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***Opuntia ficus-indica* (Nopal Extract) as Green Inhibitor for Corrosion Protection in Industrial Steels**

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Additional information is available at the end of the chapter

<http://dx.doi.org/10.5772/intechopen.72944>

Abstract

Soluble extract from *Opuntia ficus-indica* (Nopal extract) has been proposed in this chapter as a green inhibitor due to its component called mucilage, which has the ability to retain water; for this reason, it has been used as metal corrosion protection in machinery pieces, tools and other metallic components that need to be stored for short periods. In this way, three industrial carbon steels (AISI 1018, 1045 and 4140) have been exposed in sulfuric acid (H_2SO_4) to evaluate the corrosion behavior with or without Nopal extract (NE). Some electrochemical techniques have been implemented to evaluate the corrosion inhibition efficiency (IE) such as DC linear polarization resistance (LPR) and AC electrochemical impedance spectroscopy (EIS). Results indicated a considerable superficial modification of steel in terms of dielectric constant and ion charge capacity. When the NE was added, the corrosion mechanism changed from localized to general attack, decreasing the corrosion rate in all cases. More susceptibility to fail by corrosion was observed in the 1045 carbon steel in comparison with the other two studied steels; these results were confirmed by the percentage of inhibitor's efficiency of about 95%.

Keywords: green inhibitor, corrosion protection, Nopal extract, industrial carbon steel, corrosion rate, electrochemical evaluation, electrochemical impedance spectroscopy

1. Introduction

Study of corrosion inhibitors has taken a new role in recent years, since new regulations restrict the use of several components in conventional corrosion inhibitors like nitrites, benzoates,

chrome, lead, arsenic, among others, because they are toxic and dangerous to health and environment. Formulations of alternative compounds with more biocompatibility and low costs have led to the development of the *green inhibitors* also called *eco-friendly inhibitors* [1, 2]. Main sources from which these inhibitors are extracted come from plants, fruits, seeds, leaves and flowers whose components such as flavonoids, alkaloids and natural oils (pennyroyal oil, jojoba oil, etc.) are considered the inhibitor's active agent. Those extracts have been studied in low concentration and exposed to different aggressive media to protect metals (mainly carbon steels), obtaining good results at the superficial protection against corrosion during the first hours of exposure.

Figure 1 shows a general classification of the corrosion inhibitors according to its origin. Organic inhibitors are characterized by their high molecular weight structures and are polar molecules. Most organic inhibitors are adsorbed on the metal surface by displacing water molecules and forming a compact barrier. Inorganic inhibitors are salts of some metals, which have a passivation effect and reaction with the metal. Some synthetic compounds can reduce corrosion damage in carbon steels [3–5] because their oxygen, nitrogen, and sulfur heteroatoms react on the metal surface, blocking active sites where corrosion occurs.

Green corrosion inhibitors are biodegradable and do not contain heavy metals or toxic compounds. Mechanisms of the green inhibitors indicate that ions/molecules are adsorbed onto metal surface, interfering with the anodic and/or cathodic reactions and decreasing the diffusion rate for reactants to the metal surface. Usually, the electrical resistance of the metal surface is decreasing. Since their innovation, some researchers have proposed many plants as prospects to be green corrosion inhibitors. **Table 1** shows a review of several papers about green inhibitors collected from 2004 to 2017, where the carbon steels in acid media (HCl and H₂SO₄)

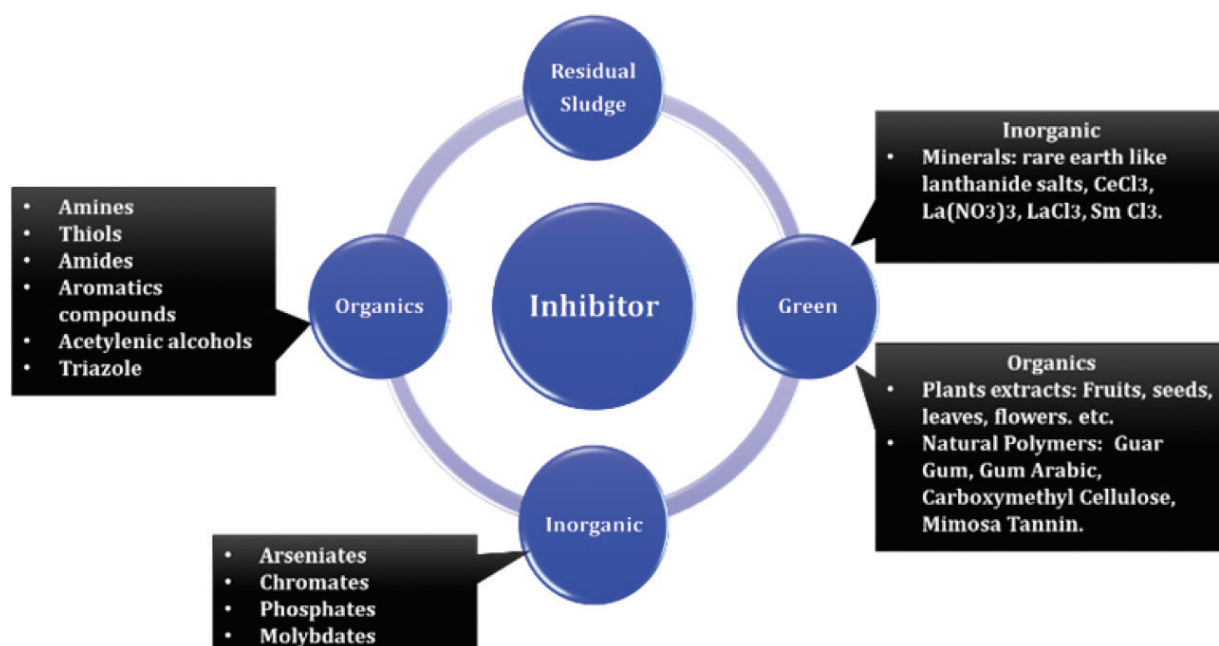


Figure 1. General classification of corrosion inhibitors.

