

MELOCACTUS CURVISPINUS (CACTACEAE) AS AN INHIBITOR OF STEEL CORROSIONS.Y. Paredes-Dugarte^{a*}, M.F. Pérez-Landáez^b, S. M. Moreno-Morales^c, H. D'Armas Regnault^b^a“Dr. Susan Tai” Institute for Biomedical and Applied Sciences Research, Materials Characterization Laboratory, University of Oriente. Cumaná, Sucre 6101, Venezuela.^bSchool of Sciences. Department of Chemistry, University of Oriente. Cumaná, Sucre 6101, Venezuela.^cYachay Tech Medicinal Chemistry Research Group (MedChem-YT), School of Chemical Science and Engineering, Yachay University for Experimental Technology and Research (Yachay Tech), Yachay City of Knowledge, Urcuquí 100119, Ecuador.*Corresponding author, E-mail: sparedes@udo.edu.ve**ABSTRACT**

This research aims to investigate the potential of *Melocactus curvispinus* pulp extract as a “green inhibitor”, focusing on the electrochemical behavior of API-N80 steel in a saline solution (3.5% m/v NaCl) and 1 mol/L H₂SO₄, with extract concentrations of 5, 10, 15 and 20% (v/v), as an environmentally friendly approach to reducing the use of toxic corrosion inhibitors, which are harmful to both the environment and human health. To achieve this, electrochemical techniques including potentiodynamic polarization, morphological characterization by SEM and photochemical analysis via FTIR with polarity fractionation were employed. The results of the electrochemical measurements showed that the extract acts as a mixed-type inhibitor in both saline and acidic media. In the saline medium achieving a maximum inhibition efficiency of 83.89% at a concentration of 15%, whereas at a concentration of 20%, a reversal of the protective effect was observed, attributed to saturation of the metal/solution interface, which accelerated the corrosion process. In the case of the acidic medium achieving a maximum efficiency of 78% at 20% extract concentration, conforming to the Langmuir isotherm with a physisorption mechanism ($\Delta G_{ads} = -3.09$ kJ/mol). SEM revealed the formation of a protective film in both media, which was denser and more compact in the saline medium than in the acidic one, correlating with the observed efficiency levels. FTIR analysis confirmed the presence of alkaloids, coumarins, saponins and pentacyclic triterpenes identified in the phytochemical tests. The research validates the *M. curvispinus* extract as a functional bio-inhibitor for N-80 steel in both aggressive environments, although its use is limited by industrial volumetric requirements.

Keywords: API N80 steel; inhibition efficiency; plant extract; potentiodynamic polarization.***Melocactus curvispinus* (cactaceae) como inhibidor de la corrosión del acero****RESUMEN**

Esta investigación tiene como objetivo estudiar el potencial del extracto de pulpa de *Melocactus curvispinus* como un “inhibidor verde”, centrándose en el comportamiento electroquímico del acero API-N80 en una solución salina (3,5 % m/v de NaCl) y 1 mol/L de H₂SO₄, con concentraciones de extracto de 5, 10, 15 y 20 % (v/v), como un enfoque respetuoso con el medio ambiente para reducir el uso de inhibidores de corrosión tóxicos, que son perjudiciales tanto para el medio ambiente como para la salud humana. Para ello, se emplearon técnicas electroquímicas que incluyen polarización potenciodinámica, caracterización morfológica por SEM y análisis fotoquímico por FTIR con fraccionamiento de polaridad. Los resultados de las mediciones electroquímicas mostraron que el extracto actúa como un inhibidor tipo mixto tanto en medio salino como en medio ácido. En el medio salino se alcanzó una eficiencia de inhibición máxima del 83,89% a una concentración del 15%, mientras que con la concentración del 20% se observó una reversión del efecto protector, atribuida a la saturación de la interfase metal/solución, lo que aceleró el proceso de corrosión. En el caso del medio ácido se alcanzó una eficiencia máxima del 78% a una concentración de extracto del 20%, conforme a la isoterma de Langmuir con un mecanismo de fisiorción ($\Delta G_{ads} = -3,09$ kJ/mol). El SEM evidenció la formación de una película protectora en ambos medios, siendo más densa y compacta en el medio salino que en el ácido, correlacionándose con los niveles de eficiencia observados. El análisis FTIR confirmó la presencia de alcaloides, cumarinas, saponinas y triterpenos pentacíclicos identificados en las pruebas fitoquímicas. La investigación valida el extracto de *M. curvispinus* como bioinhibidor funcional para el acero N-80 en entornos agresivos, si bien su uso se ve limitado por los requisitos volumétricos industriales.

Palabras claves: acero API N80, eficiencia de inhibición, extracto vegetal, polarización potenciodinámica.

INTRODUCTION

In the oil industry, internal corrosion that occurs in carbon steel pipes due to aggressive agents present in the reservoir has generally been addressed through the use of inhibitors, which, when added in moderation to the fluid, prevent the continuation of oxidation-reduction reactions in the corrosive process, slowing the rate of metal deterioration for limited periods [1].

There are different types of inhibitors used to control corrosion in carbon steel pipes caused by the acidification of oil wells. These include the condensed compound of ketone, amine, aldehyde, and alquinol; in addition to phosphate and chromate considered biotoxic organic compounds for human health and the environment [2]. As an alternative to this problem, in recent decades a wide variety of extracts from plants, fruits, peels, roots, and seeds have been tested on metal surfaces in different environments for corrosion control, as they are an economical, renewable, and safer resource for the environment [3,4].

A study on the use of plants to control corrosion in steels shows the effect of organic extracts recovered in methanol and hexane from *Curcuma longa* as a corrosion inhibitor of 1018 carbon steel in a 3% (m/v) NaCl solution [5]. Other studies have used garlic extract as a corrosion inhibitor for carbon steel in an acidic medium (0.5 M HCl) [6], coffee waste extracts as inhibitors of carbon steel corrosion in an acidic environment (HCl, 0.5 M) [7], avocado seed extract as a natural inhibitor for AISI 1010 carbon steel in a 0.5 M aqueous sulfuric acid solution [8], and guar gum as an eco-friendly corrosion inhibitor for N80 carbon steel under sweet conditions in saline solution [9].

Similarly, prickly pear (*Opuntia ficus-indica*) extract successfully inhibited the corrosion of AISI 1018 carbon steel in a 0.6 M H₂SO₄ solution, achieving 84% inhibition through the formation of a protective organic layer adsorbed onto the metal [10]. This plant belongs to the Cactaceae family, whose genera shares similar bioactive compounds, such as polysaccharides, pectins, and

flavonoids, responsible for forming protective films on metal surfaces [11]. For instance, an analysis of Cactaceae mucilage conducted in an alkaline/saline medium on steel bars embedded in concrete, evaluated using Electrochemical Noise (EN) and Linear Polarization Resistance (LPR), demonstrated a significant delay in the onset of corrosion [12].

