

Inhibition of Pipeline Steel Corrosion in 0.5 M HCL using Ethanolic Extract of *Citrus Sinensis* Seeds

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Abstract: Inhibitive action of ethanolic extract of *Citrus sinensis* seeds on the corrosion of pipeline steel in monobasic acid medium (0.5 M HCl) was studied using gravimetric method. Adsorption isotherm models were mainly used to elucidate the inhibition mechanism of ethanolic extract of *Citrus sinensis* seeds in 0.5 M HCl. Surface morphology of pipeline steel before and after inhibition were studied by scanning electron microscope (SEM). Results obtained showed that *Citrus sinensis* seed extract inhibited pipeline steel corrosion in 0.5 M HCl environment. The corrosion inhibition efficiency increased with increased concentration of the *Citrus sinensis* seed extract and decreased with raise in environmental temperature in the acidic medium. The corrosion inhibition data fitted better into Freundlich adsorption model, while the presence of the extract gave a smoother corrosion surface morphology.

Keywords: Corrosion inhibition; Plant extract; *Citrus sinensis*; Adsorption; Surface morphology

1. Introduction

Corrosion is a universal crucial problem that strongly affects natural and industrial environments.^[1,2] The challenge posed by corrosion is of enormous cost to national economies. It has been reported that corrosion costs about 5% of national GDP of any developing nation.^[1] Unfortunately, the cost is keep increasing due to the industrialization (new petroleum and oil well companies). However, the corrosion can be controlled by using various methods in which corrosion inhibitors are the most efficient and cost effective one.^[2] Understanding of corrosion and its mechanism leads to formulation of more efficient and cheap mitigation program. Corrosion is the electrochemical interaction between a metal and its environment. This interaction leads to material degradation due to the quest of materials to attain kinetic and thermodynamic stability.^[3] Practically, corrosive environments are either basic or acidic in nature. Most industrial environment is acidic due to the release of acidic gases -SO_x and -NO_x into the environment resulting in acid rain. While construction materials such as steel are used in both weak basic and weak acidic environments, most atmospheric environments are acidic. This leads to loss of structural integrity among other issues. Pipeline steel is one of the most used steel materials in building construction due to its mechanical strength, availability and competitive price. However, the metallic properties of steel makes it's susceptible to corrosion as well defines its workable corrosion mitigation procedures. Some of the corrosion mitigation activities are effective structural design,

electrical protection, as well as application of chemical inhibitors. Several researchers have reported the successful application of corrosion inhibitors as an anti-corrosion measure.^[4-7] Till date, there are plenty of heterocyclic compounds are reported as efficient corrosion inhibitors for the mild steel in acidic medium (1 M HCl and H₂SO₄).^[8-10] The heterocyclic compounds that contain all the three N, O and S atoms are found to be the most efficient corrosion inhibitors for mild steel in acidic environments.^[11] However, most of the heterocyclic compounds are synthetic compounds and in order to prepare the compounds, the system must it requires expensive starting material and difficult preparation protocol.^[12] In addition, they are not environmentally friendly in nature when compare to plant extracts. In addition, most of the efficient corrosion inhibitors are toxic and detrimental to the environment. Recently, there is a significant preference for the use of phyto extracts as corrosion inhibitors due to their cost-effectiveness and environmentally friendly nature.

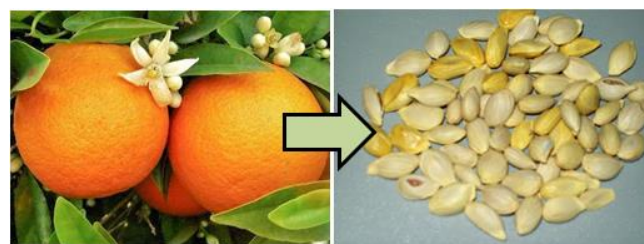


Fig. 1. Photographs of *Citrus sinensis* fruits and seeds.

