Inhibitory Effect of Ethanolic Extract of Propolis on Corrosion of Ferritic Stainless Steel in Chloride Media

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This work evaluates the inhibitory effect of ethanolic extract of propolis (EEP) on the corrosion of AISI 409 stainless steel (SS) in chloride media. Additions of 100, 200, and 500 µL of EEP in an aqueous sodium chloride solution were performed. The inhibitory effect on the SS was evaluated using electrochemical impedance spectroscopy (EIS) measurements up to 720 hours in immersion. EEP additions of 200 µL and 500 µL increased the polarization resistance of the SS. After 720 hours of immersion, the highest impedance was identified for the SS in solution with 500 µL of EEP. The maximum inhibition efficiency observed for addition of 500 µL of EEP in solution was 98.1 %, after 360 h of immersion.

Keywords
chloride, propolis, corrosion inhibitor, stainless steel, electrochemical

Introduction

Corrosion inhibitors are chemical agents that reduce or prevent corrosion reactions between the metal and the environment[1–5]. Present in small quantities, inhibitors can act on the electrode surface by adsorption, passivation or surface layer formation[1–5]. Recent developments in corrosion inhibitors are the utilization of ionic liquids[6], and rare earth organic compounds, which can provide an environmentally safe and non-toxic alternative to chromates as corrosion inhibitors for steels[7,8].

Green inhibitors are also a recent field in the literature. Green inhibitors are organic compounds, which are non-toxic, ecofriendly, obtained from plant extracts, natural fibers, fruits, and biomass, and have polar functional groups, double and triple bonds or aromatic rings acting as anchor points from the molecule to the metal surface occurring in physical or chemical adsorption[7]. The search for plant extracts and natural fibers that can act as inhibitors for the corrosion process are justified by the need for obtaining an eco-friendly and low-cost product from renewable sources[9,10]. Polyphenols, alkaloids, and flavonoids act as inhibitors through interactions between the p electrons, from aromatic rings, and non-bonding electrons from the heteroatoms. Organic inhibitor efficiency is essentially related to physical-chemical characteristics, properties of the functional groups (electronic density in the donor atom), the presence of p orbitals, molecule electronic structure, and polarizability[11].

Recent studies have reported fruits and vegetable extracts, such as orange, mango, papaya, and coffee bean, acting as corrosion inhibitors[12]. However, there are a few reports related to the bio-product inhibitor activity produced by insects, especially bees[2,9]. An interesting bio-product is propolis, due to its varied composition, including flavonoids, esters, aldehyde, ketone, terpenoids, phenylpropanoids, and other compounds[4]. Generally employed as ethanolic extract, the composition of propolis depends on the flora and climate of each location, and bee species[4,9].

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Applied in cosmetics, the use of propolis has been increasing in the last years, due to its phytotherapeutic behavior, especially in Brazil, which has the third largest production in the world[4]. The biological properties of propolis are extensively described in the literature due to its great potential, especially in antimicrobial activity[13–16]. However, research related to the inhibitory effect of ethanolic extract of propolis in solution are quite recent[4,9]. This work evaluates the potential of the ethanolic extract of propolis (EES) acting as a corrosion inhibitor on the surface of AISI 409 SS in aqueous chloride media. The selection of AISI 409 SS is that despite being a stainless steel, it has a low cost due to the low chromium content (10.5 wt.%), and a lower corrosion resistance than austenitic steels and most ferritic steels, especially in media containing chlorides, due to the low chromium content and the absence of molybdenum and nickel in its composition.

Materials and methods

AISI 409 stainless steel, designated by the Unified Numbering System (UNS) as UNS S40900, was supplied by a steel manufacturer, and steel samples of 1 cm² in area were embedded in an epoxy resin. All specimens in the study were mechanically polished using abrasive sanding papers of fineness 400, 600, and 1200. The samples were dried and degreased with acetone. The AISI 409 SS chemical composition is shown in Table 1.

The electrolyte used was 30 mL of aqueous solution of 3.5 wt.% NaCl, with EEP additions of 100 µL, 200 µL, and 500 µL. A micropipette was used for the addition of the propolis extract in the NaCl solution, and the beaker was stirred for homogenization. The samples were immersed up to 720 hours.

