CHARACTERIZATION AND EVALUATION OF THE NATURAL EXTRACTS OF POTENTIAL INTEREST FOR THE ANTICORROSIVE INDUSTRY: *Opuntia ficus indica*

Prato, M. del R.; Gonzalez, A.T.; Reyes, R.
Centro de Investigaciones Tecnológicas-CITEC. Universidad Nacional Experimental Francisco de Miranda, Coro, Falcón, Venezuela.

Abstract

Experimental results were used to investigate the application of naturally occurring substances in physical-chemical corrosion inhibition processes. Anticorrosive tests were conducted in a 3.5% saline and 1N acid solutions. The inhibition efficiencies ranging from 20-57% were obtained in saline solutions, and 80-87% in acid solutions, in the presence of 5% *Opuntia ficus indica* extracts using the "weight-loss" technique.

Resumen

Se utilizó una serie de resultados experimentales para investigar la aplicación de sustancias naturales en procesos físico-químicos inhibidores de la corrosión. Se realizaron ensayos anticorrosivos en soluciones salinas al 3.5% y soluciones ácidas 1N. Se obtuvo eficiencias del proceso inhibidor de la corrosión entre 20-57% para soluciones salinas, y entre 80-87% para soluciones ácidas, en presencia de extractos de *Opuntia ficus indica* al 5%, utilizando la técnica de "pérdida de peso" *(weight-loss).*

I. INTRODUCTION

In agreement with the reports of some authors, there exist some compounds (in plants of semiarid zones) which produce a delay action of corrosive processes [1-5]. In the present case it is assumed that the ***Opuntia*** possess the same characteristics of those encountered in the Aloe-resin.

The Falcon state is a semiarid region of Venezuela which possesses a xerophyt vegetation presenting constitution substances that have inhibitory characteristics of corrosive processes. This fact has withdrawn great interest in the world's scientific community.

The researchers currently involved in this field, have been previously studying another commercial plant of great use in the Falcon state: the ***Aloe barbadensis*** or ***Aloe vera.*** The results yielded an amazing expectancy about the anticorrosive capacities of the extract (resin), so confirming the use that this plant had been given (metal preservation).

In some regions of Venezuela there exists a high index of material damage, due to the high temperatures and humid environment. Besides, the coast extension of about 2000 Km is a factor that makes a large part of the Venezuelan territory a very corrosive environment. The importance of the studies herein reported are evident if one is going to take into account its contribution to lessen damages and costs due to environment corrosion and industrial effluents (HCl, H_2SO_4) and also, because it constitutes saving, which can converted in a profit generator and obtain a stable product of multiple use in respect to metals preservation (metallic constructions, historical monuments, industry equipment).

Actually there exists an increase of studies of this nature and basically on tropical plants [1-5]. The interest of this type of investigation was verified in the IX Ibero American Congress of Electrochemistry, where different authors showed the results of their investigations about the anticorrosive properties from natural substances, like the ***Aloe vera*** case.

The experience and success obtained in previous investigations [12-13] promoted similar investigations of extracts on plants from regions as the falconian. This also constitutes the second study in the case of ***Opuntia ficus indica*** (Spain tune) extract. This plant is found among the common plants of Venezuela [14], it is widely distributed and cultivated in all tropical and subtropical regions of the world. Right now this plant has little use, besides it is of an eminent interest to establish its potential. The results will be relevant tools to attack many corrosive problems, especially where the crop exists.

The studies realized abroad and in Venezuela, with natural extracts of any xerophyt plants that exist as wild and crop form in the Falcon State, Venezuela; case ***Aloe vera*** and ***Opuntia ficus indica***, commonly known as Spain Tune, demonstrate that these extracts possess in its constitution, substances with inhibiting characteristics to corrosive process, making it possible that experimentally we try to

replace the commercial inhibitor denominated hexamethylene tetramine ($C_6H_{12}N_4$), of a acid solution (50% HCl), by Opuntia extracts, in cleaning solutions of steels.

This solution is used for chemical cleanliness for removal of corrosion products of steel, through the standard methods of weight-loss study to determine corrosion rate, where ASTM norms are mostly used. For steel cases, it is recommended the solution denominated CLARK; there also exist other methods as the presented by the International Organization for Standardization ISO [15]. The ISO/DIS 8407 is a Norm used in this work and specifies the use of $C_6H_{12}N_4$ as inhibitor; being cheaper, due to a less acid volume used and a cleaning time of 2.5 rounds less than the used to clean steel samples.

II. METHODOLOGY

The ***Opuntia ficus indica***, extracts were obtained by the extraction process outlined in the flow diagram of the process of Fig. 1.

The physico-chemical evaluation were made by triplicate (moisture contain, pH, % of ashes, and % of proteins) using the analysis of the Association of Official Analytical Chemist A.O.A.C., and the evaluation of the density according to ASTM16.

