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Aqueous extract of *Ipomoea carnea* leaves as green corrosion inhibitor for mild steel in pickling paste

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ABSTRACT

The inhibitor efficiency increased from 50% to 66%. In mixtures of different proportions of HCl (4N) and H_2SO_4 (4N), *Ipomoea carnea* showed 61% to 68% protection. The inhibitor efficiency of Ipomoea increased with increase in time of application. The rate of dissolution increased with temperature. Results also show that inhibitor maintained its effectiveness over entire range of temperature. Corrosion current reduced with time both in uninhibited and inhibited paste. In uninhibited paste, the reduction in current was 11 ma in 1 hour while in inhibited paste it was 14 ma in 1 hour. The inhibitor has anodic polarization effect and it polarized mild steel by 10 mV whereas the cathodic polarization was less and it polarized mild steel by 6 mV. Thus, the inhibitor is mainly an anodic inhibitor.

Keywords: *Ipomoea carnea, mild steel, inhibitor, polarize*

1. INTRODUCTION

Iron and mild steel are used in large quantities for structural purposes and for fabrication of machine tools. Iron on exposure to moist air, is found to be covered with a reddish – brown coating called rust. The rust consists essentially of hydrated ferric oxide, $Fe_2O_3.3H_2O$, together with small quantity of ferrous carbonate, $FeCO_3$.Acid solutions are usually used for pickling in order to remove rust from their surface. Results indicate that metal dissolves most rapidly in pure sulfuric acid solution, somewhat more slowly in pure hydrochloric acid and slowest of all in pure phosphoric acid [1]. The dissolution of iron in H_2SO_4 is slowed down by halide ions [2].

$$Fe + 2HCl = FeCl_2 + H_2$$

The hydrogen molecule, due to slow rate of formation in some cases, penetrates the crystal lattice and deforms it leading to brittleness of metal. Organic, inorganic, or a mixture of both inhibitors can inhibit corrosion by either chemisorption on the metal surface or reacting with metal ions and forming a barrier-type precipitate on its surface [3]. Because of the toxic nature and/or high cost of some chemicals currently in use as inhibitors, it is necessary to develop environmentally acceptable and inexpensive ones. Natural products can be considered as a good source for this purpose. The aqueous extracts from different parts of some plants such as Henna, *Lawsonia inermis* [4], Rosmarinous officinalis L. [5], *Carica papaya* [6], *cordia latifolia* and curcumin [7], date palm, *phoenix dactylifera,* henna, *lawsonia inermis,* corn, *Zea mays* [8], and *Nypa Fruticans Wurmb* [9] have been found to be good corrosion inhibitors for many metals and alloys. Leaves extracts are used

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as common corrosion inhibitors. The anticorrosion activity of Meethi neem (*Murraya koenigii),* Amla (*Emblica officianilis*), Black Myrobalan (*Terminalia chebula*), soapberry (*Sapindus trifolianus*), and Shikakai (*Accacia conicianna*) was investigated. Corrosion inhibition has also been studied for the extracts of Beautiful swertia (*Swertia angustifolia*). Similar results were also shown by Eucalyptus (*Eucalyptus sp.*) leaves, Jambolan (*Eugenia jambolana*), sugar-apple (*Annona squamosa*), Babul (*Acacia Arabica*), Papaya (*Carica papaya*), Neem (*Azadirachta indica*) and Ironweed (*Vernonia amydalina*) were used for steel in acid media. Attap palm (*Nypa fructicans*) wurmb leaves were studied for the corrosion inhibition of mild steel in HCl media. Castor (*Ricimus communis*) leaves were studied for the corrosion inhibition of mild steel in acid media in addition to the use of herbs such as coriander, hibiscus, anis, black cumin, and garden cress as new type of green inhibitors for acidic corrosion of steel [10 - 15].Seeds are of great concern for corrosion inhibition studies. Tobacco (*Nicotiana*), black pepper (*Piper nigrum*), castor seeds oil (Ricinus communis), *acacia* gum, and lignin can be good inhibitors for steel in acid medium. *Papaya*, *Poinciana pulcherrima*, Fedegoso (*Cassia occidentalis*), and Datura (*Datura stramonmium*) seeds are efficient corrosion inhibitors for steel [16 - 18]. In the present work our aim is to use inhibited pickling acid in the paste form so that it can be conveniently applied on large structures as well as on small tools to be pickled / cleaned. As a contribution to the current interest on environmentally friendly, green, corrosion inhibitors, the present study investigates the inhibiting effect of leaves extract of *Ipomoea carnea*, a green inhibitor which is commonly known as The Morning Glory.

