

INVESTIGATION OF CORROSION-RESISTANT PROPERTIES OF CITRUS AURANTIUM ESSENTIAL OIL IN 1 M HCl

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The fresh peel essential oils of citrus aurantium from Eastern Morocco (Oujda) have been analyzed by gas chromatography and gas chromatography–mass spectrometry. The analyzed essential oil consist mainly of monoterpene hydrocarbons (54.38%), with limonene (52.67%) being the major constituent. The effect of addition of citrus aurantium essential oil on the corrosion of steel in 1 M HCl acid has been studied by weight loss measurements, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) measurements. The inhibition efficiency has been found to increase with inhibitor content to attain 95% (at 5 g/l). Data obtained from EIS studies have been analyzed to determinate the model inhibition process through appropriate equivalent circuit models. Inhibition efficiency obtained by the various methods is in good agreement. The adsorption of natural products on the steel surface has been found to obey the Langmuir's adsorption isotherm.

Keywords: mild steel, citrus aurantium, oil, HCl, corrosion, green inhibitor, gas chromatography–mass spectrometry.

Corrosion of materials is a natural phenomenon that causes preoccupations particularly for the industry, because it causes a total loss of millions of dollars in many industries [1]. The addition of organic inhibitors is one of the methods used to reduce a metal corrosion rate [2–4]. Such materials usually contain polar functionality with nitrogen, oxygen and/or sulfur in a conjugated system [5–8]. These functions cause adsorption on the interface of the metal, according to their nature and their charge [9–11]. However, in the past two decades, studies are directed towards the application of non toxic inhibitors called ecological or green inhibitors. The natural plants from extracts, oils or pure compounds can play a major role in protecting the environment. Green inhibitors are biodegradable and are not toxic [12–15]. The successful use of natural substances to inhibit corrosion of metals in acid and alkaline environment has been of much interest for some scientists [16–18].

In the present work, the study of the effectiveness of the essential oil extracted from the fresh peel of the sour orange as a corrosion inhibitor for steel in molar HCl medium is reported. Generally, sour orange fruits or scientifically called Citrus aurantium (family's Citrus is Rutaceae) [19] are used as desserts, in juice production and jim. The food processing and agro industrial food production give significant quantities of waste or by-products such as peels, seeds and pulp which represent 50% of gross processing valuable fruit. These by-products are considered as an able valuable source of functional ingredients such as flavonoid, fiber and essential oils [20]. Indeed, the essential oil is consi-

dered as the product of the largest citrus aurantium, often obtained from the peels [21–24]. The content of essential oil of Tunisian citrus aurantium peel is 1.24%. The qualitative and quantitative analysis allowed the identification of 10 components in the essential oil, mainly monoterpenes hydrocarbons 99.1%, with 96.86% limonene and β -pinene 1.37% being the main constituents [25]. However, essential oil of Tunisian fresh citrus aurantium peel analyzed by gas chromatography (GC) and gas chromatography – mass spectrometry (GC–MS) identified 27 constituents which the 90.25% limonene and 1.56% linalool with the major compounds [26]. The purpose of this paper is to examine the corrosion inhibition effect of citrus aurantium essential oil for mild steel in 1 M HCl solution by gravimetric method and electrochemical techniques such as linear polarization and impedance spectroscopy (EIS).

Methods and materials. Preparing sample and essential oil extraction. Citrus aurantium is collected from oranges located in the Seast of Morocco, Oujda precisely in December 2013. The fruits are washed and peeled. The fresh peels of citrus aurantium were submitted 4 h hydrodistillation. The essential oil was dried over anhydrous sodium sulphate and then stored in sealed glass vials at 4 to 5°C prior to analysis.

GC and GC–SM analysis. The essential oil was analyzed using gas chromatograph – mass spectrometer (Shimadzu QP 2010), the gas chromatography coupled to mass spectrometry. This plant was extracted by the hydrodistillation method to obtain the essential oil, which was subsequently used to study the corrosion inhibition properties of mild steel in polluted hydrochloric acid solution.

Solutions. The aggressive solutions of 1 M HCl were prepared by dilution an analytical grade 37% HCl with double distilled water. The concentration range of green inhibitor employed was 0.1...5.0 g/l.

