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ORIGINAL RESEARCH PAPER

EVALUATION OF THE CORROSION INHIBITION POTENTIAL OF *RAPHANUS SATIVUS* AND *SPINACIA OLERACEEA* EXTRACTS PART II: MILD STEEL CORROSION INHIBITION BY *RAPHANUS SATIVUS* AND *SPINACIA OLERACEA* EXTRACTS AS GREEN CORROSION INHIBITORS

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Abstract: Our study presents the investigation of the inhibitive performances of *Spinacia oleracea* (spinach) and *Raphanus sativus* (radish) hydroalcoholic leaves extracts on mild steel corrosion in acidic environments.
The spinach and radish leaves extracts were obtained under green conditions (microwave assisted extraction and ethanol as biodegradable solvent). The extracts were added in the corrosive solutions at a concentration of 0.5 $g \cdot L^{-1}$. The inhibition of the corrosion of mild steel in H_2SO_4 solution by the plant extracts has been studied using the weight loss method. Both extracts inhibit steel corrosion in 0.1 M H_2SO_4, but *Raphanus sativus* extract proved slightly higher corrosion inhibition efficiency (78 %) than *Spinacia oleracea* the extract (75 %).

Keywords: *green corrosion inhibitors, mild steel, Raphanus sativus, Spinacia oleracea, weight loss method*

INTRODUCTION

Pure metals and alloys react with corrosive medium to form a stable compound with metal loss. The formed compounds are called corrosion compounds and the metal surface becomes corroded. The corrosion can be general, the entire surface of the metal is attacked at the same rate, or local, an intense attack is visible at localized sites on the surface, the rest of the surface being corroded at lower rates.

Among the several methods of corrosion control and prevention, the use of corrosion inhibitors is very popular. Corrosion inhibitors are substances which, when added in small concentrations to corrosive media, decrease or prevent metal corrosion [1]. Inhibitors function by adsorption of ions or molecules onto the metal surface. They reduce the corrosion rate by:

- decreasing the diffusion rate for reactants to the surface of the metal;
- decreasing the electrical resistance of the metal surface.

A broad spectrum of organic compounds is available as corrosion inhibitors, however, the successful utilization of most corrosion inhibitors has been hindered by their toxic nature [2]. The known hazard effects of most synthetic corrosion inhibitors are the motivation for the orientation to natural products that can be used as green inhibitors [3].

Lately, plant extracts become the center of attention as an environmentally acceptable, readily available and renewable source for a wide range of needed inhibitors. Plant extracts are viewed as a rich source of naturally synthesized chemical compounds that can be extracted by simple procedures with low costs [3].

Several studies have been carried out using natural products as corrosion inhibitors for metals such as mild steel [4 – 10], copper [11], aluminum [12 – 16] and zinc [17, 18] and different plants: *Azadirachta indica* [19], *Allium sativum* [20], *Chamaemelum mixtum* L., *Cymbopogon proximus*, *Nigella sativa* L. and *Phaseolus vulgaris* L. [8], *Chinese gooseberry* [21], Brahmi (Bacopamonnieri) and Henna [22], green tea [23], *Lemon Balm* [24], *Saraca ashoka* [25], *Rollinia occidentalis* [26], *Lupinous albus* L. [27] or *Luffa cylindrica* [28], if is to name only some of them. Thus, it was found that inhibitors function by adsorption of ions or molecules onto the metal surface. The aromatic rings and heteroatoms (S, N, O, P, etc.) are the adsorption centers for the green corrosion inhibitors [29]. The literature mentions the corrosion inhibition effects of saponins, tannins, alkaloids, steroids, vitamins, glycosides and amino acids [1, 30, 31].

Radish (*Raphanus sativus*) and spinach (*Spinacia oleracea)* leaves present in their composition: ascorbic acid, folic acid, vitamin A or flavonoids [32, 33], compounds that have proven corrosion inhibition properties [31]. In their paper, Shivakumar *et al.* showed that the corrosion rate of mild steel decreased with the increase of the spinach leaves extract concentration in hydrochloric acid media [34]. Also, recently, papers have explored the inhibition potential of radish extracts for the protection of mild steel in 0.5 M sulfuric acid [35] and tap water [36] showing good results regarding the corrosion inhibition efficiency.

