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Inhibitive action of *Leptadenia pyrotechnica* extract on the corrosion of Mild Steel in H_2SO_4 solution

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ABSTRACT

Inhibitive action on corrosion of mild steel in acidic media has been studied using Mass Loss Technique and Thermometric Method. Corrosion of mild steel in H_2SO_4 solution can be effectively inhibited by addition of extract of *Leptadenia pyrotechnica* as green corrosion inhibitor. The results indicate that all the extracts inhibited the corrosion process in acid media by virtue of adsorption and inhibition efficiency improved with inhibitor concentration. The highest inhibition efficiency was found upto 87.04% in 1.0N H_2SO_4 with 0.8% extract. It is concluded that *Leptadenia pyrotechnica* is an effective corrosion inhibitor and can safely be used as green corrosion inhibitor without any toxic effect.

Keywords: Corrosion, Mild steel, Leptadenia pyrotechnica, Inhibition efficiency, Mass Loss Technique, Thermometric Method

INTRODUCTION

Industries depend heavily on the use of metals and alloys. Mild steel is extensively used in different industries by virtue of its good structure, properties, mechanical workability and low cost. One of the most challenging and difficult tasks for industries are the protection of metals from corrosion. Corrosion is a ubiquitous problem that continues to be of great relevance in a wide range of industrial applications and products; it results in the degradation and eventual failure of components and systems both in the processing and manufacturing industries and in the service life of many components. Mitigation of this problem of corrosion lies on the use of corrosion inhibitors, which help to prolong the life spasm of these materials. Corrosion inhibitors are organic or inorganic chemical compounds which are usually used in small concentration whenever a metal is in contact with an aggressive medium. Presence of hetero atom (S, N and O) with free electron pairs, aromatic rings with delocalized π-electrons, alkyl chains with high molecular weight and substituent group in organic compound generally improve the inhibition efficiency.[1]

Nevertheless, the known hazardous effects of most synthetic organic inhibitors and the need to develop cheap, non-toxic and environmentally benign processes have now made researchers to focus on the use of natural products. Plant extracts are viewed as an incredibly rich source of naturally occurring chemical compounds that can be extracted from herbs, spices and medicinal plants by simple procedures with low cost and are biodegradable in the environment. There are numerous naturally occurring substances like Heena,[2] Tamarind, Tea leaves, Tannin, pomegranate juice and peel,[3] Quinoline based cinchona alkaloids, and very popular ayurvedic powder Mahasudarshana churna have been reported as corrosion combating material. Corrosion inhibition efficiency of *Eugenia jambolans*,[4] *Adhatoda vasica*, *Prospis juliflora*,[5] *Datura Stromonium*,[6] Brahmi,[7] *Hibiscus Cannabinus*,[8] Garlic,[9] *Ocimum sanctum*,[10] *Cordia dichotoma*[11] have also been reported. Recently the use of naturally occurring substances like *Pennisetum glaucum*,[12] Molasses,[13] Pumpkins,[14] Black tea,[15] Bhringaraj,[16] *Araucaria columnaris*,[17] *Pergularia daemia*[18] and *Phoenix dactylifera*[19] have been evaluated as effective green corrosion inhibitors.

In the present studies the inhibitive effects of ethanolic extract of stem, fruit and root of *Leptadenia pyrotechnica* have been tested. *Leptadenia pyrotechnica* (common name – Kheep) is an erect, ascending shrub with green stem and pale green alternating bushy branches with watery sap.

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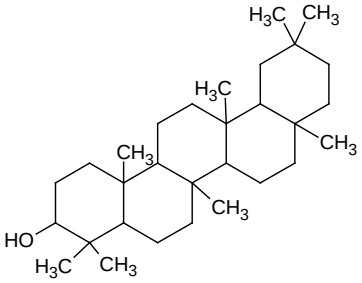
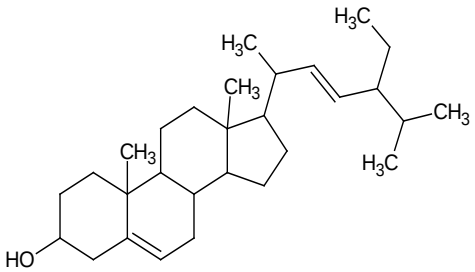
CHEMICAL CONSTITUENTS

Three terpenes (phytol, squalene and taraxerol), five sterols (cholesterol, campasterol, stigmasterol, β-sitosterol and fucosterol), fifteen fatty acids (C14-C25), eleven n-alkanol (C29-C39) has been isolated from extract of *Leptadenia pyrotechnica.*[19] Moustafa et al. reported the isolation of twenty four alkaloids and three glycosides from the aerial parts of the *Leptadenia pyrotechnica.*[19] Almost all of the alkaloids belonged to heterocyclic group including pyridine, pyrrole, pyrazine and indole types.



