



## *Gloriosa superba* Linn. Extract as Eco-friendly Inhibitor for Mild Steel in Acid Medium: A Comparative Study

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The comparative studies of various parts of the plant *Gloriosa superba* Linn. extract (leaves, flowers, stems, tubers) were carried out to investigate the corrosion protection efficiency on the corrosion of mild steel in 1 N HCl medium by using mass loss method, polarization measurements and electrochemical impedance spectroscopy at room temperature. Polarization measurements showed that the studied extract acts as mixed type inhibitor with significant reduction of cathodic and anodic current densities. Organic moieties present in the extract are found responsible for effective performance of inhibitor which is well supported by FTIR studies. On comparison, optimum inhibition efficiency was found in *Gloriosa superba* Linn. stem extracts with 99.80 % at 15 ppm concentration. The nature of protective film formed on the mild steel surface has been confirmed by SEM analysis. The surface coverage values fits well to the Temkin adsorption isotherm.

**Keywords:** *Gloriosa superba* Linn., Corrosion, Electrochemical impedance spectroscopy.

### INTRODUCTION

Corrosion is a common problem for several metals. Once corrosion initiates, it propagates rapidly, most dangerous is the loss of strength by corrosion, but it can be considerably reduced using various effects have been made corrosion control and prevention. Our modern societies and resulting losses each year are in the hundreds of billion U.S. dollars. The annual corrosion cost ranged from approximately 1-5 % of the Gross National Product (GNP) of each nation. Very popular and recent trend was used as green or eco-friendly inhibitor, is one of best practical method to protect/reducing corrosion attack [1-5]. More researchers have been reported for synthetic organics [6-10], polymer [11-17], heterocyclic compounds [18-25] plants extract [26-35] and various parts like leaf, flowers, seeds, fruits [36-39] as corrosion inhibitor for mild steel, aluminium, carbon steel, zinc, copper and alloys in various medium. The present work is designed as a contribution to the growing interest on environmentally benign corrosion inhibitory study to cheap, environmentally safe, less toxic and easily available substance and simple procedure [40-48]. In this study, the extracts of same medicinal plants but various parts like leaves, stems, tubers and flowers were used to investigate the corrosion protection efficiency on the corrosion of mild steel in 1 N HCl medium.

### EXPERIMENTAL

**Preparation of the specimen:** The composition (%) of mild steel used in this study was Mn- 0.169, Mg- 0.016, C- 0.030, Cr- 0.029, P- 0.031, S - 0.029, Ni- 0.030, Cu- 0.017, Si- 0.015 and the remainder Fe of dimension 4 cm × 2 cm × 0.1 cm were polished to a mirror finished with the emery sheet of various grade and degreased with acetone before analysis.

**Inhibitor preparation:** Freshly collected aerial parts of the medicinal plants *Gloriosa superba* Linn. leaves, stems, tubers and flowers were dried in room temperature and ground into fine powder. 10g of the fine particles was added in 150 mL di-ionized water and kept overnight. The aqueous solution was filtered and volume was made up to 250 mL.

**Mass loss method:** Mild steel strips were immersed in 200 mL of 1 N HCl acid with and without of different concentrations of the inhibitors. A 3 mm hole was drilled at the center for suspension for the weight loss coupons. The immersed (before and after) mild steel surface was weighed and inhibition efficiency (%) was calculated as follows:

$$\text{Inhibition efficiency (\%)} = \frac{W_0 - W_i}{W_0} \times 100 \quad (1)$$

$W_0$  and  $W_i$  are the weight loss with and without of the inhibitor.

**Electrochemical methods:** The polarization measurements were carried out in an electrochemical cell with a three elec-

trode cell set up was used. Mild steel (1 cm<sup>2</sup>) was used as a working electrode; Pt electrode was used as counter electrode and a saturated calomel electrode as reference electrode. The electrode setup was fully and separately immersed in the acid solution for approximately 30 min to reach open circuit potential (OCP) was attained. Anodic and cathodic polarization curves were obtained from - 0.5 mV to + 2 mV at a scan rate of 1 mV s<sup>-1</sup>.

$$\text{Inhibition efficiency (\%)} = \frac{I_{\text{corr}} - I_{\text{corr}}^*}{I_{\text{corr}}} \times 100 \quad (2)$$

where,  $I_{\text{corr}}$  and  $I_{\text{corr}}^*$  are corrosion current without and with the inhibitors.

