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Aqueous Extracts of Mango and Orange Peel as Green Inhibitors for Carbon Steel in Hydrochloric Acid Solution

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In this paper, aqueous extracts of mango and orange peels were shown to be good corrosion inhibitors for carbon steel in a 1 mol L^{-1} HCl solution. The inhibition efficiency increased as the extract concentration increased over a concentration range of 200-600 mg L^{-1}, varying from 79 to 96% (mango) and 84 to 91% (orange) using Tafel plots and from 69 to 94% (mango) and 76 to 90% (orange) using electrochemical impedance. In the presence of 400 mg L^{-1} of mango and orange peel extracts, the weight loss measurements showed an increase in the inhibition efficiency with immersion time, where the best results after 24 h of immersion were 97% and 95%, respectively. The adsorption of the extract components on the surface of the carbon steel follows the Langmuir adsorption isotherm. With the extraction procedure used in this work, it can be surmised that it is likely that the more polar heterosides in the extracts are responsible for the corrosion inhibition of carbon steel in an acid solution.

Keywords: *carbon steel, EIS, polarization, weight loss, inhibitor*

1. Introduction

Acid solutions are widely used in industry, where a few of the most important fields of application are acid pickling, chemical cleaning and processing, ore production, and oil well acidification[1]. Carbon steel is one of the most important alloys, which is frequently used in a wide range of industrial applications. Corrosion problems arise due to the interaction of aqueous solutions with carbon steel, particularly during the pickling process, in which the alloy is brought into contact with highly concentrated acids. This process can lead to economic losses due to corrosion of the alloy[2].

The use of inhibitors is one of the most practical methods to protect metal against corrosion in acidic media[3]. Inhibitors protect metals by effectively adsorbing on the surface and blocking active sites for metal dissolution and/or hydrogen evolution, which thereby hinders the overall metal corrosion in aggressive environments[4]. Currently, a large amount of research has focused on natural extracts that can replace synthetic compounds. However, there are few studies on the potential of using byproducts as green corrosion inhibitors. The following are examples of works on byproduct extracts used as corrosion inhibitors for carbon steel in acidic media: banana peels[5], aqueous extracts of fruit peels (oranges, mangos, passion fruits and cashews)[6], coffee grounds[7] and mango and orange peels[8], papaya seeds[9] and peels and seeds of papayas[10]. Indeed, it is extremely appealing to use industrial waste as corrosion inhibitors, such as peels and seeds.

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The literature shows that mango and orange peels are a rich source of antioxidants, which includes ascorbic acid, carotenoids, and phenolic compounds[11,12]. During fruit processing for juice, peels are a major byproduct. Mango peels are major by-products of the mango processing industry and constitutes approximately 15-20% of the total weight of mango fruit[13]. Because mango peels are not currently used for any commercial purposes, they are discarded as waste and become a source of pollution[12]. The orange peel, which represents roughly half of the fruit mass, contains the highest concentrations of flavonoids of citrus fruits[14].

This paper reports the effect of aqueous extracts of mango and orange peels as corrosion inhibitors for carbon steel in 1 mol L^{-1} hydrochloric acid using anodic and cathodic polarization curves, electrochemical impedance measurements and weight loss measurements. The goal of this study is to evaluate a different extraction procedure than the procedure reported in our previous work[6].

2. Experimental

2.1. Preparation of mango and orange extracts

The fruits were washed under running water and peeled; the peels were then air-dried and crushed using a blender. Then, 10 g of the crushed peels were added to a Soxhlet apparatus and extracted successively with three different solvents of increasing polarity: (a) hexane, (b) ethyl acetate,

and (c) ethanol until discoloration. The temperatures used in the extraction were equal to the boiling temperatures of the solvents used. This previous step of Soxhlet extraction was performed to eliminate nonpolar substances. Afterwards, the extracts were obtained by simple water infusion, i.e., the remaining mass of the dried peel from the Soxhlet extraction (approximately 5 grams) was added to a beaker containing 100 mL of distilled water that had been freshly boiled and then allowed to stand for 30 min without heat; this mixture was agitated sporadically. After extraction, the sample was filtered, the volume was lyophilized, and the extract was stored in a desiccator until the analysis.