The aim of the present paper was the investigation of the inhibitive performances of *Spinacia oleracea* (spinach) and *Raphanus sativus* (radish) hydroalcoholic leaves extracts on mild steel corrosion in sulfuric acid environment.

MATERIALS AND MHETODS

Reagents

Ethanol 96 % and acetone were purchased from Chemical Company (Iasi, Romania). Analytical grade sulfuric acid (H_2SO_4) and hydrochloric acid (HCl) were purchased from Sigma Aldrich (Germany).
0.1 M H_2SO_4 solution, used as corrosive media, was prepared by diluting the appropriately calculated acid volume with distilled water.
Spinach and radish extracts were prepared as previously described [37]. For the corrosion inhibition efficiency tests, 0.5 g of extract were dissolved in 1 L of 0.1 M H_2SO_4 solution.

Corrosion measurement

Gravimetric method
The gravimetric (weight loss) method is the simplest and longest-established technique to calculate the corrosion rate [31]. It is also and one of the most popular and accurate methods used for metal corrosion measurement [3, 4, 7, 26, 28, 30, 34, 35, 38]. It involves the measurement of the mass of an experimental coupon before and after its exposure to a corrosive media.
Rectangular coupons of mild steel with dimensions of 7.5 × 13 × 2 mm were used. The steel composition was: 0.16 % C, 0.59 % Mn, 0.24 % Si, 0.16 % P, 0.015 % S and the rest up to 100 % is represented by iron. They were immersed in 0.1 M H_2SO_4 in the presence and the absence (control) of the plant extracts, at room temperature, suspended with plastic wires so that they don’t touch the plastic cup’s walls or bottom (Figure 1).

Figure 1. *Experimental assembly for corrosion determination*

The coupons weight loss was monitored for a period of 7 days (168 h). Before immersion, each coupon was washed with distilled water and acetone, dried and weighted with an analytical balance Kern, ABT 220-4 M (Germany). Every 24 h a control coupon and sample coupon were extracted from the solution and cleaned by immersion in concentrated HCl for a few seconds, rinsed thoroughly with distilled water and acetone, dried and weighted.

The inhibition efficiency ($E\%$) of the plant extracts was determined by using the following formula:

$$E\% = (\Delta w_0 - \Delta w_i) / \Delta w_0 \quad (1)$$

where: Δw_0 and Δw_i represent the coupons weight loss in the absence and in the presence of the inhibitor, respectively.

The corrosion rate (R_{cor}) was measured using the following formula:

$$R_{cor} = \Delta w/(S \cdot t) \ [\mathrm{g \cdot m^{-2} \cdot h^{-1}}] \quad (2)$$

where: Δw represents the coupon weight loss [g], S is the coupon total surface [m^2] and t is the immersion time in corrosive medium [h].

The corrosion penetration index (P) was calculated according to equation (3):

$$P = 8.76 \cdot R_{cor}/\rho \ [\mathrm{mm \cdot year^{-1}}] \quad (3)$$

where: ρ is the material density [$g \cdot cm^{-3}$].

Conductivity measurement

Measurements of the conductivity of the corrosive media with and without plant extracts were realized each day after the steel coupon was extracted, using a Thermo Scientific™ Orion™ Versa Star Pro™ *p*H/Conductivity Multiparameter Benchtop Meter (Thermo Fisher Scientific, USA).

Image of metal coupons surfaces

Pictures of the metal coupons were realized with a professional digital microscope LCD Digital Microscope II (Celestron, Torrance, CA, US) that is equipped with a 5 MP digital camera that captures high-resolution images.

RESULTS AND DISCUSSION

The gravimetric method was employed to determinate the corrosion inhibition effect of the hydroalcoholic extracts of spinach and radish leaves. As it can be seen in Figure 2 in the coupons immersed in the 0.1 M H_2SO_4 solution constantly loss weight over from the first to the 7th day. Thus, in 168 h more than 9 % of the coupon weight is lost. The presence of the plant extract in the corrosive media induces an important decrease of the weight loss. Thus, in the presence of the hydroalcoholic spinach or radish leaves extract the weight loss was more than 4 times lower.

