J. Mater. Environ. Sci., 2022, Volume 13, Issue 09, Page 1025-1036

Journal of Materials and Environmental Science ISSN : 2028-2508 e-ISSN : 2737-890X CODEN : JMESCN Copyright © 2022, University of Mohammed Premier Oujda Morocco

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# The inhibition behaviour of extracts from *Plumeria rubra* on the corrosion of low carbon steel in sulphuric acid solution

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Received 01 Sept 2022, Revised 27 Sept 2022, Accepted 28 Sept 2022

- Keywords
- $\checkmark$  Corrosion,
- ✓ Inhibitor efficiency,
- ✓ Plumeria rubra,
- $\checkmark$  Adsorption,
- ✓ Acid cleaning.

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#### Abstract

The corrosion inhibitive action of extracts of flower petals (FP) and leaves (LV) of *Plumeria rubra* on corrosion of low carbon steel in 0.5 M H<sub>2</sub>SO<sub>4</sub> solution was investigated using the classical weight loss approach. Determination of temperature effects and consideration of adsorption behaviour of the extracts were also carried out. The results obtained indicate that the extracts were potent and effective as reliable inhibitors for protection of low carbon steel in the acidic environment. The inhibition efficiency increased with the dosage of the extracts. Evaluation of temperature effects revealed that inhibition efficiency decreased with rise in temperature, a trend which is usually ascribed to physical adsorption of the organic molecules in the extracts on the surface of the steel substrate. With these results, mechanism of physical adsorption of the plant extract molecules on the surface of the substrate is proposed for the inhibition gave results that were best fitted by Langmuir isotherm model.

#### 1. Introduction

Acid cleaning and descaling of steel structures have been known to be a routine industrial practice which is aimed at removing inorganic scales and other unwanted materials from the surface of steel structures and metallic substrates. However, during such industrial procedures, the substrates are exposed to acid corrosion or dissolution in the aggressive cleaning solutions [1-3]. To avoid loss of efficiency and eventual breakdown of the metallic equipment, there is need to protect metal, steel and other alloys against corrosion when in contact with corrosive media [4]. One of the numerous methods to achieve this is by introduction of chemical compounds known as corrosion inhibition additives into the corrodent solution to retard the corrosion reaction and reduce corrosion rate. Such compounds are characterized by the presence of aromatic rings and heteroatoms such as nitrogen, oxygen and sulphur in a conjugated  $\pi$  electron systems which enhance the adsorption of the molecules on the surface of the metals thereby creating a barrier that protects the substrate against corrosion [1,3,5-11].

Extensive review of literature has revealed that most inorganic chemicals such as chromatebased compounds which inhibit corrosion reactions are not only costly, but are also toxic and harmful to both humans and the immediate natural environment [12,13]. It is against this backdrop and in accordance to recent toxicity and safety standards stipulated by the Paris Commission [14,3] that attention has shifted towards the use of natural product-based organic inhibitors especially those of plant origin which have gained global reputation and acceptance because they are environmentally friendly, non-toxic, cheap and readily available [3,5,15]. The extracts from the leaves, stem-bark, flower petals, seeds, roots and fruits of most medicinal plants have been reported to comprise of a myriad of phytochemical compounds which contain the heteroatoms that enhance the mitigation of corrosion in various corrosive environments [5,16-20]. For instance, Oguzie [21] studied the effect of Sansevieria trifasciata leaf extract on aluminium corrosion in KOH and HCl environments. In a related study, Oguzie and co-workers [22] assessed the influence of Ocimum basilicum on the corrosion of aluminium in both KOH and HCl media. Satapathy et al. [23] investigated the inhibiting effect of Justicia gendarussa on the corrosion of mild steel in hydrochloric acid solution. Okafor et al. [24] studied the effect of *Phyllanthus amarus* on the inhibition of corrosion of mild steel in acidic media. Ahanotu and his team [2] investigated the leaves extract of *Pterocarpus santalinoides* as a sustainable and potent inhibitor for low carbon steel in a simulated pickling medium. Khoshsang and Ghaffarinejad [25] studied the effect of Saffron flower petal extract as an eco-friendly corrosion bioinhibitor for carbon steel in HCl solution. Elgyar et al. [26] studied the inhibition action of Viscum album extract on the corrosion of carbon steel in an acid solution. Sedik et al. [15] reported on the experimental and theoretical insights into copper corrosion inhibition using protonated amino acids. A host of similar reports have been documented and the potency of these plant extracts can be accounted for by the presence in them of alkaloids, phenolic compounds such as tannins and other secondary metabolites.

