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Aqueous Extract Gloriosa Superb a Leaf as a Corrosion Green Inhibitor on Mild Steel in 1.0m HCL Medium

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Abstract: *The influence of aqueous extract Gloriosa superba leaf towards the corrosion on mild steel in 1.0M HCl has been investigated by weight loss method, polarization and impedance technique. The protective film adsorbed on the metal surface is confirmed by Fourier transform infrared (FTIR) spectroscopy. The results found shows that Gloriosa superba is a good inhibitor for mild steel in this medium. The inhibition efficiency of the compound was found to differ with the concentration of the inhibitor (2% to10 % v/v), immersion time 2hr and temperature 303-333K.The inhibition efficiency was found high at (10% v/v) concentration of the inhibitor for 2hours (94.7%). Potentiodynamic polarizations were confirmed that the studied inhibitor behaves as a mixed type inhibitor.*

Keywords: *- Inhibitor, corrosion, adsorption isotherm, potentio dynamic polarization1.0M HCl.*

I. Introduction

Plant extracts have become more important as eco-friendly acceptable, easily available and renewable source for wide range of corrosion inhibitors. In general, the plant extracts are of inhibitors with highest inhibition efficiency and of non-toxicant. A number of natural green products have been used as inhibitors on corrosion for different metals in different media. Corrosion inhibitors usually contain polar groups with hetero atoms such as nitrogen, sulphur and oxygen.[1] Correspondingly inhibitors include a wide list of organic and inorganic compounds containing the functional groups such as aldehydes, amines, amino acids, nitro compounds, amides, ester, phosphates, phosphates, ketones and carboxylic acids.

The present work investigated the inhibition efficiency of an aqueous extract of plant material, Gloriosasuperba extract, in controlling corrosion of mild steel (MS) immersed in 1.0 M HCl in the absence and presence of inhibitor, using mass loss method, analyzed the protective film by Fourier transform infrared (FTIR) spectroscopy, based on the results of the above studies and potentiodynamic polarization and the AC impedance spectra.

II. Experimental Methods

Gloriosa superb leaves:

Aqueous extract of **Gloriosa superba** leaves is used as corrosion inhibitor in this work. The given below **figure-1** shows the leaf of plant **Gloriosa superba.** Results of phytochemical screening of aqueous extract of Gloriosa superba leaves was given in **table-1.**

Plant description:

Kingdom: Plantae
Order: Liliales
Family: Colchicaceae
Genus: Gloriosa
Species: Gloriosa Superba
Botanical name: Gloriosa Superba
Commonname: glorylily, firelily, flamelily.

Preparation of Extract

A crude extract of **Gloriosa superba** leaves was prepared by using soxhlet extraction process. About 100g of dried powdered leaf of **Gloriosa superba** were uniformly packed into the thimble and extracted by using 1000ml of double distilled water to get 10% extract of the inhibitor. The method of extraction process was continued for a day or up to the solvent present siphon tube of an extracting sample getting colorless. Since the extract was captured in a beaker and kept overnight for cooling [2,3]. The cooled extract was stored as a stock solution for the use of corrosion inhibition studies.

Figure1: Gloriosa superba

Table 1: Preliminary phytochemical screening of aqueous leaf extract of Gloriosa superb[3]

Phytochemical Constituents	**Gloriosa superba**	**Phytochemical Constituents**	**Gloriosa superba**
Alkaloids	+	Terpenoids	+
Carbohydrates	-	Glycosides	+
Flavonoids	+	Steroids	+
Tannins	+	Anthraquinones	-
Phenols	+	Phenols	+

Weight-loss method:

For the weight-loss measurements, mild steel rods of chemical composition, C- 0.046%, Mn-0.248%, Si-0.029%, P- 0.012%, S-0.019%, Cr-0.05% and the remainder Fe and of size 1*5cm2 were used. The specimens were polished with different grade emery papers, cleaned with acetone, washed with double distilled water and finally dried and kept in desiccators [4-7]. The cleaned specimens were weighed before and after immersion in 1.0MHCl for immersion period 2hours in the absence and presence of the various concentrations (2% to 10% v/v) of the inhibitor and in the temperature range 303 to 333K. The corrosion rate and inhibition efficiency were determined from the loss in weight of the steel samples.