Another observation that can be made is that the corrosion rate of the control sample (in the absence of leaf extracts) strongly decreases after 96 h, the weight loss tending to a constant value. This can be explained by the fact that in the first 96 h the global corrosion process is kinetically controlled, the reaction between H^+ ions and metal being faster than the diffusion of ionic components through the liquid boundary layer. In the absence of stirring, in time, corrosion products accumulate on the metals surface, and Fe^{2+} ions concentration in the boundary layer increases enough to slow down the acidic attack, and the global process will be controlled by ions diffusion in the liquid layer from the vicinity of the sample.

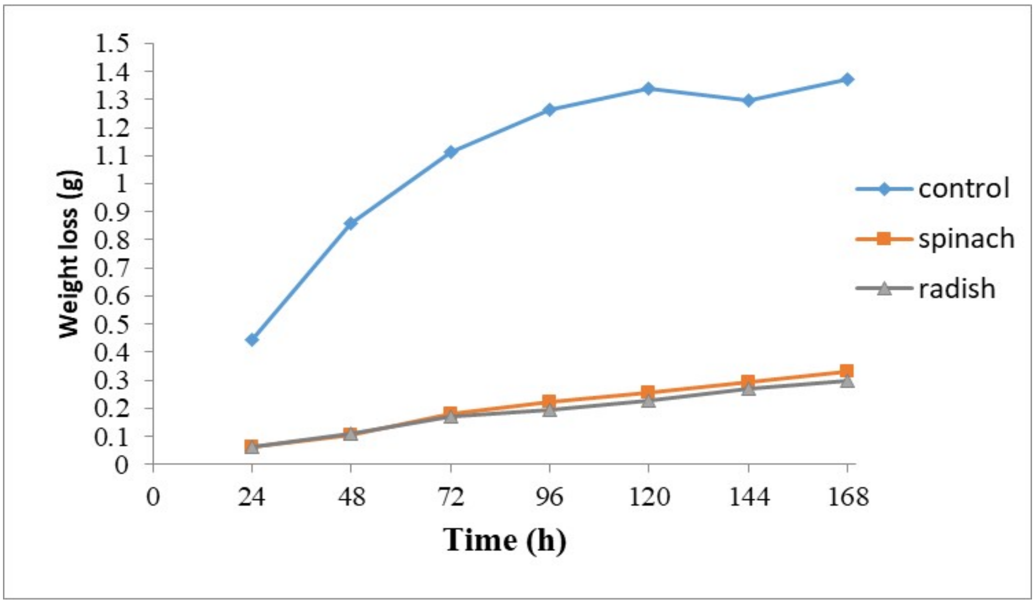

Figure 2. *Weight loss evolution in the presence and the absence of the plant extract*

Figure 3 presents the aspect of the coupon surface after 7 days of immersion in the corrosive media in the absence (Figure 3 a) and in the presence (Figure 3 b and c) of the plant extracts. As it can be observed, in the absence of inhibitors, the surface roughness of the coupons increased, denoting a higher material loss due to corrosion processes.

a. without inhibitor ***b.*** with spinach extract ***c.*** with radish extract

Figure 3. *Image of coupon surface after 168 hours of immersion in 0.1 M* H_2SO_4

Table 1. *Evolution of the corrosion inhibition efficiency (%) over 168 hours*

Time [h] / Extract type	24	48	72	96	120	144	168
0.5 g spinach extract	85.6	87.6	83.7	82.2	80.8	77.3	75.7
0.5 g radish extract	85.5	87.4	84.7	84.6	82.8	79.1	78.4

The calculated corrosion inhibition efficiencies of the extracts of spinach and radish leaves are presented in Table 1. For the first 72 h, the values of the efficiency of the two extracts were almost identical (83.7 % in case of spinach extract versus 84.7 % in case of radish extract) and after 96 h a slightly better protective effect is observed for the radish extract. We can see that for both extracts the inhibition efficiency constantly decreases from the first to the last day of the experiment. The extracts stability in the acidic media for 4 weeks has been proven by our previous work [37].