2.2. Specimen preparation

Working electrodes were prepared from steel specimens with a composition (in wt. %) of C: 0.17-0.24, P: 0.04, Mn: 0.30-0.60, S: 0.05, and Fe: balance. Coupons cut in dimensions of 4.0 cm × 2.0 cm × 0.15 cm were used for the weight loss measurements, whereas specimens, which were prepared by embedding steel rods in epoxy resin with 1 cm^2 of exposed surface area, were used as the working electrodes for the polarization and EIS measurements. The exposed area was mechanically abraded with 400, 500 and 600 grade emery paper, washed with double distilled water, degreased with acetone and finally dried before each experiment.

2.3. Solution preparation

The electrolyte was a 1 mol L^{-1} HCl solution, which was prepared using 37% m/v HCl (Merck Co., Darmstadt, Germany) and double distilled water. All chemicals were of analytical-grade. The experiments were performed under non-stirred, naturally aerated conditions. The concentration range of the mango and orange peel extracts varied from 200 to 600 mg L^{-1} in the electrolyte solution. The addition of the fruit peel extract did not change the pH of the hydrochloric acid solution.

2.4. Electrochemical procedure

Electrochemical measurements were performed using a conventional three-electrode cylindrical glass cell at a temperature of 25 ± 2 °C. A saturated calomel electrode (SCE) and a large-area platinum wire were used as the reference and auxiliary electrodes, respectively.

Before each electrochemical measurement, the open circuit potential (OCP) was recorded as a function of time up to 30 min. After this time, a steady-state OCP, which corresponds to the E_{corr} of the working electrode, was obtained. Electrochemical impedance measurements were performed using AC signals with a 10 mV peak-to-peak amplitude in the frequency range of 10 kHz to 10 mHz. The impedance diagrams are given in the Nyquist representation. After the electrochemical impedance measurements, anodic and cathodic polarization curves were obtained separately at a scan rate of 0.333 mV s^{-1} in the anodic and cathodic directions ($E = E_{corr} \pm 300$ mV), respectively. The above procedures were repeated for each inhibitor concentration.

The electrochemical experiments were performed using a computer-controlled instrument, Autolab Potentiostat/Galvanostat (PGSTAT30) with GPES and FRA software provided by Autolab, which were used for the polarization curves and impedance measurements, respectively.

The inhibition efficiency (n%) was calculated from both the potentiodynamic polarization curves and the electrochemical impedance diagrams, as described by Equations 1 and 2, respectively:

$$n\% = \frac{j_{corr,0} - j_{corr}}{j_{corr,0}} \text{ x100} \quad (1)$$

where $j_{corr,0}$ is the corrosion current density in the absence of an inhibitor (blank), and j_{corr} is the corrosion current density in the presence of an inhibitor, which is obtained from Tafel plots.

$$n\% = \frac{R_{ct} - R_{ct,0}}{R_{ct}} \text{ x100} \quad (2)$$

where $R_{CT,O}$ and R_{CT} are the charge transfer resistances in the absence (blank) and presence of an inhibitor, respectively.

2.5. Weight loss measurements

Duplicate specimens were immersed in the acid test solutions in the absence and presence of 400 mg L^{-1} of mango and orange peel extracts for 1, 4 and 24 h at room temperature (25 °C). The specimens were then removed, rinsed in water and acetone, dried and finally stored in a desiccator. The weight loss was determined using an analytical balance with a precision of 0.1 mg. The inhibition efficiency (n%) was obtained using the following equation:

$$n\% = \frac{W_0 - W}{W_0} \text{ x100} \quad (3)$$

where W_0 and W are the corrosion rate in mils penetration per year (mpy) in the absence (blank) and presence of the extract, respectively.

$$W\,(mpy) = \frac{K\,M}{A\,t\,\rho} \quad (4)$$

where K is a constant (3.45×10^6), M is the weight loss in grams, A is the specimen area in cm^2, t is time in hour and ρ is the specific mass of carbon steel (7.86 g/cm^3).

The weight loss measurements were obtained according to ASTM G31-72[15], which is the standard methodology for this technique in the laboratory.