Electrochemical Polarization studies:

For the electrochemical measurements, the cell used was a conventional three electrode system with platinum auxiliary electrode, saturated calomel as reference electrode and polished mild steel specimen as working electrode. The working electrode was lacquered so as to expose 1sq.cm area to contact with the system solution. The potentiodynamic curves were recorded by changing with the electrode potential. Anodic and cathodic branches of the polarization curves were recorded and joined by a computer into a Tafel diagram [8-12]. The least square method was used to extrapolate Tafel straight lines and to determine electrochemical parameters.

Electrochemical impedance spectroscopic studies:

Electrochemical impedance spectroscopy was used over the frequency 100 to 100000HZ and with AC signal amplitude 0.005V. The measurements were recorded after the electrode reaching a steady value of E_{corr}. The experiments were carried out at a constant temperature of 30°C. The charge transfer resistance R_{ct} was obtained from the plot (Z'/ ohm Vs.-Z"/ohm) by calculating the difference in the impedance values at low and high frequencies [13- 17]. The C_{dl} is calculated from the relationship

$$\mathbf{C_{dl}=1/2\pi f_{max} X\ R_{ct}}$$

Where,

$\mathbf{C_{dl}}$=double layer capacitance.

$\mathbf{R_{ct}}$= charge transfer resistance.

$\mathbf{f_{max}}$=frequency at Z' value maximum.

The inhibition efficiency was determined from Nyquist plot using charge transfer resistance values by the formula,**(Tsuruet al, 1978).**

I.E(%)=$R_{ct(inh)}-R_{ct(blank)}/R_{ct(inh)}\times 100$

Where,
$R_{ct(inh)}$=charge transfer resistance in the presence of in hibitor.
R_{ct}=charge transfer resistance in the absence of in hibitor.

III. Surface Analysis:

FT-IR spectral Analysis:

FT–IR spectrophotometer was used to record the FT-IR spectrum from 4000 to 400cm^{-1}. The adsorption bands of the plant leaf extract and metal surface have been analyzed by FT- IR spectra [18]. FT-IR spectrum of protective layer scratched from the mild steel surface after having immersed in 1.0 M HCl in the presence of leaf extract as inhibitor for 2 hours at room temperature has been examined.

IV. Results and Discussion

Mass loss method:

Table -2 gives the values of inhibition efficiency calculated from mass loss measurement for various concentrations of the inhibitor in 1.0 M HCl time of immersion. From the calculated weight loss Values the inhibitive efficiency (I.E %) for each concentration was calculated using the equation [19]

I.E%$=$Wo –W_i/ Wo*100
Where, W_o and W_i are weight loss in theabsence and in the presence of inhibitorrespectively.

Table 2: Inhibition action on mild steel corrosion in 1.0M HCl by aqueous extract ofGloriosasuperba leaves(303K)

Inhibitors	**Concentration of in hibitor(%v/v)**	**Corrosion rate(mmd)**	**Inhibition efficiency(%)**
Blank	_	615.2	_
GS	2	197.9	67.82
	4	165.8	73.04
	6	117.7	80.86
	8	80.2	86.95
	10	32.1	94.78

The inhibition efficiency increases with increasing inhibitor concentration. This behavior may be attributed to the increase of the surface area covered by the adsorbed molecules of **Gloriosa superba** with the increase in concentration of the inhibitor. Organic compounds present in **Gloriosa superba** can easily be adsorbed on the corroding metal giving rise to such inhibition and the inhibition may be due to their synergistic action [20]. The maximum I.E (94.78%) of the inhibitor was achieved at (10 % v/v) of the concentration of inhibitor for 2hours of exposure in the corrosive acid medium.

Effect of temperature:

It is clear from table-3 that in the temperature range examined the I.E (%) decreases with the increase of inhibitor concentration. In this case, the corrosion efficiency and corrosion rate is determined for all the inhibitor system from 2-10% with the temperature ranging from 303- 333K. The efficiency of inhibition and rate of corrosion at this condition are indicated **(Table-3).**

The efficiency is decreases from 94 to 70% and the corrosion rate is increases from 32 to 363as the rise in temperature from 303-333Kin 1.0M HCl at the higher concentration (10%) for **Gloriosa superba.** The temperature effect is increased on the dissolution process of metal and the partial desorption of the plant extract inhibitor from the surface of metal [21].