As a contribution to the growing interest on environmentally benign, green, and potent corrosion inhibitors, the present study seeks to determine the inhibition performance and adsorption behavior of extracts of flower petal and leaves of *Plumera rubra* on low carbon steel corrosion in 0.5 M sulphuric acid solution using the classical weight loss technique. *Plumeria rubra*, popularly called red frangipani is a deciduous popular garden and park plant species that belongs to the family of Apocynaceae. It is originally native Mexico, Central America, Colombia and Venezuela [27]. In this study, the effects of temperature on the corrosion rate, inhibition efficiency, as well as adsorption characteristics that govern metal corrosion have also been investigated.

## 2. Methodology

## 2.1 Sourcing and Preparation of Materials

Mature leaves and flower petals of *Plumeria rubra* were locally harvested from the live plant in the former site of Imo State Polytechnic at Umuagwo-Ohaji. The plant samples were botanically authenticated in the botanical garden of the institution. The harvested plant materials were washed with water, air-dried under shade at room temperature and then ground to powder.

A low carbon steel sheet was procured from the Materials and Metallurgical Engineering Workshop of the Federal University of Technology, Owerri, Nigeria. They were mechanically cut into small test coupons of dimension 30 mm  $\times$  15 mm  $\times$  1 mm. The elemental composition of the steel is presented in Table 1. A small open cylindrical hole of diameter 2.5 mm was drilled on the largest surface of each coupon to allow suspension into the aggressive solution with polymeric thread in the weight loss experiments. The test coupons were then abraded using emery paper, washed in absolute ethanol to remove oil and grease particles, dried using acetone and then stored in a moisture-free desiccator prior to weight loss experiments [28].

Table 1: Weight composition of the test coupons

Element	Mn	С	S	Р	Si	Ni	Al	Fe
Wt. (%)	0.43	0.16	0.033	0.02	0.16	0.008	0.007	99.182

#### 2.2 Preparation of Corrodent and Inhibitor Test Solutions

The aggressive acidic solution (corrodent) used in this study was  $0.5 \text{ M H}_2\text{SO}_4$  solution, prepared from research grade sulphuric acid with certified percent purity of 98%, specific gravity of 1.84 and molar mass of 98.097 gmol<sup>-1</sup>. Exactly 27.2 ml of the stock acid was taken and diluted to mark using double-distilled water in a standard 1.0 litre volumetric flask according to equation (1) below;

$$V_{\rm s} = \frac{C_d V_d M}{10\rho\gamma} \tag{1}$$

where  $V_s$  = required volume of the concentrated (stock) acid,  $C_d$  = required molarity of the dilute acid,  $V_d$  = required volume of the dilute acid, M = molar mass of the acid,  $\rho$  = density of the acid, and  $\gamma$  = percent purity of the acid.

Each powdered plant sample (20.0 g) was put into 500 ml of the corrodent in a round bottom flask. The resulting mixture was heated under reflux for 2 hours and left to cool to room temperature. It was thereafter filtered and the extract stock solution was used to prepare five inhibitor test solutions of concentration 0.1 g L<sup>-1</sup>, 0.2 g L<sup>-1</sup>, 0.3 g L<sup>-1</sup>, 0.4 g L<sup>-1</sup> and 0.5 g L<sup>-1</sup>.