**Electrochemical impedance method (EIS):** The same three electrode cell assembly was used to carry out the Electrochemical impedance studies. A plot of  $Z'$  versus  $Z''$  was made. The double layer capacitance ( $C_{\text{dl}}$ ) was determined using formula:

$$C_{\text{dl}} = \frac{1}{2\pi} f_{\text{max}} R_{\text{ct}} \quad (3)$$

where,  $R_{\text{ct}}$  is charge transfer resistance and  $C_{\text{dl}}$  is double layer capacitance.

## RESULTS AND DISCUSSION

**Mass loss method:** The weight loss process is undoubtedly the most commonly used method of primary calculation. The weight loss data are listed in Table-1. It was noted from the table that as the concentrations of the inhibitor increases the weight loss as well as corrosion rate are decreases and higher inhibition efficiency was found (99.80 % for stems) at 15 ppm. This examined at the optimum concentration (15 ppm) of all four inhibitors and the sequence of the inhibition efficiency was found to be *Gloriosa superba* Linn. stems > leaves > tubers > flowers.

Parts of GSL plant	Conc. of the extract (ppm)	Weight loss (g)	Corrosion rate (mmpy)	IE (%)
Leaves	Blank	0.1107	64.258	–
	5	0.0011	0.638	99.00
	10	0.0017	0.986	84.58
	15	0.0013	0.754	94.12
	20	0.0015	0.870	87.80
Stems	Blank	0.0445	25.830	–
	5	0.0046	4.817	86.21
	10	0.0040	2.318	89.88
	15	0.0001	0.116	99.79
	20	0.0035	2.031	92.13
Flowers	Blank	0.0350	20.310	–
	5	0.0083	4.817	76.28
	10	0.0002	0.116	99.42
	15	0.0005	0.290	98.57
	20	0.0040	2.321	88.57
Tubers	Blank	0.0849	49.281	–
	5	0.0089	5.166	89.51
	10	0.0003	0.170	99.64
	15	0.0120	6.965	88.86
	20	0.0110	6.385	87.04

GSL = *Gloriosa superba* Linn.

The pretreated specimens were fully and separately immersed in 100 mL of 1 N HCl at room temperature. The inhibition efficiency of *Gloriosa superba* Linn. extract on mild steel as a function of time was presented in Table-2. Hence more adsorption takes place on the mild steel surface, the inhibition efficiency increases with an increase in immersion time and inhibitive properties of the *Gloriosa superba* Linn. extract are fairly good for studied situation.

Parts of GSL plant	Conc. of the extract (ppm)	Inhibition efficiency (%)					
		1 h	3 h	5 h	7 h	9 h	12 h
Leaves	Blank	–	–	–	–	–	–
	5	68.10	71.53	66.76	64.39	54.32	45.56
	10	78.10	80.25	78.54	76.76	60.12	68.72
	15	87.50	89.99	86.16	87.96	82.49	86.95
	20	90.2	92.16	93.15	90.37	91.30	94.10
Stem	5	70.31	74.90	80.33	86.16	85.78	90.12
	10	75.08	86.59	87.88	87.72	86.88	94.78
	15	78.85	88.90	89.19	89.15	87.22	96.91
	20	84.93	92.03	93.05	94.09	90.78	95.01
Flowers	5	72.81	78.90	76.14	74.21	83.89	78.90
	10	75.95	80.16	85.33	76.78	87.28	89.13
	15	78.84	84.56	87.60	82.59	87.99	92.98
	20	93.21	91.89	92.69	90.17	93.89	94.16
Tubers	5	78.90	74.93	80.23	83.78	86.87	89.21
	10	86.98	80.54	85.33	84.98	88.96	90.56
	15	87.98	83.44	86.59	88.98	89.18	92.21
	20	92.62	90.34	94.25	92.09	91.54	94.60

**FTIR Measurement:** The FTIR spectroscopy is not capable to firm exactly the main structure of the extract, but evident what it's the more abundant chemical composites. FTIR spectra of the *Gloriosa superba* Linn plants of various parts like leaves, stems, tubers and flowers extract was shown in Fig. 1. For *Gloriosa superba* Linn, which contain bands (leaves, stems flowers and tubers) corresponding 3301.30, 3272.56, 3396.15, 3170.23 cm<sup>-1</sup> can be assigned to hydroxyl group respectively. Strong peak at 1645.55 cm<sup>-1</sup> correspond to carbonyl group. The presence of aliphatic stretching of C-H is shown at frequency 2921.34, 2923.07, 2923.33, 2967.78 cm<sup>-1</sup> (leaves, stems, flowers and tuber) respectively. The absorbance at 1597.46 and 1648.18 cm<sup>-1</sup> as well as several band between 1328.34 to 1040.72 cm<sup>-1</sup> that indicates the presence of aromatic ring.