2. EXPERIMENTAL SECTION

Mild steel (Fe 99.30%, C 0.076%, Si 0.026%, Mn 0.192%, P 0.012%, Cr 0.050%, Ni 0.050%, Al 0.023%, and Cu 0.135%) panels of size 10 cm * 7.5 cm of pickled cold rolled closed annealed mild steel (18 SWG) cut from a single sheet were used in all experiments. For identification of specimens all were numbered and a suspension hole of about 2 mm diameter near upper edge was made. The specimens were polished to mirror finish with emery paper. They were cleaned with cotton to remove powder and traces of adhered metal, and then they were degreased with sulfur – free toluene followed by cleaning with methanol before experiments. All the acid and chemicals used in the experiment were of AR grade quality. Distilled water was used for the preparation of solution. In the study of mixture of HCl and H_2SO_4, 4N solutions of each acid was prepared and mixed in different proportions 0 – 100 to 100 – 0. Clay – soil was collected, washed, dried, powdered and sieved. 100 gm sieved soil was taken in a plastic glass with a hole at the bottom. This glass was put over uninhibited and inhibited acids. Soil soaked acid uniformly and thus pickling paste was prepared. 100 gm soil soaked 31.3 cc acid. Polished and weighed panels were suspended by a V-shaped hook made of capillary over 100 % humidity for 6 months at room temperature. In 6 months, heavy rust appeared on the panels. Panels were re-weighed to get the amount of rust. Pickling paste was applied over weighed rusted panels under different conditions. After the experiment, paste was removed by washing with saturated sodium bicarbonate solution. The panels were again washed with water and dried with hot air. The panels were finally weighed to get the amount of rust dissolved. Experiments were conducted in triplicate and mean value is reported in the Table. The leaves of *Ipomoea carnea* were crushed and squeezed. Liquid, thus obtained was used as inhibitor. 1 cc of extracted liquid was added to 100 cc of acid for the preparation of inhibited pickling paste. The inhibitor efficiency was calculated from the following equation:

$$\%IE = \frac{W_u - W_i}{W_u} X\ 100$$

Where, %IE = Inhibitor efficiency; W_u = Wt. loss without inhibitor; W_i = Wt. loss with inhibitor

Corrosion current was measured using ammeters of ma range by making galvanic couples of mild steel and platinum. Mild steel and platinum couple was put in pickling paste (inhibited and uninhibited), they were connected through ammeter to record the corrosion current flowing through couple. Corrosion current as a function of time was measured. Polarization measurements were made using following instruments: Transistorized current source (CV/CC 30 V-/A with voltmeter and ammeter); Equitronics Digital Potentiometer 4 ½ Digit; Equitronics Digital Current Meter; Decade Resistance Box; Saturated Calomel Electrode. Current was supplied from power source through available resistance. Current was measured using a multimeter. Steel was used as working electrode and platinum foil as auxiliary electrode. Variables Studied: Concentration of inhibitor; Concentration of acid; Period of Exposure; Temperature; Corrosion Current; Polarization (Anodic & Cathodic).

3. RESULTS SECTION

3.1. Effect of concentration of *Iopomoea carnea* in HCl + H_2SO_4. The effect of concentration of *Ipomoea* 0.01% to 5.0% in paste containing 4N HCl (40 parts) + H_2SO_4 (60 parts) on its inhibitive efficiency for mild steel at room temperature is shown in table 1 and figure 1. Results show that when 0.01% *Ipomoea* was added to paste, weight loss reduced from 20.4 mg/dm^2/hr to 10.2 mg/dm^2/hr, as the concentration of *Ipomoea* was further increased weight loss was further decreased. At 5% concentration, the weight loss obtained was 6.9 mg/dm^2/hr. Inhibitor efficiency was 50% which continuously increased with increase in concentration of inhibitor upto 66%.