On the other hand, *Melocactus curvispinus* is a small, dark green, conical or globular cactus with pink flowers and club-shaped fruits; it grows in open areas on rocky or sandy substrates [13] and is used empirically for human and animal consumption (fresh fruit), as well as in the treatment of certain diseases and as an ornamental plant in gardens [14,15,16]. This species also belongs to the Cactaceae family; however, published scientific studies (primarily in Colombia and Venezuela) focus on its use as a water biocoagulant/clarifier (due to its mucilage) or on its ecology and germination [17], rather than on electrochemistry or metal corrosion inhibition; therefore, it is important to analyze its potential use as a green corrosion inhibitor.

In this context, the purpose of our research was to determine the chemical composition of the pulp extract of *M. curvispinus* and evaluate its corrosion inhibition capacity in API N-80 steel pipe test tubes exposed to aggressive media, as a contribution to the current interest in environmentally friendly corrosion inhibitors, providing a local ecological alternative to the corrosion processes that occur in oil transport pipelines.

MATERIALS AND METHODS

Material

The material used was taken from an API 5CT N-80 grade steel pipe, in commercial condition, supplied by “Petróleos de Venezuela, S.A.” (PDVSA) - Morichal District, located in the south of Monagas State. Chemical composition: C = 0.248 wt%, Si = 0.1990 wt%, Mn = 1.385 wt%, S = 0.012 wt%, P = 0.013 wt%, Cr = 0.010 wt%, Ni = 0.003 wt% and Fe remaining.

Extract preparation

M. curvispinus plants were collected in La Llanada Vieja, Cumaná, Sucre state, Venezuela. They were then washed with plenty of distilled water to ensure the elimination of microorganisms, after which the shell was removed and the pulp was cut into small pieces, which were mechanically pressed in a very fine, previously sterilized tulle-type cloth to obtain the crude extract in liquid phase, which was kept refrigerated for 72 hours. *M. curvispinus* (Cactaceae family) is locally known as “pichigüey” or “melón de cerro”.

Electrochemical tests

To evaluate the efficiency of the *M. curvispinus* pulp extract in both saline and acidic media, the potentiodynamic polarization technique was used due to its ability to drive and accelerate redox reactions at the metal-electrolyte interface. This provides immediate quantitative information on the corrosion rate, corrosion current density (i_{corr}), and corrosion potential (E_{corr}) [18], which are direct indicators for evaluating the corrosion rate and the material's performance under the conditions assessed in this study. However, the use of electrochemical impedance spectroscopy (EIS) will be considered in future research to support the mechanisms of adsorption and the formation of protective films.

For this purpose, a standard three-electrode electrochemical cell, was used [19]: an auxiliary graphite electrode, a saturated calomel reference electrode (SCE), and a working electrode consisting of the material under study, which were connected to a Gamry Reference 600 potentiostat-galvanostat/ZRA.

To prepare the working electrodes, samples measuring 1 cm² x 1 cm (pipe thickness) were cut from the API N-80 steel pipe, and an electrical contact was made on the outer face of the pipe with a copper wire, which was then V_{corr} was used to determine the percentage of corrosion inhibition efficiency of the extract in the corrosive medium using the following equation (1):

protected with a glass tube. Subsequently, the sample/wire system was embedded in epoxy resin, leaving only the surface that will be exposed to the corrosive medium uncovered, in this case the inner face of the pipe, which was subjected to a polishing process, starting with roughing using silicon carbide (SiC) sandpaper of different grits (from 240 to 1200), followed by polishing with 5.0, 1.0, and 0.3 μm alumina, respectively, until a smooth, well-finished surface is achieved, free of scratches and polishing marks, in accordance with the recommendations of the international standard ASTM E3 [20].

Two electrolyte media were used for the electrochemical tests: 3.5% (m/v) NaCl and H₂SO₄ (1 mol/L); with each of them, five 350 mL solutions were prepared with 0; 5, 10, 15, and 20% (v/v) of the crude extract of *M. curvispinus* pulp, respectively.

Prior to recording the open-circuit potential, the three electrodes were left to stand immersed in the solution for 15 hours to ensure greater system stability. The potential was then recorded under static conditions for one hour, until the steady-state open-circuit potential of the sample in contact with the corrosive medium was reached. Next, a sweep of ± 250 mV around the equilibrium potential was performed at a rate of 2 mV/s to record the linear polarization curves (Tafel curves). This rate was selected to bring the system close to a pseudo-steady state, minimizing the capacitive current of the electric double layer and allowing for a clear delineation of the anodic and cathodic slopes without unduly prolonging the experiment [18.] Gamry-Framework software was used to linearly extrapolate the anodic and cathodic branches of the Tafel curves to the corrosion potential and to record electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), and corrosion rate (V_{corr}) [21]. The experiments were performed in triplicate to ensure reproducibility of the results.

$$(\%)IE = \frac{V_{\text{o-corr}} - V_{\text{corr-inh}}}{V_{\text{o-corr}}} \times 100 \quad (1)$$

Where V_{o-corr} represents the corrosion rate in the absence of the inhibitor and $V_{corr-inh}$ represents the corrosion rate in the presence of the inhibitor.

Immersion test

To carry out the immersion tests, samples measuring 1 cm² x 1 cm (pipe thickness) were cut from the steel pipe, which were roughened and polished on the inner surface of the pipe, using the same procedure described above, in order to maintain consistent polishing conditions across all experiments. The samples were then immersed in the corrosive media, both with and without the extract, for 10 days without agitation at room temperature, in accordance with the procedure outlined in the ASTM G31 standard [22]. In this test, the extract concentrations that showed the highest corrosion inhibition efficiency were used.

Scanning electron microscopy / Energy dispersive X-ray spectroscopy (SEM/EDX)

Once the immersion test was completed, the test specimens were washed with distilled water, dried, and examined in a FEI-Model Quanta FEG 250 analytical scanning electron microscope coupled to an EDAX-Apollo X spectrometer.

Qualitative chemical tests

To detect the different families of compounds, present in the *M. curvispinu* extract, specific chemical tests were carried out, following the reported phytochemical analysis methodology [23,24].

Fourier Transform Infrared Spectroscopy (FTIR)

For FTIR analysis, the *M. curvispinus* pulp extract was fractionated with five solvents in ascending order of polarity: hexane, chloroform, acetone, ethyl acetate, and methanol, in order to investigate the phytochemical complexity of the extract. From each fraction, a pellet was

prepared with KBr, compacted in a press to obtain translucent discs, all of which were analyzed in a Perkin Elmer spectrometer, model Frontier FT-IR16 PC, with a resolution of 2cm⁻¹ and 24 scans in the spectral range between 4000 and 450cm⁻¹.