Weight loss measurements. Coupons were cut into 1.5×1.5×0.05 cm³ specimens having composition (%): (0.09 P; 0.01 Al; 0.38 Si; 0.05 Mn; 0.21 C; 0.05 S and Fe balance) used for weight loss measurements. Prior to all measurements, the exposed area was mechanically abraded [with 180; 400; 800; 1000; 1200 grades] of emery papers. The specimens were washed thoroughly with bidistilled water, degreased and dried with ethanol. Gravimetric measurements were carried out in a double wall glass cell equipped with a thermostat cooling condenser. The solution volume is 100 cm³. The immersion time for the weight loss was 6 h at (308±1) K. In order to get a good reproducibility, the experiments were carried out in a duplicate. The average weight loss was obtained. The corrosion rate (w_{corr}) was calculated using the following equation:

$$w_{\text{corr}} = \frac{\Delta m}{St}, \quad (1)$$

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

$$E_w = \frac{w_{\text{corr}} - w_{\text{inh}}}{w_{\text{corr}}} \cdot 100\%, \quad (2)$$

where w_{inh} and w_{corr} are the values of corrosion rate with and without inhibitor, respectively.

Electrochemical tests. The electrochemical study was carried out using a potentiostat PGZ100 piloted by Voltmaster soft-ware. This potentiostat was connected to a cell with three electrode thermostats with a double wall. A saturated calomel electrode (SCE) and platinum electrode were used as the reference and auxiliary electrodes, respectively. Anodic and cathodic potentiodynamic polarization curves were plotted at a polarization scan rate 0.5 mV/s. Before all experiments, the potential was stabilized at free potential during 30 min. The polarisation curves were obtained from –800 mV to

-200 mV at 308 K. The solution test was carried out after deaeration by bubbling nitrogen. Inhibition efficiency (E_p , %) was defined as Eq. 3, where i_{corr_0} and $i_{corr_{inh}}$ represent corrosion current density values without and with inhibitor, respectively.

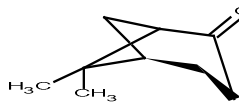
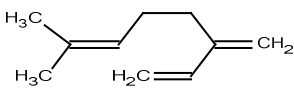
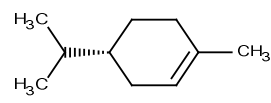
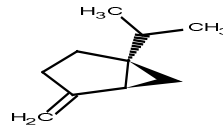
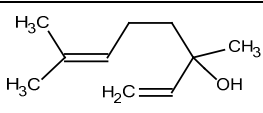
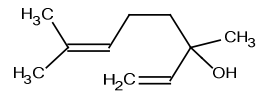
$$E_p = \frac{i_{corr_0} - i_{corr_{inh}}}{i_{corr}} \cdot 100\% . \quad (3)$$

The EIS measurements were carried out with the electrochemical system, which included a digital potentiostat model Voltalab PGZ100 computer at E_{corr} after immersion in the solution without bubbling. After determination of the steady-state current at a corrosion potential, sine wave voltage (10 mV) peak to peak, at frequencies between 100 kHz and 10 mHz were superimposed on the rest potential. Computer programs automatically controlled the measurements performed at the rest potentials after 0.5 h of exposure at 308 K. The impedance diagrams are given in the Nyquist representation. Inhibition efficiency (E_R , %) is estimated using relation 4, where R_{t_0} and $R_{t_{inh}}$ are the charge transfer resistance values in the absence and presence of inhibitor, respectively:

$$E_R = \frac{R_{t_{inh}} - R_{t_0}}{R_{t_{inh}}} \cdot 100\% . \quad (4)$$

Results and discussion. Characterization and chemical composition of essential oils. Compounds of citrus aurantium peel essential oil, their retention indices, percentages and structures, are listed in Table 1. Nine components were identified in the Citrus aurantium peel essential oil, amounting to 61.75% of the total essential oil. Peel essential oil was dominated by monoterpene hydrocarbons (54.38%), and limonene was the major constituent (52.38%) followed by β -myrcene (0.71%). β -Linalool was the main oxygenated monoterpenes (2.99%) of the essential oil.

Table 1. Essential oil composition of citrus aurantium peel

Compounds	Time, min	Percentage, %	Structures
β -pinene	3.541	0.48	
β -myrcene	3.784	0.71	
D-Limonene	4.801	52.67	
(+)-sabinene	5.779	0.52	
β -Linalool	6.685	2.99	
Linalyl alcohol	7.043	2.62	

α -terpineol	9.044	0.63	
(+)-caran-3-beta-ol	8.896	0.55	
6-methyl-2-(oxiranyl)-5-hepten-2-ol	9.044	0.63	

Previous compositional studies regarding the peel oil constituents of citrus aurantium showed similar results proving that limonene was the major volatile compound [22, 25, 26].

Weight loss measurements. Values of the inhibition efficiency and corrosion rate obtained from the weight loss measurements of mild steel for different concentrations of citrus aurantium essential oil (CAO) in 1 M HCl at 35°C after 6 h of immersion are given in Table 2. The corrosion rate decreases with the increase of concentration of the tested inhibitor and in turn the inhibition efficiency (E_w , %) increases to attain 95% (at 5 g/l). From the weight loss measurement, we can conclude that CAO is the excellent inhibitor.