#### 2.3 Weight Loss Experiments and Corrosion Rate

The low carbon steel coupons were weighed and this initial weight was recorded as  $W_0$ . With polymeric threads, the test coupons were fully immersed inside the six corrosion cells containing 150 ml of the corrodent at 27<sup>o</sup>C which were then covered with air-tight lids in order to stimulate an anaerobic environment. The test coupons were retrieved at 24 hours intervals for 4 days. On each retrieval the coupons were appropriately washed with the aid of brittle brush inside water to remove corrosion products, degreased with absolute ethanol, dried in acetone and re-weighed, and the final weights after 4 days were recorded as  $W_1$ . The weight loss was deduced as  $W_0 - W_1$ . This procedure was repeated at an elevated temperature of 60<sup>o</sup>C to determine the temperature effect on the corrosion rate. The weight loss measurements were performed in triplicates to ensure reproducibility and the average weight losses were taken and used in subsequent calculations [24,29,30].

Each average weight loss was converted to corrosion rate (R) in mm/yr using equation (2) as follows;

$$R = \frac{K\Delta W}{\rho A t} \tag{2}$$

where K is a constant with a value of 8.76 x  $10^4$ ,  $\Delta W$  is the average weight loss in g of the test coupon at the end of test, A is the total surface area of one test coupon to the nearest 0.01  $cm^2$ , t is the duration of immersion in *hour*, and  $\rho$  is the density of the material of the test coupon in g  $cm^{-3}$ .

#### 2.4 Surface Coverage and Inhibitor Efficiency

With the corrosion rate data, the degree of surface coverage ( $\theta$ ) were calculated according to equation (3) below;

$$\theta = \left[\frac{R_0 - R_i}{R_0}\right] \tag{3}$$

where  $R_0$  and  $R_i$  are the corrosion rates of the uninhibited and inhibited systems respectively. The inhibitor efficiency ( $\eta$ %) was then deduced as expressed in equation (4);

$$\eta\% = 100\theta \tag{4}$$

#### 2.5 Adsorption Considerations

The adsorption behavior of the *Plumeria rubra* extracts was investigated by testing different models of adsorption isotherms to ascertain which one fits the experimental results well. The tested isotherms are the Freundlich, Langmuir, Temkin and Frumkin adsorption isotherm models.

#### 3. Results and Discussion

#### 3.1 Weight Loss, Corrosion Rate, Surface Coverage and Inhibitor Efficiency

The variation of weight loss of the test coupons with time over a period of 4 days in  $0.5 \text{ M H}_2\text{SO}_4$  solution without and with various concentrations of the two PR extracts and at different temperatures is presented in Table 2(a,b) and Table 3(a,b), and graphically illustrated in Figure 1(a,b) and Figure 2(a,b).

**Table 2a:** Variation of weight loss by low carbon steel in  $0.5 \text{ M H}_2\text{SO}_4$  solution without and with PR flower petal extract for 96 h exposure at  $27^{\circ}\text{C}$ 

Extract		Weight I	Losses (g)		Total	R	θ	η%
conc.	24 h	48 h	72 h	96 h	Weight	(mm/yr)		
(g L <sup>-1</sup> )					Lost,			
					$\Delta \mathbf{W}$ (g)			
0.0	0.72	0.68	0.53	0.48	2.41	21.100	_	_
0.1	0.06	0.05	0.04	0.02	0.17	1.574	0.925	92.5
0.2	0.06	0.04	0.03	0.02	0.15	1.377	0.935	93.5
0.3	0.05	0.03	0.01	0.00	0.09	0.839	0.960	96.0
0.4	0.05	0.02	0.01	0.00	0.08	0.744	0.965	96.5
0.5	0.04	0.02	0.01	0.00	0.07	0.698	0.967	96.7

**Table 2b:** Variation of weight loss by low carbon steel in  $0.5 \text{ M H}_2\text{SO}_4$  solution without and with PR leaf extract for 96 h exposure at  $27^{0}\text{C}$ 