RESULTS AND DISCUSSION

Characterization of the starting material

Scanning electron microscopy inspection of the polished surface of N80 steel revealed the presence of a high density of pores or microcavities scattered throughout the matrix, as shown in Figure 1a. Using the Digimizer image analysis software, the analyzed microcavities on the surface of the polished steel were found to have an average area of 20.28 μm², a perimeter of 16.08 μm, and a length of 5.80 μm. Figure 1a shows the parameters of the three highlighted microcavities. Figures 1b-c show a globular inclusion together with EDX chemical microanalysis that reveals the presence of nitride-type inclusions in the N80 grade steel under study. In the case of the polished steel surface attacked with 3.5% Nital reagent, a martensitic microstructure characteristic of this type of carbon steel is revealed (Figure 1d). According to the literature, the main non-metallic inclusions that form in carbon steels are sulfides, nitrides, and oxides of different sizes and morphologies, which contribute to the improvement of mechanical properties. However, they can affect its corrosion behavior in different aggressive environments, acting as active sites and precursors of the pitting corrosion process, a highly destructive form of targeted attack [25].

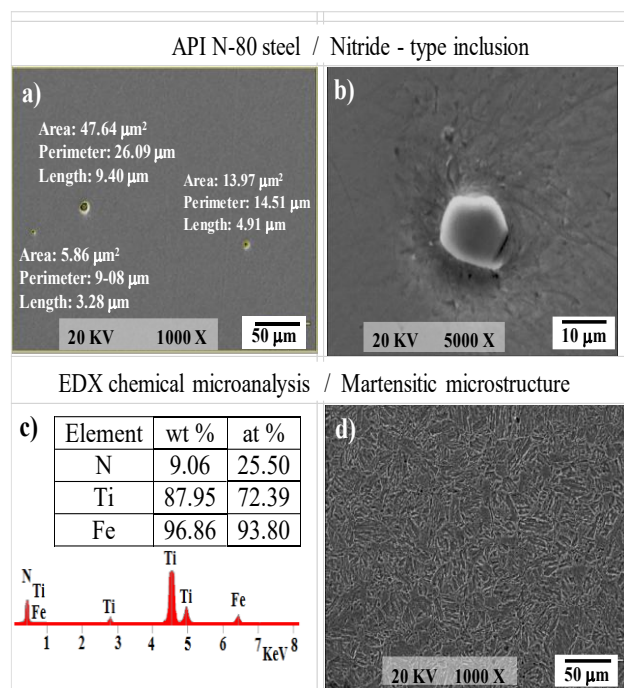


Fig. 1. SEM micrographs of: (a) polished surface, (b-c) nitride-type inclusion together with EDX microanalysis, and (d) martensitic microstructure

Electrochemical tests

Potentiodynamic polarization curves - Saline medium

The electrochemical parameters derived from the polarization curves (such as corrosion current density (i_{corr}), corrosion potential (E_{corr}), and corrosion inhibition efficiency (%IE)) are presented in Table 1. Analysis of these data shows that the addition of 5% and 20% extract shifts the corrosion potential (E_{corr}) toward more noble (anodic) values. However, i_{corr} decreases only slightly with the 5% concentration, whereas with the 20% concentration it increases significantly compared to the control, suggesting a process of accelerated metal dissolution rather than a protective effect. This behavior could be attributed to the fact that, at a concentration of 20%, the solution becomes saturated; this prevents the inhibitor molecules from adsorbing effectively onto the metal surface and forming a protective barrier against aggressive ions, resulting in a high corrosion rate—even higher than that of the control. In contrast, the 10% and 15% concentrations shift the (E_{corr}) values toward cathodic regions, with a decrease in current densities,

which led to a notable decrease in the corrosion rate compared to the control of 131.70×10^{-3} to 39.70×10^{-3} and 21.17×10^{-3} at concentrations of 10% and 15%, respectively, reflecting the strong inhibitory effect of the extract in saline solution.

This behavior indicates that a 15% concentration of the extract is the critical threshold for completely saturating the metal surface. Furthermore, the maximum shift in corrosion potential from 44 mV, as measured when the value changed from (-755 mV) in the control solution to (-799 mV), indicates that the extract acts as a mixed-type inhibitor with predominantly cathodic efficiency. Generally, a shift of less than 85 mV in the corrosion potential relative to the reference solution indicates that the inhibitor acts as a mixed-type inhibitor, simultaneously affecting both anodic and cathodic reactions without altering the dominant corrosion mechanism [26].

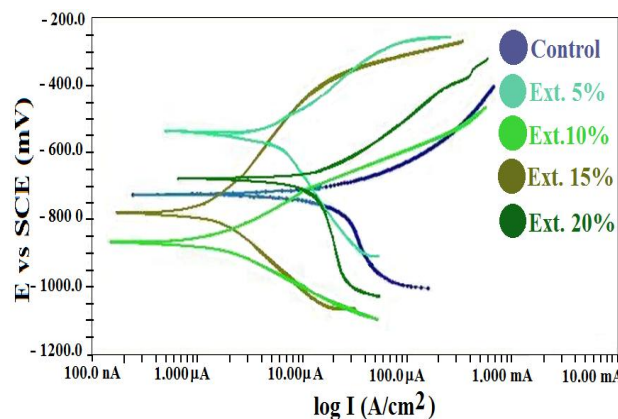


Fig. 2. Polarization curves of N-80 steel in saline solution with different concentrations of crude extract from the pulp of *M. curvispinus*.

Furthermore, Table 1 shows that the inhibition efficiency does not follow a linear trend as the extract concentration increases, due to system collapse at 20%; moderate efficiencies ranging from 60% to 80% were obtained within the optimal ranges of 10% and 15% extract concentration.

Table 1. Electrochemical parameters obtained from polarization measurements of N80 steel in the absence and presence of crude extract from the pulp of *M. curvispinus* in saline medium.

Inhibitor (% v/v)	I_{Corr} (A/cm^2)	E_{Corr} (mV)	V_{Corr} (mmpy)	Efficiency (%IE)
0	11.30×10^{-6}	-755.0	131.70×10^{-3}	-
5	8.78×10^{-6}	-533.0	102.00×10^{-3}	22.55
10	3.42×10^{-6}	-851.0	39.70×10^{-3}	69.86
15	1.82×10^{-6}	-799.0	21.17×10^{-3}	83.89
20	16.10×10^{-6}	-678.0	187.50×10^{-3}	-42.36

Potentiodynamic polarization curves - Acid medium

The polarization curves and electrochemical kinetic parameters of API N80 steel in (H_2SO_4) solution, obtained using the Tafel extrapolation method before and after adding different concentrations of *M. curvispinus* pulp extract, are presented in Figure 3 and Table 2, respectively.

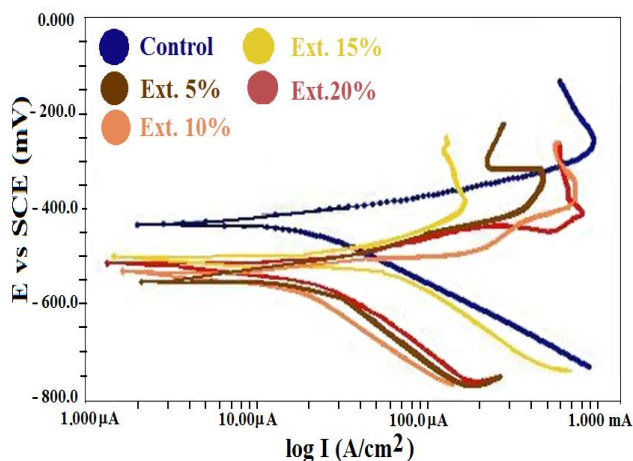


Fig. 3. Polarization curves showing the effect of different concentrations of crude extract of *M. curvispinus* on the potentiodynamic behaviour of N-80 steel in an acidic medium.