Inhibitor	Metal	Medium	IE (%)	Year	References
<i>Zanthoxylum armatum</i>	Mild steel	H ₃ PO ₄	> 90	2004	[8]
<i>Simmondsia chinensis</i>	Carbon steel	HCl	98	2004	[9]
<i>Artemisia vulgaris</i>	Carbon steel	HCl	85	2004	[10]
<i>Datura metel</i>	Carbon steel	HCl and H ₂ SO ₄	84~97	2005	[11]
<i>Mentha pulegium</i>	Carbon steel	HCl	80	2006	[12]
<i>Ammi visnaga</i>	SX316	HCl	99.3	2006	[13]
<i>Carica papaya</i>	Mild steel	H ₂ SO ₄	94	2007	[14]
<i>Gongronema latifolium</i>	Aluminum	HCl, NaOH	> 90	2007	[15]
<i>Opuntia ficus-indica</i> (Cactus)	Aluminum	HCl	94	2007, 2012	[16, 17]
<i>Phyllanthus amarus</i>	Mild steel	HCl and H ₂ SO ₄	> 90	2008	[18]
Black pepper	Carbon steel	HCl and H ₂ SO ₄	98	2008	[19]
Zallouh root	Carbon steel	H ₂ SO ₄	90	2008	[20]
<i>Justicia gendarussa</i>	Mild steel	HCl	93	2009	[21]
Clove oil	Nickel, inconel 600, 690	HCl	88~92	2009	[22]
<i>Lupinus albus</i>	Carbon steel	HCl and H ₂ SO ₄	86	2009	[23]
<i>Gossypium hirsutum</i>	Aluminum	NaOH	97	2009	[24]
<i>Ananas sativum</i>	Aluminum	HCl	96	2010	[25]
<i>Murraya koenigii</i>	Mild steel	HCl	~ 94	2011	[26]
Marine Alga <i>Caulerpa racemosa</i>	Mild steel	H ₂ SO ₄	~85	2012	[27]
<i>Spirulina Platensis</i>	Mild steel	HCl and H ₂ SO ₄	~80	2012	[28]
<i>Aloe vera</i>	Zinc, Galvanized iron	HCl, H ₂ SO ₄	88.5	2014	[29]
<i>Tagetes erecta</i>	Mild steel	H ₂ SO ₄	98.07	2014	[30]
Watermelon rind	Mild steel	HCl, H ₂ SO ₄	~86	2014	[31, 32]
<i>Mangifera indica</i> (Mango) and Orange	Mild steel, Carbon steel	H ₂ SO ₄ , HCl	97	2014	[33]
Anise	Carbon steel	HCl	94	2014	[34]
Coconut coir	Aluminum, Mild Steel	HCl and H ₂ SO ₄	80	2012,2014	[35–37]
<i>Musa paradisiaca</i> (banana)	Mild steel, SS304	H ₂ SO ₄ , NaCl	82.7	2011,2014	[38, 39]

Inhibitor	Metal	Medium	IE (%)	Year	References
<i>Ficus hispina</i>	Mild steel	HCl	90	2015	[40]
<i>Nicotiana tabacum</i>	Mild steel	H ₂ SO ₄	94	2015	[41]
<i>Litchi chinensis</i>	Mild steel	H ₂ SO ₄	97.8	2015	[42]
<i>Capsicum annuum</i> , <i>Citrus aurantium</i> , <i>Moringa oleifera</i>	Copper	HNO ₃	60–80	2015	[43]
<i>Valoniopsis pachynema</i>	Brass	H ₃ PO ₄	96.06	2015	[44]
<i>Azadirachta indica</i>	Al, Mild steel, Tin, SS, Carbon steel	HCl, H ₂ SO ₄ , HNO ₃	> 85	2015	[45]
<i>Petroselinum crispum</i> (Parsley), <i>Eruca sativa</i> (Arugula) <i>Anethum graveolens</i> (Dill)	Carbon steel	HCl	92	2015	[46]
<i>Morinda citrifolia</i>	AISI 1045, AISI 8620	HCl	> 86	2015,2016	[47, 48]
Pomegranate	Carbon steel, Mild steel, α -Brass	HCl	> 90	2015,2013,2017	[49–51]
<i>Gentiana olivieri</i>	Mild steel	HCl	92	2016	[52]
Roasted coffee	Carbon steel	HCl	94	2016	[53]
Curcumin	Aluminum	NaCl	80	2016	[54]
<i>Calotropis procera</i> leaves	Mild steel	NaCl	> 90	2016	[55]
<i>Ocimum basilicum</i>	Carbon steel, Mild steel	NaCl + Na ₂ S, H ₂ SO ₄	88.5	2016,2012	[56, 57]
Tilia	Mild steel	HCl	79	2017	[58]
<i>Cedrus atlantica</i>	Lead	Na ₂ CO ₃	~ 70	2017	[59]

Table 1. Review of several papers related with green inhibitors from 2004 to 2017.

were the most studied, possibly because the total annual cost of metallic corrosion in a country is associated mainly with the corrosion of carbon steels of different industrial sectors. Most of these works were focused on demonstrating the efficiency of these extracts as well as their behavior with respect to temperature, identifying the absorption mechanism from the extract components [6, 7], however, other factors like cost and amount are not mentioned in detail.

Evaluation of corrosion inhibition efficiency was based on the implementation of electrochemical and gravimetric methods; the electrochemical techniques consist of linear polarization resistance (LPR) and electrochemical impedance spectroscopy (EIS). With LPR was obtained information about the inhibition reaction with the compound, classifying the inhibitor action as anodic, cathodic or mixed. EIS allowed to detect the resistance changes on the surface and how the active sites are reordered through changes at the interfacial capacitance of metal-electrolyte.