Extract		Weight I	Losses (g)		Total	R	Θ	η%
conc.	24 h	48 h	72 h	96 h	Weight	(mm/yr)		
(g L <sup>-1</sup> )					Lost,			
					$\Delta \mathbf{W}$ (g)			
0.0	0.74	0.63	0.53	0.49	2.39	22.380	-	-
0.1	0.07	0.06	0.04	0.03	0.20	1.881	0.916	91.6
0.2	0.07	0.05	0.03	0.02	0.17	1.610	0.928	92.8
0.3	0.06	0.05	0.04	0.01	0.16	1.529	0.932	93.2
0.4	0.05	0.04	0.03	0.01	0.13	1.248	0.944	94.4
0.5	0.04	0.04	0.01	0.00	0.09	0.847	0.962	96.2

Extract		Weight I	Losses (g)		Total	R	θ	η%
conc.	24 h	48 h	72 h	96 h	Weight	(mm/yr)		
(g L <sup>-1</sup> )					Lost,			
					$\Delta \mathbf{W}$ (g)			
0.0	0.75	0.71	0.59	0.53	2.58	22.495	-	-
0.1	0.07	0.06	0.06	0.04	0.23	2.075	0.908	90.8
0.2	0.06	0.05	0.05	0.03	0.19	1.740	0.923	92.3
0.3	0.05	0.04	0.04	0.01	0.14	1.236	0.945	94.5
0.4	0.04	0.04	0.03	0.00	0.11	0.999	0.956	95.6
0.5	0.04	0.03	0.02	0.00	0.09	0.828	0.963	96.3

**Table 3a:** Variation of weight loss by low carbon steel in  $0.5 \text{ M H}_2\text{SO}_4$  solution without and with PR flower petal extract for 96 h exposure at  $60^{\circ}\text{C}$ 

**Table 3b:** Variation of weight loss by low carbon steel in  $0.5 \text{ M H}_2\text{SO}_4$  solution without and with PR leaf extract for 96 h exposure at  $60^{\circ}\text{C}$ 

Extract		Weight I	Losses (g)		Total	R	Θ	η%
conc.	24 h	48 h	72 h	96 h	Weight	(mm/yr)		
(g L <sup>-1</sup> )					Lost,			
					$\Delta \mathbf{W}$ (g)			
0.0	0.77	0.67	0.58	0.45	2.47	23.077	-	-
0.1	0.09	0.08	0.04	0.03	0.24	2.252	0.902	90.2
0.2	0.08	0.06	0.04	0.02	0.20	1.864	0.919	91.9
0.3	0.07	0.05	0.05	0.02	0.19	1.767	0.923	92.3
0.4	0.06	0.05	0.04	0.01	0.16	1.485	0.936	93.6
0.5	0.06	0.05	0.01	0.00	0.12	1.121	0.951	95.1

It is observed from Figures 1(a,b) and 2(a,b) that there is a huge weight loss by the test coupon immersed in the blank system where extract concentration is zero compared to other corrosion cells that contain the PR extract at the different temperatures investigated. A cursory observation of the variation of weight loss with time for the two systems reveals that weight loss decreases as the immersion duration increases at the various extract concentrations. This behavior could be interpreted to imply that the extracts became more potent to suppress corrosion reaction as the exposure time is increased.

Figure 3 reveals that the rate of corrosion reaction of low carbon steel steel in the hostile solution was dependent on the extract concentration as low corrosion rates were recorded as extract concentration was increased. With PR flower petal extract, a mass of 2.41 g was lost in the blank system after 96 hour immersion which resulted to a high corrosion rate of value 21.100 mm/yr. But with the introduction of 0.1 g L<sup>-1</sup> of the PR flower petal based inhibitor into the second cell, there is a drastic drop in weight loss to only 0.17 g, resulting to a corresponding decrease in corrosion rate to 1.574 mm/yr. Hence, the inhibition efficiency obtained at 0.1 g L<sup>-1</sup> of the extract was as high as 92.5% (see Figure 4). A similar observation was made at that temperature with the PR leaf based inhibitor which dropped the corrosion rate from 22.380 mm/yr in the blank system to 1.881 mm/yr with 0.1 g L<sup>-1</sup> of the extract, resulting to inhibitor efficiency of 91.6% (Figure 4).