Table 2. Corrosion parameters, in the absence and presence of crude extract from *M. curvispinus* pulp, in an acidic medium

Inhibitor (% v/v)	I_{Corr} (A/cm^2)	E_{Corr} (mV)	V_{Corr} (mmpy)	Efficiency (%IE)
0	33.30×10^{-6}	-450.0	375.9×10^{-3}	-
5	32.30×10^{-6}	-538.6	254.4×10^{-3}	32.32
10	23.30×10^{-6}	-538.0	154.4×10^{-3}	58.93
15	11.60×10^{-6}	-529.0	105.3×10^{-3}	71.99
20	10.30×10^{-6}	-538.0	84.4×10^{-3}	77.55

These results show how the presence of the extract in the acidic medium shifts the corrosion potential in the cathodic direction as the inhibitor concentration increases, indicating that cathodic interactions are more effective than anodic ones against steel corrosion [27]. Table 2 also shows a slight variation in the corrosion potentials, which remain within a range of 538 mV to 529 mV in the presence of the inhibitor. Furthermore, the graphs show that the cathodic domains are closely parallel, suggesting that the mechanism of the cathodic reactions is not affected. Likewise, as the inhibitor concentration increased, the shift in the anodic and cathodic branches, accompanied by a progressive reduction in corrosion current density, resulted in a noticeable decrease in the corrosion rate, which fell from 375.9×10^{-3} mmpy (control) to 84.4×10^{-3} mmpy with the addition of 20% of the extract, suggesting the formation of an adsorbed barrier layer that restricted the dissolution of the steel in the aggressive H_2SO_4 solution. Since the corrosion potential shift is 79 mV relative to the control (from -450 mV to -529 mV), the extract is classified as a mixed-type inhibitor with a cathodic predominance under the experimental conditions.

According to the criterion mentioned above: if the shift in E_{Corr} is < 85 relative to the control, the inhibitor is of the mixed type [26]. This 79 mV difference in the (E_{Corr}) potential reinforces the mixed nature of the inhibitor,

suggesting that the extract can control the aggressiveness of the acidic medium through a combination of anodic and cathodic protection mechanisms. In turn, the inhibition efficiency shows an upward trend as the extract concentration increases, reaching a maximum of 78% at a concentration of 20%.

In summary, under the experimental conditions, the electrochemical results obtained validate the potential of *M. curvispinus* extract as a moderately effective green bioinhibitor for N-80 steel in both environments, significantly reducing the corrosion rate in absolute terms in the acidic medium from 375.9×10^{-3} to 84.4×10^{-3}

mmpy. However, the most efficient and stable relative performance is observed in the saline medium at concentrations of 10% and 15%, where the minimum metal degradation rates of 39.70×10^{-3} mmpy and 21.17×10^{-3} mmpy, respectively, are achieved.

Furthermore, a comparative analysis of *M. curvispinus* with recently reported mixed-type green inhibitors for high-strength API-grade steels used in the oil industry in aggressive environments is presented in Table 3.

Tabla 3. Comparación del extracto de la pulpa del *M. curvispinus* con otros inhibidores.

Plant extract	Category of steel	Corrosive media	Inhibitor Efficiency (%IE)	Type of adsorption mechanism	Testing electrochemical	Reference
<i>Melocactus curvispinus</i>	API N80	NaCl (3,5%)	83.89 15% (v/v)	Competitive adsorption	Potentiodynamic Polarization	This study
		1M H ₂ SO ₄	77.75 20% (v/v)	Physisorption (Langmuir)		
<i>Caña panelera</i>	API N80 Q	CO ₂ -saturated saline solution	99.62 500 ppm	*	Potentiodynamic Polarization	28
<i>Guar Gum</i>	API N80	CO ₂ -saturated saline solution	84.68 0.4 g/ L (25 °C) 81.04 0.4 g/ L (50 °C)	Electrostatic or physical (Temkin /Dubinin Radushkevich)	Potentiodynamic Polarization	9
<i>Sclerocarya birrea leaf extract</i>	API 5L X42	1M H ₂ SO ₄	93 20 g/L at 303 K	Physisorption (Temkin)	Weight-loss	29
<i>Solanum villosum</i>	API N80	1M HCl	93.6 100 ppm	physicochemical (Langmuir)	Weight-loss	30
<i>Moringa oleifera Leaves</i>	API 5L X52	1 M HCl	99 0.5 g/L at 60 °C	Physisorption (Langmuir)	Potentiodynamic Polarization	31
<i>Aloe vera Gel</i>	API 5L-B	1 M HCl	42.3 100 ppm 52.0 100 ppm	*	Potentiodynamic Polarization (EIS)	32
<i>Gum arabic</i>	API 5L X60	1 M HCl	90,67 2g/L	*	Potentiodynamic Polarization	33
* Not reported: Adsorption model and inhibitor type						

When comparing the 83.89% efficiency of *M. curvispinus* in a chlorinated medium (3.5% NaCl) with the efficiency of sugarcane (99.62% in CO₂-saturated saline solution)

and guar gum (81.04%–84.68% in CO₂-saturated saline solution), variations can be observed that are influenced by the chemical nature of the extracts and the mechanisms

of surface interaction. An inhibition rate exceeding 80% indicates greater adsorption of organic molecules from the natural extract onto the active metal sites, forming an effective physical barrier against chloride ions (Cl^-).

The high efficiency of sugarcane has been attributed to the concentration of active organic compounds that form a protective film on API N80 Q steel, although the study in question does not specify the adsorption model. Guar gum, for its part, exhibits performance very similar to that of *M. curvispinus* but through a process of physisorption or electrostatic adsorption governed by Temkin and Dubinin–Radushkevich isotherms. In contrast, the present study attributes the inhibition of *M. curvispinus* to specific competitive adsorption between chloride ions (Cl^-) and the organic molecules in the extract on API N80 steel. In acidic media (H_2SO_4 and HCl), *M. curvispinus* exhibits an efficiency of 77.75%, a yield that is higher than that of *Aloe vera gel* (42.3%–52.0%), and below that of other natural extracts such as *Sclerocarya birrea* (93% in H_2SO_4), *Solanum villosum* (93.6% in HCl), *Gum arabic* (90.67% in HCl), and *Moringa oleifera* (99% in HCl). These differences in yield and behavior are explained by the specific phytochemical composition of each extract and its affinity for the metal surface [34]. In this regard: while *M. curvispinus* and extracts of *Sclerocarya birrea* or *Moringa oleifera* clearly exhibit physisorption processes governed by isotherms (such as Langmuir or Temkin), in which organic molecules block active corrosion sites, other studies, such as those on *A. vera* and *gum arabic*, report low efficiencies, even lower than those obtained with *M. curvispinus*, although they do not characterize the adsorption model. These reports confirm that the effectiveness of corrosion inhibition depends on the strength of the adsorption bonds, the coverage of the metal surface by the inhibitor's photochemical molecules, and the stability of the protective layer in the corrosive electrolyte [34].

