% error.

Ultra-violet spectroscopy (UV) and FTIR analysis

FTIR analysis of the plant extracts was carried by FTIR 8400S Shimadzu in the spectral region between 4000 and 400 cm^{-1}. The optical studies were measured using the ultra-violet-visibleV-670 that measures the absorption spectra at a wavelength of 800–200 nm at room temperature.

Results and discussion

Open circuit potential measurements (OCP)

Figure 1 reveals that the OCP of mild steel in 0.5 M HCl solutions in the absence and presence of 0.4 g L^{-1} leaf extracts is attained after 20 min of immersion. It is clearly observed that the variation of OCP after 20 min is within 2 mV min^{-1}, indicating that the mild steel electrode reached its equilibrium state at this time. The OCP of mild steel electrode was shifted towards less negative values in the presence of the leaf extracts. Such positive shift of the corrosion potential indicates the influence of these extracts on the anodic process [27].

Potentiodynamic polarization data measurements

The potentiodynamic polarization curves of mild steel shown in Fig. 2 indicate that the addition of leaf extracts

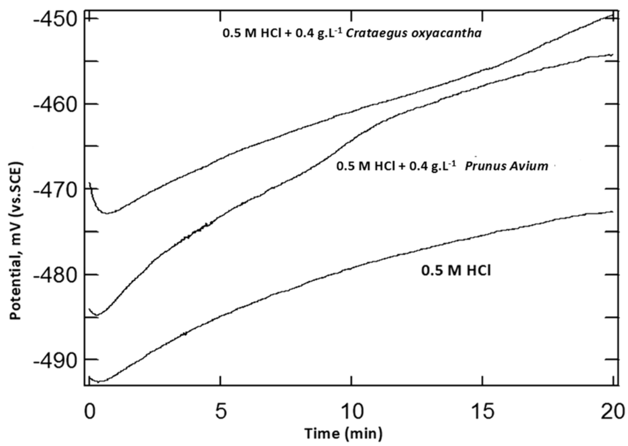

Fig. 1 The variation of open circuit potential as a function of time for mild steel in 0.5 M HCl solution in the absence and presence of *Crataegus oxyacantha* and *Prunus Avium* leaf extracts at 30 °C

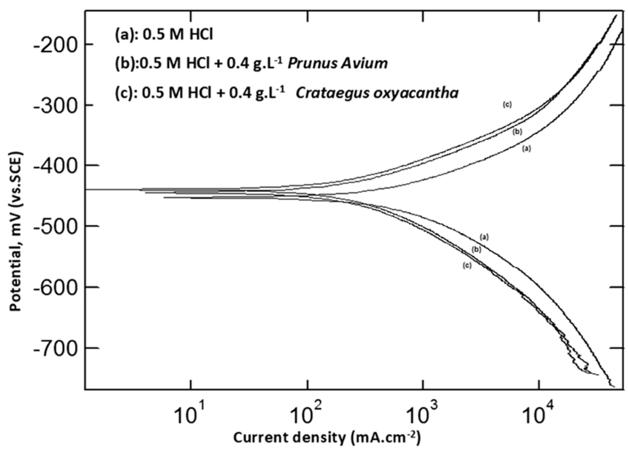

Fig. 2 Potentiodynamic polarization curves for mild steel in 0.5 M HCl in the absence and presence of 0.4 g L^{-1} *Crataegus oxyacantha* and *Prunus Avium* leaf extracts at 30 °C

suppresses both anodic metal dissolution and the cathodic hydrogen evolution reactions indicating that they could be classified as mixed type inhibitors.

The electrochemical parameters including the corrosion current density (i_{corr}) that is obtained from the intersection of the extrapolation of anodic and cathodic Tafel lines together with percentage of inhibition efficiency (%P) are given in Table 1. The %P was calculated from polarization measurements using the relation: $\%P=[(i_0 - i)/i_0]\times 100$

$$\%P = \left[\left(i_0 - i\right)/i_0\right] \times 100$$

where i_0 and i are the corrosion current densities in the absence and the presence of plant leaf extracts, respectively.

The displayed data showed that i_{corr} decreases with increasing *Crataegus oxyacantha* and *Prunus Avium* leaf extracts concentrations accompanied with an increase in %P. The slight variations in anodic and cathodic Tafel slopes, β_a and β_c, in the presence of these extracts indicate that the inhibiting action is taking place the simple blocking of existing cathodic and anodic sites on the metal surface [28]. The studied leaf extracts could be classified as pickling type inhibitors since they approximately have no effect on the corrosion potential (E_{corr}) [29].