Taraxerol



Stigmasterol

EXPERIMENTAL

Mild steel having composition of 0.14% C, 0.11% Si, 0.35% Mn, 0.75%Ni, 0.025% P, 0.03% S and the rest of Fe, specimens used in the mass loss experiments were mechanically cut from commercially available mild steel samples into coupons of 2.5cm x 1.55cm x 0.02cm with a small hole of about 2mm diameter near the upper edge. Specimens were cleaned by buffing to produce spotless finish and then degreased. Different concentration solutions of hydrochloric acid were prepared using double distilled water.

The extract of stem, fruit and root of *Leptadenia pyrotechnica* were obtained by drying, then finely powdered and extracted with boiling ethanol.[20,21] The solvent is distilled off and the residue is treated using inorganic acid, where the bases are extracted as their soluble salt. The free bases are librated by the addition of any bases and extracted with various solvents, e.g. ether, chloroform etc. Each specimen was suspended by a glass hook and immersed in a beaker containing 50 mL of test solution with or without inhibitor at room temperature and left exposed to air. Evaporation losses were made up with distilled water. Duplicate experiments were performed for each and mean values of mass loss were calculated. The percentage inhibition efficiency was calculated as-

$$\text{Inhibition efficiency } (\eta\ \%) = \frac{100(\Delta M_u - \Delta M_i)}{\Delta M_u} \quad (1)$$

Where, ΔM_u and ΔM_i are the mass loss of the specimen in uninhibited and in inhibited solution respectively.

The degree of surface coverage (θ) can be calculated as-

$$\text{Surface coverage } (\theta) = \frac{(\Delta M_u - \Delta M_i)}{\Delta M_u} \quad (2)$$

The corrosion rates in mmpy can be obtained by the following equation-

$$\text{Corrosion rate (mmpy)} = \frac{\text{Mass loss x } 87.6}{\text{Area x Time x Metal density}} \quad (3)$$

Where mass loss is expressed in mg. Area is expressed in cm^2. Exposed time is expressed in hours and metal density is expressed in gm/cm^3.

Inhibition efficiency was also calculated using thermometric method. This involves the immersion of specimen (dimension 2.5cm x 1.55cm x 0.02cm) in an insulating reaction chamber having 50 mL of test solution at an initial room temperature. Temperature change was observed at regular intervals using a thermometer with a precision of 0.1^0C. Initially the increase in temperature was slow, then rapid, attaining a maximum value and then decreased. The maximum temperature was noted. The inhibition efficiency was calculated as

$$\text{Inhibition efficiency } (\eta\ \%) = \frac{100(RN_f - RN_i)}{RN_f} \quad (4)$$

Where RN_f and RN_i are the reaction number in the free solution and inhibited solution respectively.

Reaction number RN ($K\ min^{-1}$) is given as:

$$RN = \frac{T_m - T_i}{t} \quad (5)$$

Where, Tm and Ti are the maximum temperature of solution and in initial temperature of solution respectively. t is time required (in minutes) to attain maximum temperature.

The coefficient of correlations (r) between the inhibitor concentration and inhibition efficiency can be calculated by using the formula-

$$r = \frac{N\sum dx\ dy - (\sum dx)(\sum dy)}{\sqrt{N\sum dx^2 - (\sum dx)^2[N\sum dy^2 - (\sum dy)^2]}} \quad (6)$$

Where x is inhibitor concentration and y is inhibition efficiency. N is number of test sample.