Table 1. Elemental Composition of the Pipeline Steel Coupon

Element	C	S	P	Si	Mn	Al	Fe
Composition (wt%)	0.28	0.024	0.006	0.21	1.53	0.03	Balance

Indeed, the plant extracts contain polar atoms such as S, N, O, P etc. Because of this nature, the lone pair of electrons present on these atoms is pumped on to the metal surface; loss of electrons from the metal surface can be avoided. Thus corrosion inhibition takes place. Because of adsorption of inhibitor molecules on metal surface, protective film is formed, thus control the corrosion of steel. To date, various plant extracts were studied for the corrosion inhibition of steel in HCl.^[13,14] Fruits (Punica granatum,^[15] Ziziphus mauritiana Fruit Extract,^[16] Unripe fruit peel extract of Musa acuminata (Cultivar variety - Nendran) (MNP)^[17], leaves (acid extract of Ephedra alata leaves,^[18] Morindacitrifolia leaves extract,^[19] ethanolic extracts from leaves (LV), bark (BK) and roots (RT) of Nauclealatifolia^[20] extract of dodonaeviscosa leaves (DLVE),^[21] aqueous extract leaves of Larrea tridentate (AELL),^[22] and RosmarinusOffic inalis L. leaves extract^[23], roots (Anacyclus pyrethrum L.^[24] and Nauclealatifolia^[25]), barks (bark-extract of Rhizophora mangle L^[26] and Cassia Auriculata (CAF) plant^[27]), flowers (air dried flowers of Nerium oleander and Tecomastans.,^[28] and acid extract of flowers of Cassia Auriculata (CAF) plant.^[27]), and seeds (roasted coffee seed (Coffea Arabica) extract^[29] and acid extract of dry azadirachta indica seed^[30]) have been used as green corrosion inhibitors.

Many parts of plant resources have been investigated and found to act as good inhibitors of metallic corrosion.^[31] All though several plant parts are used for the extraction, plant seeds are less studied. In particular, parts of different citrus species have been successfully investigated for its corrosion inhibition properties.^[32] However, the seeds of these plants though useful for its propagation are mostly discarded arbitrarily causing environmental and waste treatment concerns. However, there are very few reports on the corrosion inhibition characteristics of citrus seed.^[33] Fig. 1. shows the photographs of *Citrus sinensis* fruits and seeds. In this work, ethanolic extract of *Citrus sinensis* seeds was prepared. The resultant extract was then used as corrosion inhibitor for the inhibition of Pipeline steel corrosion in acidic environment using gravimetric method. Adsorption isotherm models were mainly used to elucidate the inhibition mechanism of ethanolic extract of *Citrus sinensis* seeds in 0.5 M HCl. Surface morphology of pipeline steel before and after inhibition were studied by scanning electron microscope (SEM). We believe that the use of extract of *Citrus sinensis* seeds may reduce the waste disposal challenges and generate baseline data for seeds corrosion inhibition.

2. Materials and Methods

2.1. Preparation of Citrus Sinensis Seed extract

The materials used for this study were Pipeline steel obtained from the Mechanical engineering workshop of University of Port Harcourt, Nigeria. The *Citrus sinensis* seeds were identified by the Plants and Biotechnology, Department of University of Port Harcourt. The sheets were mechanically cut into rectangular coupons of

dimensions 4 cm x 5 cm x 0.34 cm. The elemental composition (C, S, P, Si, Mn, Al and Fe) was tabulated in Table 1. The coupons were polished with successive grits of silicon carbide abrasive paper, rinsed with distilled water, degreased in absolute ethanol, deemed in acetone and dried to a constant weight. Prior to use, the coupons were stored in an active dissector in order to avoid rusting. All other reagents were of analytical grade and used without further purification.

2.2. Preparation of Citrus sinensis seed extract

The procedure for extracting the seed extract has been previously reported.^[34] The HCl was used to prepare serial diluents solutions of 0.5 M HCl and the *Citrus sinensis* extract. At first, the seeds were in an oven below 40°C and pulverized using electric blender to ensure enhanced extraction. A 500 g of both pulverized powders were soaked in separate 1000 ml volumetric flasks using 99% ethanol for 72 hours. After soaking the extract was filtered and the filtrate was concentrated using rotary evaporator and transferred to a sterile air tight analytical container.

2.3. Gravimetric Method

The pre-cleaned and weighed coupons were immersed with the aid of synthetic thread into 170 mL of test solutions. The higher temperature tests were conducted by placing the test cells in thermostatic water baths (P Selecta, 6001197 France) maintained at 303, 313 and 333 K. The coupons were retrieved after 4 hours, washed with the help of a brittle brush, degreased in absolute ethanol, immersed in acetone and allowed to dry till constant weight and weighed. The weight was recorded. The weight loss ΔW was calculated by subtracting the initial weight before immersion W_i and the final weight after drying to constant weight W_f . The serial dilutions of the 0.5 M HCl and extract concentrations were 1 g/dm³, 2 g/dm³, 3 g/dm³, 4 g/dm³, 5 g/dm³ and blank/control without the extract. The tests were conducted in triplicate and the mean values of the weight loss obtained at each temperature and were used to determine the following parameters.

$$\text{Weight loss (W)} = (W_i - W_f) \text{ g} \quad (1)$$

Where W = weight loss of pipeline steel coupon, W_i = Initial weight of the pipeline steel coupon, W_f = Final weight loss of pipeline steel coupon in grams.