We also realized qualitative tests on detecting the presence of tannins, alkaloids and phenol compounds, utilizing methodology according to Marcano and Hasegawa [17].

Corrosion tests by gravimetry were used at two intervals of time (one and two weeks), to evaluate the anti corrosive effect of the Opuntia extracts. The tests were made on six samples of steel an approximately area of 5 cm^2, changing periodically the corrosive environment (HCl 1N and H_2SO_4 1N) and NaCl solution 3.5%.

The chemical cleanliness tests by the Isocorrag method, were used incorporating the Opuntia extracts to replace $C_6H_{12}N_4$ in the cleaning of 80 cm^2 steel samples previously corroded.

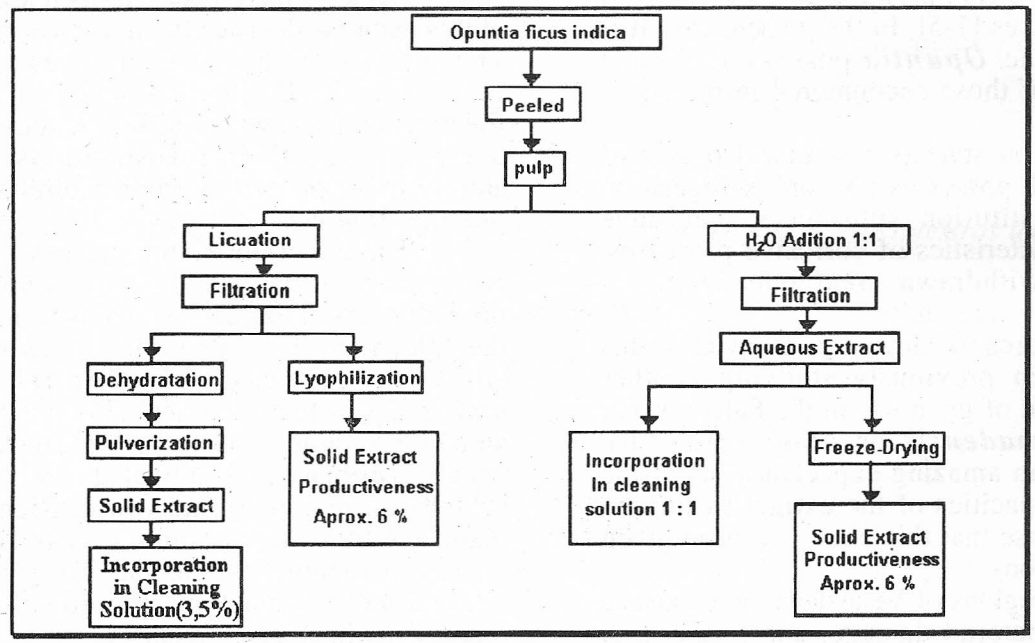

Fig 1. Flow diagram of the process to obtain the extract of ***opuntia ficus indica***.

The methodology employed consisted basically in obtaining the appropriate extract for the corrosion tests, followed by tests on steel, where diverse corrosive solutions (HCl, H_2SO_4, NaCl and NaOH) were introduced with the extract in very low amounts, making it more competitive with others inhibitors of commercial use according to specifications of ASTM 16 or ISO/DIS8407 [15].

III. RESULTS

The diverse extracts obtained are described in Table 1. Table 2 presents the results of the physico-chemicals tests; it is observed that the extract have acidity character, showing the qualitative test of tannins, alkaloids and phenol compounds.

Table 1. Description of the diverse extracts of ***Opuntia*** for use as inhibitor in the cleaning solution of steel.

EXTRACTS OF ***Opuntia Ficus indica***	**DESCRIPTION**
AQUEOUS EXTRACT	Liquid extract obtained by extraction and filtrate in water to room temperature.
SOLID EXTRACT	Dry extract obtained by extraction of the pulp, mixed (without addition of water) filtrate, dried and pulverized.
LYOPHILIZED EXTRACT	Extract obtained by freeze-drying of the solid and aqueous extracts.

Table 2. Physico-Chemical results of the ***Opuntia ficus indica*** extracts.

PARAMETERS	**TYPES OF EXTRACTS**		
	AQUEOUS	**SOLID**	**LYOPHILIZED**
DENSITY g.cm-3	0.99	0.84*	-
MOIST %	99.55	9.53	9.89
pH	4.65	4.60*	-
ASHES %	13.64	24.10	21.34
PROTEINS	-	0.23	-
	QUALITATIVE TESTS		
TANNINS	+	+	+
ALKALOIDS	+	+	+
PHENOL COMPOUNDS	+	+	+

*In solution 6% P/V.