Electrochemical impedance spectroscopy results

The Nyquist plots shown in Fig. 3 consist of depressed semicircles of capacitive type signifying that the dissolution process of mild steel occurs under activation control [30]. The depressed capacitive loop is ascribed to dispersion effects, which have been attributed to roughness and inhomogeneities on the surface during corrosion [28, 31].

The obtained Nyquist impedance plots were examined by fitting the experimental data to a simple equivalent circuit model, Fig. 4, which includes the solution resistance R_s and the constant phase element (CPE) together with the charge transfer resistance element R_{ct}. The R_{ct} value is a measure of electron transfer across the surface and is inversely proportional to corrosion rate.

Table 1 The electrochemical polarization parameters for the corrosion of mild steel in 0.5 M HCl containing different concentrations of *Crataegus oxyacantha* and *Prunus Avium* leaf extracts, respectively, at 30 °C

Plant leaf extract	Conc (g L^{-1})	$-E_{corr}$ (mV vs. SCE)	β_a	β_c	i_{corr} (mA cm^{-2})	%P
			mV/decade			
Crataegus oxycantha	Blank	450	91	124	0.721	–
	0.1	445	89	113	0.478	34
	0.2	435	78	109	0.308	57
	0.3	440	74	105	0.274	62
	0.4	435	76	108	0.247	66
Prunus avium	0.1	450	78	104	0.416	42
	0.2	445	87	117	0.3443	52
	0.3	446	85	125	0.301	58
	0.4	444	81	110	0.285	61

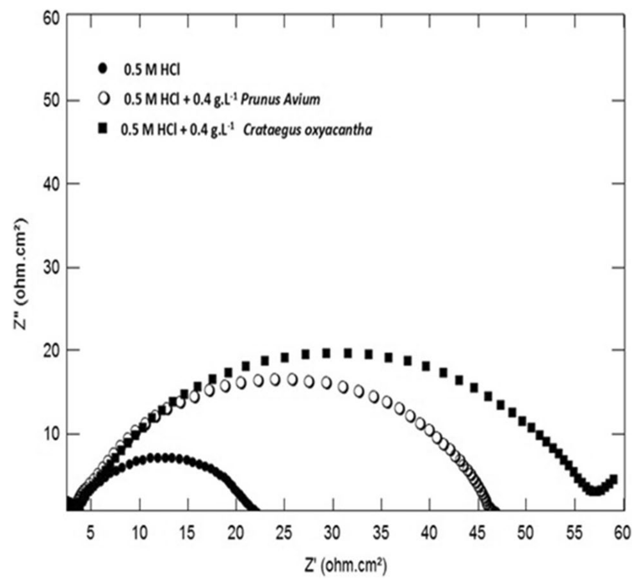

Fig. 3 Nyquist impedance plots for mild steel in 0.5 M HCl in the absence and presence of 0.4 g L^{-1} *Crataegus oxyacantha* and *Prunus Avium* leaf extracts at 30 °C

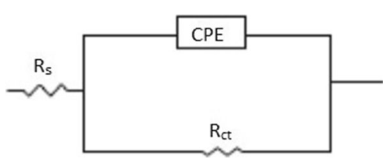

Fig. 4 Schematic for the equivalent circuit model

To compensate for non-homogeneity in the system, the capacitances were implemented as a constant phase element (CPE) that is defined by two values, the non-ideal double layer capacitance and n.

The impedance, Z, of CPE is presented by

$$Z_{CPE} = Q^{-1}(i\omega)^{-n}$$

where $i=(-1)^{1/2}$, ω is the frequency in rad s^{-1}, $\omega=2\Pi f$, and f is the frequency in Hz. If n equals 1, the value of Q present in the above equation is identical to that of ideal capacitor C. Then, the $Z_{CPE}=(i\omega C)^{-1}$. In this case, the Q that is equal to C has units of capacitance, i.e., μF/cm^2, and represents the capacity of the interface. However, for a non-homogeneous system, where n values are different from 1, Q is equal to the CPE admittance (Y_0) and has units of $\mu s^n/\Omega\ cm^2$. In this case, the system shows behavior that has been attributed to surface heterogeneity or to continuously distributed time constants for charge transfer reactions [32–35]. The double layer capacitance (C_{dl}) could be calculated using the following equation [36]:

$$C_{dl}=\frac{(Y_o \times R_{ct})^{1/n}}{R_{ct}}.$$

The fitting of the spectrum to the equivalent circuit model permits the evolution of the elements of the circuit analog. The experimental and computer fitting results of the Nyquist plot of 0.3 g L^{-1} *Crataegus oxyacantha* in 0.5 M HCl at 30 °C are demonstrated in Fig. 5.