Table 2. Corrosion parameters for mild steel in 1 M HCl in absence and presence of different concentrations of CAO obtained from weight loss measurements at 35°C for 6 h

Inhibitors	C , g/l	w , $\text{mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$	E , %
1 M HCl	–	0.82	–
CAO	0.5	0.19	77
	1.0	0.12	<u>85</u>
	2.0	0.11	87
	3.0	0.09	89
	5.0	0.04	95

Adsorption isotherm. Adsorption of organic inhibitor molecules from the aqueous solution can be regarded as a quasi-substitution process between the organic compounds in the aqueous phase Org_{sol} and water molecules at the electrode surface $\text{H}_2\text{O}_{\text{ads}}$ [27, 28].

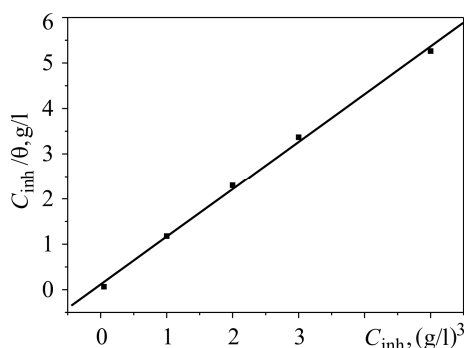
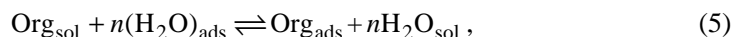


Fig. 1. Langmuir isotherm of steel in the 1 M HCl solution in presence of CAO, calculated by the gravimetric method.

where Org_{sol} and Org_{ads} are the organic species dissolved in the aqueous solution and adsorbed onto the metallic surface, respectively, $\text{H}_2\text{O}_{\text{ads}}$ is the water molecule adsorbed on the metallic surface and n is the size ratio representing the number of water molecules replaced by one organic adsorbate, of adsorption the fractional surface coverage values as a function of inhibitor concentration must be obtained. It is found that the adsorption of studied inhibitors on the steel surface obeys the Langmuir adsorption isotherm equation [29, 30]:

$$\frac{C}{\theta} = \frac{1}{k} + c, \quad (6)$$

where C is the concentration of inhibitor, k the adsorption equilibrium constant, and θ is the surface coverage expressed by the ratio $E/100\%$.

The surface coverage values θ were tested graphically for fitting a suitable adsorption isotherm. The plot of C/θ versus C yielded a straight line with a slope 1.04. This was observed clearly proving that the adsorption of the citrus aurantium oil from acid solutions on the mild steel surface obey the Langmuir adsorption isotherm (Fig. 1).

Potentiodynamic polarization curves. The parallel cathodic Tafel curves in Fig. 2 suggested that the hydrogen evolution was activation-controlled and the reduction mechanism was not affected by the presence of the inhibitor. The results in Table 2 showed that the inhibition efficiency increased, while the corrosion current density decreased with increasing CAO concentrations.

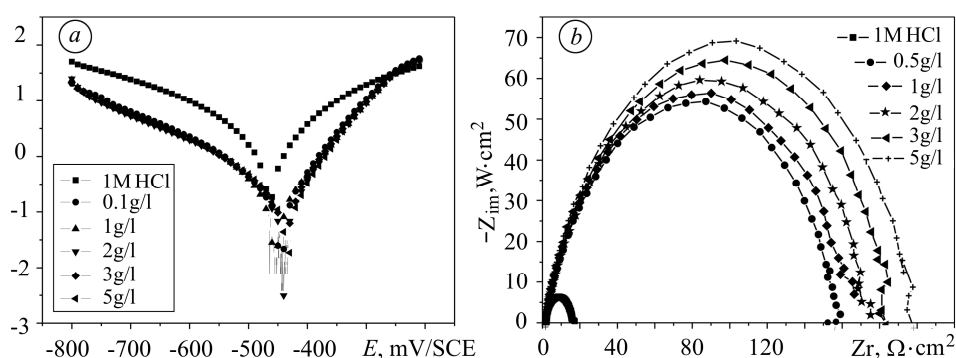


Fig. 2. Tafel plot of mild steel (a) and Nyquist plot (b) with different concentrations of CAO in 1 M HCl solution.