Electrochemical impedance spectroscopy (EIS) measurements were performed after 1 h, 96 h, 360 h, 600 h, and 720 h of immersion. EIS analysis was performed at the corrosion potential in a frequency range from 100 kHz to 1 mHz, with an applied AC amplitude of 10 mV, using a three-electrode cell: Pt as a counter electrode and Ag/AgCl as a reference electrode. Fitting results were made using Zview software version 2.1. The electrochemical tests were performed using Autolab PGSTAT 100 N potentiostat.

The iron content (mg L⁻¹) in solution and in corrosion products released from the steel was analyzed by atomic absorption spectrometry (AAS) after 720 h of immersion to determine the global inhibition efficiency of EEP. The solution was analyzed using AAS, dried at 100 °C, and weighed; the corrosion products were dissolved in hydrochloric acid, and this solution was also analyzed by atomic absorption spectrometry. The equipment used was Hitachi Z8200.

The SEM characterization was performed using a FEG Scanning Electron Microscope with FIB Nanofabrication System – Quanta FEG 3D FEI. The acceleration voltage was 15 V.

Results and discussion

Nyquist diagrams of the AISI 409 SS in solutions with addition of EEP after different immersion times: (a) 1 hour, (b) 96 hours, (c) 360 hours, (d) 600 hours, and (e) 720 hours are shown in Fig. 1.

Results of steel in electrolytes with the EEP addition of 100 µL and with absence of EEP were fitted using the circuit with two-time constants (Fig. 2). The first element, R_1, is related to the resistance of the electrolyte, R_2 and CPE are the resistance and the constant phase element associated with the corrosion product layer. The R_3 and C1 are the resistance and capacitance associated with processes occurring at the layer/metal interface.

Impedance results for the SS in solutions with additions of 200 µL and 500 µL of EEP showed different behavior. The equivalent circuit, shown in Fig. 3, was used to fit the experimental data. The R_1 is the resistance of the solution, R_2 is the resistance of the external layer of propolis, R_3 is the resistance associated with the oxide layer of SS, and R_4 is the resistance at the metal/oxide layer. R_p is considered the sum of values of R_2, R_3, and R_4 impedance. Table 2 shows the electrochemical parameters obtained using the data fitting with equivalent circuits.

After the first hour of immersion, a small difference was observed between the values of steel impedance; all results showed the same magnitude order, indicating insufficient inhibitory action of EEP on steel. After 96 h of immersion, the highest impedance of SS was observed in the solution with 200 µL of EEP, but this impedance decreased as time increased. After 360 h, 600 h, and 720 h of immersion, the highest impedance was obtained for the steel in solutions with 500 µL of EEP.