**Potentiodynamic polarization studies:** The Tafel parameters for mild steel in the absence and presence of an inhibitor concentration of *Gloriosa superba* Linn. extract in 1 N HCl are presented in Table-3 and its polarization curve are shown in Fig. 2. Fig. 2 showed that the addition of *Gloriosa superba* Linn. inhibitor did not affect the values of  $E_{\text{corr}}$  large extent but both anodic dissolution of mild steel and cathodic reduction reaction was observed, indicating that the composite could be classified as mixed type inhibitor. It was observed from the table that the corrosion current density ( $I_{\text{corr}}$ ) decreases with increasing inhibitors concentration. The maximum inhibition efficiency detected at higher inhibitor concentration shows that more inhibitor molecules are adsorbed on the metal surface,

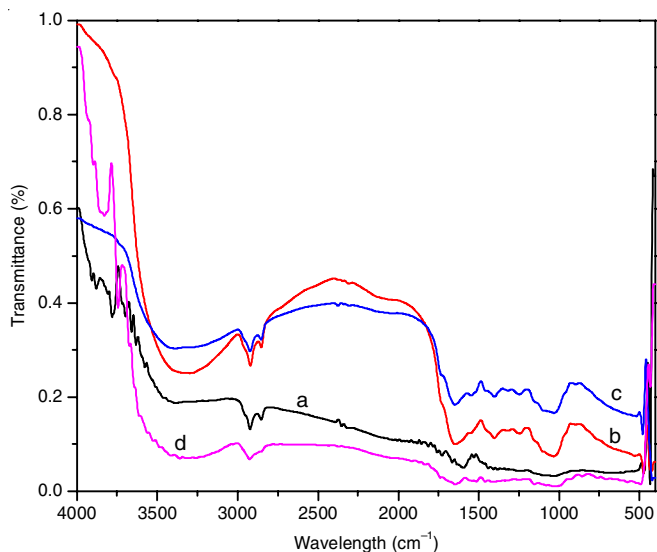


Fig. 1. FTIR spectra of *Gloriosa superba* Linn. (a) leaves, (b) barks, (c) fruits and (d) tubers extracts

which provides more surface coverage for the active sites of mild steel where direct attack occurs and migrates the corrosion

attack. From these findings, the maximum inhibition efficiency of 99.80 % was observed for *Gloriosa superba* Linn. stems extract at 15 ppm.

**Electrochemical impedance studies (EIS):** Impedance spectroscopy is one of the most simple and consistent techniques and also used to study the characterization of electrode (surface) behaviour in 1 N HCl solution in the absence and presence of the plants extracts [49-52]. Fig. 3 shows the Nyquist plots of various parts of *Gloriosa superba* Linn plants like leaves, stems, tubers and flowers at various concentrations. The different corrosion parameters derived from EIS measurement are presented as Table-4. The impedance spectra showed a single semicircle and as the concentration of inhibitor increases diameter of the semicircle increases, indicating that the charge-transfer process mainly controls the corrosion of mild steel surface and retards the electron transfer reaction and form strong protective film. It was noted from the table that the significant increase in  $R_{ct}$  and decrease in the  $C_{dl}$  values with increase in concentration of inhibitor indicated the increased inhibition efficiency of the inhibitor [53-56].