The surface coverage (θ) of *Citrus sinensis* seed extract on pipeline steel in 0.5 M HCl at different concentrations was obtained by using the equation below.

$$\theta = 1 - \frac{W_i}{W_o} \quad (2)$$

Where W_o is Corrosion rates in the absence of inhibitor; W_f = Corrosion rates in the presence of inhibitor.

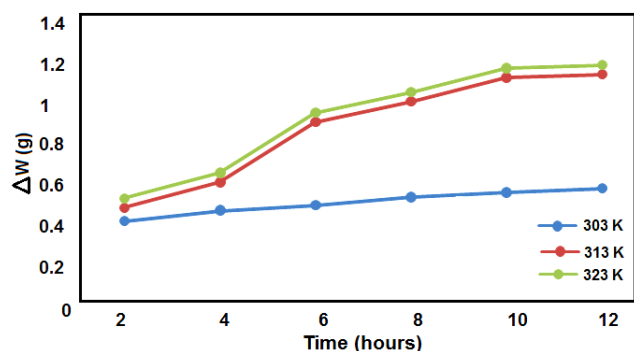


Fig. 2. Variation of weight loss with time for the corrosion of pipeline steel in 0.5 M HCl environment with and without various concentrations of *Citrus sinensis* seed extract.

The corrosion inhibition efficiencies were calculated using the formula;

$$\%IE = 1 - \frac{W_i}{W_0} \times 100 \quad (3)$$

Where W_0 and W_i are weight losses in grams of metal coupon in the presence and absence of various concentration of the citrus seed extract, respectively.

The total surface area of pipeline steel coupon immersed in the solution was calculated adding the areas of all the surfaces:

$$A = 2KM + Kt + 2Mt + 2\pi rt - 2\pi r^2 \quad (4)$$

Where A - Total surface area of the coupon immersed in solution, K - Length of coupon, M - Width of coupon, T - Thickness of coupon, R - Radius of the hole drilled on coupon.

The corrosion rate, half-life and rate constant were obtained as showed below.

$$\text{Corrosion rate (mpy)} = \frac{87.6 \Delta w}{DAT} \quad (5)$$

Where: ΔW = Change in weight loss
 D = Density of specimen
 A = Surface area of specimen
 T = Exposure time.

$$\text{Half-life: } t_{1/2} = \frac{0.693}{k} \quad (6)$$

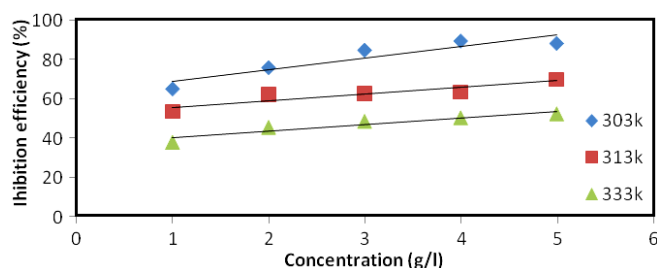


Fig. 3. Plot of inhibition efficiency against concentration of *Citrus sinensis* seed extract in 0.5 M HCl at different temperature.

2.4. Surface analysis

The pre-cleaned pipeline steel coupons were subjected to SEM studies by immersing one in 0.5 M HCl and another in 5 g/dm³ solution of HCl and *Citrus sinensis* seed extract for 12 hours. The coupons were retrieved, dried and tested to study the surface morphology of the steel. Surface morphology of pipeline steel before and after inhibition was studied by scanning electron microscope (SEM).