Table 1 : Effect of concentration of inhibitor(*Ipomoea carnea*)on the rate of attack of mild steel by paste [40 HCl(4N) +60 H_2SO_4(4N);1 hr.;3.0 gm paste/dm^2 = coating thickness]

Concentration of inhibitor(%)	Weight loss(mg/dm^2)	Inhibitor Efficiency(%IE)
Nil	20.4	Nil
0.01	10.2	50
0.1	9.1	55
0.5	8.7	57
1	8.1	60
2	7.2	65
5	6.9	66

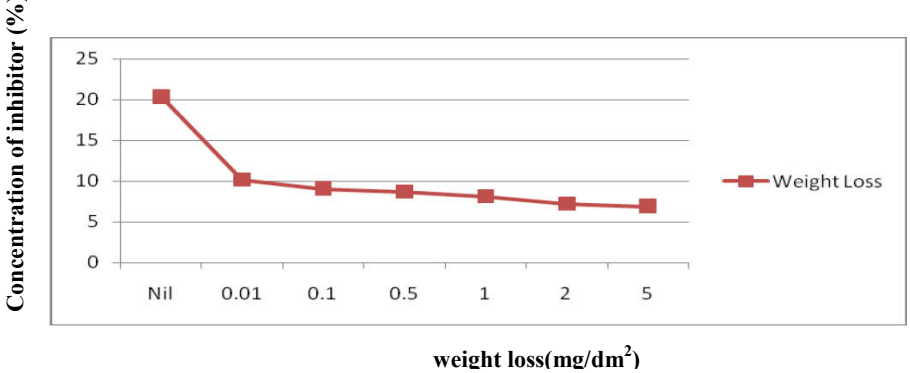


Figure 1a : Effect of concentration of inhibitor (*Ipomoea carnea*) on the rate of attack of mild steel by paste [40 HCl(4N) +60 H_2SO_4(4N);1 hr.;3.0 gm paste/dm^2 = coating thickness]

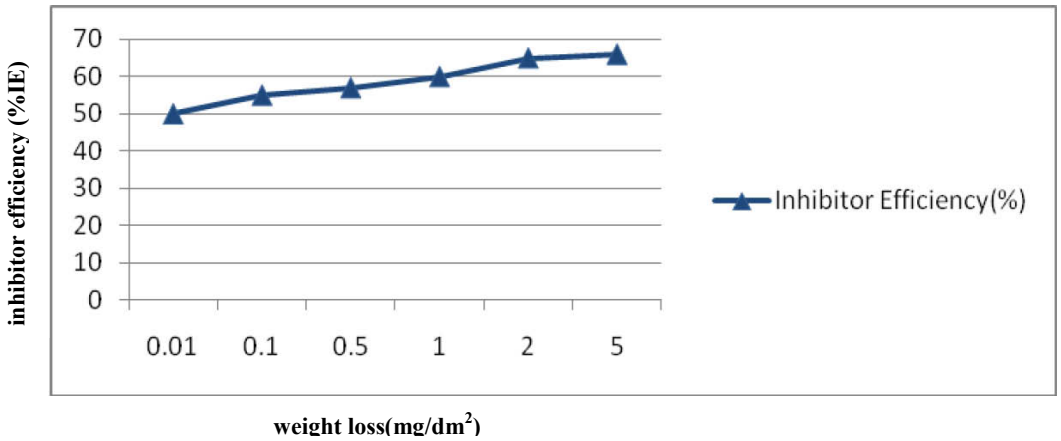


Figure 1b : Effect of concentration of inhibitor (*Ipomoea carnea*) on its inhibitive performance [40 HCl(4N) +60 H_2SO_4(4N);1 hr.;3.0 gm paste/dm^2 = coating thickness]

3.2. Effect of change in proportion of HCl and H_2SO_4. 4N HCl and 4N H_2SO_4 were mixed in different proportions from 0-100 to 100-0 and results of attack due to uninhibited and inhibited paste is given in table 2 and figure 2. Attack due to inhibited paste having different proportions of HCl and H_2SO_4 did not show much variation in weight loss, however the weight loss was minimum in HCl :H_2SO_4 :: 40:60. The inhibitor efficiency varied from 61% to 68%.

Table 2 : Effect of change of ratio of HCl(4N) and H_2SO_4(4N) in inhibited paste on the rate of attack of mild steel [RT;1 hr.;3.0 gm paste/dm^2 = coating thickness; *Ipomoea carnea* = 1%]

Volume of acid (c.c) HCl + H_2SO_4	Weight loss (mg/dm^2) Un.	In.	Inhibitor Efficiency (%IE)
0 + 100	27.9	8.9	68
10 + 90	26.1	8.6	67
20 + 80	24.3	8.2	66
40 + 60	20.5	7.1	65
60 + 40	22.9	8.3	64
80 + 20	23.1	8.5	63
90 + 10	24.2	9.1	62
100 + 0	24.8	9.6	61