3. Results and Discussion

3.1. Electrochemical experiments

3.1.1. Potentiodynamic polarization curves

Figures 1a and 1b present the anodic and cathodic polarization curves of carbon steel in 1 mol L^{-1} HCl solution in the absence and presence of extracts from mango and orange peels, respectively. The electrochemical parameters, i.e., the corrosion potential (E_{corr}), corrosion current density (j_{corr}), and the anodic (ba) and cathodic (bc) Tafel constants, shown in Table 1, were collected from the Tafel plots.

It can be observed from the potentiodynamic polarization curves that the presence of extract caused a decrease in both the anodic and cathodic current densities. These results could

be explained by the adsorption of organic compounds that are present in the extracts at the active sites of the electrode surface, which also retards the metallic dissolution and hydrogen evolution and consequently, slows the corrosion process. It can be observed from Table 1 that the corrosion current density (j_{corr}) decreases with the presence and concentration of the inhibitor. Additionally, in the presence of the extracts from mango and orange peels, the E_{corr} shifted slightly to more anodic potentials compared with that of the blank (maximum shifts of 25 and 16 mV, respectively), which demonstrates that this extract acts as a mixed-type inhibitor with predominantly anodic characteristics. The cathodic Tafel slopes (bc) did not change significantly with the addition of the extracts (Table 1), which indicates that the adsorbed inhibitor molecules did not affect the hydrogen evolution reaction, i.e., the hydrogen evolution was diminished exclusively by the surface blocking effect. Regarding the anodic region, the anodic Tafel slopes (ba) also did not change significantly with the addition of the inhibitor, which reveals that the inhibitor, which adsorbed to the carbon steel, did not affect the metal dissolution reaction. The inhibition efficiency calculated from the j_{corr} values obtained in the absence and presence of mango and orange peel extracts varied from 79 to 96% and from 84 to 91% over a concentration range of 200-600 mg L^{-1}.

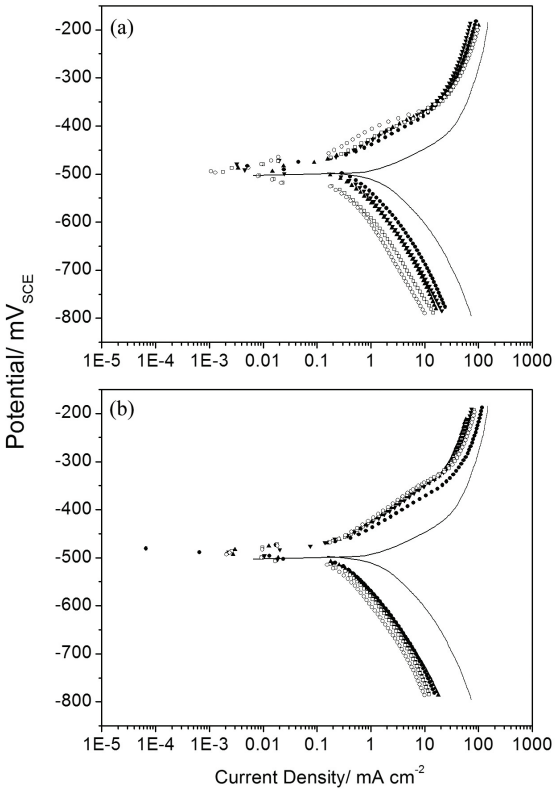

Figure 1. Polarization curves of carbon steel in 1 mol L^{-1} HCl in the absence (□) and presence of mango (a) and orange (b) peel extracts: 200 (●), 300 (▲), 400 (▼), 500 (□) and 600 mg L^{-1} (○).

3.1.2. Electrochemical Impendence Spectroscopy (EIS)

The electrochemical impedance diagrams for carbon steel in 1 mol L^{-1} HCl solution in the absence and presence of increasing extract concentrations of aqueous extracts from mango and orange peels are shown in Figures 2a and b. Table 2 summarizes the impedance data. The electrochemical impedance diagrams show only one depressed capacitive loop, which is attributed to a single time constant, in the absence and presence of the fruit peels extracts, which indicates two significant effects: the charge transfer resistance significantly increases and f_{max} decreases in the presence of the extracts. These effects decrease the capacitance value, which may be caused by reduction in the local dielectric constant and/or by an increase in the thickness of the electrical double layer. These results show that the presence of the extracts modifies the electric double-layer structure, which suggests that the inhibitor molecules act by adsorption at the metal/solution interface. Deviations from a perfect circular shape indicate frequency dispersion of the interfacial impedance. In the literature, this anomalous phenomenon is attributed to the heterogeneity of the electrode surface arising from the surface roughness or interfacial phenomena[16,17]. The intersection of this semicircle with the real axis at high frequencies produced

Table 1. Kinetic parameters obtained from Tafel plots for carbon steel in 1 mol L^{-1} HCl in the absence and presence of aqueous garlic peel extract at the following concentrations: 100, 200, 300 and 400 mg L^{-1}.