3. Results and Discussions

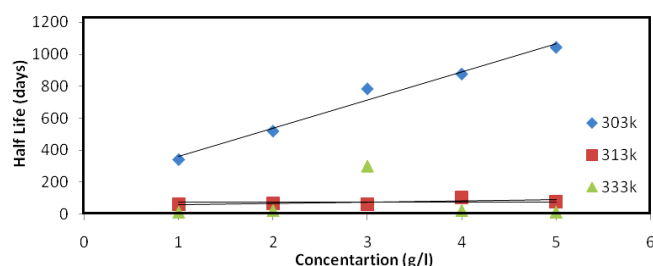
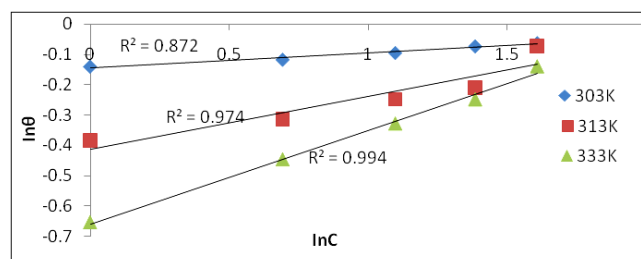
3.1. Weight loss and Corrosion inhibition efficiency

The weight loss pattern of pipeline steel in HCl environment was studied in the presence and absence of *Citrus sinensis* seed extract (Fig. 2). Fig. 2. shows the variation of weight loss with time (2, 4, 6, 8, 10 and 12 hours) and temperature (303, 313 and 333 K) in the corrosion of pipeline steel in 0.5 M HCl environment with and without various concentrations of *Citrus sinensis* seed extract. Fig. 2 showed a gradual increase in the rate of weight loss of pipeline steel in 0.5 M HCl with time at all the studied temperatures (without the presence of seed extract). However, the presence of the seed extract significantly reduces the rate of weight loss of the pipeline steel. This behaviour was noticed to be increased with the rise in temperature. The rate of weight loss is inversely related to increase in concentration of the additives and directly proportional to the temperature. The reduction in rate of weight loss in the presence of the extract is mainly attributed to the formation of protective layer by the inhibitor molecules by the *Citrus sinensis* seed extract. The presence of O, C and aromatic groups are the main reason for the protection of pipeline steel in 0.5 M HCl. This phenomenon is previously reported by several researchers.^[35] For instance, Karthiga et al.,^[14] noticed that the weight loss of steel significantly reduced when the steel exposed to 0.5 M HCl in the presence of inhibitor. In the present case, it is due to the presence of efficient phytochemical constituents present in the seed extract.

Based on the results obtained from the weight loss data, the inhibition efficiency of *Citrus sinensis* seed extract was calculated for the corrosion of pipeline steel in 0.5 M HCl in the presence of various concentration of inhibitor at difference temperatures (303, 313 and 333 K). Equation 3 was used to calculate the IE% of *Citrus sinensis* seed extract. Fig. 3. Shows plot of inhibition efficiency against concentration of *Citrus sinensis* seed extract in 0.5 M HCl at different temperature. This behaviour has been previously noticed and concluded that the inhibitor to effectively inhibits the corrosion of metal in a corrosive environment.^[36-38] However, increasing the temperature strongly affect the inhibition efficiency of the seed extract. In general, most of the synthetic inhibitors like benzimidazole and its derivatives show better inhibition efficiency at high temperatures when compare to low temperature. In fact, the can get adsorbed on metal surface via chemisorption at elevated temperature. The coordination site strongly assists the coordination of heteroatom to Fe-surface. However, most the plant extracts show decrease in the inhibition efficiency at elevated temperature. This is mainly due to the physisorption of inhibition molecules on the metal surface.^[39] Five different concentrations such as 1, 2, 3, 4 and 5 g/l

Table 2. Corrosion rate, inhibition efficiency and degree of surface coverage

	Corrosion Rate			Inhibition Efficiency (%IE)			Degree of surface coverage (θ)		
	30°C	40°C	60°C	30°C	40°C	60°C	30°C	40°C	60°C
Blank	5.07	6.59	9.98						
1	4.34	6.19	8.93	87.05	68.28	52.18	0.87	0.68	0.52
2	3.68	5.79	8.47	89.01	73.37	63.8	0.89	0.73	0.64
3	2.87	4.79	7.98	90.56	78.27	71.99	0.91	0.78	0.72

**Fig. 4.** Variation of material half-life of Pipeline steel at 303, 313 and 333 K in the presence of *Citrus sinensis* seed extract in 0.5 M HCl environment.**Fig. 5.** Freundlich adsorption isotherm for pipeline steel corrosion cotyledon in 0.5M H₂SO₄ at 30°C, 40°C and 60°C.