**Figure 1a:** Variation of weight loss with time for low carbon steel in 0.5 M  $H_2SO_4$  without and with PR flower petal extract for 96 h immersion at  $27^{0}C$ 



Figure 2a: Variation of weight loss with time for low carbon steel in 0.5 M  $H_2SO_4$  without and with PR flower petal extract for 96 h immersion at  $60^{0}C$ 



Figure 3: Variation of corrosion rate of low carbon steel in 0.5M H<sub>2</sub>SO<sub>4</sub> without and with various concentrations of PR extracts for 96 h immersion at 27  $^{0}$ C and 60  $^{0}$ C



**Figure 1b:** Variation of weight loss with time for low carbon steel in 0.5 M  $H_2SO_4$  without and with PR flower leaf extract for 96 h immersion at  $27^{0}C$ 



**Figure 2b**: Variation of weight loss with time for low carbon steel in 0.5 M  $H_2SO_4$  and with PR flower leaf extract for 96 h immersion at  $60^{0}C$ 



**Figure 4:** Variation of inhibitor efficiency ( $\eta$ %) with various concentrations of PR-based inhibitors for low carbon steel corrosion in 0.5M H<sub>2</sub>SO<sub>4</sub> for 96 h immersion at 27<sup>o</sup>C and 60<sup>o</sup>C

With increasing concentration of the extracts from 0.1 g L<sup>-1</sup> to 0.5 g L<sup>-1</sup> in each case, the corrosion rate decreased tremendously to 0.698 mm/yr and 0.847 mm/yr respectively, while percent inhibition efficiency increased proportionally to 96.7% and 96.2% respectively. This shows that these two extracts of *Plumeria rubra* are potent in protecting the substrates from acid attack. Table 5 shows the various phytochemical constituents of the Plumeria rubra extracts studied. The higher inhibition efficiency recorded at higher extract concentrations may have been occasioned by the synergistic or antagonistic effects of these various organic constituents contained in the studied extracts [31-32] and this suggests that most of the inhibitor molecules are adsorbed on the surface of the test coupons thereby increasing the degree of surface coverage,  $\theta$ , of the coupons. The larger the degree of surface coverage resulting from this phenomenon, the greater the protection efficiency of the extract. The layer of adsorbed inhibitor molecules creates a barrier that hinders the transfer of charge and mass between the low carbon steel and the aggressive acid solution, thereby protecting the specimens from further acid attack [33]. The effect of elevated temperature on the performance of these plant-based inhibitors could be observed by comparing the data in Table 2(a,b) with those in Table 3(a,b). This study has revealed that at elevated temperature, the corrosion rate increased while the inhibitor efficiency decreased proportionally both without and with the different concentrations of the plant-based inhibitors. This trend is often ascribed to physisorption involving electrostatic interaction between charged adsorbed inhibitor molecules and the atoms/ions on the surface of the substrate (carbon steel) [33-35]. The two extracts functioned effectively as corrosion inhibitors for the substrate at the two temperatures under study and their protection efficiencies slightly decreased by the temperature rise as no more than 1.7% efficiency was lost in the two systems at the elevated temperature. However, *Plumeria rubra* flower petal extract performed better than the leaf counterpart. It is pertinent to state that discussing the adsorption characteristics of these extracts in terms of thermodynamic parameters such as standard free energy of adsorption, heat of adsorption as well as activation energy would not be wise given the fact that the studied extracts contained infinite number of secondary metabolites whose molecular masses are not determined [23,32,36,37].