In summary, *M. curvispinus* has been shown to be a competitive inhibitor compared to other natural extracts,

particularly in saline and moderately acidic environments, with specific mechanisms such as physisorption according to the Langmuir model in acidic media and specific competitive adsorption against chloride (Cl^-) ions in saline media.

Adsorption isotherm

Acid medium

The inhibition efficiency is directly related to the adsorption of molecules from the *M. curvispinus* extract onto the metal surface. To determine the type of adsorption, the fraction of surface area covered (θ) was calculated for different concentrations of the extract using Equation (2) [35]:

$$\theta = \frac{\%IE}{100} \quad \text{ecuación (2)}$$

Where, %IE is the inhibition efficiency percentage reported in Table 2.

The linear relationship between C/θ as a function of C , shown in Figure 4, suggests that the adsorption of the extract followed the Langmuir model adsorption isotherm, which can be expressed by the equation [35]:

$$\frac{C}{\theta} = \frac{1}{K_{\text{ads}}} + C \quad \text{equation (3)}$$

Where C is the concentration of the extract, K_{ads} is the adsorption equilibrium constant, which can be calculated from the intersection of the straight line with the C/θ axis. The value of the linear regression coefficient ($R^2 = 0.9518$) and the determination of a reduced adsorption equilibrium constant ($K_{\text{ads}} = 0.06269$) suggest a weak interaction between the organic molecules and the active sites of N80 steel.

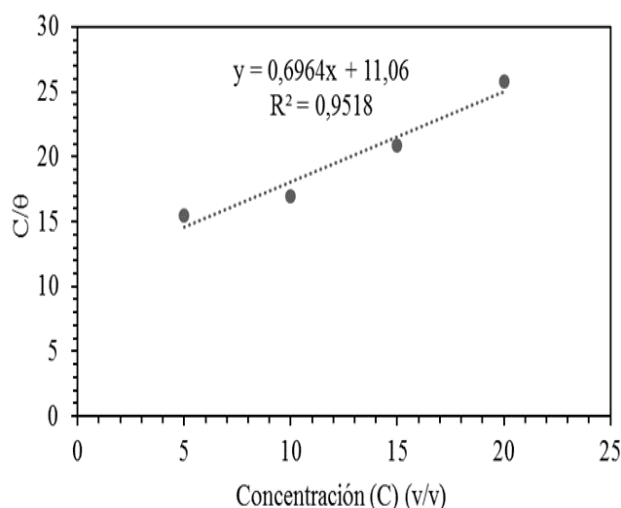


Fig. 4. Langmuir adsorption isotherm plot for the *M. curvispinus* extract in an acidic medium (H_2SO_4) on the surface of N80 steel.

The free energy of adsorption (ΔG_{ads}) of the extract, meanwhile, allows us to determine the nature of the bond between the inhibitor and the metal; this is calculated using the fundamental equation derived from the Langmuir isotherm [35]:

$$\Delta G_{\text{ads}} = -RT \ln(55.5 K) \quad \text{equation (4)}$$

R is the universal gas constant (8.314 J/mol·K), T is the standard test temperature of 298.15 K (25 °C) and the value 55.5 corresponds to the molar concentration of water in the solution. This yields a negative and extremely low value for ΔG_{ads} ($\Delta G_{\text{ads}} \approx -3.09$ kJ/mol), which is consistent with the low value of the equilibrium constant obtained ($K_{\text{ads}} = 0.06269$), indicating spontaneous but weak adsorption of the inhibitor molecules onto the metal surface, which explains the moderate maximum efficiency achieved of just 78%.

According to the literature, values of ΔG_{ads} below the threshold of -20 kJ/mol are associated with physical adsorption or physisorption [35]. Thus, the low value of ΔG_{ads} (≈ -3.09 kJ/mol) obtained confirms that the inhibition process in an acidic medium is governed by physisorption, whereby the molecules of the *M. curvispinus* extract adhere to the surface of N80 steel via weak electrostatic forces (Van der Waals forces) and not via covalent bonds (chemisorption). Therefore, it could be

inferred that the extract protected the steel substrate by forming a protective monolayer.

Saline medium

As shown in Table 2, due to the lack of linearity (the fact that at 20% the extract loses its inhibitory effect and becomes a corrosion promoter) the data do not show a statistically valid correlation for fitting to thermodynamic adsorption isotherm models (e.g., Langmuir or Temkin), which require that the fraction of surface area covered by adsorbed molecules (θ) increases with the concentration of the adsorbate [36]. This behavior suggests that adsorption involves competition between chloride ions and the organic molecules in the extract on the metal surface, which makes it difficult to directly apply the ideal Langmuir model.

Characterization by scanning electron microscopy complemented by energy-dispersive X-ray spectroscopy (SEM/EDX)

Saline medium

Figure 5 shows scanning electron micrographs of the surface of N-80 steel after 10 days of immersion in a saline solution, in the absence and presence of the optimal concentration (15%) of the crude extract of *M. curvispinus*. In the absence of the inhibitor, the surface exhibits continuous deterioration, characterized by the heterogeneous accumulation of corrosion products and scattered pitting (Figure 5a). Some of these pitting defects are associated with inclusions in the steel, as detailed previously and shown in Figure 5b. The micrograph also shows the approximate morphology of the pitting defects in terms of area, perimeter, and length. The elemental chemical analysis of the matrix and the inclusion are presented in Figures 5c and 5d, respectively. At a concentration of 15%, a highly efficient protective film is formed that physically blocks the mass transport of key corrosive agents, such as oxygen, water, and chloride ions, to the metal substrate. Furthermore, this film prevents the

initial oxide or rust crystals from adhering to the substrate during the early stages of the metal's degradation process. The SEM micrograph shown in Figure 5e confirms the microscopic existence of this continuous protective layer. Microstructural analysis at higher magnification, shown in Figure 5f, reveals that the protective layer exhibits morphological fluctuations and microdomains of lower electron density (dark regions indicating areas of lower atomic mass), characterized by variations in the film's thickness.

This lack of uniformity suggests that the metal/solution interface is governed by strong dynamic competition between chloride ions and the organic molecules in the

extract, which limits the formation of a uniform structure. Despite this local heterogeneity, the protective layer provides remarkable long-term resistance, allowing the steel to withstand prolonged periods of immersion without suffering severe chemical degradation. In turn, inspection at higher magnification reveals the presence of a few pitting lesions in the less dense areas of the protective layer (dark areas). The area ($448.40 \mu\text{m}^2$), perimeter ($99.38 \mu\text{m}$), and length ($30.11 \mu\text{m}$) of the pitting highlighted in Figure 5f are smaller than the average values recorded for the area ($1688.99 \mu\text{m}^2$), the perimeter ($125.28 \mu\text{m}$), and the length ($41.30 \mu\text{m}$) of several bites analyzed in the absence of the inhibitor.