Medium	Inhibitor Concentration (mg L^{-1})	E_{corr} (mV)	J_{corr} (mA cm^{-2})	ba (mV/dec)	bc (mV/dec)	n%
Blank	0	–504	1.00	56.3	–74.3	–
Mango peel	200	–482	0.207	61.5	–74.3	79
	300	–479	0.147	64.0	–94.8	85
	400	–484	0.145	64.0	–69.2	86
	500	–485	0.0893	53.8	–97.3	91
	600	–479	0.0443	51.2	–76.8	96
Orange peel	200	–491	0.161	65.8	–92.2	84
	300	–488	0.127	69.2	–79.4	87
	400	–490	0.127	71.7	–79.4	87
	500	–489	0.115	69.2	–87.1	89
	600	–489	0.0946	64.0	–89.6	91

an ohmic resistance (Rs) of approximately 1.5 Ω cm^2 of the solution. The solution resistance (R_s) is identical in the absence and presence of the fruit peel extracts. The charge-transfer resistance (R_{CT}) values were calculated from the difference in impedances at lower and higher frequencies. The double-layer capacitance (C_{DL}) was calculated using the following equation:

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}} \quad (5)$$

where f_{MAX} is the frequency at which the imaginary component of the impedance is maximal. A C_{DL} value of 52.8 μF cm^{-2} was found for the carbon steel electrode at 1 mol L^{-1} HCl. From Table 2, it is clear that the R_{CT} values increased and that the C_{DL} values decreased with increasing inhibitor concentration, which indicates a decrease in the active surface area caused by the adsorption of the inhibitors on the carbon steel surface, and suggests that the corrosion process became hindered. This hypothesis is corroborated by the anodic and cathodic polarization curves results. The inhibition efficiency calculated from the R_{ct} values obtained in the absence and presence of mango and orange peel extracts varied from 69 to 94% and from 76 to 90% over a concentration range of 200-600 mg L^{-1}. The best result for the inhibition efficiency of these extracts was obtained with a mango and orange peel extract concentration of 600 mg L^{-1} with an efficiency equal to 94% and 90%, respectively.

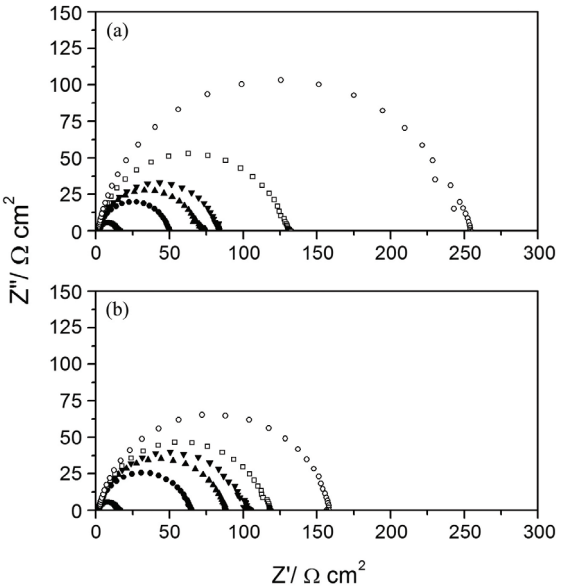

Figure 2. Impedance diagrams obtained at the corrosion potential for carbon steel in 1 mol L^{-1} HCl solution in the absence (■) and presence of mango (a) and orange (b) peel extracts: 200 (●), 300 (▲), 400 (▼), 500 (□) and 600 mg L^{-1} (○).