Table 1. Mass loss data for mild steel in H_2SO_4 with alcoholic extracts of plant Leptadenia pyrotechnica at 299 ±0.1K. [Area of exposure- 7.75 cm^2]

	0.5N H_2SO_4 (48 hrs)				1.0N H_2SO_4 (48hrs)			
Inhibitor Concentration (%)	Mass Loss (Δm) Mg	Inhibition Efficiency (η %)	Corrosion Rate (mmpy)	Surface Coverage (θ)	Mass Loss (Δm) mg	Inhibition Efficiency (η %)	Corrosion Rate (mmpy)	Surface Coverage (θ)
Stem extract								
Blank	325		9.7482		463		13.8875	
.2	130	60.00	3.8993	0.6000	108	76.67	3.2394	0.7667
.4	113	65.23	3.3894	0.6523	89	80.78	2.6695	0.8078
.6	88	72.92	2.6395	0.7292	76	83.58	2.2796	0.8358
.8	72	77.85	2.1596	0.7785	60	87.04	1.7997	0.8704
Fruit extract								
Blank	325		9.7482		463		13.8875	
.2	137	57.85	4.1092	0.5785	119	74.30	3.5693	0.7430
.4	116	64.31	3.4794	0.6431	106	77.11	3.1794	0.7711
.6	91	72.00	2.7295	0.7200	88	80.99	2.6395	0.8099
.8	76	76.62	2.2796	0.7662	71	84.67	2.1296	0.8467
Root extract								
Blank	325		9.7482		463		13.8875	
.2	141	56.62	4.2292	0.5662	123	73.43	3.6893	0.7343
.4	120	63.08	3.5993	0.6308	118	74.51	3.5394	0.7451
.6	105	67.69	3.1494	0.6769	95	79.48	2.8495	0.7948
.8	87	73.23	2.6095	0.7323	80	82.72	2.3996	0.8272
	1.5N H_2SO_4 (72 hrs)				2.0 N H_2SO_4 (72 hrs)			
Stem extract								
Blank	1159		23.1758		1730		34.5937	
.2	324	72.04	6.4788	0.7504	467	73.01	9.3383	0.7301
.4	317	72.65	6.3388	0.7608	452	73.87	9.0383	0.7387
.6	305	73.68	6.0989	0.7806	439	74.62	8.7784	0.7462
.8	290	74.98	5.7989	0.8502	424	75.49	8.4784	0.7549
Fruit extract								
Blank	1159		23.1758		1730		34.5937	
.2	325	71.96	6.4988	0.7196	473	72.66	9.4582	0.7266
.4	321	72.30	6.4188	0.7230	460	73.41	9.1983	0.7341
.6	316	72.74	6.3188	0.7274	444	74.34	8.8784	0.7434
.8	309	73.34	6.1786	0.7334	425	75.50	8.4984	0.7550
Root extract								
Blank	1159		23.1758		1730		34.5937	
.2	340	70.66	6.7987	0.7066	480	72.25	9.5982	0.7225
.4	329	71.61	6.5788	0.7161	465	73.12	9.2983	0.7312
.6	322	72.22	6.4388	0.7222	451	73.93	9.0183	0.7393
.8	313	72.99	5.2588	0.7299	428	75.26	8.5584	0.7526

RESULTS AND DISCUSSIONS

Mass loss and inhibition efficiency for mild steel in various concentration of acid inhibitor are shown in table 1. It is observed that the inhibition efficiency increases with increase in the concentration of inhibitor and also increases with increase in acid strength. The corrosion rate decreases with increase in concentration of inhibitor. All the inhibitor has reduced the corrosion rate to a significant extent.

Stem, fruit and root extract of *Leptadenia pyrotechnica* exhibit maximum inhibition efficiency up to 87.04%, 84.67% and 82.72% respectively in 1.0 N H_2SO_4. The result reveals that inhibition efficiency increases with increase in inhibition concentration from 0.2% to 0.8%.