Corrosion inhibition efficiency can be calculated through the coverage degree “ θ ” on the metal surface by the following equations:

$$\theta = \frac{\frac{1}{R_{ct}(\text{blank})} - \frac{1}{R_{ct}(\text{inhibitor})}}{\frac{1}{R_{ct}(\text{blank})}} \quad (1)$$

$$\theta = \frac{i_{\text{corr}(\text{blank})} - i_{\text{corr}(\text{inhibitor})}}{i_{\text{corr}(\text{blank})}} \quad (2)$$

$$\theta = \frac{V_{\text{corr}(\text{blank})} - V_{\text{corr}(\text{inhibitor})}}{V_{\text{corr}(\text{blank})}} \quad (3)$$

$$\theta = \frac{\text{Weight loss in blank} - \text{Weight loss in inhibitor}}{\text{Weight loss in blank}} \quad (4)$$

where R_{ct} is the charge transfer resistance, i_{corr} is the corrosion current and V_{corr} is the corrosion rate. Expressions (1), (2) and (3) are obtained from electrochemical techniques like LPR and EIS, and Eq. (4) is calculated using the gravimetric method. With any of those equations, the efficiency is obtained with:

$$EI(\%) = \theta \times 100 \quad (5)$$

In general, a good inhibitor must have an efficiency greater than 90%.

Efficiency of an organic compound as a corrosion inhibitor depends mainly on the anchor capacity by chemical or physical interactions on the metal surface, forming multilayers of adsorbed organic molecules that block the active sites of corrosion and retarding the anodic and/or cathodic reactions. Thermodynamic studies with adsorption isotherms (Temkin, Frumkin, Langmuir, Freundlich, among others) have allowed to estimate the stability of the layer adsorbed on the surface. These depend on the physicochemical properties of the active molecules related to the inhibitor functional groups like aromatic rings and amino acids with $-\text{NH}_2$ groups, which are readily protonated in acid media and adsorbed on the metal surface through acid anions (Cl^- , SO_4^{2-}) negatively charged. Electron density from the donor atoms, and the interaction between the orbital π from inhibitory and the orbital d of the iron, play an important role in the adsorption type [60–62].

Investment in the use of inhibitors for industrial scale to prevent corrosion represents a small percentage (1%) worldwide compared with the costs in the use of paints, surface treatments and materials selection, which represent 80% of the investment. The main sectors investing in inhibitors such as petrochemicals (production, synthesis, and refining), water treatment systems, and chemical and food processes, have reported an efficiency of up to 90% in the mitigation of corrosion, saving billions of dollars. However, despite its effectiveness, many of the commercial synthetic inhibitors that are currently used generate problems of toxicity and contamination to the environment. Environmental Protection Agency (EPA) regulations have determined that inorganic inhibitor compounds such as

salts of chromates, phosphates and molybdates, as well as organic inhibitors like phosphates, amides or thiols, are dangerous. Due to this problem, green inhibitors open an opportunity for the replacement of these compounds (**Table 1**) where the synthesis process of natural extracts is of low cost and can be applied in closed systems as pipelines and packaging of pieces for transport. Nowadays, the limitation in the industrial staggering of these green inhibitors is in their preservation, since it has been shown that after 24 h, the natural extract begins to decompose which would imply the use of large volumes of extract to serve the industry [63].

1.1. *Opuntia ficus* plant

One plant that has caused interest about their potential as a green inhibitor is *Opuntia ficus-indica* (Nopal), which is a traditional vegetable in Mexico with a high soluble fiber content. Nopal is classified as a wild plant that survives in desert regions and does not require much water for cultivation. It is said that it has an important ecological performance, as it stops the degradation of the deforested soil, making the arid land productive because of its capacity to retain water. There are about 1600 species in 122 genera of the cactus family, from which comes the Nopal. It has fruits that are edible and are known with the name of Tunas. The soluble fiber of Nopal known as *mucilage*, which has the capacity to retain up to 30 times its weight in water. For this particular compound, it has been promoted as a good candidate for the production of an ecological inhibitor for the protection of the steel against corrosion [64, 65]. Chemical analysis of mucilage indicates that it contains a large amount of pectin as well as minerals such as calcium, potassium and sodium whose quantities depend on the age of the cladodes (leaves). The presence of these minerals suggests that this plant may remain stable over long periods in alkaline media.

Mucilage is similar in consistency with Aloe vera (slimy) and has been used for the conservation of building materials. Chandra [66] and Torres Acosta [17] studied the effects of dehydrated Nopal and Aloe vera powder in the electrochemical performance of reinforcing steel in chloride contaminated concrete. Preliminary results suggest that adding these powders in small concentrations might be suitable for enhancing corrosion resistance of steel in concrete. Therefore, this investigation deals with the performance of Nopal mucilage as a corrosion inhibitor for industrial steels exposed in H_2SO_4 .

2. Experimental details

2.1. Specimens preparation

Three types of carbon steels were evaluated in this research: AISI 1045, 1018 and 4140. The composition of each metal is shown in **Table 2**. Rods of each steel were cut mechanically to obtain specimens of 25 mm of diameter and 5 mm of thickness for the electrochemical evaluations. The surface preparation was grinding with SiC paper through numbers 80, 240, 320, 400, 600 and 1500 grits until a mirror finish. Finally, the specimens were washed with distilled water, degreasing with acetone and drying with hot air.

Element	Wt. %		
	1045	1018	4140
C	0.45	0.2	0.4
Mn	0.7	0.9	0.85
P	0.04	0.04	0.04
S	0.05	0.05	0.05
Fe	98.76	98.81	97.31
Mo	*****	*****	0.2
Cr	*****	*****	0.9
Si	*****	*****	0.25

Table 2. Metal composition with AISI classification.

2.2. Preparation of Nopal extract

Nopal extract was obtained through the selection of tender cladodes, a cleaning process and removal of thorns, cut and disinfected with 2 ml of chlorine +1 ml of Cu_2SO_4 , assuring the elimination of microorganism and fungi that degrade the extract. Subsequently, it was crushed in an extractor where the pulp was removed from the juice; this last one was heated for 1 h at 78°C with a volume ratio of 2:1 in distilled water. Finally, the concentrated liquid was filtered through a fine sieve to ensure separation of solid particles larger than $180\ \mu\text{m}$, obtaining the final product as shown in **Figure 2**.