It is important to note that although in this work, the extracts were obtained by a different extraction procedure compared with our previous work[6], the inhibition efficiency did not significantly increase. For example, in the presence of 400 mg L^{-1} mango and orange peel extracts obtained by simple infusion, the inhibition efficiencies were 83 and 95%, respectively, whereas for the same concentration of mango and orange peel extracts obtained using this new extraction procedure, the inhibition efficiencies were 81 and 85%, respectively. Therefore, the infusion method for these fruit peel extracts is a good method to produce inhibitors due to its simplicity and advantages compared with extraction procedures using organic solvents. In addition to these results, note that the substances responsible for the inhibition action of both extracts obtained from the different extraction procedures are likely the same substance.

The interactions between the extracts and the carbon steel surface can be examined by the adsorption isotherm. The inhibition efficiency is directly proportional to the fraction of the surface covered by adsorbed molecules (θ), which was calculated in this case using the equation, θ = n/100. The most frequently used adsorption isotherms are Langmuir, Temkin, Frumkin and Flory-Huggins. Therefore, each of these adsorption isotherms was tested in terms of their descriptions of the adsorption behavior of extracts on carbon steel surface in an HCl solution. The correlation coefficient, R^2, was used to choose the isotherm that best fitted the experimental data. The linear

Table 2. Impedance data for carbon steel in 1 mol L^{-1} HCl in the absence and presence of mango and orange peel extracts.

Medium	Inhibitor Concentration (mg L^{-1})	E_{OCP} (mV)	R_{ct} (Ωcm^2)	f_{max} (Hz)	C_{dl} (μF cm^{-2})	n%
Blank	0	–500	15.6	193.1	52.8	–
Mango peel						
	200	–493	50.0	62.5	51.0	69
	300	–479	74.4	62.5	34.2	79
	400	–485	83.3	47.1	40.6	81
	500	–485	131.3	35.6	34.1	88
	600	–492	254.0	20.2	31.0	94
Orange peel						
	200	–485	64.9	62.5	39.3	76
	300	–488	88.3	47.1	38.3	82
	400	–486	104.2	47.1	32.4	85
	500	–489	118.2	47.1	28.6	87
	600	–492	158.0	35.6	28.3	90

relationships of C/θ vs. C, as shown in Figure 3, suggest that the adsorption of mango and orange extracts on carbon steel obeyed the Langmuir adsorption isotherm. This isotherm can be represented as

$$\frac{C}{\theta} = \frac{1}{K} + C \quad (6)$$

where C is the concentration of the extract, and K is the adsorption constant.

Figure 3 depicts linear plots with high correlation coefficients of 0.9958 and 0.9997 and a slope of 0.8853 and 1.0159 for the mango and orange peel extracts, respectively. The values of the adsorptive equilibrium constants at 25 °C are 0.0083 and 0.01617 L mg^{-1} for the mango and orange peel extracts, respectively. Unfortunately, the comparative analysis between mango and orange extracts is not possible yet due to the unknown nature of the substances responsible for the adsorption process.

3.2. Weight loss measurements

Table 3 presents the results of the corrosion rates measurements for carbon steel in 1 mol L^{-1} HCl solution in the absence and presence of 400 mg L^{-1} of aqueous extracts from mango and orange peels for different immersion times (1, 4 and 24 h) at 25 °C. These critical assays demonstrate the extracts' stability with time. The carbon steel corrosion rate (W_{CORR}) was greatly reduced upon the addition of mango and orange peel extracts for all immersion times. This behaviour reflects the inhibitory effect of these extracts against carbon steel corrosion in an acid solution, which corroborates the results obtained from electrochemical impedance diagrams and polarization curves. It is also noted that the inhibition efficiency increased with time in the presence of mango and orange peel extracts, from 72% after 1 h of immersion to 97% after 24 h for mango peel extract and from 83% to 95% for orange peel extract, which indicates that the inhibition efficiency was enhanced after longer periods of immersion.