The electrolyte showed a low resistance varying from 1 Ω cm² up to 13 Ω cm². As the concentration of propolis in solution increased, the conductivity decreased and the solution resistance increased, due to the organic and low polarity char-
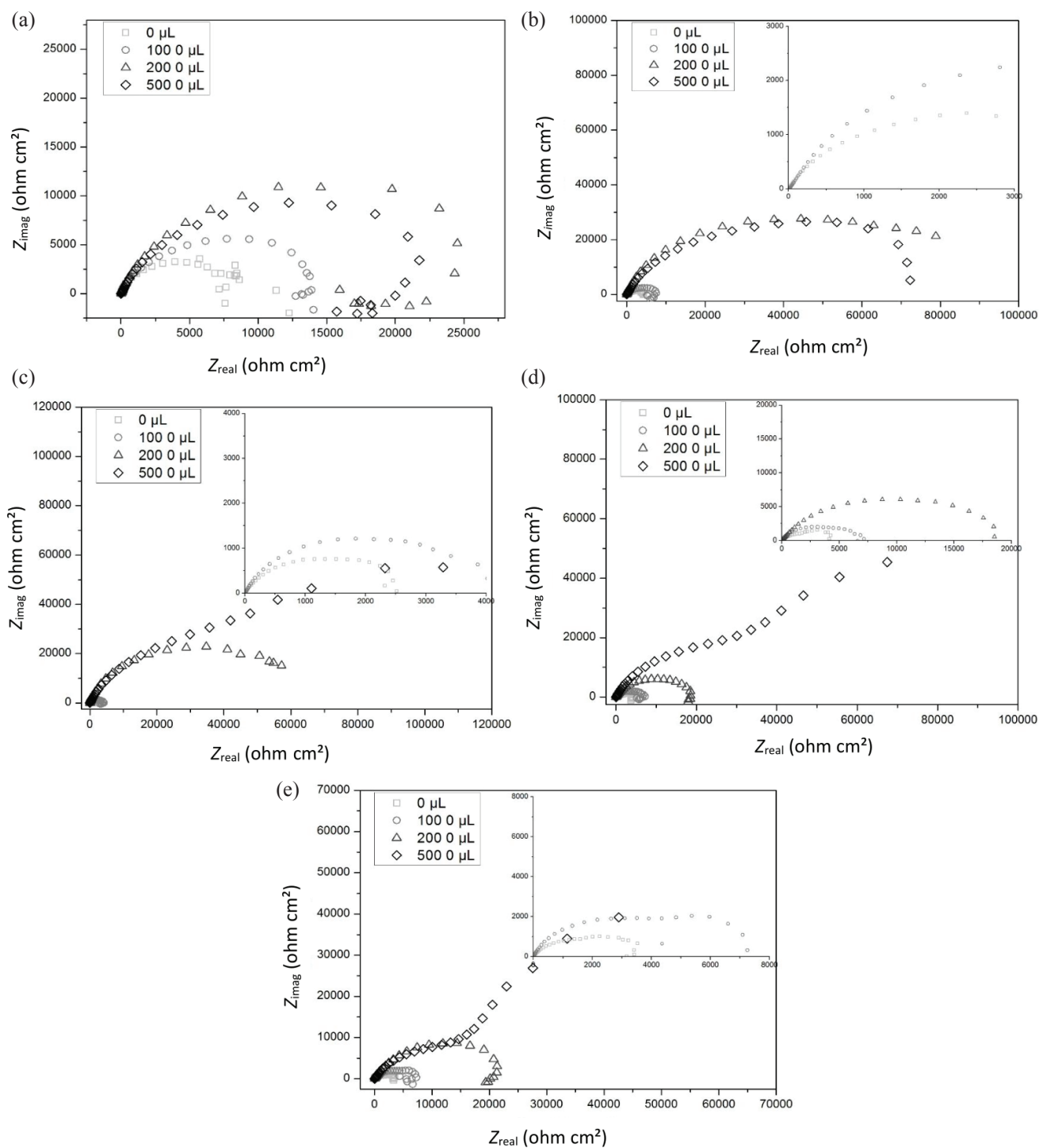
Table 1 – *Chemical composition of the AISI 409 SS (wt.%)*

Steel	Cr	Ni	Ti	C	Si	Mn	P	S
AISI 409	10.5	–	0.75	0.08	1.00	1.00	0.045	0.045

Fig. 1 – *Nyquist diagram at different immersion times: (a) 1 hour, (b) 96 hours, (c) 360 hours, (d) 600 hours, and (e) 720 hours*
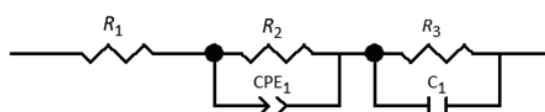

Fig. 2 – *Equivalent circuit for steel corrosion in a saline solution with 100 µL of EEP and in a saline solution without EEP*
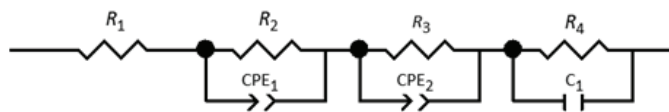
Fig. 3 – *Equivalent circuit for steel corrosion in saline solutions with addition of 200 µL and 500 µL of EEP*

Table 2 – *Electrochemical parameters obtained using fitting with equivalent electrical circuits*