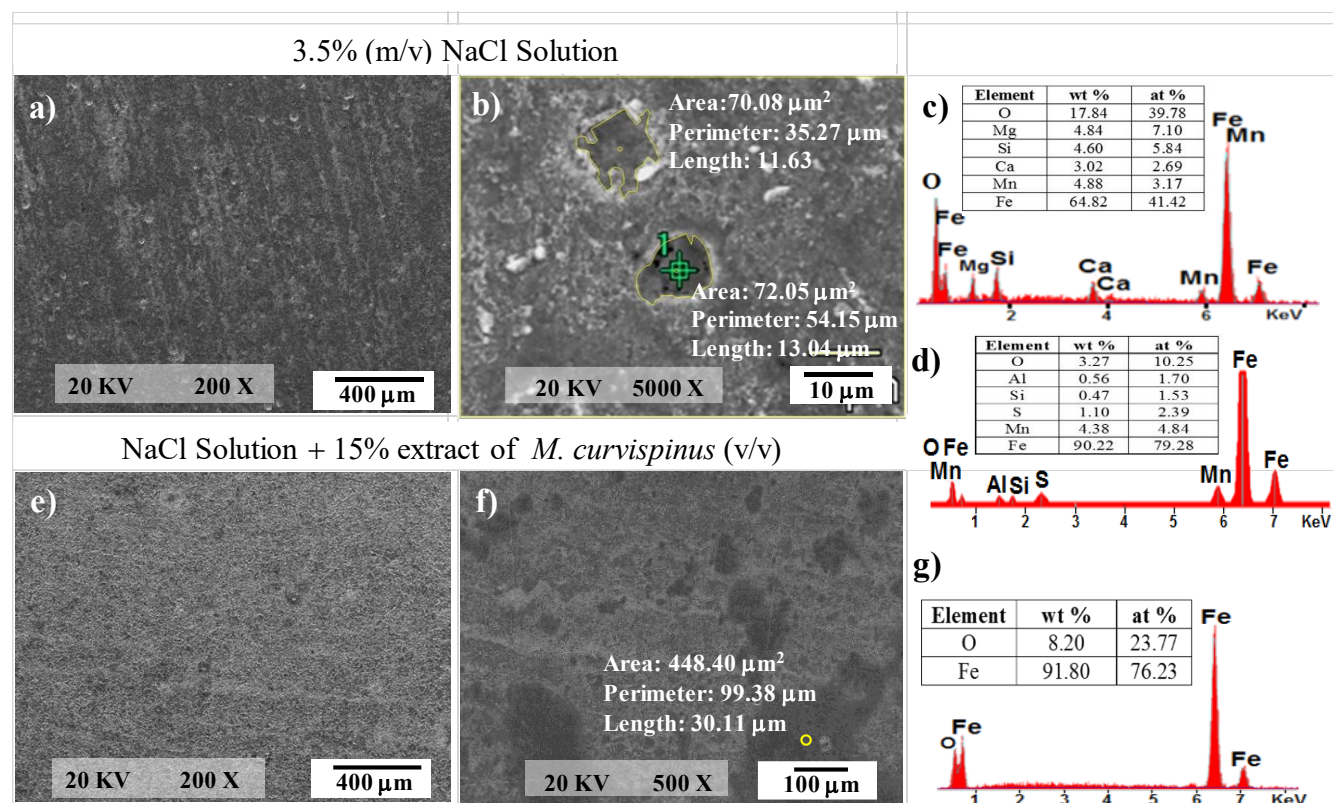


Figure 5. SEM micrographs and EDX chemical microanalysis of the surface of N-80 steel immersed for 10 days in saline solution: (a-d) without inhibitor and (e-g) with 15% extract of *M. curvispinus* pulp.

Acid medium

Figure 6 presents the comparative morphological analysis of the N-80 steel surface after ten days of immersion in an acidic medium (H_2SO_4), evaluated in the absence and presence of the optimal concentration (20% v/v) of the crude extract of *M. curvispinus*. In the absence of the

inhibitor (Figures 6a and 6b), severe degradation of the metal substrate is observed, characterized by corrosion products with a rough morphology, as well as deep and extensive pitting.

This phenomenon results from the synergistic action of the intrinsic aggressiveness of sulfate ions and the kinetics of

the hydrogen evolution reaction in highly acidic environments, which causes rapid surface degradation of the steel. In contrast, the micrographs in Figures 6d and 6e reveal the protective effect of the crude extract of *M. curvispinus*. A thin protective film was observed to form, along with a dramatic reduction in the size of the puncture wounds compared to the control group, with a decrease in the average area of 25011.90 μm^2 , perimeter of 1017.36 μm , and length of 381.03 μm for the puncture wounds analyzed in the control group, to average values of 2372.72 μm^2 in area, 242.36 μm in perimeter, and 77.97

μm in length for the bites in the presence of the inhibitor. This suggests that the coating formed by the components of the extract stabilizes the metal/solution interface and significantly suppresses the corrosion process. This is because the organic molecules are easily protonated, promoting an ordered monolayer arrangement on the metal surface. This barrier effect translates quantitatively into a 77.5% reduction in the corrosion rate, which drops from 375.9×10^{-3} mmpy to 84.4×10^{-3} mmpy. Finally, the elemental composition profiles, obtained via EDX, are presented in Figures 6c (control) and 6f (with inhibitor).