Table- 2. Reaction Number (RN) and Inhibition efficiency (η %) for mild steel in H_2SO_4 at 299±0.1 K with alcoholic extracts of plant Leptadenia pyrotechnica. [Area of exposure- 7.75 cm^2]

Inhibitor concentration	**1N H_2SO_4**		**2N H_2SO_4**		**3N H_2SO_4**	
Stem extract	(RN)	(η %)	(RN)	(η %)	(RN)	(η %)
Blank	0.1836	-	0.1929	-	0.2103	-
.2	0.0543	70.42	0.0518	73.15	0.0535	74.56
.4	0.0512	72.11	0.0466	75.84	0.0464	77.94
.6	0.0436	76.25	0.0422	78.12	0.0413	80.36
.8	0.0319	82.63	0.0282	85.38	0.0279	86.73
Fruit extract						
Blank	0.1836	-	0.1929	-	0.2103	-
.2	0.0601	67.27	0.0635	67.08	0.0619	70.53
.4	0.0548	70.15	0.0521	72.79	0.0511	75.50
.6	0.0466	74.62	0.0455	76.41	0.0435	79.32
.8	0.0346	81.15	0.0350	81.36	0.0347	83.50

Root extract						
Blank	0.1836	-	0.1929	-	0.2103	-
.2	0.0570	68.95	0.0595	69.05	0.0632	69.95
.4	0.0504	72.55	0.0521	72.99	0.0531	74.75
.6	0.0451	75.44	0.0462	76.05	0.0470	77.65
.8	0.0382	79.19	0.0369	80.87	0.0357	83.02

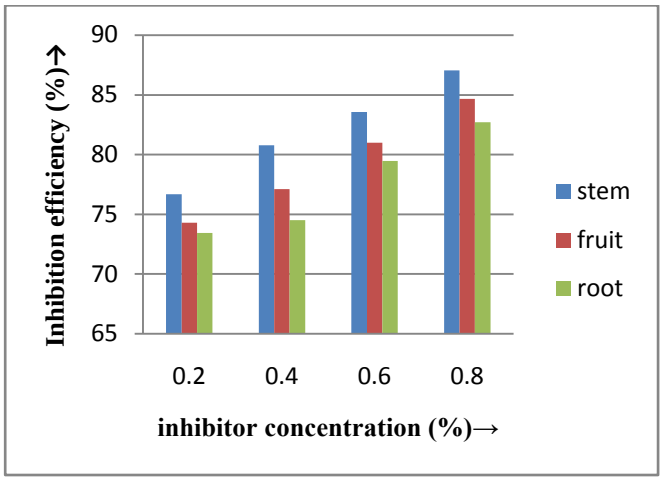


Figure 1. variation of inhibition efficiency (η %) with inhibitor concentration (%) for mild steel in 1.0 N H_2SO_4 of *Leptadenia pyrotechnica* (72 hrs)

Inhibition efficiency was also determined using the thermometric method. Reaction numbers (RN) and inhibition efficiency in various concentrations of H_2SO_4 (1N, 2N and 3N) are summarized in table- 2.

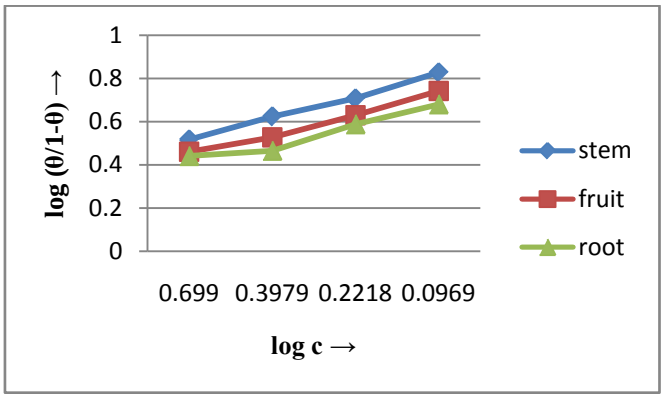


Figure 2. Langmuir adsorption isotherm for mild steel in 1.0 N H_2SO_4 with alcoholic extracts of *Leptadenia pyrotechnica* (72hrs)

The maximum inhibition efficiency obtained with the highest concentration (0.8%) of inhibitor and with the highest concentration of H_2SO_4 (3.0N). The maximum inhibition efficacies observed by thermometric measurement are 86.73%, 83.50% and 83.02% for the stem, fruit and root respectively. The variation in reaction number (RN) with inhibitor concentration is depicted graphically in Fig.-2.

The value of correlation coefficient (r = 0.9954, 0.9925 and 1.0000) indicate that there is a high degree positive correlation between concentration and inhibition efficiency (η %), which proves that the inhibition efficiency increases with increase in the inhibitor concentration.