Immersion (h)	Inhibitor addition	R_s ($\Omega\,cm^2$)	R_2 ($\Omega\,cm^2$)	CPE1 ($\mu F\,s^n\,cm^{-2}$)	n_1	R_3 ($\Omega\,cm^2$)	CPE2 ($\mu F\,s^n\,cm^{-2}$)	n_2	R_4 ($\Omega\,cm^2$)	C1 ($\mu F\,cm^{-2}$)	R_p ($\Omega\,cm^2$)	Inhibition efficiency
1	0 µL	6.2	5766	85	0.87	2148				538	7914	–
	100 µL	12.1	6580	184	0.83	7993				497	14573	46 %
	200 µL	5.8	1400	676	0.60	1330	293	0.80	23481	151	26211	70 %
	500 µL	8.4	6774	405	0.64	4280	237	0.80	10816	291	21870	64 %
96	0 µL	6.9	2606	427	0.73	1652				412	4258	–
	100 µL	8.7	5835	533	0.61	1986				503	7821	46 %
	200 µL	10.0	7213	275	0.67	1964	361	0.82	71109	122	80286	95 %
	500 µL	12.7	1442	265	0.69	6833	252	0.76	64721	159	72996	94 %
360	0 µL	4.5	2145	472	0.72	533				125	26778	–
	100 µL	9.6	3544	430	0.72	462				107	4006	33 %
	200 µL	2.5	58961	198	0.81	4122	122	0.56	48.25	129	63131	96 %
	500 µL	2.1	41	670	0.44	50858	140	0.73	88324	187	139223	98 %
600	0 µL	5.9	2059	492	0.72	2397				460	4456	–
	100 µL	6.7	5679	403	0.73	1244				369	6923	36 %
	200 µL	1.2	43	126	0.34	18440	141	0.72	1402	200	19885	78 %
	500 µL	2.8	70	138	0.57	87756	133	0.77	38800	245	126626	96 %
720	0 µL	4.8	2116	550	0.71	1433				900	3549	–
	100 µL	5.3	6695	579	0.66	1464				277	8159	57 %
	200 µL	5.1	33	307	0.49	10143	558	0.70	12400	212	22576	84 %
	500 µL	3.2	59	159	0.57	83987	142	0.75	18962	276	103008	97 %

acter of the compounds such as flavonoids, esters, aldehyde, ketone, terpenoids, and phenylpropanoids, which are constituents of propolis. In electrolytes with EEP additions of 200 µL and 500 µL, for immersion times above 100 h, the solution resistance decreased (Table 2) due to the steel surface coverage by EEP, which was removed from solution. Effects like ethanol addition or evaporation, as well as metal dissolution can also result in R_e change. Data fitting with different models led to slightly different R_s values, which was probably the reason for the sudden change in R_s values obtained for 100 µL and 200 µL solutions.

Corrosion inhibitors act on steel surfaces due to adsorption, and this process cannot be instantaneous, depending on the diffusion coefficient of the inhibitor in the electrolyte. After 96 hours of immersion, a great variation of the polarization resistance was observed, and the highest R_p was found for the steel in solutions with 200 µL and 500 µL of EEP (Table 2). Grudić *et al.*[17] investigated the possibility of corrosion inhibition of copper by propolis extract in 0.51 mol dm^{-3} NaCl solution. The results of the study also showed that with increasing propolis extract concentration in the solution there was a slight increase in open circuit copper potential to positive values, an increase in polarization resistance, and a decrease in the corrosion current density.

After 96 h of steel immersion in a saline solution with 100 µL of EEP, the R_p value increased slightly compared to the steel in a saline solution without EEP. The surface of AISI 409 SS, after 1 hour of immersion in a saline solution with 100 µL EEP addition, showed a partial inhibitor coverage (Fig. 4).

Organic inhibitors decrease the corrosion rate due to superficial phenomena by providing a protective layer. Inhibitor adsorption occurrs as a substitutional process, and the water molecules present in the metal surface are displaced by the organic molecules[18]. Inhibition efficiency is usually defined as a function of one or more measurable indicators in the experiments performed. In this work, the iron content present in immersion residue was considered an element defining inhibition efficiency, but it was not the only one; an electrochemical parameter, R_p, was also adopted to evaluate the inhibition efficiency, present in equation (1).
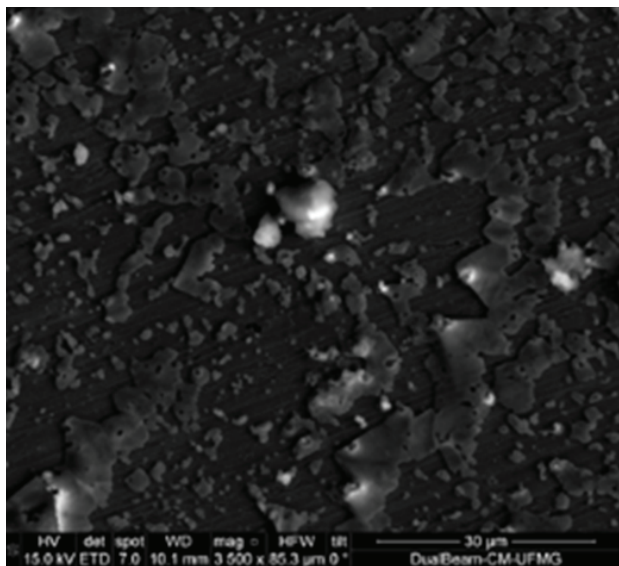
F i g . 4 – *SEM micrograph of steel surface after one hour of immersion in a saline solution with 100 μL of EEP*