The anticorrosive effect of aqueous and solid extracts of Opuntia is, in all cases, in acid environment, not occurring in similar way for the NaCl solution. The percentage of inhibition to the anticorrosive action of the extracts of Opuntia, was obtained in all acid environments between a 80% to 87% of efficiency; and in saline environment, between a 20% to 57%. This inhibition efficiency of the extract is visualized in Fig. 2 and Fig. 3, being present for both periods of one and two weeks and in a comparative form the anticorrosive action as the inhibition percentage. A totally of thirty corrosion tests were realized in the conditions described before.

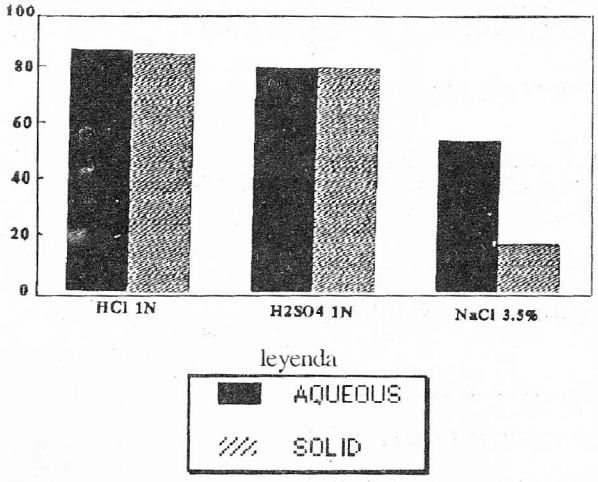

Fig.2 Valoration of inhibitive power of Opuntia during one week of exposition.

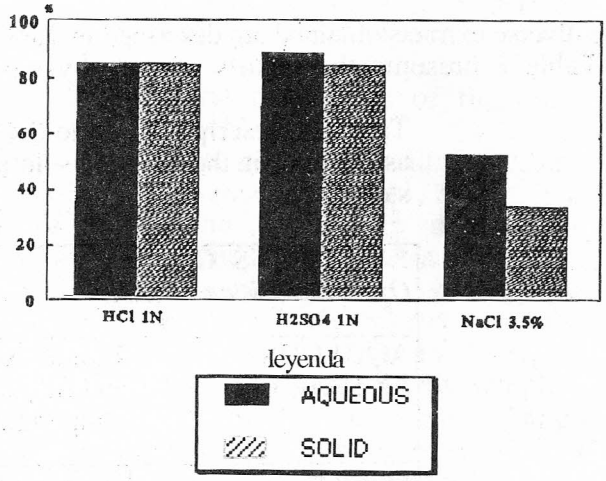

Fig.3 Valoration of inhibitive power of Opuntia extract during two weeks of exposition.

IV. RESULTS OF THE CHEMICAL CLEANLINESS PROCESS FROM THE CORRODED STEEL

The cleaning solutions for corroded steel used with Opuntia extract acting as inhibitor, are described in Table 3. The Opuntia extracts were used in identical conditions as above, replacing the $C_6H_{12}N_4$ inhibitor of commercial use.

Table 3. Description of cleaning solution.

CLEANING SOLUTION	DESCRIPTION
STANDARD SOLUTION	500 ml HCl concentrated 3.5 g $C_6H_{12}N_4$ completed with water up to 1 liter
SOLUTION WITH SOLID OPUNTIA EXTRACT	500 ml HCl concentrated 3.5 g solid Opuntia completed with water up to 1 liter
SOLUTION OF AQUEOUS OPUNTIA EXTRACT	500 ml HCl concentrated 500 ml aqueous Opuntia extracts
SOLUTION WITH LYOPHILIZED OPUNTIA EXTRACT	500 ml HCl concentrated 3.5 g solid Opuntia lyophilized completed with water up to 1 liter

Figure 4 presents the results of a chemical cleanliness cycle according to method ISO/DIS8407 [15]. It is observed the change the steel weight. It is obtained a smaller value with the extracts, related to a minor drag from the metal base (steel). It was also observed for each case of chemical cleanliness, a metallic surface more polished from corrosive products and a better appearance from the metal base, indicating a lesser attack by the HCl used in the cleaning.

Figure 4 Weight-loss from the corroded steel using standard solution and replacing C6H12N4 by Opuntia extract.