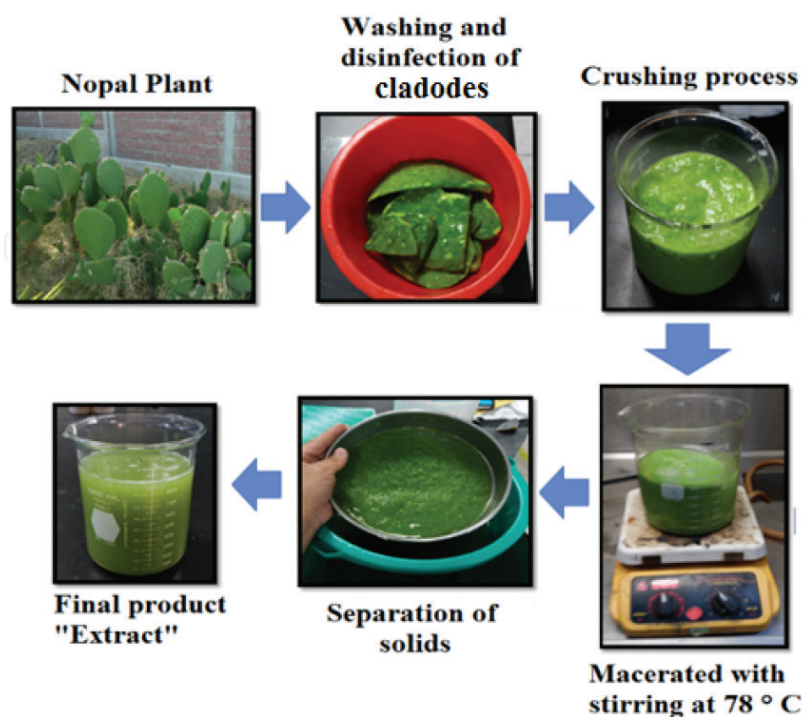


Figure 2. Nopal extract preparation process.

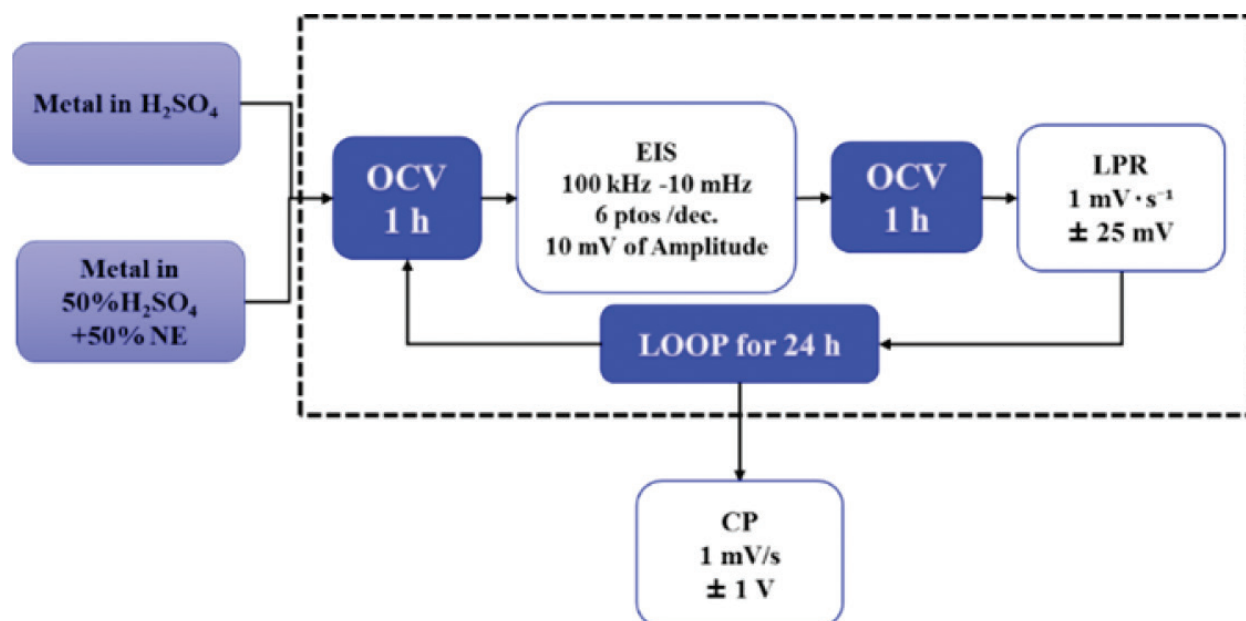


Figure 3. Experimental sequence.

2.3. Experimental sequence

Electrochemical characterization was carried out in an acrylic cell for flat probes [65], assembled with an arrangement of three electrodes. Steels 1018, 1045 and 4140 were placed as working electrode (WE), a grid of platinum was placed as a counter electrode (CE) and a calomelane electrode was used as reference electrode (RE). Solution of H₂SO₄ (0.6 mol·l⁻¹) was used as the test electrolyte, prepared with analytical grade reagents and deionized water. All experiments were performed under standard conditions of temperature and pressure. **Figure 3** shows the experimental sequence where the steels in H₂SO₄ with and without Nopal extract (NE) were evaluated. The relation of Nopal extract and H₂SO₄ ratio was 50:50 volume %. Open circuit potential (OCP) was monitored to ensure the system stability during 1 h, followed by the electrochemical impedance spectroscopy (EIS) technique, applying a sweep of frequencies from 100 kHz to 10 mHz with 10 mV amplitude. OCP measurement was run again for 1 h to continue with linear polarization resistance (LPR) with an overpotential range of ±25 mV and a sweep speed of 1 mV·s⁻¹. Cycle OCP-EIS-OCP-LPR was repeated for 24 h until record the final behavior with a polarization curve (CP) applying an overpotential of ±1 V at a scan rate of 1 mV·s⁻¹. Corrosion rates, capacitances and resistances for all steels were calculated.