The percentage inhibition efficiency (%P) can be obtained from impedance measurements according to the equation:

$$\%P = \left[\left(R_{ct} - R_{ct0}\right)\right]/R_{ct} \times 100$$

where R_{ct0} and R_{ct} are the values of the charge transfer resistance ($\Omega\ cm^2$) in the absence and the presence of leaf extracts, respectively.

The values of electrochemical impedance parameters obtained from fitting the experimental data to the used equivalent model and the values of %P are presented in Table 2. The data indicate that increasing plant leaf extracts concentrations increases the charge transfer resistance and a decrease in the ideal double layer capacitance C_{dl} values. Such reduction is due to the increase in the thickness of the electrical double layer suggesting that the leaf extracts' molecules act by adsorption at the metal/solution interface [31, 37].

Linear polarization resistance (LPR)

The inhibition efficiency (%P) was calculated from polarization resistance (R_p) values obtained from LPR measurements using the following equation:

$$\%P = \left[\left(R_p - R_{p0}\right)\right]/R_p \times 100$$

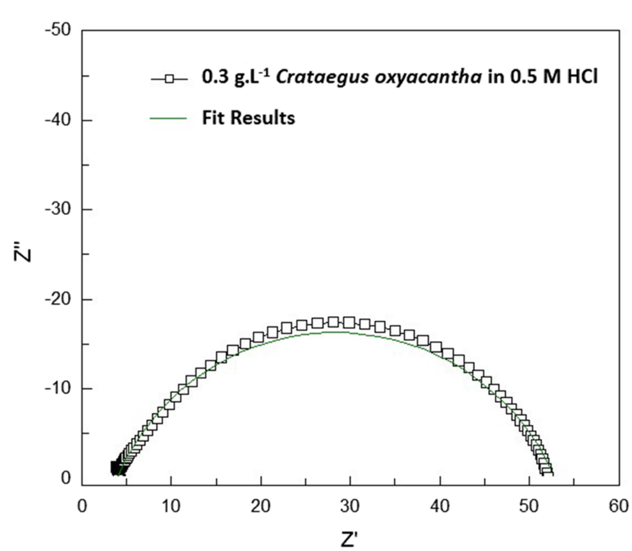

Fig. 5 The experimental and computer fitting results of Nyquist plot of 0.3 g L^{-1} *Crataegus oxyacantha* in 0.5 M HCl at 30 °C

Table 2 The electrochemical for the corrosion of mild steel in 0.5 M HCl containing different concentrations of *Crataegus oxyacantha* and *Prunus Avium* leaf extracts, respectively, at 30 °C

Plant leaf extract	Conc. (g L^{-1})	*R*s (Ω cm^2)	R_{ct} (Ω cm^2)	C_{dl} (μF/cm^2)	Y_o (μs^n/Ωcm^2)	*n*	%*P*
Crataegus oxycantha	Blank	2.40	19	530	92	0.81	–
	0.1	2.98	30	430	78	0.82	36
	0.2	2.90	40	359	77	0.84	52
	0.3	3.22	48	283	46	0.81	60
	0.4	3.24	54	349	65	0.83	65
Prunus avium	0.1	3.33	27	331	70	0.83	30
	0.2	2.99	36	293	88	0.87	46
	0.3	3.81	38	285	49	0.81	49
	0.4	3.63	43	270	50	0.82	55