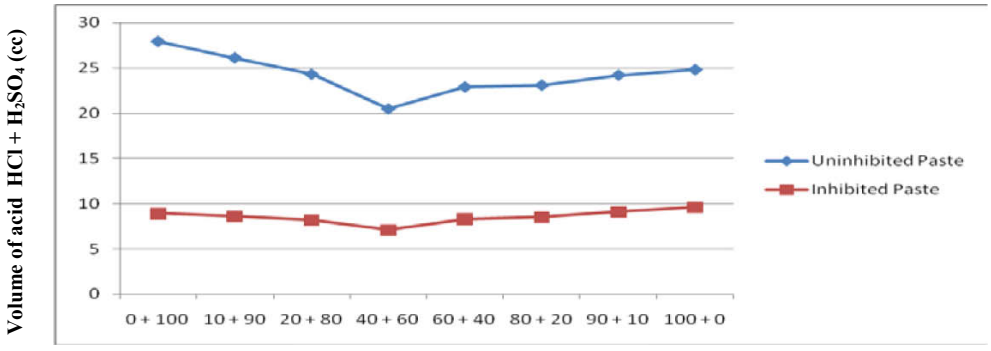


Figure 2a : Effect of change of ratio of HCl(4N) and H_2SO_4(4N) in inhibited paste on the rate of attack of mild steel [RT;1 hr.;3.0 gm paste/dm^2 = coating thickness; *Ipomoea carnea* = 1%]

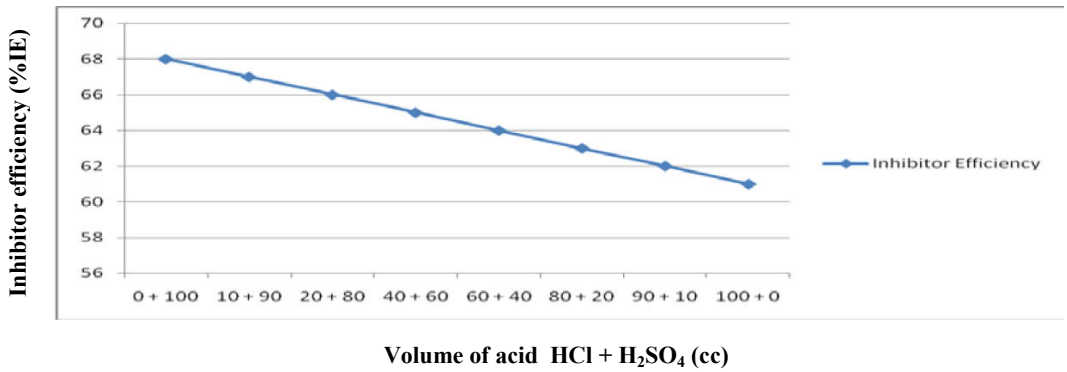


Figure 2b : Effect of change of ratio of HCl(4N) and H_2SO_4(4N) in inhibited paste on inhibitor efficiencyof *Ipomoea carnea* [RT;1 hr.;3.0 gm paste/dm^2 = coating thickness; *Ipomoea carnea* = 1%]

3.3. Effect of time of application. Table 3 and figure 3 shows weight loss of mild steel specimens for 10 minutes to 180 minutes in uninhibited paste varied from 20.0 mg/dm^2 to 41.2 mg/dm^2 and in inhibited paste varied from 7.8 mg/dm^2 to 12.3 mg/dm^2. The inhibitor efficiency varied from 61% to 70%.

Table 3: Effect of time of application of inhibited paste on the rate of attack of mild steel[40 HCl(4N) + 60 H_2SO_4(4N);RT;3.0 gm paste/dm^2 = coating thickness; *Ipomoea carnea* = 1%]

Time of Application (min.)	Weight loss (mg/dm^2)		Inhibitor Efficiency (%IE)
	Un.	In.	
10	20	7.8	61
30	26.1	9.3	64
60	34.3	11.6	66
120	39.5	12.2	69
180	41.2	12.3	70