**Phytochemical screening method:** Phytochemical screening of the aerial parts of plant's powder (aqueous) extract

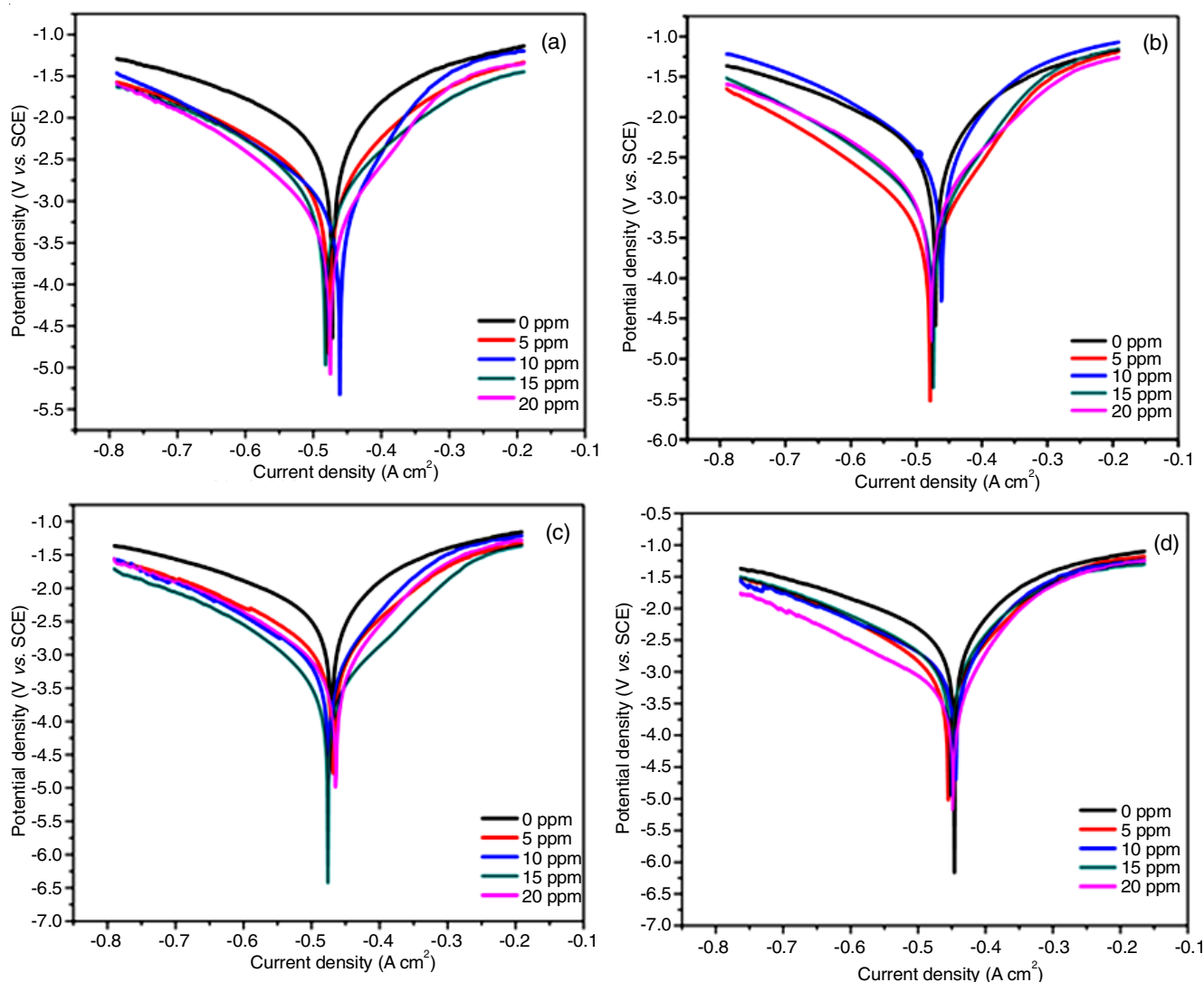


Fig. 2. Potentiodynamic polarization (Tafel) curves for mild steel in 1 N HCl solution in the absence and presence of different concentration of *Gloriosa superba* Linn. extracts of (a) leaves (b) barks (c) fruits (d) tubers

TABLE-3  
POLARIZATION MEASUREMENT AND CALCULATED VALUES OF IE (%) AT  
DIFFERENT CONCENTRATION OF *Gloriosa superba* Linn. EXTRACT

Parts of <i>Gloriosa superba</i> Linn. plant	Conc. (ppm)	$E_{\text{corr}}$ (mV/SCE)	$I_{\text{corr}}$ (mA/cm <sup>2</sup> )	$b_c$ (mV/dec.)	$b_a$ (mV/dec.)	IE (%)
Leaves	Blank	-0.471	$4.706 \times 10^{-3}$	208.85	153.42	*
	5	-0.468	$1.067 \times 10^{-3}$	160.20	115.54	99.77
	10	-0.475	$7.255 \times 10^{-4}$	165.34	90.17	84.58
	15	-0.476	$3.237 \times 10^{-4}$	131.50	100.19	93.12
	20	-0.465	$6.079 \times 10^{-4}$	146.04	90.48	87.08
Flowers	Blank	-0.471	$4.706 \times 10^{-3}$	208.85	153.42	*
	5	-0.455	$8.821 \times 10^{-4}$	155.71	93.51	76.00
	10	-0.444	$1.394 \times 10^{-3}$	191.57	104.42	99.63
	15	-0.451	$1.516 \times 10^{-3}$	174.97	116.42	99.59
	20	-0.448	$5.559 \times 10^{-4}$	188.46	87.366	85.29
Stems	Blank	-0.471	$4.706 \times 10^{-3}$	208.85	153.42	*
	5	-0.477	$8.990 \times 10^{-4}$	166.66	86.26	86.00
	10	-0.461	$1.642 \times 10^{-3}$	179.53	129.33	99.79
	15	-0.482	$1.235 \times 10^{-3}$	160.79	138.77	99.80
	20	-0.475	$5.827 \times 10^{-4}$	143.18	94.62	91.01
Tubers	Blank	-0.471	$4.706 \times 10^{-3}$	208.85	153.42	*
	5	-0.479	$4.501 \times 10^{-4}$	153.61	84.14	90.44
	10	-0.462	$3.672 \times 10^{-3}$	178.99	128.65	99.21
	15	-0.474	$7.388 \times 10^{-4}$	156.96	87.98	84.50
	20	-0.477	$1.087 \times 10^{-3}$	163.11	122.91	99.00