3. Results and discussion

Corrosion potential results are shown in **Figure 4**, where only the steels exposed in acid medium reported positive potentials with a tendency to move toward more negative (active) potentials when the inhibitor is present, indicating a change in the surface activity of the metal. **Figure 5** shows the behavior of the resistance between the metal in the acid medium and the medium with inhibitor (H₂SO₄ + NE). A considerable growth of the Nyquist semicircle was observed with the addition of NE for the three steels, being the widest semicircle for the 4140 steel, which reaches a value of charge transfer resistance (R_{ct}) of 1400 ohms at the beginning

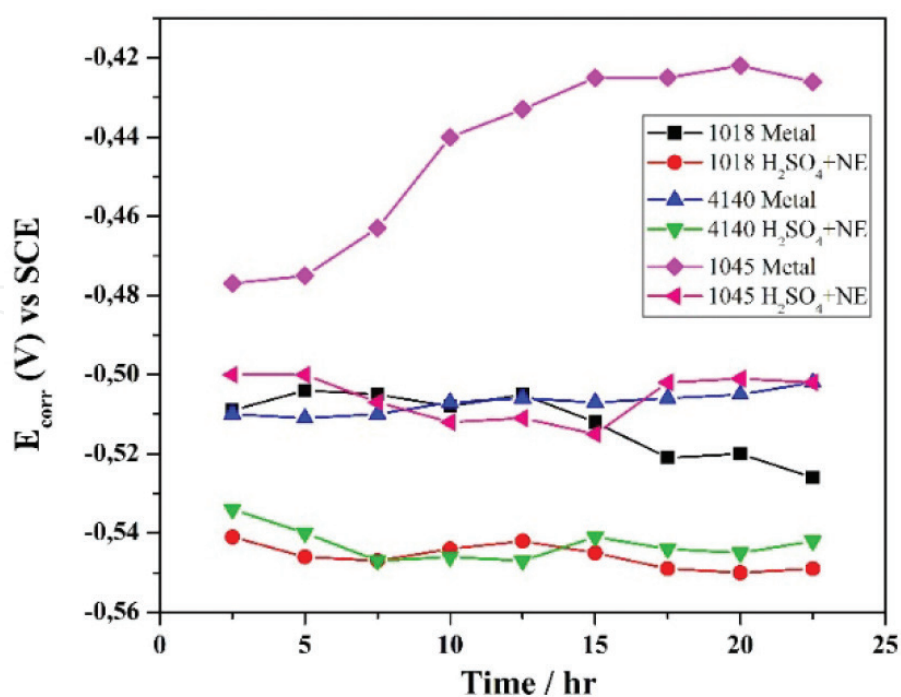


Figure 4. Potential corrosion behavior of metals for 24 h.

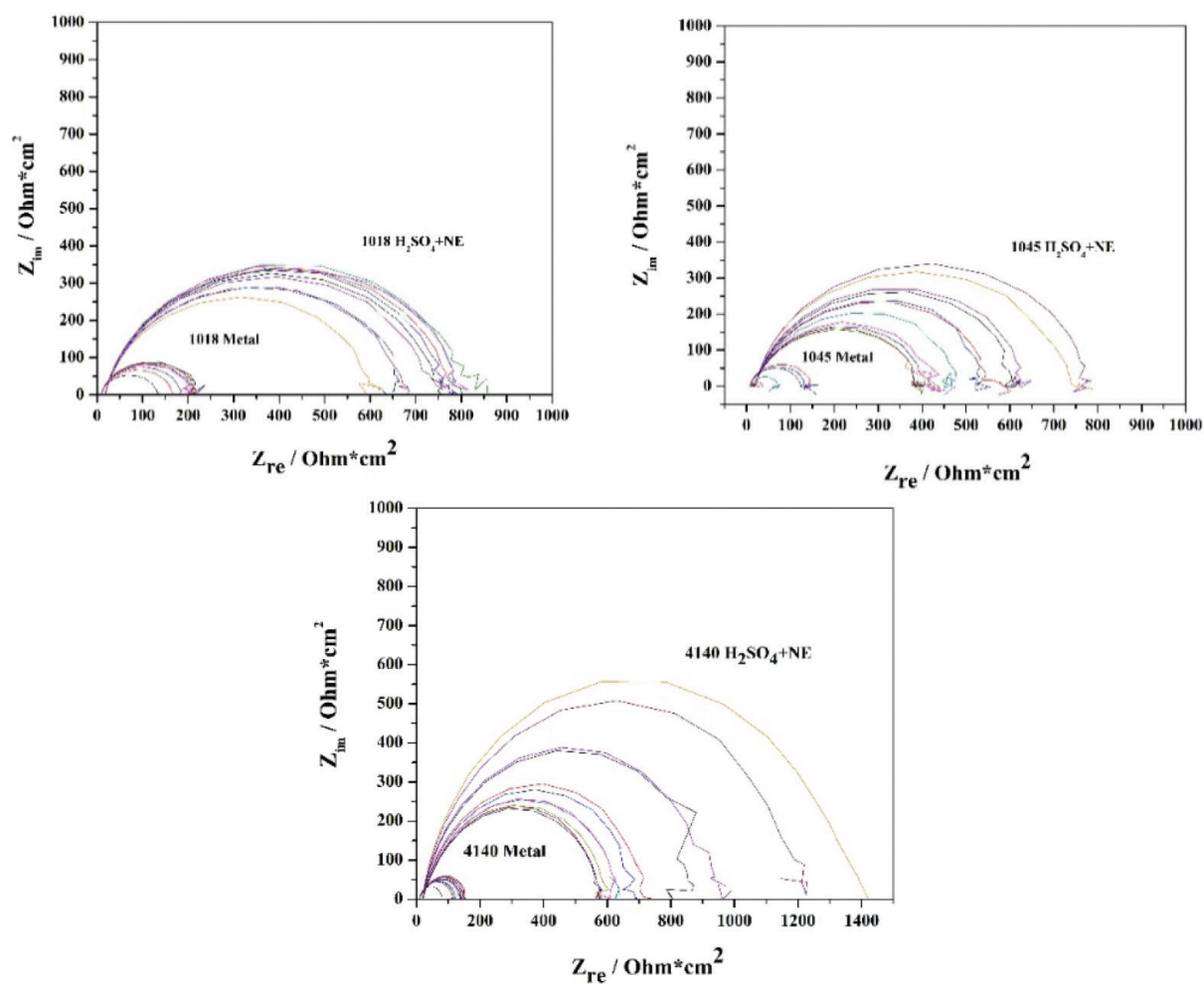


Figure 5. Nyquist plots corresponding to the three steels in H_2SO_4 and H_2SO_4 + Nopal extract.