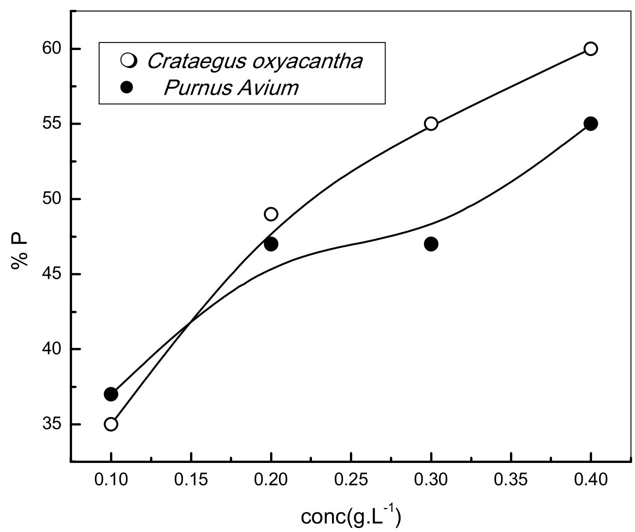

Fig. 6 Variation of %*P* obtained from R_p values as function of different concentrations *of Crataegus oxyacanth a*and *Prunus Avium* leaf extracts in 0.5 M HCl at 30 °C

where R_{p0} and R_p are the values of the polarization resistance (Ω cm^2) in the absence and the presence of leaf extracts, respectively.

Figure 6 shows the variation of %*P* obtained from R_p values as function of concentration of *Crataegus oxyacantha* and *Prunus Avium* leaf extracts in 0.5 M HCl at 30 °C. It is clearly observed that as the concentration of *Crataegus oxyacantha* and *Prunus Avium* leaf extracts increases, the values of %*P* increase up to 55–60%.

The values of %*P* are in quite good agreement with the results obtained previously from potentiodynamic polarization curves and impedance measurements

Adsorption isotherms

To discuss adsorption isotherms, the degrees of surface coverage values were obtained from AC impedance measurements using equation ($\theta = \%P/100$). Theoretical fitting of different isotherms, Langmuir, thermodynamic-kinetic model and Florry–Huggins isotherm was tested to describe the mode of inhibitors' adsorption on mild steel surface.

Langmuir isotherm is given by [38]

$$[\theta/(1-\theta)] = K[C]$$

where *K* is the equilibrium constant of the adsorption process, *C* is the inhibitor's concentration and the Florry–Huggins isotherm [39] which is given by

$$K \cdot C = [\theta/(1-\theta)^x]\exp(1-x)$$

"*x*" is the size parameter and is a measure of the number of adsorbed water molecules substituted by a given inhibitor molecule.

The kinetic-thermodynamic model [40] is given by

$$\log[\theta/(1-\theta)] = \log K' + y\log C$$

where "*y*" is the number of inhibitor molecules occupying one active site; in other words, "1/*y*" is the number of surface active sites occupied by one inhibitor molecule. The binding constant K is given by

$$K = K'^{(1/y)}$$

Figure 7a–c shows the application of the above-mentioned models to the results of adsorption of the used extracts on mild steel surface in 0.5 M HCl. The parameters obtained from the fitting these isotherms are depicted in Table 3.

It was found that the experimental data fitted all the applied adsorption isotherms except Langmuir. Such observation indicates the non-ideal behavior of these plant leaf extracts. The number of active sites occupied by a single inhibitor molecule, 1/y, is greater than unity indicating that each molecule of the leaf extracts was adsorbed onto more than one active site. Thus, the adsorbed molecules are bulky [41].

Moreover, it is known that the inhibition efficiency is a function of the value of inhibitor's binding constant K_{ads}, which represents the strength between adsorbed species and metal surface. The large values of K clarify stronger interaction, whereas small values of K signify that the interaction is