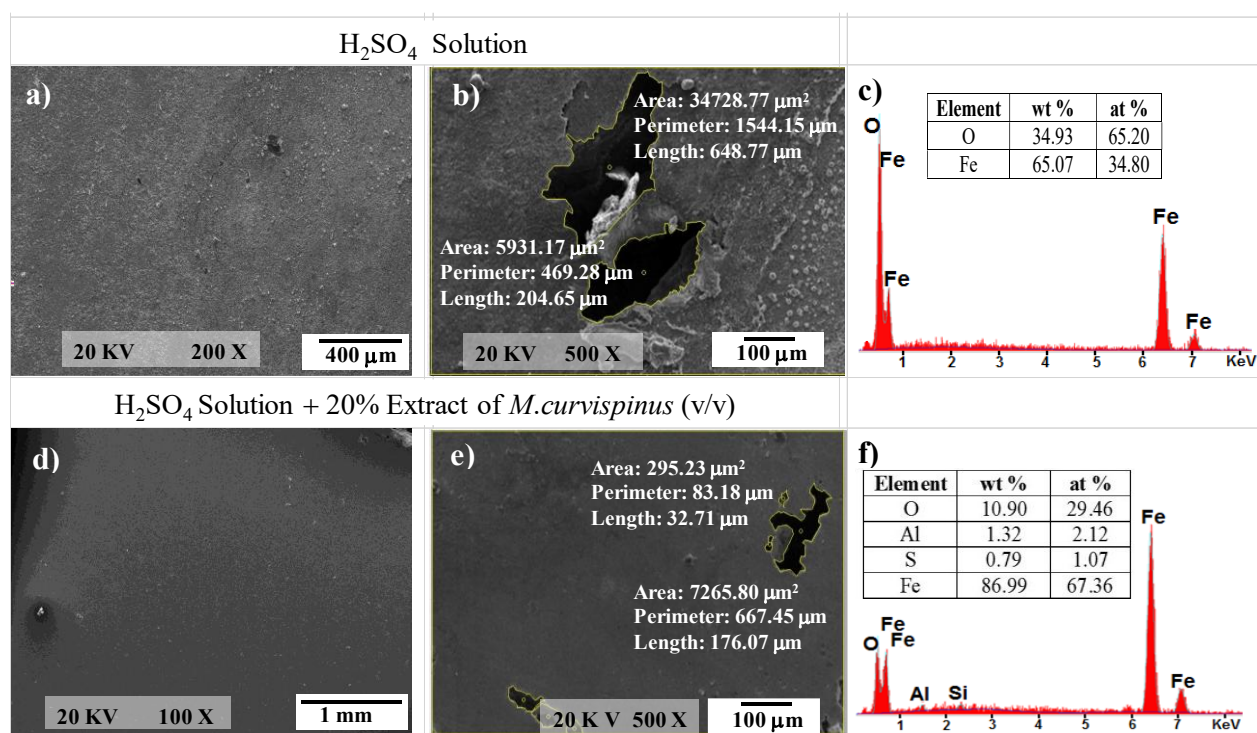


Fig. 6. SEM micrographs and EDX chemical microanalysis of the surface of N-80 steel immersed for 10 days in H₂SO₄ solution: without inhibitor (a-c) and with 20% *M. curvispinus* pulp extract (d-f).

Table 4 reports the EDX analysis obtained from the examined surfaces and the Fe/O ratio. In the presence of the inhibitor, the Fe/O ratio reflects the degree of protection provided by the adsorbed films against aggressive species in the medium. The highest value for this ratio was obtained in the saline medium, followed by the acidic medium. This is because an increase in the thickness of the protective film reduces its oxygen content. Thus, it is confirmed that the dense, compact layer formed in the saline environment

provides better protection for the steel than that achieved in the acidic environment. Conversely, in the absence of the inhibitor, the lower Fe/O ratio indicates greater deterioration of the metal surface. Under these conditions, the lowest value was obtained in the acidic medium. This behavior was expected, given that this medium is more aggressive than the saline medium and causes greater pitting corrosion damage to the steel. It also promotes the formation of a denser layer of corrosion products, as

evidenced by the scanning electron micrographs shown in Figures 6a and 6b.

Table 4. EDX analysis of surfaces exposed to corrosive medium

Solution	Elements (% wt)			Relation Fe/O (%)
	O	Fe	Balance	
NaCl	17.8 4	64.8 2	Mg: 4.84 Si: 4.60 Ca: 3.02 Mn: 4.88	3.63
NaCl + 15% Extract	8.20	91.8 0	--	11.20
H ₂ SO ₄	34.9 3	65.0 7	--	1.81
H ₂ SO ₄ + 20% Extract	10.9 0	86.9 9	Al: 1.32 S: 0.79	7.98

SEM/EDX characterization reveals differences in thickness and morphology in the protective films formed in both aggressive media in the presence of the inhibitor. The cause of these characteristics has been linked to the solubility of the inhibitor in the corrosive medium and the dynamics of the interface, where the adsorption of organic molecules requires the prior desorption of water molecules [37]. In a saline medium, the dense protective film adsorbed on the metal surface indicates that the inhibitor is more soluble than in an acidic medium. In this case, the lower surface charge density facilitates more efficient desorption of water, allowing a greater fraction of molecules from the extract to adsorb on the metal surface, resulting in a thicker protective film with a heterogeneous relief [38]. In contrast, in the acidic medium, intense solvation and strong competition for active sites limit the displacement of water molecules, resulting in a thinner and more uniform adsorbed film, which is consistent with the nature of weak physisorption and the low value of the equilibrium constant (K_{ads}) obtained by fitting the Langmuir model. These results support the interpretation of the electrochemical

measurements: the denser the protective film, the lower the corrosion rate, confirming that organic adsorption films play a crucial role in the degradation of steel in aggressive environments.

Fourier transform infrared spectroscopy (FT-IR)

The FT-IR spectra recorded between 4000 and 450 cm^{-1} of the fractions obtained from the crude extract are shown in Figure 7. Comparison of the signals from each spectrum with the average theoretical FTIR values [39], allows specific relationships to be established with the functional groups of each type of metabolite identified in the extract studied.

The broad band at 3401 cm^{-1} in the acetone and methanol fractions is indicative of carboxylic acids. The peaks around 2925 and 2854 cm^{-1} identified in all spectra correspond to characteristic stretching and twisting of C-H methylene groups. The observed bands at 2319 and 2355 cm^{-1} are signals of nitrile groups and are consistent with the identification of alkaloid compounds in preliminary chemical tests, in the spectrum of the extract obtained with ethyl acetate.

A band between 1741.27 and 1716.44 cm^{-1} representative of the carboxylic group C=O is also evident in all spectra, except for the fraction separated with chloroform. Another characteristic peak of the methylene group is reflected at 1638.18 cm^{-1} in the chloroform fraction and at 1642.64 cm^{-1} in the acetone extract. The signal present only in the methanol extract at 1607.84 cm^{-1} is indicative of C=C or C=N groups. Similarly, the band at 1457.06, 1463.53, and 1457.87 cm^{-1} in the chloroform, hexane, ethyl acetate, and acetone extracts, respectively, is assigned to the methylene group.

A new band belonging to carboxylic acids appears at 1377.96, 1384.16, and 1393.82 cm^{-1} in the hexane, acetone, and ethyl acetate fractions, respectively. Meanwhile, the bands appearing at 1079.94 cm^{-1} ; 1076 cm^{-1} and 1042.70 cm^{-1} ; 1076.30 cm^{-1} , and 1045.05 cm^{-1} in the spectra for the chloroform, methanol, and acetone fractions, respectively, corroborate the existence of C-O bonds of alcoholic groups

and indicate the presence of C-O bonds of sugar units (carbohydrates), which were mentioned in association with saponins detected in the preliminary chemical analysis. Finally, the peaks recorded at 721.18 cm^{-1} and between 620.78 cm^{-1} and 637.35 cm^{-1} in the fractions separated with chloroform, acetone, and ethyl acetate correspond to the vibrations of N-H.