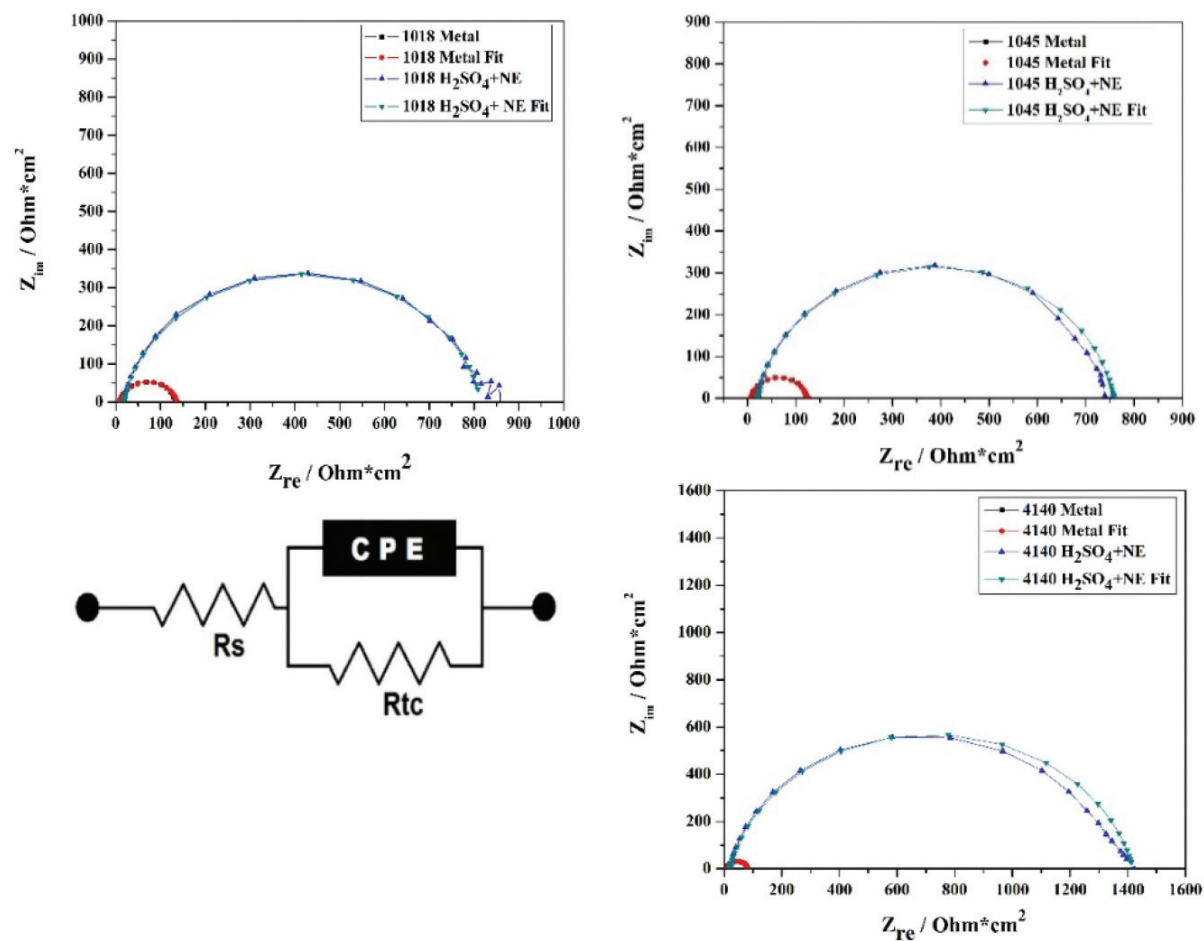


Figure 6. EEC and fit of Nyquist plots corresponding to metals in H_2SO_4 and H_2SO_4 + Nopal extract.

of the evaluation. As the exposure time increases, the semicircle shows a tendency to decrease slightly as a result of the alteration of the protection film formed by the inhibitor. At the end of the monitoring, the R_{ct} values from the inhibitor films do not approach the R_{ct} values of the blank, conserving some protection capacity with respect to the metal without inhibitor.

Semicircle adjustment was performed for each impedance cycles by taking an equivalent electrical circuit (EEC) as shown in **Figure 6**. Due to the shape of the Nyquist semicircle, the basic configuration called EEC Randles model was selected, which is formed by a resistance in series (solution resistance, R_s) coupled to a constant phase element (CPE) that is in parallel with another resistance attributed to the metal/electrolyte interface (R_{ct}).

To obtain the capacitance values (C_{dl}) from the constant phase element (CPE), it was necessary to obtain the maximum frequency of the Nyquist semicircle ($\omega_{\theta_{max}}$) as well as the α exponent that was calculated during the CPE adjustment, since $CPE = F \cdot s^{(\alpha-1)}$ where F and s indicate Faradios and seconds, respectively. Results in **Table 3** are shown in $\mu\text{F cm}^{-2}$. Equation (6) shows the calculation to C_{dl} :

$$C_{dl} = CPE * \omega_{\theta_{max}}^{(\alpha-1)} \quad (6)$$

Metal	Medium	LPR		EIS		
		J_o (A/cm ²)	E_{corr} vs. SCE (V)	R_p (Ohm · cm ²)	R_{ct} (Ohm · cm ²)	C_{dl} (μF · cm ²)
1018	H ₂ SO ₄	2.92E-05	-0.511	268.182	187.538	179.36
	H ₂ SO ₄ + NE	7.85E-06	-0.545	1015.013	717.162	155.96
4140	H ₂ SO ₄	4.74E-05	-0.506	179.379	121.76	1202.42
	H ₂ SO ₄ + NE	7.71E-06	-0.543	1085.28	815.98	185.14
1045	H ₂ SO ₄	3.34E-04	-0.439	64.865	50.78	492.49
	H ₂ SO ₄ + NE	1.15E-05	-0.505	729.44	514.16	120.41

Table 3. Electrochemical results.