are tested. At the concentration of 5 g/L, ethanolic extract of *Citrus sinensis* seeds showed excellent 90.6% of inhibition efficiency against corrosion of pipeline steel in 0.5 M HCl at 303 K. This is due the formation of protective layer by the inhibitor molecules on the surface of pipeline steel. However, inhibition efficiency was found to be decreased at elevated temperature such as 313 and 333 K (Fig. 2). This phenomenon is due to the desorption of inhibitor molecules from the metal surface at elevated temperature. Further increasing the concentration of ethanolic extract of *Citrus sinensis* seeds did not show increase in the inhibition efficiency.

3.2. Surface coverage and Corrosion rate

The degree of surface coverage for the molecular constituents of *Citrus sinensis* seed ethanol extract on pipeline steel in 0.5 M HCl at varying temperatures was calculated using Equation 2, and result is presented in Table 2. The values in Table 2 clearly show that the degree of the surface coverage increased with the addition of the extract and decreased with increase in temperature, this trend corroborates earlier hypothesis that there are desorption of inhibitor molecules at high temperature. Also, the corrosion rate considerably reduced in the presence of the extract. Worthy of note is that the corrosion rate decreased with increase in concentration of the extract and decreased with rise in temperature of the corrosion /inhibitor cell.^[40] The better corrosion rate value of 2.87 was calculated for the inhibition of *Citrus sinensis* seed ethanol extract against the corrosion of pipeline steel in 0.5 M HCl. Blank experiment showed very high corrosion rate values (5.07, 6.59 and 9.98) due to the aggressive attack of the HCl and there is no protection for the pipeline steel. Similarly, high degree of surface coverage ($\theta = 0.91$) was found out for the inhibition of *Citrus sinensis* seed ethanol extract against the corrosion of pipeline steel in 0.5 M HCl.

3.3. Material half-life $t_{1/2}$

The material half-life for Pipeline steel in 0.5 M HCl with *Citrus sinensis* seed ethanol extract was calculated using Equation 6. The results obtained were plotted against concentration of the extract; the results are presented in Fig. 4. Fig. 4. shows the variation of half-

life of Pipeline steel at 303, 313 and 333 K in the presence of *Citrus sinensis* seed extract in 0.5 M HCl environment. The material half-life of the pipeline steel in HCl was higher in the absence of the extract compared to the half-life in the presence of the extract and increased with increase in the concentration *Citrus sinensis* seed extract. This confirms the corrosion inhibiting capacity of the present extract. Also the $t_{1/2}$ increased with decrease in temperature of the extract. This is in good agreement with the corrosion rate data for the extract.^[14,41]

3.4. Adsorption

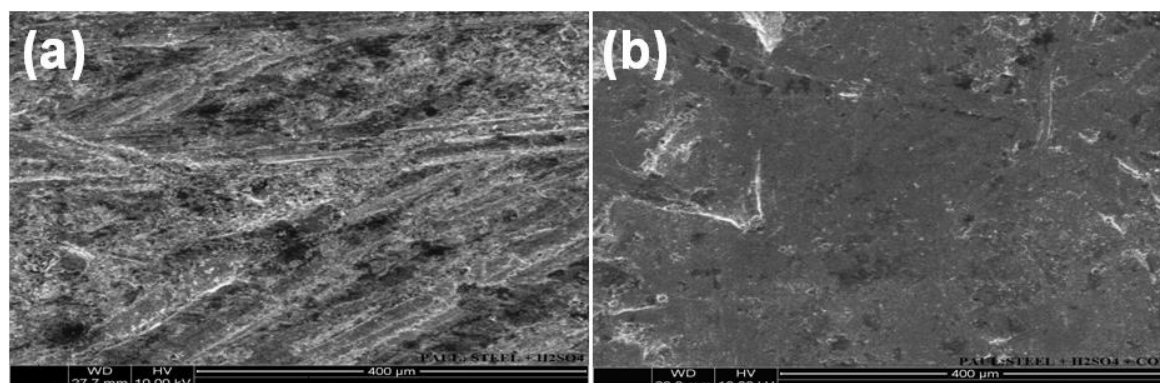
The surface coverage of molecules of *Citrus sinensis* seed extract data was fitted into various adsorption models to find the best fit model for the corrosion inhibition process. The model of the adsorption process helps corrosion professionals gain insight into the behaviour of inhibitor molecules on the surface of the metal. The mode of adsorption is dependent on physio-chemical properties of the metallic surface, the inhibitor, the thermodynamic and kinetics of the corrosion inhibition process. These factors primarily affect the degree of inhibitor molecular coverage (θ) which consequently determines the corrosion inhibition efficiency. Different adsorption models were tested for the inhibitive action of *Citrus sinensis* seed extract on the corrosion of Pipeline steel in HCl. The best fitted was Freundlich adsorption isotherm. Freundlich equation is Equation 7, and the linear form plotted using Equation 8. Plots of $\ln\theta$ against $\ln C$ are shown in Fig. 5. It shows that the inhibition mechanism of inhibition molecules on the steel surface in 0.5 M HCl obeys Freundlich adsorption isotherm model. The correlation coefficient (R^2) value was calculated to be more than 0.872, which was better fitted at 60°C than 30°C (Table 3). The negative values for the calculated ΔG_{ad} values suggest spontaneous adsorption of the extract molecules on the metal surface, though not strong enough to form chemical bonds.