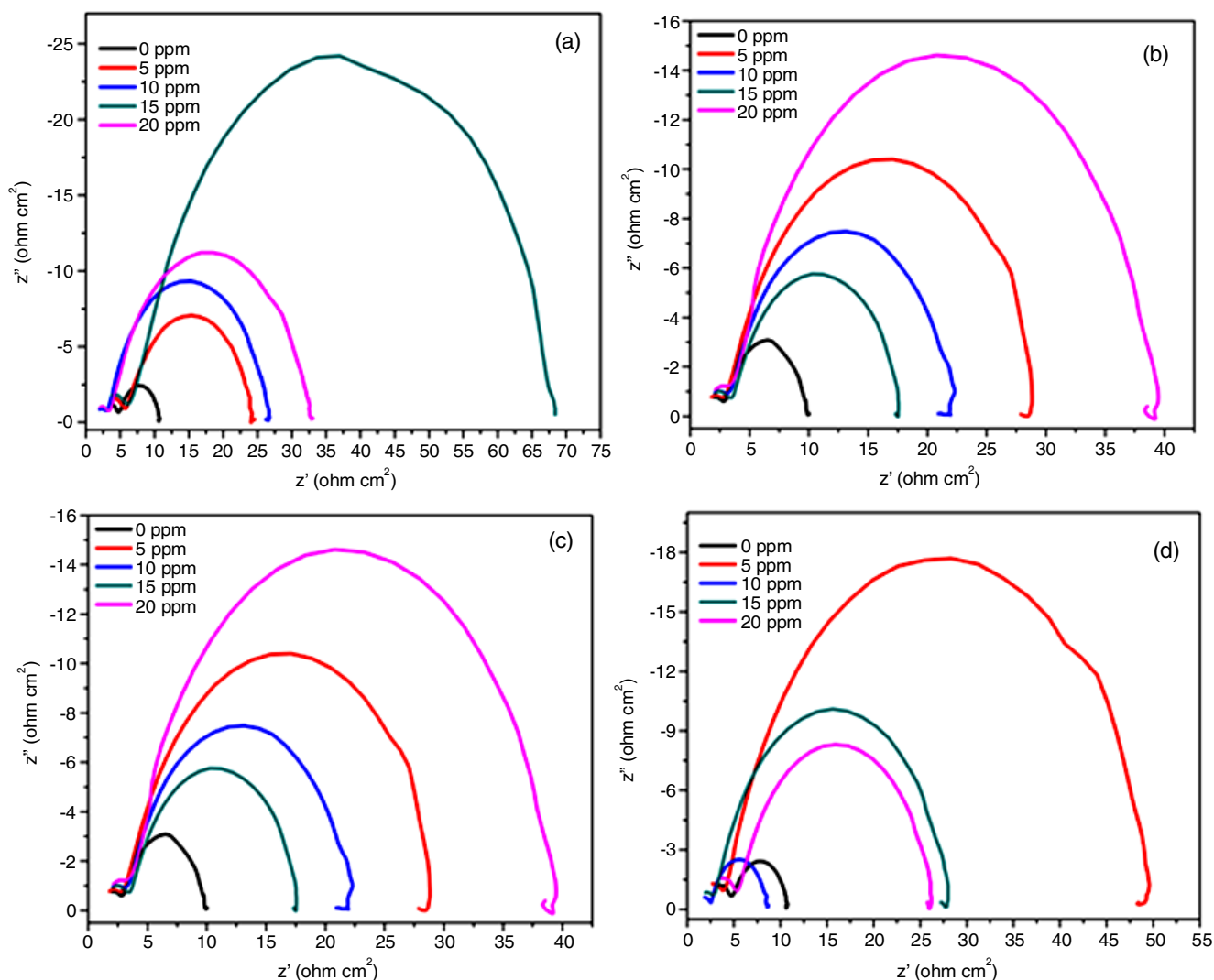


Fig. 3. Nyquist plots for mild steel in 1 N HCl acid solution without and with presence of different concentration of *Gloriosa superba* Linn. extract of (a) leaves (b) bark (c) fruits (d) tubers