Table 3.Value of coefficient of correlation (r) between Inhibition efficiency (η %) and Inhibitor concentration of *Leptadenia pyrotechnica* for mild steel in 1.0 N H_2SO_4 [Time- 24 hrs]

Inhibitor concentration	**Inhibition efficiency**	**X- $\bar{X}$**	**Y-$\bar{Y}$**	**$(dx)^2$**	**$(dy)^2$**	**dx x dy**
			Stem			
0.2	67.19	-0.3	-6.7225	0.09	45.1920	2.0168
0.4	70.36	-0.1	-3.5525	0.01	12.6203	0.3552
0.6	76.28	0.1	2.3675	0.01	5.6051	0.2368
0.8	81.82	0.3	7.9075	0.09	62.5286	2.3723
$\bar{X}$ =0.5	$\bar{Y}$=73.91	∑ X- $\bar{X}$=0	∑ Y-$\bar{Y}$=0	∑dx^2=0.2	∑dy^2=125.9459	∑ dx x dy =4.9811
			r =0 .9954			
			Fruit			
0.2	60.08	-0.3	-9.3875	0.09	88.1252	2.8163
0.4	64.43	-0.1	-5.0375	0.01	25.3764	0.5037
0.6	73.91	0.1	4.4425	0.01	19.7358	0.4443
0.8	79.45	0.3	9.9825	0.09	99.6503	2.9948
$\bar{X}$ =0.5	$\bar{Y}$=69.47	∑ X- $\bar{X}$=0	∑ Y-$\bar{Y}$=0	∑dx^2=0.2	∑dy^2=232.8877	∑ dx x dy =6.7591
			r = 0.9925			
			Root			
0.2	58.10	-0.3	-9.29	0.09	86.3040	2.7870
0.4	62.84	-0.1	-4.55	0.01	20.7025	0.4550
0.6	71.15	0.1	3.76	0.01	14.1376	0.3760
0.8	77.47	0.3	10.08	0.09	101.6064	3.0240
$\bar{X}$ =0.5	$\bar{Y}$=67.39	∑ X- $\bar{X}$=0	∑ Y-$\bar{Y}$=0	∑dx^2=0.2	∑dy^2=222.7506	∑ dx x dy =6.6420
					r =1.0000	

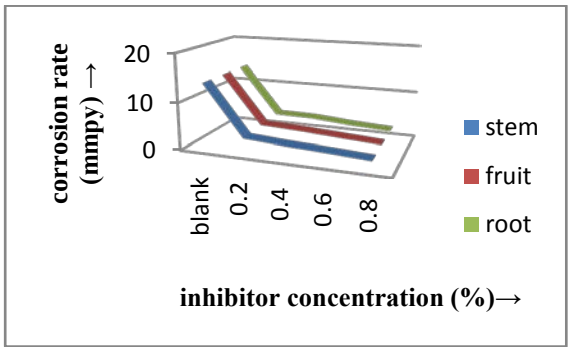


Figure 3. Variation of corrosion rate with inhibitor concentration (%) of plant *Leptadenia pyrotechnica* for mild steel in 1.0N H_2SO_4 (72hrs)

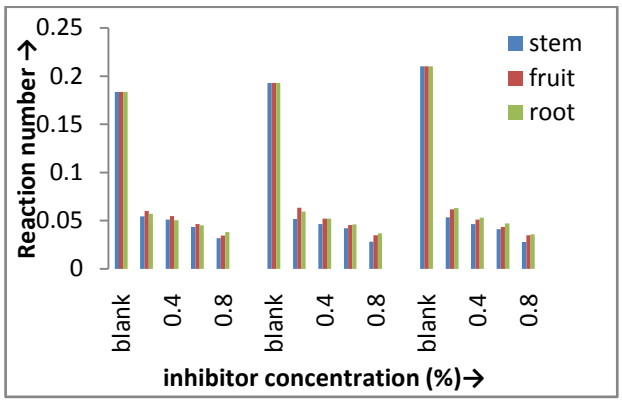


Figure 4. Variation of reaction number with Inhibitor concentration for mild steel in 1.0 N H_2SO_4 (24 hrs)

CONCLUSIONS

The alcoholic extracts of *Leptadenia pyrotechnica* are found to be effective inhibitor in acid media giving up to 87.04% efficiency and can safely be used without any corrosion damage, toxic effect and pollution.

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