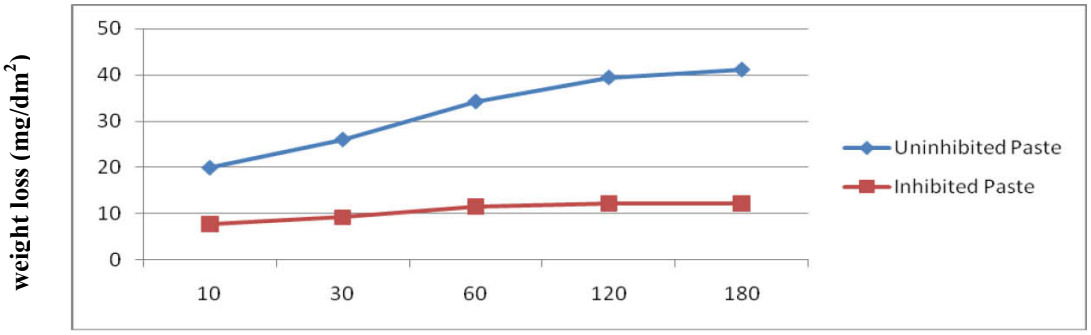


Figure 3a: Effect of time of application of paste on the rate of attack of mild steel[40 HCl(4N) + 60 H_2SO_4(4N);RT;3.0 gm paste/dm^2 = coating thickness; *Ipomoea carnea* = 1%]

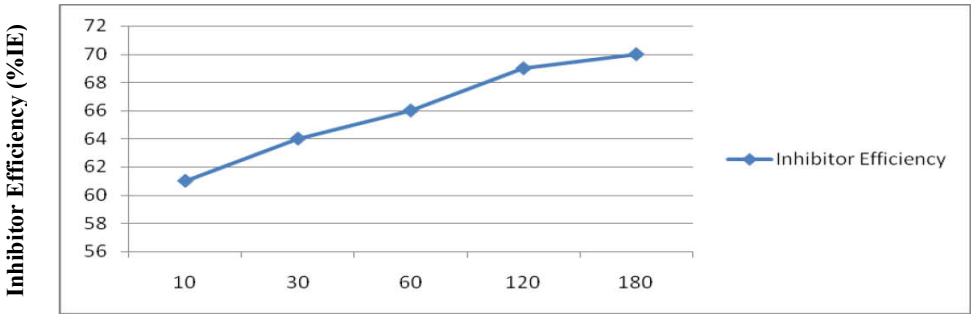


Figure 3b: Effect of time of application of paste on the inhibitive performance of *Ipomoea carnea*[40 HCl(4N) + 60 H_2SO_4(4N);RT;3.0 gm paste/dm^2 = coating thickness; *Ipomoea carnea* = 1%]

3.4. Effect of temperature. Table 4 and figure 4 show the effect of temperature (30°C-60°C) on attack of mild steel due to paste containing 4N HCl (40 parts) + 4N H_2SO_4 (60 parts) with and without 1.0% Ipomoea. In uninhibited paste, the weight loss of mild steel specimens varied from 19.7 mg/dm^2/hr to 44.1 mg/dm^2/hr in a temperature range of 30°C to 60°C. In inhibited paste, the weight loss varied from 7.0 mg/dm^2/hr to 14.9 mg/dm^2/hr. The inhibitor efficiency varied from 64% at 30°C to 66% at 60°C. Arrhenius plots have been drawn showing the dependence of log corrosion rate on 1/T for uninhibited paste and inhibited paste. The linear nature of both the curves indicates that they obey the Arrhenius Equation.

Table 4 : Effect of Temperature on the rate of attack of mild steel [40 HCl(4N) + 60 H_2SO_4 (4N);1 hr.;3.0 gm paste/dm^2 = coating thickness; *Ipomoea carnea* = 1%]

Temp. (°C)	1/T * 10^4	Weight loss (mg/dm^2)				Inhibitor Efficiency (%IE)
		Un.	log Weight loss	In.	log Weight loss	
30	33	19.7	1.2945	7	0.8451	64
35	32.4	21.2	1.3263	7.8	0.8921	63
40	31.9	26.1	1.4166	9.6	0.9823	63
45	31.4	31.3	1.4955	10.9	1.0374	65
50	30.9	35.6	1.5514	12.8	1.1072	64
55	30.4	39.4	1.5955	13.7	1.1367	65
60	30	44.1	1.6444	14.9	1.1732	66