Polarization curves of carbon steel samples exposed in H₂SO₄ with inhibitor are shown in **Figure 7(A)**, where a decrease in current corrosion of the three steels studied is observed.

Anodic slope shows a change near the 400 mV, associated with a change of their activation by the presence of a coating from the inhibitor's organic molecules, limiting the ionic interchange between the metal and electrolyte.

Figure 7(A) shows the behavior of the polarization in which there is a decrease in the flow of current on the three steels exposed to the inhibitor. **Figure 7(B)** shows the corrosion behavior for all the steels; these values were obtained by extrapolation of Tafel, where the steels show a lower corrosion rate to 1018, 4140 and 1045, respectively; these values remain stable throughout the 24 h of exposure.

Table 3 shows the values obtained with the electrochemical techniques LPR and EIS; a correspondence was observed between the values of resistance for both techniques; however,

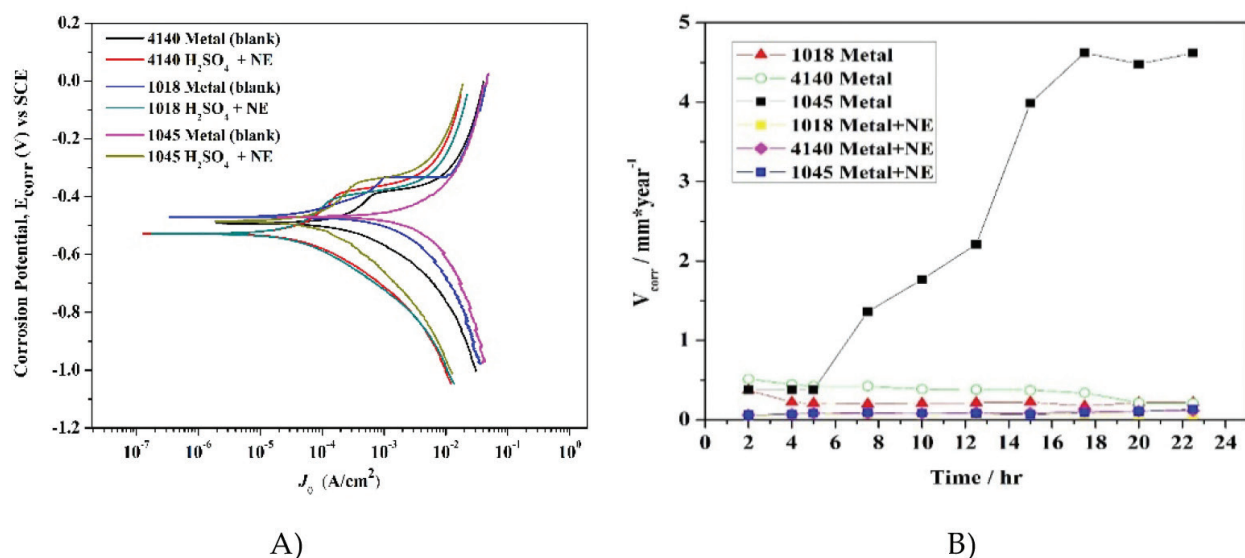


Figure 7. (A) Polarization curve for steels in H₂SO₄ and H₂SO₄ + NE medium and (B) corrosion rate of metals for 24 h.

the resistance values by the impedance technique are lower because they are depurated due to the contribution of the resistance to the solution that has been separated with the adjustment of the EEC. The current indicates that the 1018 and 4140 steels have low corrosion rate. Capacitance values indicate a decrease with the presence of the inhibitor and are lower for 1045 and 1018 steels; these values tend to decrease due to a difficulty of ionic exchange.

Figure 8 shows the metallography obtained for all steels on a macroscopic scale, where for (a)–(c), the area exposed to H_2SO_4 shows that the main mechanism for the three metals is a combination between pitting and generalized corrosion, being more notable the pitting attack for the 1018 and 1045 steels before the NE addition.