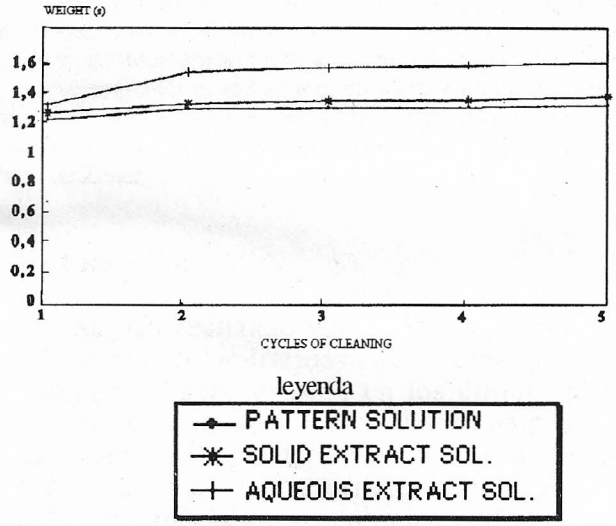

Fig. 4 Weight-loss from the corroded steel using standard solution and replacing C6H12N4 by Opuntia extract.

V. CONCLUSIONS

The extracts of ***Opuntia ficus indica*** produce a delay effect of the corrosion rate of steel in acid environments. The inhibition effect produced was 87% in solutions 1N of HCl and 1N of H_2SO_4.

Even for the case of saline solutions the effect is smaller than in acid (up to 56%), indicating that in this environment the extract would also have a use as an anticorrosive.

The extracts even in solid form or as aqueous extracts produce a similar corrosion inhibition efficiency as in the case of acid environments. However, for saline environments, the aqueous extract presents twice the efficiency with respect to the extract as solid form. It is believed that it is due to absorption of a thin film over the metallic surface.

Extract the ***Opuntia ficus indica*** can be used as a substitute inhibitor in cleaning solutions of steels, based on:

- more drag of corrosive products in each cleanliness cycle and a lower attack or drag from the metallic base. A better appearance of the metallic surface (white metal) is also obtained.
- the efficiency with respect to the hexamethylene tetramine in the final cleaning, is found to be between 20 to 25% using the extracts;
- it is not a volatile inhibitor and does not have inclination to create foam or emulsions (in acid environments);
- has less cost of cleaning process.

VI. ACKNOWLEDGMENTS

We appreciate the Investigation Council of the Experimental National University Francisco de Miranda (CTI-UNEFM), and the Foundation for Science and Technology Development (FUNDACITE-Falcon) for the logistic support conferred. We also want to give recognition to Mr. Rafael Medina for his technical contribution in this work.

VII. REFERENCES

1. R. M. Saleh, A. A.Ismail and A. A. El Hosary, *Br. Corros. J.* **17**, 131-135 (1982).

2. L.L. Shreir, *Corrosion*, 2nd Edn. **2**, 18.13 (1977).

3. A. Nordal and G. Ogner, *Acta Chem. Scand.* **18**, (1979).

4. M. Tezuka, Y. Tomoe and S. Fujii, *7th Int. Cong. Metall .Corrosion,* Brasil, 1850 (1978).

5. E.G. Sola, G. Niveyro, y M. Voldez, *7th Int. Congr. Metall .Corrosion,* Brasil,1901 (1978).

6. M. del R. Prato de Arias, M. Fernandez, S. Llanos, and A. Rincón, *Memorias II Congreso Iberoamericano de Corrosión y Protección,* **1**, 413. Maracaibo (1986).

7. M. del R. Prato de Arias, M. Fernández, and A. Rincón, *Latin American Journal of Metallurgy and Materials,* **14**, 1 (1994).

8. M. del R. Prato de Arias, M. Fernandez, A. Rincón, *Latin American Journal of Metallurgy and Materials,* **4**, 2 (1984).

9. M. del R. Prato de Arias, *Memoria del III Congreso Iberoamericano de Corrosión y Protección,* **3**, 1658. Rio de Janeiro (1989).

10. M. del R. Prato de Arias, Memorias IX Congreso Iberoamericano de Electroquímica, **571**, Tenerife (1990).

11. R. Arias, H. Petit, M. del R. Prato de Arias, A.T. González, Memoria del IX Congreso Iberoamericano de Electroquímica, **568**, Tenerife (1990).

12. M. del R. Prato de Arias, A.T. González, R. Reyes, *Memorias II Coloquio sobre Revestimientos Industriales*, Maracaibo, Venezuela (1990).

13. M. del R. Prato de Arias, A.T. González, Memoria III Jornadas de Corrosión, **14**, Caracas (1988).

14. L.Schnee, Colección de Ciencias Biológicas, Ed. Biblioteca UCV, **706**, Caracas (1984).

15. ISO/DIS8407 Draft International Standard.

16. Annual Book of ASTM Standards, **03.02** ASTM (1987).

17. D. Marcano and M. Hasegawa, Fitoquímica Orgánica, Ed. UCV, **20-23**, Caracas, Venezuela (1991).

18. M. del R. Prato, A.T. González, Revista Técnica de Ingeniería, Universidad de Zulia, **16:1**, 59-62 (1993).