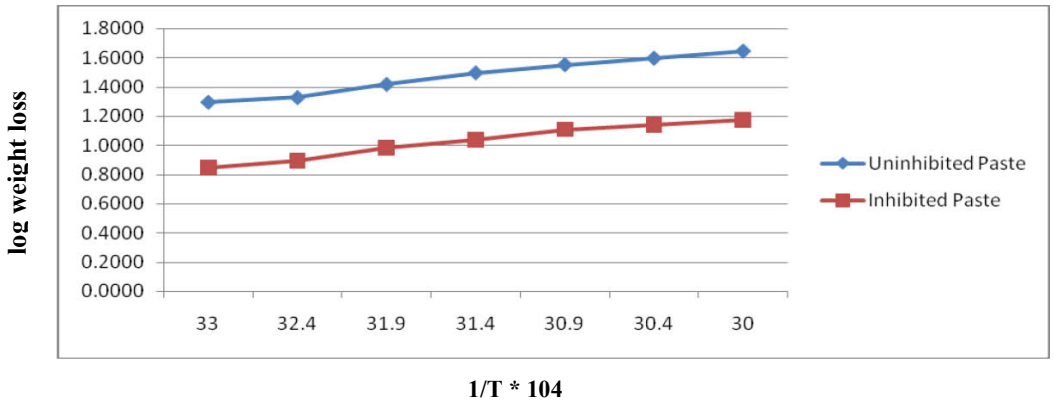


Figure 4a: Effect of Temperature on the rate of attack of mild steel [40 HCl(4N) + 60 H_2SO_4 (4N);1 hr.;3.0 gm paste/dm^2 = coating thickness; *Ipomoea carnea* = 1%] [Arrhenius Plot]

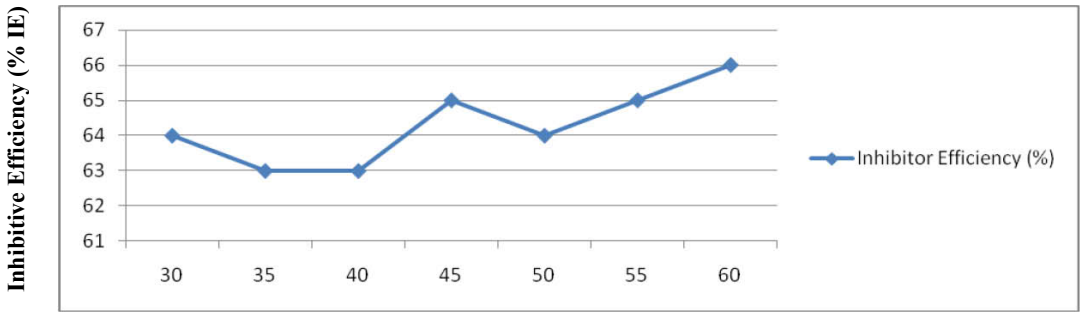


Figure 4b: Effect of Temperature on inhibitive performance of *Ipomoea carnea* [40 HCl(4N) + 60 H_2SO_4 (4N);1 hr.;3.0 gm paste/dm^2 = coating thickness; *Ipomoea carnea* = 1%]

3.5. Corrosion Current as a function of time for mild steel-platinum couple. Pickling paste with 4N HCl (40 parts) + 4N H_2SO_4 (60 parts) with and without 1.0% Ipomoea was prepared. Mild steel was connected to platinum and both were placed in pickling paste. Results given in table 5 and figure 5 show that in uninhibited system, when steel was connected to platinum, the starting current was 80 ma. The current gradually decreased with time. In inhibited system, the starting current was 63 ma which gradually reduced to 49 ma.

Table 5 : Corrosion Current in steel platinum couple placed in paste [40 HCl(4N) + 60 H_2SO_4 (4N);RT; *Ipomoea carnea* = 1%]

Time (min.)	Current (ma)	
	Uninhibited	Inhibited
0	80	63
10	79	63
20	76	61
30	73	58
40	71	54
50	70	51
60	69	49

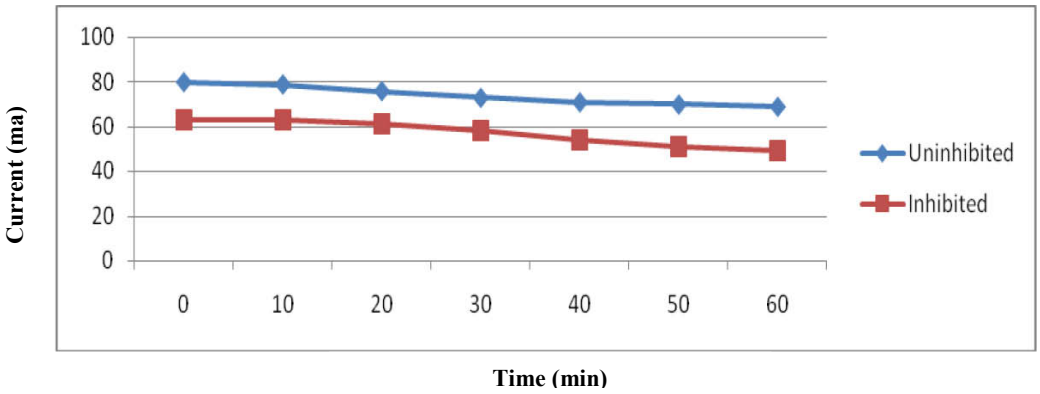


Figure 5: Corrosion Current in steel platinum couple placed in paste [40 HCl(4N) + 60 H_2SO_4 (4N);RT; *Ipomoea carnea* = 1%]