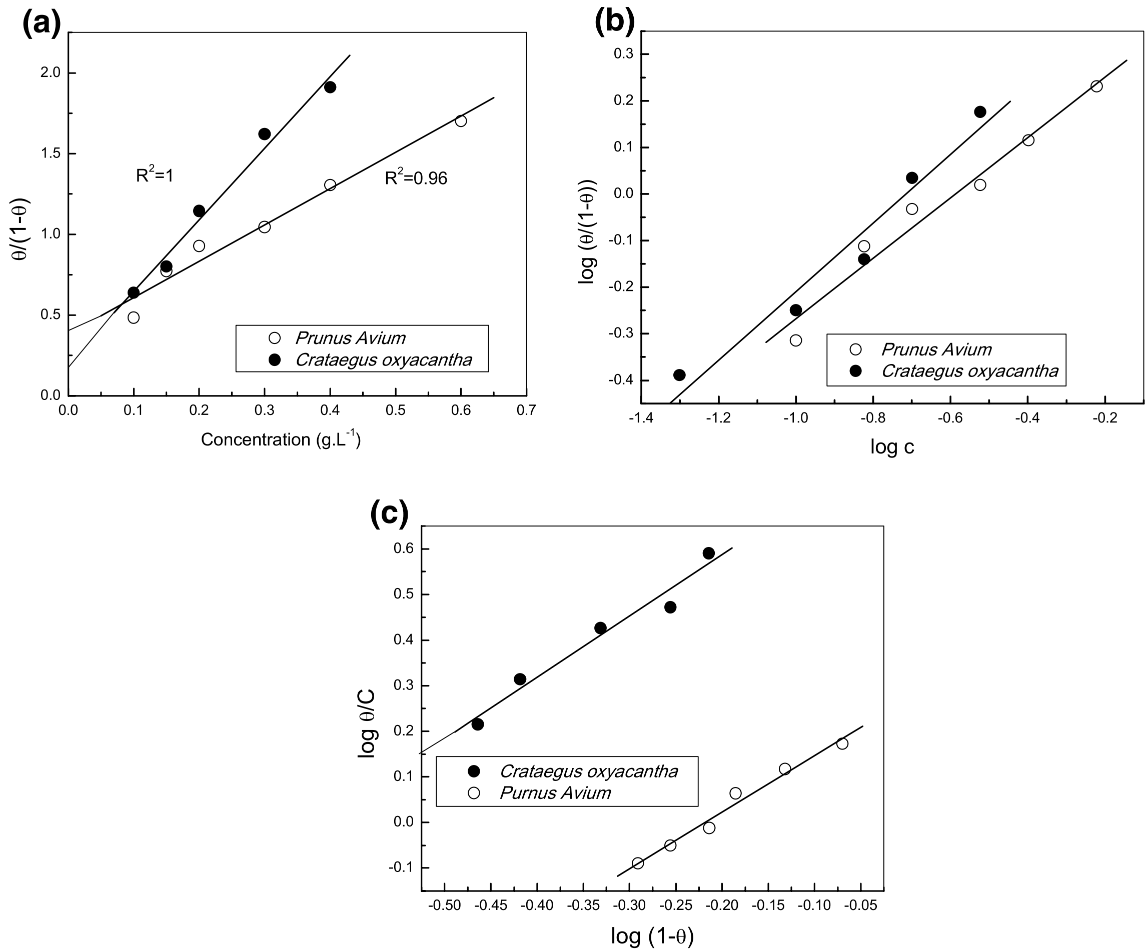

Fig. 7 **a** Application of Langmuir adsorption isotherm to the results of adsorption of *Crataegus oxyacantha* and *Prunus Avium* on mild steel surface in 0.5 M HCl. **b** Application of Kinetic–thermodynamic model to the results of adsorption of *Crataegus oxyacantha* and *Prunus Avium* on mild steel surface in 0.5 M HCl. **c** Application of Florry–Huggins model to the results of adsorption of *Crataegus oxyacantha* and *Prunus Avium* on mild steel surface in 0.5 M HCl

Table 3 Linear fitting parameters of *Crataegus oxyacantha* and *Prunus Avium* leaf extracts according to all isotherms in 0.5 M HCl at 30 °C

Inhibitor	Kinetic model			Florry–Huggins		
	K	1/*y*	R^2	*K*	*x*	R^2
Crataegus oxycantha	5.26	1.37	0.94	5.00	1.55	0.95
Prunus avium	3.81	1.54	0.96	3.99	2.00	1

weaker [17]. Hence, according to the numerical values of K obtained from the three models, the inhibition efficiency of *Crataegus oxyacantha* is better than *Prunus Avium.*

However, the equilibrium constant *K* is related to the standard free energy of adsorption (ΔG_{ads}) according to the equation:

$$K_{ads} = 1/55.5e^{(-\Delta Gads/RT)}$$

where *K* is the binding constant obtained from kinetic–thermodynamic model, *R* is the molar gas constant, *T* is the absolute temperature in Kelvin and 55.5 is the concentration of water in solution expressed in molar [26].

The calculated ΔG_{ads} values from kinetic–thermodynamic model were − 36 and − 31 kJ.mol^{-1} for Crataegus *Oxycantha* and *Prunus Avium* in 0.5 M HCl solutions, respectively. Such values are an indication of the spontaneity of the adsorption process of both leaf extracts, and the stability of the adsorbed layers on the mild steel surface in 0.5 M HCl solution. The obtained ΔG_{ads} values reveal that the adsorption takes place through physisorption mechanism [26].