#### 3.2 Adsorption Behaviour of Plumeria rubra Extracts

It is a general knowledge that acid corrosion inhibitors act by adsorption on the surface of the substrate. In order to understand the adsorption behavior of the PR based-inhibitors on carbon steel in corrodent solution, various adsorption isotherm models were tested. However, the Langmuir isotherm model was found to give the best fit with the experimental data. The Langmuir adsorption isotherm linear equation [23] is given as follows;

$$\frac{C_e}{\theta} = \frac{1}{K_{ads}} + C_e \tag{5}$$

where  $\theta$  is the degree of surface coverage,  $C_e$  is the extract concentration and  $K_{ads}$  the equilibrium constant for adsorption which is evaluated from the intercept of the plots and is related to the standard free energy of adsorption [21]. Figure 5(a,b) shows the plots of  $C_e/\theta$  versus  $C_e$  for both extracts at both temperatures and the graphs produced straight lines, indicating that the adsorption of the PR extract molecules followed Langmuir adsorption isotherm and this is evident in the correlation coefficients. The correlation coefficients ( $\mathbb{R}^2$  values) for the two extracts under study at both temperatures are very close to unity, indicating a strong agreement with Langmuir adsorption isotherm. Table 4 gives the parameters deduced from the Langmuir isotherm plots for the two plant extracts.

Temp. (K)	Extract	$R^2$	Intercept	Slope	K <sub>ads</sub>
300 K	PR flower petal	0.99986	0.0079	1.015	126.58
	PR leaf	0.99907	0.0067	1.039	149.25
333 K	PR flower petal	0.99982	0.0096	1.022	104.17
	PR leaf	0.99952	0.0095	1.039	105.26

Table 4: Parameters from the Langmuir isotherm model for PR based inhibitors



**Figure 5a:** Langmuir isotherm plots for the PR based inhibitors adsorbed on low carbon steel surface in  $0.5 \text{ M H}_2\text{SO}_4$  solution at  $27^{0}\text{C}$ 



**Figure 5b:** Langmuir isotherm plots for the PR based inhibitors adsorbed on low carbon steel surface in  $0.5 \text{ M H}_2\text{SO}_4$  solution at  $60^{\circ}\text{C}$ 

As the natural extract contains contain infinite compounds at different concentrations, as well as its concentration is expressed by mL/L or mg/L or g/L and the free enthalpy in kJ/M, safely, the evaluation of free enthalpy is not conducted as pointed out by several authors [38-44]. In this way, inhibitory action occurs is probably due to the synergistic intermolecular effect of the various constituents of the aqueous extract studied [42-46].

Plant part	Phytochemicals						
PR flower petals (PRFP)	Alkaloids, flavonoids, phenolics, tannins, terpenoids, glycosides						
PR leaves (PRL)	Alkaloids, flavonoids, tannins, steroids, terpenoids, saponins, carbohydrates proteins						
	carbonydrates, proteins						

Table 5: Major phytochemical constituents of the Plumeria rubra extracts

# [47-50]

# Conclusions

- 1. Extracts of *Plumeria rubra* flower petals (FP) and *Plumeria rubra* leaves (LV) function as inhibitor for corrosion of low carbon steel in  $H_2SO_4$  solution and inhibition efficiencies followed the trend: FP > LV.
- 2. Inhibition efficiency of both extracts increases with increase in concentration of extract and decreases with increase in temperature suggesting physical adsorption.
- 3. The high efficiency of inhibition is probably due to the synergistic effect of the studied extract.
- 4. The corrosion reaction is suppressed by adsorption of FP and LV extracts on the low carbon steel surface following the Langmuir isotherm.

**Acknowledgements :** The provision, cutting and drilling of holes in the test coupons by the Engineering Worskshop of the Department of Materials and Metallurgical Engineering, Federal University of Technology, Owerri, Nigeria are acknowledged.

**Disclosure statement:** *Conflict of Interest:* The authors declare that there are no conflicts of interest. *Compliance with Ethical Standards:* This article does not contain any studies involving human or animal subjects.

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