$$\theta = KC^n \text{ where } 0 < n < 1 \quad (7)$$

$$\ln\theta = \ln K + n \ln C \quad (8)$$

Table 3. Calculated parameters from Freundlich adsorption isotherm plot for adsorption of the extract on pipeline steel in 0.5 M HCl.

Temp(K)	0.5 M HCl + pipeline steel + Seed extract			
	K_{ads}	ΔG_{ads}	n	R^2
303	1.146	-10.539	0.563	0.974
313	1.032	-10.746	0.786	0.872
333	0.866	-11.00	0.963	0.994

**Fig. 6.** SEM images of pipeline steel: (a) immersed in 0.5 M HCl (no inhibitor) and (b) immersed in 0.5 M HCl with the presence of *Citrus sinensis* seed extract.

Where C and K both represent the concentrations of the extracts and equilibrium constant.

3.5. Surface Analysis

The surface protection of pipeline steel surface in 0.5 M HCl using *Citrus sinensis* seed extract was investigated by SEM analysis. Fig. 6a shows SEM images of pipeline steel surface immersed in 0.5 M HCl (without the presence of *Citrus sinensis* seed extract). The image showed that the pipeline steel surface is highly rough with number of cracks and pits, which is mainly due to the aggressive attack of the 0.5 M HCl solution. The content of Cl⁻ may be higher in the surface of pipeline steel immersed in 0.5 M HCl. Interestingly, the surface of pipeline steel immersed in 0.5 M HCl with the presence of *Citrus sinensis* seed extract showed very smooth (Fig. 6b). This is due to the formation of protective layer by the *Citrus sinensis* seed extract on the pipeline steel surface. It can be noted that the surface of pipeline steel (after immersion in 0.5 M HCl with the presence of *Citrus sinensis* seed extract) shows some cracks, which may be due to uneven distribution of the extract molecules over the surface of the pipeline steel. It is also possible that the active sites on the pipeline steel are not uniform or do not possess quite equal attractions for the active molecules of the extract. In this case, the adsorption of some particles on a portion of the pipeline steel occur differentially and prevent the acid from attacking that sections by micelle-like conformation or by steric hindrance. This implies adsorption of the extract on the pipeline surface forming a protective layer that inhibits corrosion.^[41]

4. Conclusions

Ethanollic extract of *Citrus sinensis* seed was investigated against the corrosion of Pipeline steel in 0.5 M HCl medium. The inhibition activity of *Citrus sinensis* seed extract was tested under various conditions such as different inhibitor concentration, time and

temperature. The ethanolic extract of *Citrus sinensis* seed considerably reduced the dissolution of the steel in 0.5 M HCl. Based on the result, it is concluded that the inhibitive action of the extract is mainly attributed to its adsorption of inhibitor molecules on the steel surface. The adsorption mechanism of inhibitor molecules on steel surface in 0.5 M HCl obeys Freundlich adsorption model. The inhibitive action of the extract was further shown by the SEM observations, which showed a smoother surface under influence of the extract (formation of protective layer by the inhibitor molecules on the steel surface). The extract of *Citrus sinensis* proved to be a good inhibitor of Pipeline steel corrosion in 0.5 M HCl environment. In addition, the extract of *Citrus sinensis* is green, cost-effective and easily available.

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Conflicts of Interest

The authors declare no conflict of interest.

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