TABLE-4  
EIS PARAMETER FOR MILD STEEL IN 1 N HCl ACID  
SOLUTION WITHOUT AND WITH THE VARIED  
CONCENTRATION OF PLANT EXTRACT

Parts of GSL plant	Conc. (ppm)	$R_{ct}$ (ohm $cm^2$ )	$C_{dl}$ ( $\mu F/cm^2$ )	IE (%)
Leaves	Blank	10.622	6.2385	*
	5	23.091	$9.72 \times 10^{-4}$	55.96
	10	25.416	$6.57 \times 10^{-4}$	59.99
	15	66.849	$9.82 \times 10^{-5}$	84.78
	20	32.213	$4.43 \times 10^{-4}$	68.43
	Blank	10.622	6.2385	*
Flowers	5	29.125	$5.29 \times 10^{-4}$	70.36
	10	22.899	$9.35 \times 10^{-4}$	62.30
	15	14.960	1.8530	42.29
	20	38.800	$2.78 \times 10^{-4}$	77.75
	Blank	10.622	6.2385	*
	5	18.093	1.6131	63.88
Stems	10	25.926	$7.29 \times 10^{-4}$	74.79
	15	28.411	$7.51 \times 10^{-4}$	77.00
	20	40.866	$2.85 \times 10^{-4}$	84.01
	Blank	10.622	6.2385	*
Tubers	5	49.722	$1.81 \times 10^{-4}$	78.63
	10	17.856	3.5388	40.51
	15	28.342	$5.67 \times 10^{-4}$	62.52
	20	25.597	$7.51 \times 10^{-4}$	58.50
	Blank	10.622	6.2385	*

GSL = *Gloriosa superba* Linn.

was tested in order to find the presence of various chemical constituent included alkaloids, carbohydrates, proteins, saponins, triterpenoids and tannins and the outcomes are listed in Table-5.

**Surface examination studies:** The morphologies of the mild steel immersed in blank 1 N HCl and with optimum concentration of the inhibitor for 24 h was analyzed by scanning electron microscopy (SEM) and are shown in Fig. 4(a-e). SEM images (Fig. 4b-e) revealed that the plants extract which was adsorbed on the metal surface decreased the metal surface from corrosion attack. From this it was clearly shown that *Gloriosa superba* Linn. plants extract act as an excellent green inhibitor in 1 N HCl medium.

TABLE-5  
PHYTOCHEMICAL SCREENING TEST OF  
EXTRACT OF *Gloriosa superba* Linn. PLANT

Phytochemical test	Leaves	Flowers	Tubers	Stem
Alkaloids	Presence	Presence	Presence	Presence
Carbohydrates	Presence	Presence	Absence	Presence
Proteins	Presence	Presence	Presence	Absence
Saponins	Absence	Absence	Presence	Presence
Thiols	Presence	Absence	Absence	Absence
Tannins	Absence	Absence	Presence	Absence
Flavanoids	Absence	Presence	Presence	Presence
Phenol	Absence	Presence	Presence	Absence
Glycosides	Absence	Presence	Presence	Presence

**Influence of temperature:** To assess the effect of temperature on corrosion and corrosion inhibition process, mass loss methods were performed at different temperature (303-323 K) in the absence and presence of various concentration of the inhibitor during 3 h of immersion. The results are given in Table-6.

**Adsorption studies:** Adsorption studies are used to investigate the mode of adsorption and its characteristics as an inhibitor on the mild steel surface. In present study the Temkin adsorption isotherm is investigated. The straight line (Fig. 5) clearly indicated that the inhibitor obey Temkin adsorption isotherm satisfactorily.

**Mechanism of inhibition:** The possible mechanism of inhibition can be described on the center of adsorption method and the structure of the components present in the *Gloriosa superba* Linn. plant extracts. The leading constituent of *Gloriosa superba* Linn. plant extract is colchicine and colchides whose structures are given in Figs. 6 and 7 having multiple bonds though which they get adsorbed on the metal surface. The compounds have to block the vigorous corrosion positions on the mild steel surface and hence the adsorption occurs by the bonding of the free electron of inhibitor with the metal. Phytochemical analysis showed the presence of glycosides, flavonoids, saponins, steroids, tannins and alkaloids. Above organic