A study by Li *et al.* [35] reported an inhibitory efficiency of radish leaves extract of 93 % after 12 h. Their study was done in slightly different conditions than ours (another mild steel type and other leaves extract concentration), but the results are comparable. By interpolating the results from Table 1, an efficiency after 12 h of 88.2 % for radish leaves hydroalcoholic extract, and of 88.5 % for spinach leaves extract has been calculated. In long term, after 168 h, efficiency decreased to 78.4 % for radish leaves extract and to 75.7 % for spinach leaves extract.

The corrosion inhibition effect of the spinach and radish extracts was also evidenced by the calculated values of the corrosion penetration index (*P*). Thus, the penetration index in the absence of the extracts was of 8.38 mm·year^{-1} and significantly lower in the presence of the spinach extracts (2.35 mm·year^{-1}) and radish extracts (2.08 mm·year^{-1}).

The conductivity of the corrosive media of each coupon was measured immediately after the coupons were extracted, and its variation is presented in Figure 4.

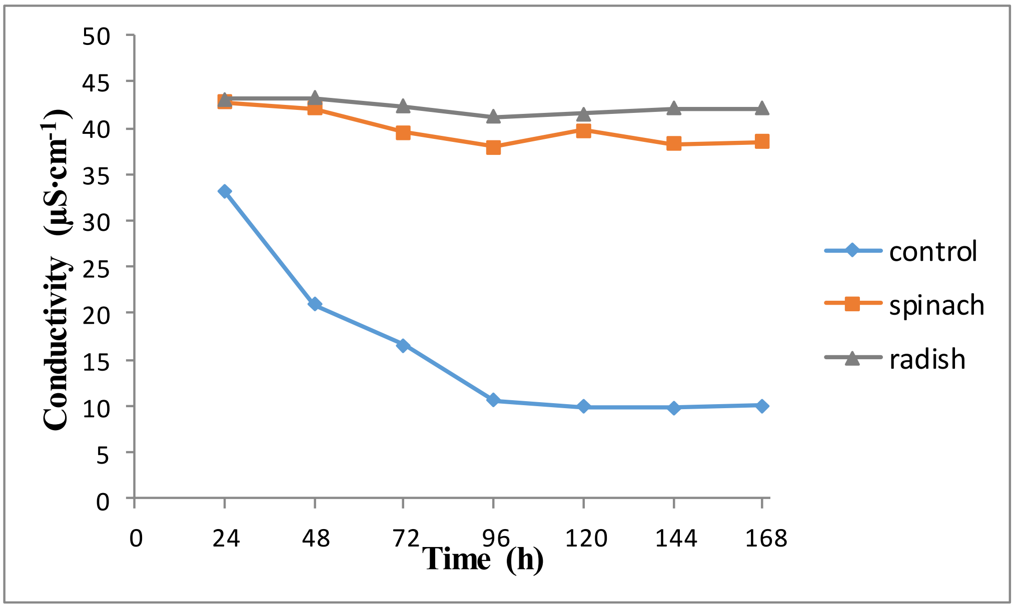

Figure 4. *Evolution of the corrosive media conductivity after coupon immersion*

As can be seen in Figure 4, in the absence of the plant extract, a significant decrease of the control solution (0.1 M H_2SO_4) conductivity was noticed (from 34 to 10 µS·cm^{-1}) while for the acidic media with spinach leaves extract the variation of conductivity was minor (from 43 to 38 µS·cm^{-1}), and practically absent for the acidic media with radish leaves extract (from 43 to 42 µS·cm^{-1}).