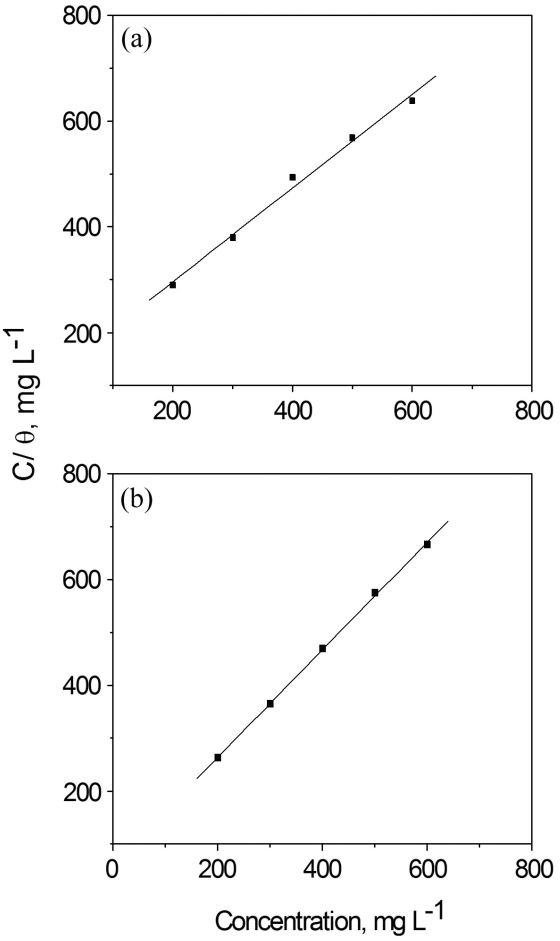

Figure 3. Langmuir adsorption isotherms of mango peel extracts (A) and orange peel extracts (B).

3.3. Inhibition mechanism

Adsorption of inhibitor molecules on a metal surface is either physical or chemical. For the physical adsorption (physisorption) of inhibitors on a metal surface, there must be interactions between the metal surface and the molecules *via* relatively weak interactions, e.g., dipole-dipole interactions, whereas chemical adsorption (chemisorption) occurs by the sharing of electrons between the inhibitor molecules and metals or the transfer of charges/electrons from the inhibitor molecules to the metal surface.

The complex chemical compositions of these extracts make it rather difficult to attribute the inhibitory action to a particular constituent or group of constituents. Mango and orange peels are rich sources of antioxidant compounds, such as polyphenols, carotenoids and vitamins C and E[11,12]. Phenolic compounds, particularly flavonoids, have been shown to possess significant antioxidant activity, which is primarily based on their structural characteristics (number and position of phenolic hydroxyls, other groups, conjugation)[14]. Mango and orange peels are also abundant sources of pectin, which is a structural heteropolysaccharide[18,19]. Flavonoids can act as chelating agents, where several authors have suggested that chelating activity is due to its binding to the ion metal (M^{n+}) at two sites on the molecule: in the ortho-diphenolic group of ring B and ring C, as shown in Figure 4[20]. In this case, the inhibitor molecules could affect the metal dissolution reaction, but it

Table 3. Carbon steel corrosion rate data in 1 mol L^{-1} HCl in the absence and presence of 400 mg L^{-1} of mango and orange peel extracts with 1, 4 and 24-h immersion times at 25°C.

Immersion time (hours)	Medium	W_{corr} (mpy)	Inhibition efficiency (%)
1	Blank	926.7	-
	Mango peel	255.1	72
	Orange peel	156.4	83
4	Blank	865.1	
	Mango peel	73.1	92
	Orange peel	75.4	91
24	Blank	763.5	
	Mango peel	20.5	97
	Orange peel	34.9	95

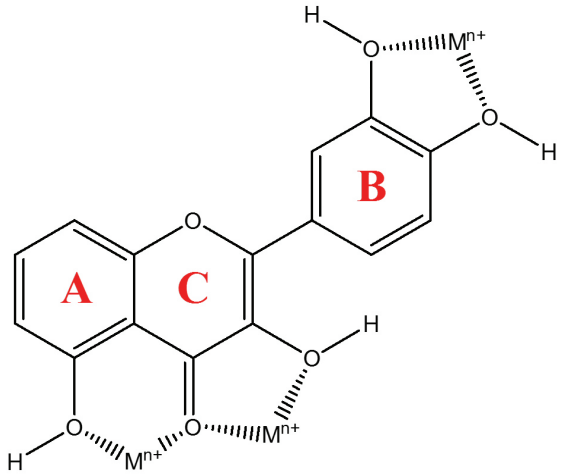

Figure 4. Binding sites in flavonoids for the transition metal.