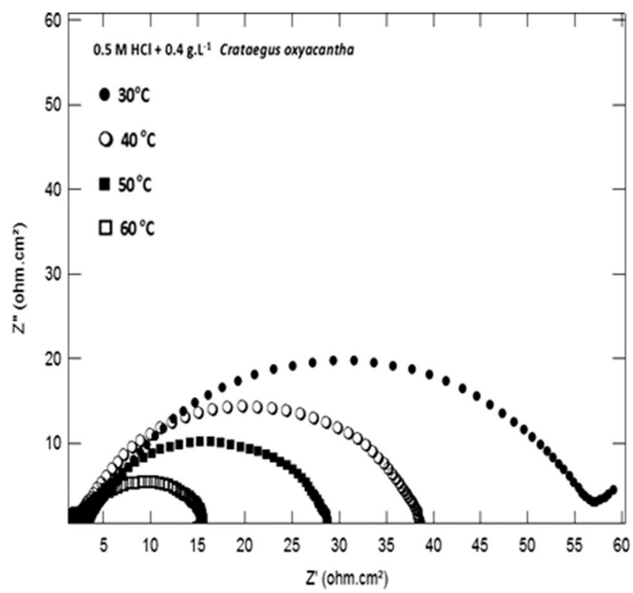

Fig. 8 Nyquist Impedance plots for mild steel in 0.5 M HCl in the presence of 0.4 g L^{-1} *Crataegus oxyacanth*a leaf extract at different temperatures

Activation Parameters

Figure 8 represents the Nyquist Impedance plots for mild steel in 0.5 M HCl in the presence of 0.4 g L^{-1} *Crataegus oxyacantha* leaf extract at different temperatures. As seen, increasing the temperature decreases the size of the depressed semicircles indicating a decrease in the charge transfer resistance (R_{ct}) and, thus, an increase in the corrosion rate. Such behavior confirms the desorption of plant extract molecules from the metal surface at elevated temperatures.

The activation parameters for mild steel in 0.5 M HCl in the absence and presence of 0.4 g L^{-1} plant extracts were obtained from the linear square fitting of ln(υ) and ln (υ/T) data vs. ($1/T$), by applying Arrhenius and transition state equations [25].

The corrosion rates (υ) were taken as the reciprocals of the charge transfer resistance (R_{ct}) which were obtained from the Nyquist plots of different temperatures. The apparent activation energy, E_a, activation entropies, ΔS^* and activation enthalpies, ΔH^*, in the absence and presence of plant extracts are depicted in Table 4.

Table 4 revealed that E_a and ΔH^* values increase in the presence of both plant leaf extracts, indicating a higher protection efficiency [29]. The positive values of ΔH^* indicate that the formation of the activated complex is endothermic process. However, the negative value of ΔS^* implies that the activated complex represents an association rather than a dissociation step. This means that a decrease in disordering takes place on going from reactants to the activated complex [26, 42].

Table 4 The thermodynamic parameters of mild steel in 0.5 M HCl in the absence and presence of 0.4 g L^{-1} *Crataegus oxyacantha* and *Prunus Avium* leaf extracts

Plant extract	E_a (kJ mol^{-1})	ΔH^* (kJ mol^{-1})	ΔS^* (J mol^{-1} K^{-1})
–	31	28	− 179
Crataegus oxycantha	36	33	− 167
Prunus avium	31	28	− 183

Spectrophotometric and FTIR analysis

Several studies used FTIR analysis as a tool to determine functional groups present in any extract [11, 43, 44]. Figure 9a, b shows the FTIR spectra of Crataegus *oxyacantha* and *Prunus Avium* plant leaves' extracts. IR spectrum for *Crataegus oxyacantha* showed absorption bands for C–H stretching vibrations, generally in the range of 2923–2800 cm^{-1}; a broad –OH group (3420 cm^{-1}); and a C=C vibration for aromatics (1653.83–1463.83 cm^{-1}). Likewise bands were obtained for *Prunus Avium* leaf extract in addition to C=O at 1739 cm^{-1} that is attributed to esters. By matching these spectra with the literature in order to determine the active chemical ingredients for these plant leaves extracts under study [45, 46], it was found that catechin may be the major active chemical ingredient in *Crataegus oxyacantha* leaf extract, whereas methylvanillate in *Prunus Avium* extract.