(a)

(b)

→ **Interaction with Π electrons**

→ **Interaction with p electrons**

F i g . 5 – *(a) acid 3,5-diprenyl-4- hydroxycinnamic, Artepillin C®, (b) interactions between Artepillin C® and the metal surface*

$$n = \frac{R_p - R_{p0}}{R_p} \cdot 100 \qquad (1)$$

where n is inhibition efficiency, R_p and R_{p0} are the polarization resistances with and without addition of EEP, respectively. The inhibition efficiencies of EEP are shown in Table 2.

Inhibition efficiency of 100 μL EEP was lower than 60 %, and it decreased until 360 hours of immersion; then there was a slight increase, reaching 56.5 % after 720 h. The low inhibition efficiency agreed with the low steel surface coverage presented in Fig. 4 in solution with 100 μL of EEP. For 200 μL and 500 μL EEP additions, the inhibition efficiency increased up to 360 h of immersion, and thus decreased especially for the steel in solution with 200 μL of EEP. For the 200 μL EEP solution, after 360 hours of immersion, propolis desorption or propolis loss by the spalling of corrosion product occurred. A different process was observed for steel in 500 μL EEP solution; inhibition efficiency remained practically constant over time because of EEP's great effectiveness acting as a corrosion inhibitor on AISI 409 SS.

The maximum inhibition efficiency, calculated by R_p, was 98.1 %. Gaspari *et al.*[2] presents the efficiency for different authors who worked with honey derivates acting as a corrosion inhibitor. In this study, the efficiency observed was higher than all recent values reported in the literature[2,18–22], suggesting that ethanol extract of propolis acts as a strong corrosion inhibitor.

Propolis is composed of more than 160 compounds. The major bioactive compound present in Brazilian green propolis is 3,5-diphenyl-4- hydroxycinnamic acid, Artepillin C®, reported by Dolabella *et al.*[4] using high performance liquid chromatography (HPLC). Artepillin C®'s structure, shown in Fig. 5(a), has bioactive, antitumoral, antibacterial, and antioxidant properties[23]. The corrosion inhibitor process depends on the interaction between the principal organic compound, Artepillin C®, and the steel surface. Initially, the organic molecules diffuse through the saline solution toward the steel surface. Organic inhibitors interact with the steel surface, displacing water molecules and adsorbed ions. The displacement is favored when the interaction occurs by non-bonding electrons, from p orbitals and π electrons, from aromatic rings. Artepillin C® has p electrons from oxygen atoms and π electrons from an aromatic ring, being able to interact with metal d orbitals, promoting a coordinate bonding involving electron transfer from the organic molecule to the metal surface. Chemisorption strength depends on electric density of the donor atom and the polarizability of the chemical group[11]. Fig. 5(b) shows possible interactions between Artepillin C® and the metal surface.
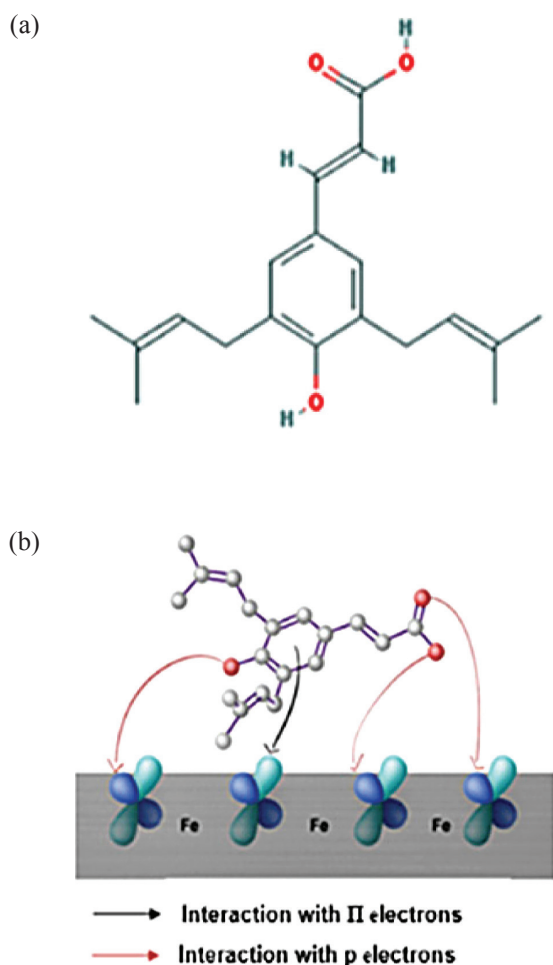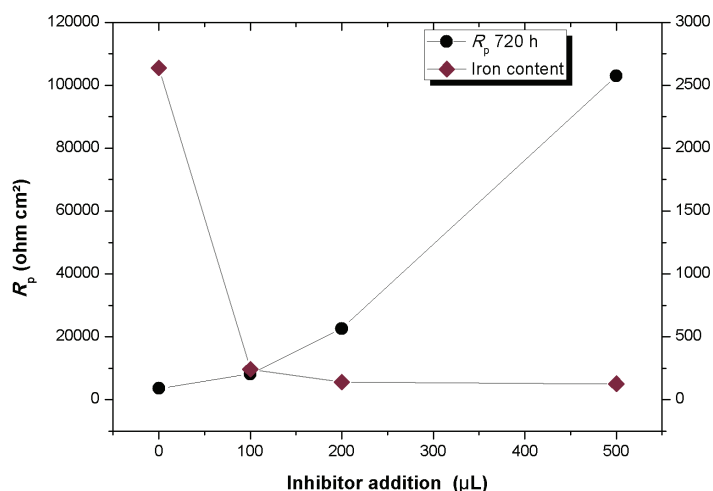
F i g . 6 – *Iron content in corrosion product and polarization resistance after 720 hours of immersion*