Table 3. Tafel polarization parameters obtained at different concentrations of CAO

Inhibitors	C , g/l	$-E_{\text{corr}}$, mV/SCE	I_{corr} , $\mu\text{A}/\text{cm}^2$	$-\beta_c$	β_a	E_p , %
1 M HCl	–	464	1386	164	135	–
CAO	0.5	439	183	149	80	87
	1	455	178	144	80	87
	2	441	132	142	96	90
	3	434	104	135	66	92
	5	454	95	112	77	93

From Fig. 2 and Table 3, it is clear that the addition of inhibitor causes a decrease in the current density. The values I_{corr} of the mild steel in the inhibited solution are smaller than that in the inhibitor free solution. The values of corrosion potential (E_{corr}) were found to be almost identical at all CAO concentrations, indicating that it acted as a mixed-type inhibitor [31].

Electrochemical impedance spectroscopy. The corrosion behavior of steel in acidic solution in the presence and absence of inhibitor was investigated by EIS at 298 K after 30 min of immersion (Fig. 3). The impedance parameters derived from these investigations are mentioned in Table 4.

Generally, Fig. 3 showed that the impedance spectra exhibited one single depressed semicircle, and the diameters of semicircle increased with the inhibitor concentra-

tion. The single semicircle can be attributed to the charge transfer that takes place at the electrode/solution interface, and the transfer process controls the corrosion reaction of the mild steel and the presence of inhibitor does not change the mechanism of mild steel dissolution [32].

Table 4. Impedance parameters for mild steel in 1 M HCl in the absence and presence of different CAO concentrations

Inhibitor	C, g/l	R_t , $\Omega\cdot\text{cm}^2$	f_{max} , Hz	C_{dl} , $\mu\text{F}/\text{cm}^2$	E, %
1 M HCl	–	14.57	54.64	200	–
CAO	0.5	156	17.48	58.41	91
	1	166	16.73	57.33	91
	2	175	16.27	55.92	92
	3	183	15.97	54.48	92
	5	198	16.74	48.04	93

The values of the polarization resistance were calculated by subtracting the high frequency intersection from the low frequency intersection [33]. Double layer capacitance values were obtained at the maximum frequency (f_{max}), at which the imaginary component of the Nyquist plot is the maximum and calculated using the following equation.

$$C_{dl} = 1/2\pi f_{\text{max}} R_t, \quad (7)$$

where C_{dl} is the double layer capacitance, $\mu\text{F}\cdot\text{cm}^{-2}$; f_{max} is the maximum frequency, Hz and R_t is the charge transfer resistance, $\Omega\cdot\text{cm}^2$. The R_t values increased with the increase of the concentration of CAO. The maximum values of the double layer capacitance are also brought down to the maximum extent in the presence of inhibitors and the decrease in the values of C_{dl} follows the order similar to that obtained for Icorr in this study. The results obtained by the polarization technique in the acidic solution were in good agreement with those obtained by EIS with a small variation.

CONCLUSION

CAO is an efficient corrosion inhibitor in 1 M HCl. Polarization studies showed that CAO was a mixed inhibitor and its inhibition efficiency increased with the inhibitor concentration increase. Impedance method indicates that CAO adsorbs on the mild steel surface with increasing transfer resistance and decreasing double-layer capacitance. The adsorptions of CAO on the mild steel in 1 M HCl solution obey the Langmuir adsorption isotherm with a high correlation coefficient. The inhibitor efficiency determined by the electrochemical and gravimetric methods is in good agreement.

РЕЗЮМЕ. Проаналізовано склад оливи зі свіжих шкірок цитрусових за допомогою газової хроматографії та хроматомас-спектрометрії. Олива містить вуглеводні монотерпenu (54,38%) та лімонену (52,67%). Гравіметричним та поляризаційним методами, а також імпедансної спектроскопії вивчено вплив її додатків на корозію сталі у розчині 1 M HCl. Ефективність інгібітора зі збільшенням вмісту до 5 g/l зростала на 95%. За результатами імпедансної спектроскопії побудовано еквівалентні моделі інгібування. Встановлено, що адсорбцію оливи на поверхні сталі описує ізотерма Ленгмюра.

РЕЗЮМЕ. Проанализировано содержание масла из свежих шкурок цитрусовых с помощью газовой хроматографии и хроматомасс-спектрометрии. Масло содержит углеводороды монотерпена (54,38%) и лимонена (52,67%). Гравиметрическим и поляризационным методами, а также импедансной спектроскопии изучено влияние ее добавок на коррозию стали в растворе 1 M HCl. Эффективность ингибитора с ростом концентрации до

5 g/l увеличивалась на 95%. По результатам импедансной спектроскопии построены эквивалентные модели процесса ингибирования. Выявлено, что адсорбцию масла на поверхности стали описывает изотерма Ленгмюра.

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