Mechanism of inhibition

It is clearly observed from the UV spectra presented in Fig. 10 that there is no shifting in the absorption bands of plant extract (PE) in the presence of 10^{-3} M $FeSO_4$. Such observation is quite an indication of the absence of $[Fe\text{-}PE]^{2+}$complex and that the inhibition takes place through simple physical adsorption of extract molecules on the surface according to the following equation:

$$HCl \rightarrow H^+ + Cl^-$$

$$PE + H^+ \rightarrow HPE^+$$

$$Fe + Cl^- \rightarrow (FeCl_{ads})^-$$

$$HPE^+ + (FeCl_{ads})^- \rightarrow (FeCl_{ads})HPE$$

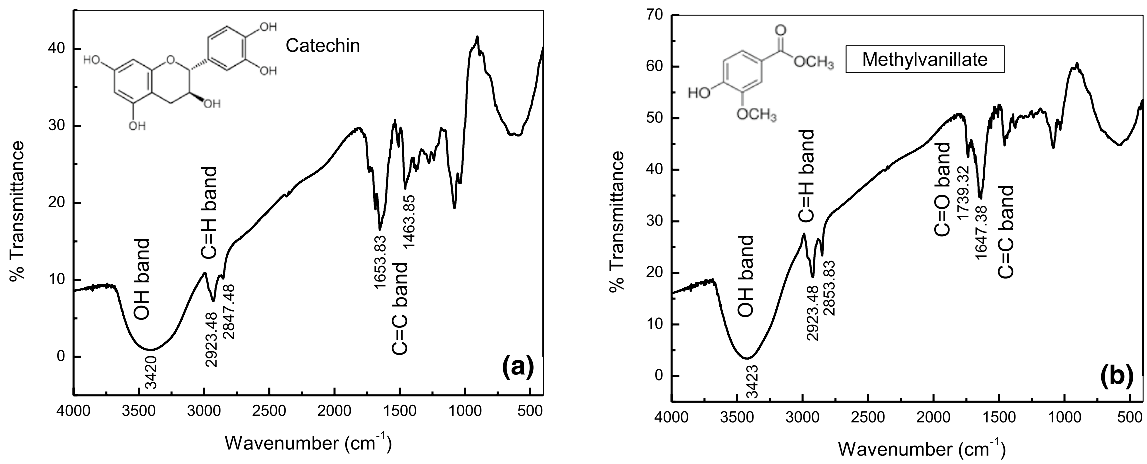

Fig. 9 **a** FTIR spectra of *Crataegus oxyacantha* plant leaf extract. **b** FTIR spectra of *Prunus Avium* plant leaf extract

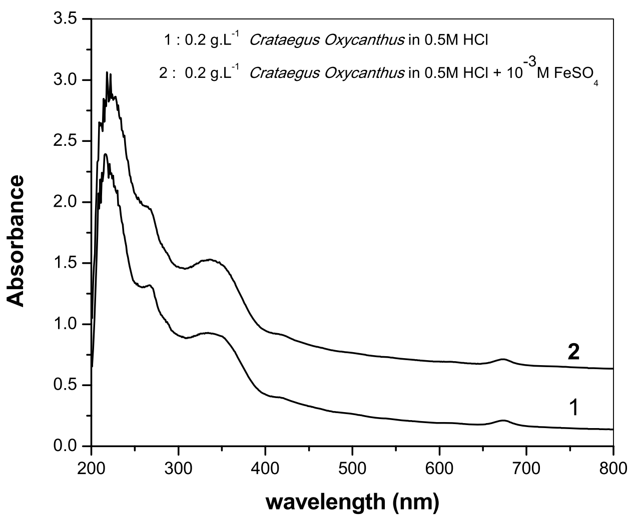

Fig. 10 UV absorption spectra obtained of 0.2 g L^{-1} *Crataegus oxyacantham* extract in 0.5 M HCl in the absence and presence of 10^{-3} M $FeSO_4$

Conclusion

Crataegus oxyacantha and *Prunus Avium* leaf extracts acted as good corrosion inhibitors for mild steel in 0.5 M HCl solution. An excellent agreement between the inhibition efficiencies calculated using different electrochemical techniques was obtained. Such inhibition of these plant leaf extracts depends on the physical adsorption of the chemical constituents of the extract on mild steel surface rather than forming a complex with Fe^{2+} ions.

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