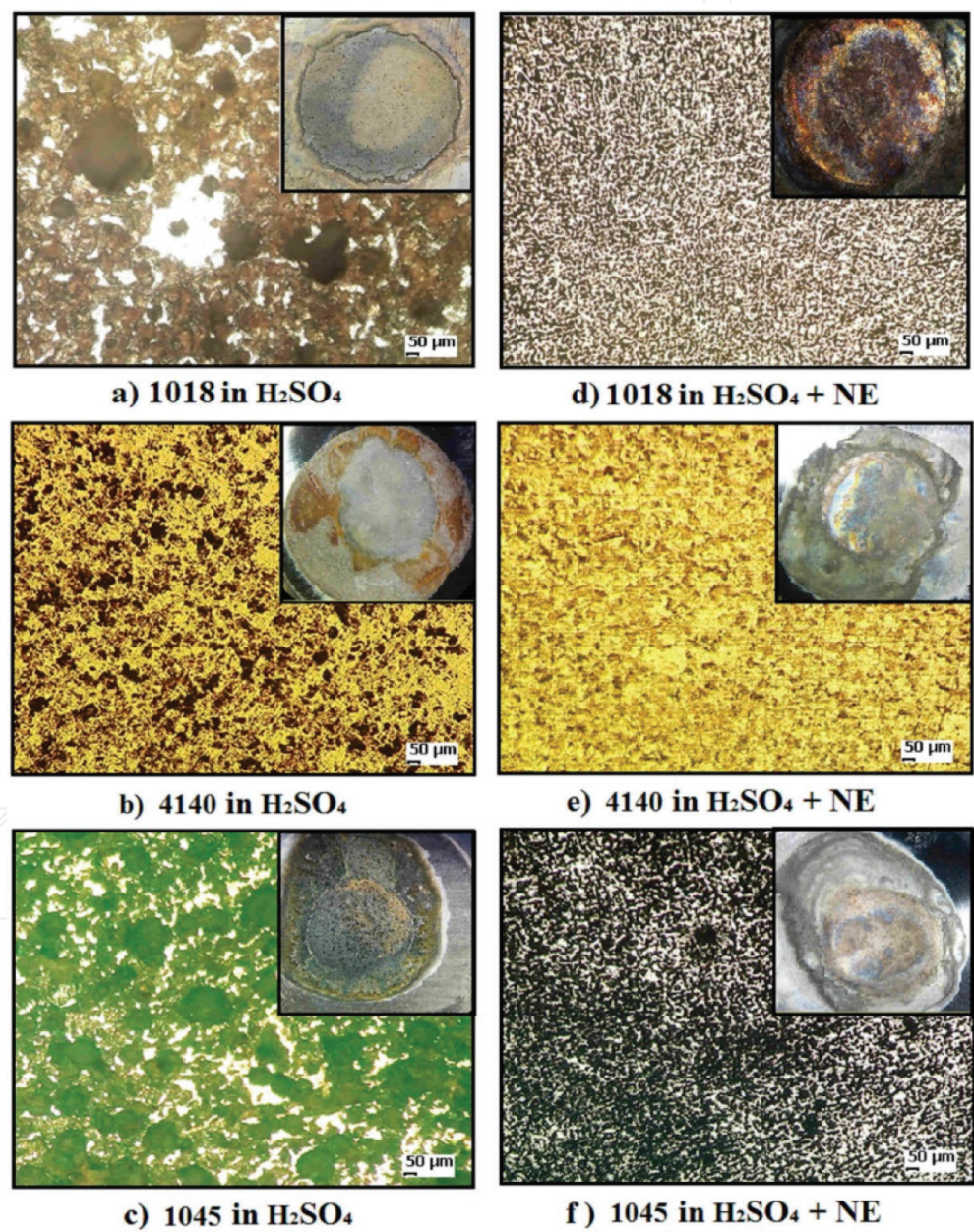


Figure 8. Metallographic obtained of metals after exposition in H_2SO_4 and H_2SO_4 + Nopal extract.

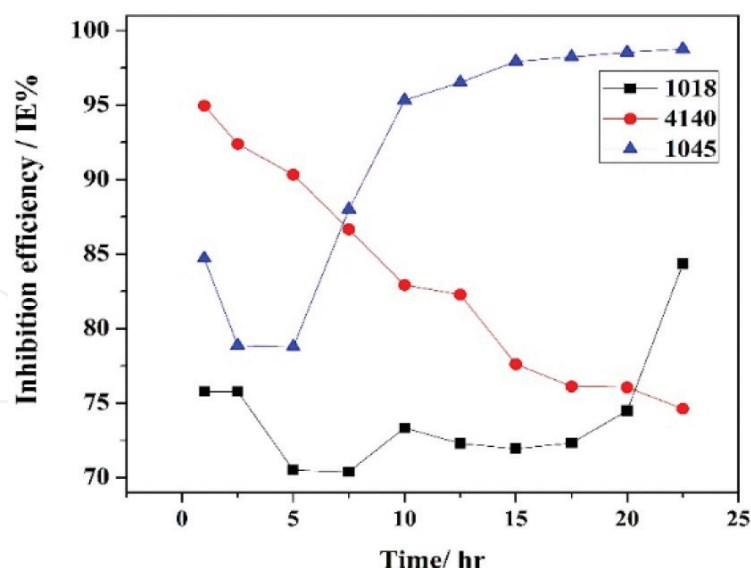


Figure 9. Inhibition efficiency for the three metals in H_2SO_4 + Nopal extract after 24 h.

Figure 8(d)–(f) shows the behavior of the steels after exposed to the acid mixture plus the Nopal extract. Change of the corrosion mechanism was observed with the addition of the NE from a localized attack (pitting) to a generalized attack due to the redistribution of the active sites by the presence of the organic molecules from the inhibitor, having better behavior in the steel 1045 where the corrosion was considerably diminished.

Figure 9 shows the efficiency calculated for the three steels, observing the performance of the Nopal extract during the 24 h of exposure. It was observed that steel 1045 obtains the highest percentage of efficiency, which is associated with the inhibitor coverage, and remains constant from the 10th hour of exposure, while the other two steels (1018 and 4140) do not achieve sufficient efficiency values to guarantee their protection.

4. Conclusions

Opuntia ficus-indica (Nopal extract) was used as a green inhibitor to evaluate the corrosion behavior in the protection of three industrial steels (AISI 1018, 1045 and 4140), exposed in sulfuric acid (H_2SO_4) plus the addition of NE in the same proportion (50/50 volume %). Corrosion potential showed a tendency to be more negative when the NE was added, forming a compact barrier on the metal surface that increased the surface resistance, according to the growth of the Nyquist semicircles and with respect to the steel substrates.

Charge transfer resistance (R_{ct}) was reduced as the exposition time increased due to the deterioration of the protective film. A 4140 steel reported less corrosion rate with respect to the 1018 and 1045 steels, respectively. Polarization resistance (R_p) showed the same behavior with the best behavior for the steel 4140 after 24 h.

Change of the corrosion mechanism was observed with the addition of the NE from a localized attack (pitting) to a generalized attack due to the redistribution of the active sites by the presence of the organic molecules from the inhibitor, having better behavior in the steel 1045 where the corrosion was considerably diminished.

The NE addition changed the corrosion mechanism in the steels from a localized attack (pitting) to a generalized attack due to the adsorbed inhibitor molecules on the metal surface, reducing the anodic and/or cathodic reactions and as a consequence the corrosion rate.

Alloyed steel (4140) showed a decrease in the inhibitor efficiency with the exposition time, showing better tendency (above 90%) for the 1045 steel. This is possible because the chemical composition of the steel is interacting with the adsorbed organic molecules, increasing or reducing the active sites for corrosion. Difference in corrosion rate for the 4140 steel with the others was the combination of the green inhibitor and their alloyed elements (Mo and Cr).

Acknowledgements

The authors wish to thank our institution (CIDETEQ) and (UAEM) for the facilities and the National Council of Science and Technology (CONACYT) for financial support. We appreciate the participation of the student Luis E. Almazán Corona, who actively participated in the experimental development through the program UAEM-SIyEA (Secretary of Investigation and Advance Studies) 3817/2014/CID.

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