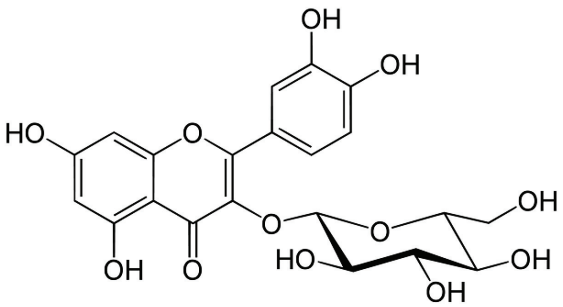

Figure 5. Heterosides structure.

was shown in the present work that they did not affect the metal dissolution reaction.

The typical method for the extraction of flavonoids using increasingly polar solvents consists of the following steps: 1) a first extraction is usually performed with a nonpolar solvent to remove oil, grease, pigments and sterols, which facilitates the subsequent extraction of flavonoids; 2) a second extraction is performed with slightly more polar solvents (chloroform, methylene chloride or di-ethyl ether), which allows the slightly polar free aglycones, such as flavones, flavanones, aglucone isoflavones and other species with a high degree of methylation, to be recovered. By increasing the polarity of the solvent (acetone, methanol, water), it is possible to extract the aglycone, flavones and more polar flavonols, such as aurones and chalcones. Finally, hot water extraction will extract the more polar heterosides (Figure 5), such as polyglycosides, flavanodiols, catechin and procyanidins, and sugars[21]. Thus, the inhibitory effect observed in the polarization curves, electrochemical impedance diagrams and weight loss measurements is likely to occur via the adsorption of the heterosides (more polar compounds) present in the mango and orange peel extracts on steel surfaces.

In a simple infusion, the extraction procedure used in the previous work, could extract several less polar substances even in water[6]. Thus, the present work was performed to show if such substances (more polar heterosides) could be responsible for the corrosion inhibition of these extracts. The inhibition efficiency obtained from this new extraction procedure, which contains more polar heterosides, is extremely similar to that obtained from simple infusion. Therefore, the more polar heterosides are likely responsible for the corrosion inhibition in both extracts, i.e., those obtained from a simple infusion and from the new extraction procedure used in this work.

Such substances contain hydroxyl groups that also can be adsorbed on metal surfaces through H-bonding, which involves the displacement of water molecules from the metal surface[22].

$$Org_{(sol)} + xH_2O_{(ads)} \rightarrow Org_{(ads)} + xH_2O_{(sol)} \qquad \text{(Reaction 1)}$$

where x is the number of water molecules replaced by an organic inhibitor.

It is important to note that the discussion of the adsorption isotherm behaviour using natural product extracts as inhibitors in terms of thermodynamic parameters (such as the standard free energy of adsorption value, ΔG^0_{ads}) is impossible because the molecular mass of the extract component responsible for the adsorption process is unknown. Others authors have noted the same limitation in their works[6,7,23-25].

4. Conclusions

Mango and orange peel extracts act as corrosion inhibitors for carbon steel in 1 mol L^{-1} HCl solutions. The inhibition efficiency of carbon steel in 1 mol L^{-1} HCl increased with the concentration of mango and orange peel extracts over a concentration range of 200-600 mg L^{-1} based on both electrochemical and weight loss measurements. All electrochemical results, including the slight displacement of the corrosion potential, the inhibitory action in both anodic and cathodic polarization curves, and the results of the electrochemical impedance measurements showed that the examined extracts acted as adsorption inhibitors on the carbon steel surface. The inhibitory effect was performed via the adsorption of compounds present in the fruit peel extracts on the steel surface. The inhibition efficiency obtained from this new extraction procedure, which contains more polar heterosides, is extremely similar to that obtained from a simple infusion. Thus, the more polar heterosides are likely responsible for the corrosion inhibition in both extracts. Due to the hydroxyl presence in these molecules, the adsorption on the metal surface could occur through H-bonding, which involves the displacement of water molecules from the metal surface The adsorption of the fruit peel extracts followed a Langmuir adsorption isotherm.

Acknowledgements

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