In the absence of the plant extract, the conductivity of the corrosive environment decreases with more than 70 % in the first 96 h, after that being stabilized at about 10 µS·cm^{-1}. This is in good concordance with the weight loss measurements that indicate a higher corrosion rate in the first 4 days of the experiment. In this period of time, the very mobile and small H^+ ions from the solution are replaced by more massive, less mobile Fe^{2+} ions, at a rate of 1 iron ion for 2 hydrated protons. Thus, the ionic concentration of the environment decreases along with ions' mobility, leading to

the conductivity decrease. In the presence of plant extract inhibitors, this phenomenon occurs at a lower rate, the components of the leaves extract hindering or blocking the electrochemical reactions at the liquid – metal interface.
If it is analyzed the correlation between conductivity and weight loss for the first 96 hours of the process (after that time assuming that the process is controlled by external diffusion), it can be observed that there is a strong linear dependence between weight loss (y) and conductivity (x), as shown in Figure 5.

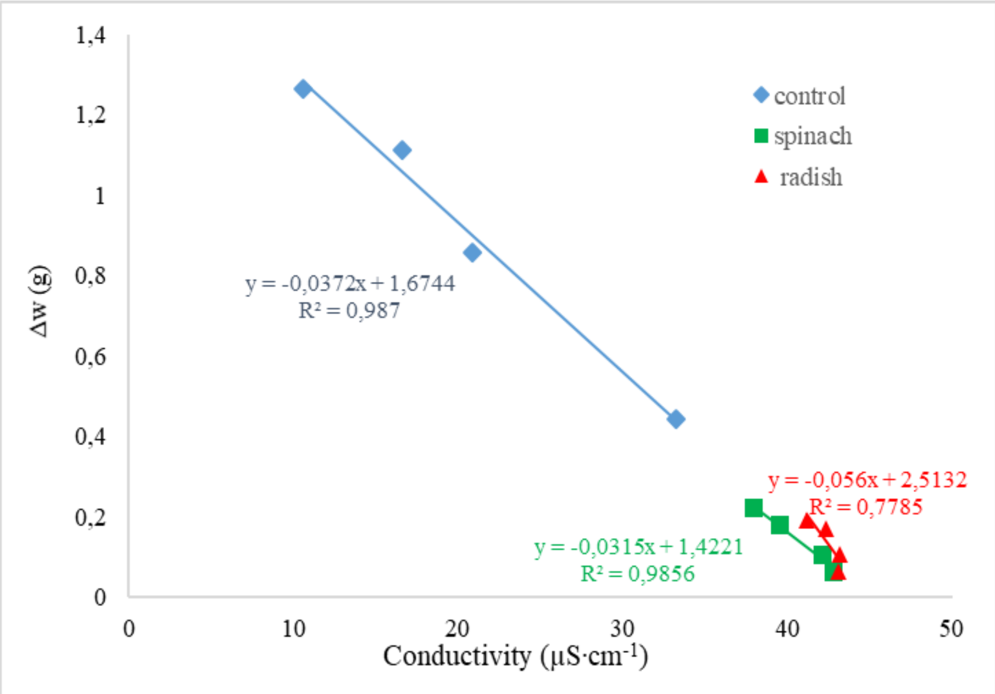

Figure 5. *Correlation between weight loss and the environment conductivity in the first 96 hours of the experiment*

CONCLUSIONS

The inhibitory effect of the hydroalcoholic extract of the radish and spinach leaves obtained using a green extraction methodology was investigated by weight loss measurements on mild steel corrosion in 0.1 M H_2SO_4 solution.
It has been observed that in the absence of inhibitors the corrosion of mild steel in acidic environments and static conditions is kinetically controlled in the first 96 hours, after that the process being controlled by diffusion. The addition of hydroalcoholic extracts of radish or spinach leaves in the corrosive environments decreases the corrosion rates with a factor of 3.5 to 4, from 8.38 mm·year^{-1} to 2.35 mm·year^{-1} (with spinach leaves extract), respectively 2.08 mm·year^{-1} (with radish leaves extract).
Both tested extracts, spinach and radish leaves, showed good corrosion inhibition efficiency on long term (168 hours), respectively more than 75 %, for mild steel in 0.1 M H_2SO_4 environments.
It was also found that there is a strong correlation between the corrosion process expressed in terms of coupon weight loss and the decrease of corrosive environment

conductivity, especially when the process occurs under kinetically controlled conditions: the higher the weight loss, the lower the conductivity. The conductivity decrease of the corrosive environment is strongly hindered by the presence of the used green inhibitors.

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