3.6. Polarization for mild steel.

3.6.1. Anodic Polarization. Table 6 and figure 6 shows anodic polarization data for mild steel exposed to pickling paste containing 4N HCl (40 parts) + 4N H_2SO_4 (60 parts) with and without 1.0% Ipomoea. Results show that when current was raised from $3.2*10^{-3}$ ma to $47.8*10^{-3}$ ma the potential increased from -620 mV to -600 mV for uninhibited system. In inhibited system, the potential varied from -610 mV to -580 mV. Thus, Ipomoea polarized anode to some extent.

Table 6 : Anodic Polarization data for mild steel placed in paste[RT; 40 HCl(4N) + 60 H_2SO_4 (4N) ; *Ipomoea carnea* = 1%]

Current Density (ma/cm^2)	**log CD**	**Potential Uninhibited (mV vs SCE)**	**Potential Inhibited (mV vs SCE)**
$3.2 * 10^{-3}$	-3.5051	-620	-610
$4.3 * 10^{-3}$	-3.6335	-620	-610
$6.4 * 10^{-3}$	-3.8021	-620	-609
$10.1 * 10^{-3}$	-2.0043	-619	-609
$15.0 * 10^{-3}$	-2.1761	-618	-608
$23.3 * 10^{-3}$	-2.3674	-615	-605

31.7 * 10^{-3}	-2.5011	-612	-600
40.9 * 10^{-3}	-2.6117	-606	-587
47.8 * 10^{-3}	-2.6803	-600	-580

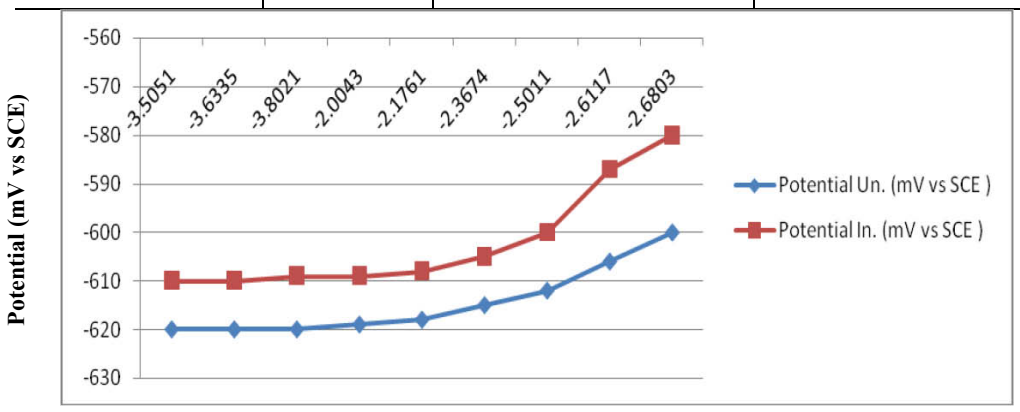


Figure 6: Anodic Polarization data for mild steel placed in paste[RT; 40 HCl(4N) + 60 H_2SO_4 (4N) ; *Ipomoea carnea* = 1%]

3.6.2. Cathodic Polarization. Table 7 and figure 7 show cathodic polarization data for mild steel exposed to pickling paste containing 4N HCl (40 parts) + 4N H_2SO_4 (60 parts) with and without 1.0% Ipomoea. Results show a potential drop, ΔV of 42 mV when current was raised from 1.2*10^{-3} ma to 15.6*10^{-3} ma for uninhibited system. For inhibited system, at minimum current density (1.2*10^{-3} ma/cm^2) potential was -521 mV which decreased to -569 mV at maximum current density (15.6*10^{-3} ma/ cm^2). Thus, a potential drop, ΔV of 48 mV was observed suggesting that Ipomoea polarized cathode to a little extent.