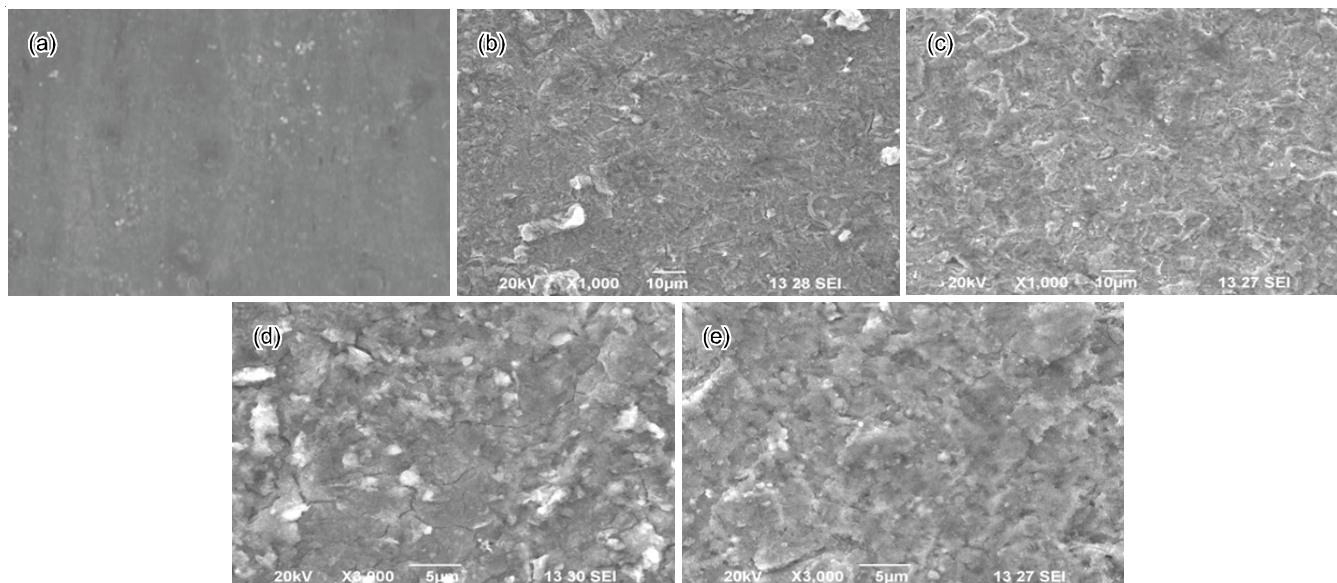


Fig. 4. SEM images of the surface of mild steel immersed in 1 N HCl after one day at room temperature (a) after immersion without inhibitor and with immersed optimum concentration of *Gloriosa superba* Linn. plant extracts from (b) leaves, (c) barks (d) fruits and (e) seeds