Conclusions

Inhibitory activity of ethanolic extract of propolis (EEP) was evaluated in the corrosion process of AISI 409 steel in a NaCl 3.5 wt.% solution using electrochemical impedance spectroscopy up to 720 hours of immersion. The 100-µL addition of EEP showed no inhibitory action on corrosion of AISI 409 steel, in terms of polarization resistance. EEP additions of 200 µL and 500 µL increased the polarization resistance of the SS. After 96 h of immersion, the highest polarization impedance was observed for steel in 200 µL of EEP in solution, but the impedance decreased as time increased. After 720 h, the highest impedance was identified for the SS in solution with 500 µL of EEP.

Maximum inhibition efficiency observed for addition of 500 µL of EEP in solution was 98.1 %, after 360 h of immersion. This inhibition efficiency is higher than the value found in literature for bio-products derived from honey.

Inhibition efficiency was also evaluated by measuring the iron content in solution and in corrosion products released from the steel, using atomic absorption spectroscopy. The global maximum efficiency of 95.2 % was observed for the addition of 500 µL of EEP, but all measured efficiencies were superior to 90 %, indicating great potential of EEP application as a green corrosion inhibitor.

Inhibition efficiency generally decreased as time increased when the adsorption process occurred by physical nature. Some works have reported cases where inhibition efficiency increases as time increases, which is attributed to the stability of the adsorbed species on the metal surface[24]. Different inhibition efficiencies observed were related to the amount of EEP present in solutions.

The global inhibition efficiency, after immersion time, was evaluated by using iron content in solution after 720 hours of immersion, according to equation (2)[12].

$$n = \left(\frac{W_0 - W}{W_0} \right) \cdot 100 \qquad (2)$$

where, W_0 and W are the iron content in the absence and presence of EEP, respectively, measured by atomic absorption spectroscopy.

Global inhibition efficiency observed for additions of 100 µL, 200 µL, and 500 µL of EEP are 90.8 %, 94.7 %, 95.2 %, respectively, indicating that EEP acts as a corrosion inhibitor of AISI 409 SS in NaCl 3.5 wt.% solution.

Fig. 6 shows that polarization resistance increased, and iron content decreased with increasing EEP addition. Experimental data showed that an addition of 100 µL of EEP was sufficient to reduce the corrosion process, even though it did not promote full surface coverage. The iron content was lower than it was for the electrolyte with no EEP addition, indicating that EEP can act as corrosion inhibitor for AISI 409 SS in NaCl 3.5 wt.% in all contents studied.

The highest polarization resistance and the lowest iron content in solution results obtained after 720 hours steel immersion assay in 500 µL EEP had shown the highest corrosion resistance of steel in this medium.

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