The identification of these functional groups confirms the presence of triterpene metabolites, alkaloids, coumarins, and saponins in the *M. curvispinus* extract detected in the phytochemical analysis; these compounds are characterized by the presence of heteroatoms (O, N) and conjugated double-bond systems. It is likely that the oxygen-containing functional groups of coumarins and saponins interact with the empty iron orbitals by donating pairs of free electrons, while hydrogen bonds may help stabilize the adsorbed layer.

For their part, pentacyclic triterpenes may promote an increase in the hydrophobicity and compactness of the protective film, which would limit the diffusion of acidic species and ions (Cl^-) toward the metal surface. Nitrogen-containing alkaloids tend to enhance this protective effect by promoting adsorption through potential donor-acceptor

interactions with the metal substrate [34, 40]. In this context, it is difficult to attribute the anticorrosive effect to a specific metabolite. Therefore, our laboratory will conduct further research to isolate the bioactive compounds and evaluate their inhibitory capacity.

In summary, the study validates the pulp extract of *M. curvispinus* as a functional bio-inhibitor for N-80 steel in the environments analyzed, although its use is limited by the nature of its physical adsorption and by industrial volume requirements. Therefore, future research will focus on optimizing the dosage by studying synergistic effects with inorganic halides or low-toxicity polymeric surfactants, as well as isolating the active components of the extract and testing their inhibitory capacity through the use of polymeric nano-encapsulation techniques for their controlled release, as pathways to transform this natural resource into a competitive corrosion technology aligned with green chemistry standards and offering superior economic viability in large-scale systems, characterized by its simplified, solvent-free extraction process that minimizes initial processing costs and the associated environmental impact.

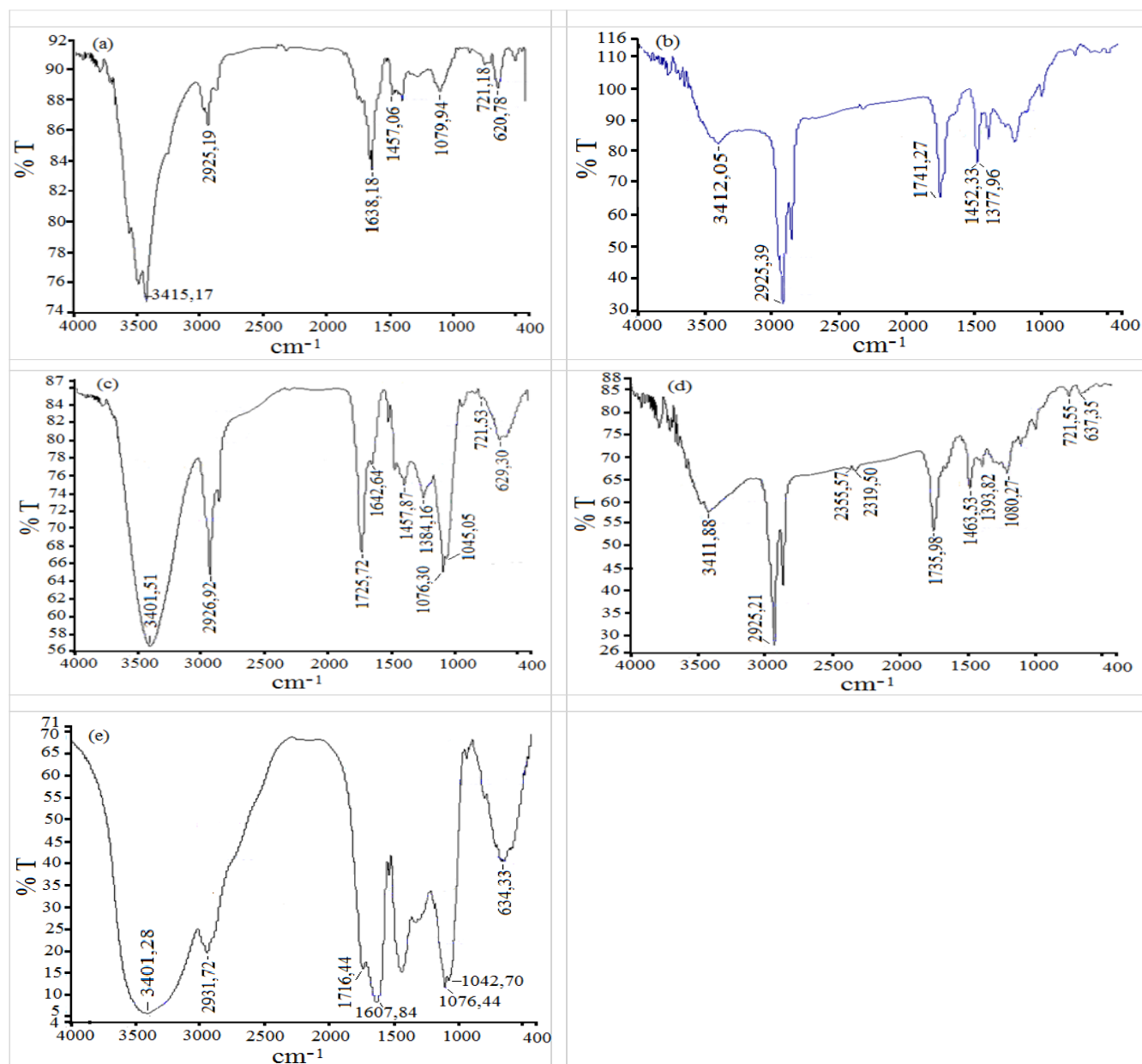


Fig. 7. FTIR spectra of the fractions of the *M. curvispinus* pulp extract obtained with organic solvents: (a) chloroform, (b) hexane, (c) acetone, (d) ethyl acetate, and (e) methanol.

CONCLUSIONS

The results obtained from the electrochemical tests shows that the crude extract of *M. curvispinus* pulp acted as an inhibitor of API N-80 steel corrosion in NaCl and H₂SO₄ solutions, through mixed inhibition mechanisms.

An absolute reduction in the corrosion rate in an acidic medium was observed, from 375.9×10^{-3} to 84.4×10^{-3} mmpy, while in a saline medium the lowest rate of degradation (21.17×10^{-3} mmpy) was recorded at a concentration of 15% v/v.

In the saline medium, maximum efficiency was achieved at 15% v/v, with the system becoming saturated at a concentration of 20%, thus defining the operational limit of the crude extract in this environment. In contrast, in the acidic medium, maximum inhibition efficiency was obtained at a concentration of 20%, conforming to the Langmuir isotherm via a physisorption mechanism.

SEM observations revealed the formation of a dense, compact film with a heterogeneous surface in the saline

medium, whereas in the acidic medium the adsorbed film was thin and uniform.

FTIR analysis confirmed the presence of triterpenes, alkaloids, coumarins and saponins, which had been identified by phytochemical tests.

M. curvispinus extract represents a promising ‘Green Chemistry’ approach for protecting N80-grade steel in both aggressive environments; however, its large-scale industrial viability requires optimization strategies such as nano-encapsulation to reduce current volume requirements.

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