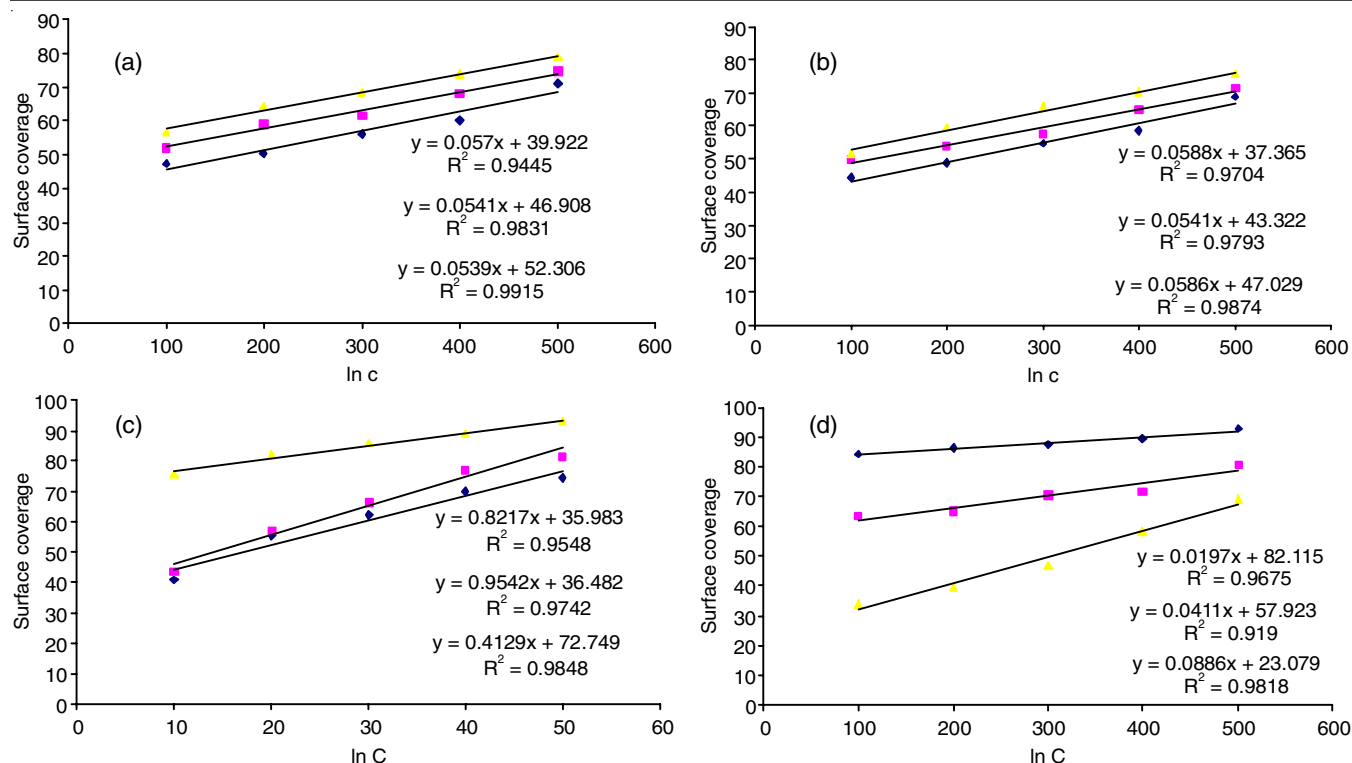


Fig. 5. Temkin adsorption isotherm plot for mild steel in 1 N HCl containing different concentration of *Gloriosa superba* Linn plant extracts (a) leaves (b) barks (c) fruits and (d) tubers

Parts of GSL plant	Conc. of extract (ppm)	IE (%)		
		303 K	313 K	323 K
Leaves	Blank	—	—	—
	5	44.10	49.12	66.76
	10	57.60	57.67	72.54
	15	62.28	63.95	80.16
	20	76.65	82.12	93.15
Stem	5	66.66	65.50	64.55
	10	71.11	69.40	68.54
	15	81.48	75.97	74.76
	20	82.22	79.05	77.79
Flowers	5	44.35	49.85	51.67
	10	48.69	53.97	59.59
	15	54.55	57.64	70.29
	20	68.76	71.48	75.65
Tubers	5	47.40	52.15	56.81
	10	50.37	59.13	64.37
	15	60.01	68.17	68.36
	20	71.10	74.70	79.03

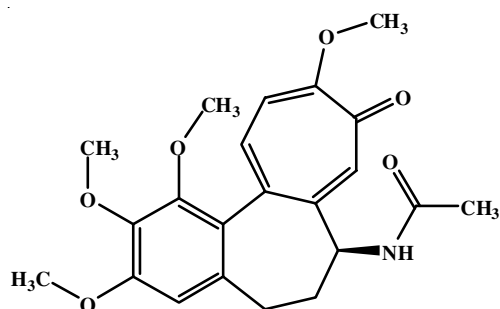


Fig. 6. Chemical structure of colchicine

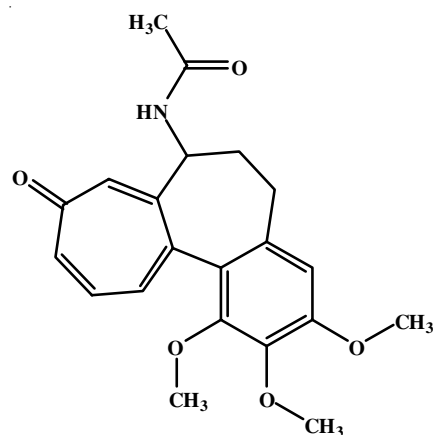


Fig. 7. Chemical structure of colchides

fragments grows adsorbed on the metal surface developing a protecting film and difference in inhibitory properties of inhibitor is closely related to the difference in molecular structure [57-59].

## Conclusion

The medicinal plants of *Gloriosa superba* Linn. extract can acts as an excellent eco-friendly green inhibitor, cost effective and easily available for the corrosion of mild steel in 1 N HCl. The extracts of *Gloriosa superba* Linn. plants showed maximum efficiency of 99.80 % stem at the optimum concentration of 15 ppm for one day immersion time at room temperature. The best correlations among the experimental outcomes showed in weight loss method have good agreement with the electrochemical methods. The *Gloriosa superba* Linn. plants extracts act as a mixed type inhibitor; they suppressed both cathodic and anodic process. The adsorbed film over the

mild steel surface has been confirmed by SEM analysis. The adsorption of *Gloriosa superba* Linn. plants extract at mild steel – acid solution interface followed the Temkin adsorption isotherm.

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