Table 7: Cathodic Polarization data for mild steel placed in paste[RT; 40 HCl(4N) + 60 H_2SO_4 (4N) ; *Ipomoea carnea* = 1%]

Current Density (ma / cm^2)	log CD	Potential Un. (mV vs SCE)	Potential In. (mV vs SCE)
1.2 * 10^{-3}	-3.1004	-512	-521
1.7* 10^{-3}	-3.233	-513	-522
2.5* 10^{-3}	-3.4048	-515	-526
4.1* 10^{-3}	-3.6149	-516	-533
5.9* 10^{-3}	-3.7716	-522	-540
8.1* 10^{-3}	-3.909	-527	-546
9.2* 10^{-3}	-3.9643	-531	-550
12.8* 10^{-3}	-2.1072	-546	-558
15.6* 10^{-3}	-2.1931	-554	-569

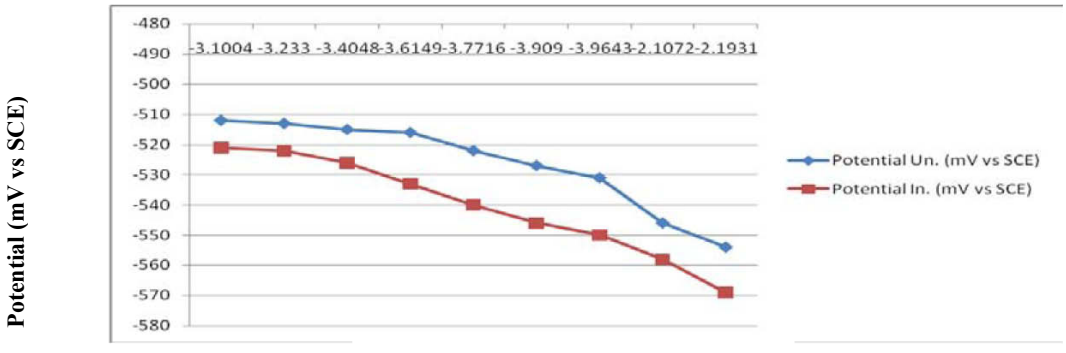
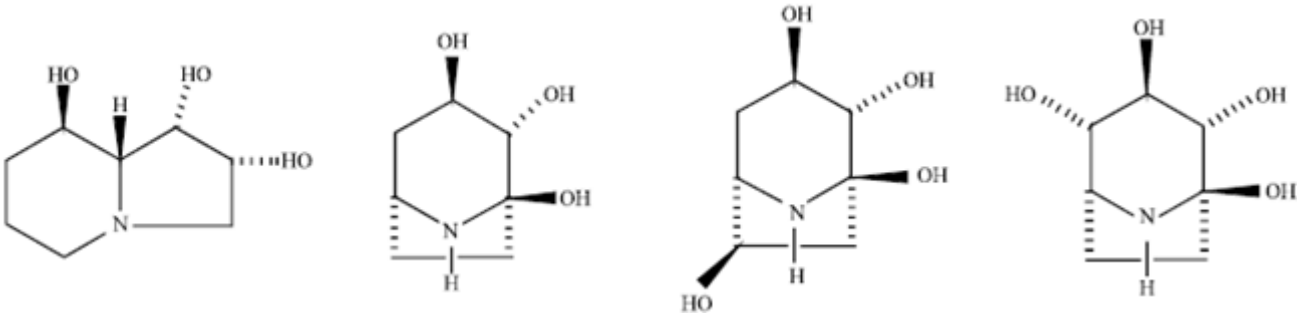


Figure 7: Cathodic Polarization data for mild steel placed in paste[RT; 40 HCl(4N) + 60 H_2SO_4 (4N) ; *Ipomoea carnea* = 1%]

4. CONCLUSIONS

The adsorption of *Ipomoea carnea* leaves extract is uniform over the surface. The leaves extract acted mainly as anodic type inhibitor. The inhibition is due to the formation of the film on the metal/acid solution interface through adsorption of *Ipomoea carnea* leaves extract molecules. Cromatographic separation of the leaf extract resulted in the isolation of swainsonine, 2-epi-lentiginosine, calystegine A_3,B_1, B_2, B_3 and C_1 and N-methyl-trans-4-hydroxy-1-proline (19,20). Swainsonine and calystegine A_3,B_1 and $B_2$are potent inhibitors (21). Structure of alkaloids present in *Ipomea carnea*: Swainsonine (1); Calystegines A_3 (2), B_1 (3), B_2 (4).



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