Table-3: Inhibition efficiency of aqueous leaf extract of GS on the corrosion of mild steelin1.0 M HCl at different temperatures

Temperature (K)	**Concentrationof inhibitor(%v/v)**	**Corrosionrate (mmd)**	**Inhibition efficiency(%)**
303	**Blank**	615.2	-
	2	197.9	67.82
	4	165.8	73.04
	6	117.7	80.86

	8	80.2	86.95
	10	32.1	94.78
313	**Blank**	979.0	-
	2	353.1	63.93
	4	310.3	68.30
	6	262.2	73.22
	8	214.0	78.14
	10	155.1	84.15
323	**Blank**	1181.5	-
	2	508.2	54.54
	4	433.4	61.24
	6	395.9	64.59
	8	326.3	70.81
	10	262.1	76.55
333	**Blank**	1251.9	-
	2	663.4	47.00
	4	631.3	49.57
	6	524.3	58.11
	8	476.2	61.96
	10	363.8	70.94

Polarization measurement:
The electrochemical parameters used in this study were obtained from Potentiodynamic polarization curves and presented in **table-4** and **figure-2**. The percentage inhibition efficiency was calculated from relationship,
I.E (%)= Io–I /Io *100

Where Io and I are corrosion current densities in the absence and presence of inhibitor respectively and the polarization resistance R_p was calculated from Stern- Geary equation,

$$Rp= \beta a\ \beta c/ 2.3\ (\beta a + \beta c)\ I_{corr}$$

βa and βc - the values of cathodic and anodic Tafel slopes , I_{corr} – the corrosion current density.
The lower corrosion density (I_{corr}) values in the presence of the **Gloriosa superb** extract without causing significant changes in the corrosion potential (E_{corr}) suggest that it is a mixed type inhibitor [22-24].

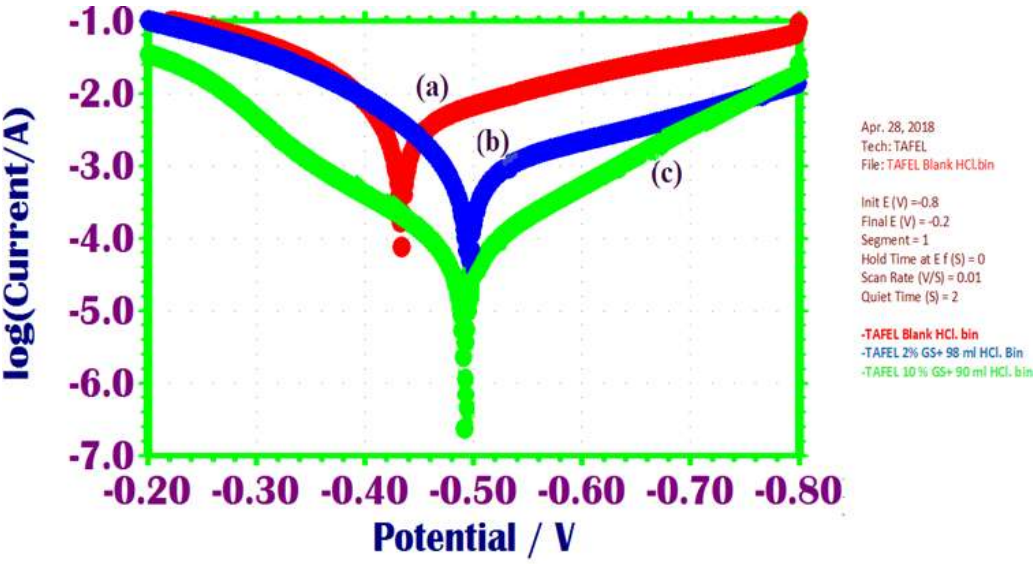

Figure -2: Potentiodynamic polarization curves for corrosion of mild steel in 1.0M HCl (a)without inhibitor (b)with2%and (c)with 10% aqueous leafextract ofGS

Table-4: Potentiodynamic polarization parameters for the corrosion of mild steel in 1.0 MHCl without and with various concentrations of GS leaf aqueous extract

Concentration of the leaves extract(% v/v)		Tafel slope				Inhibition efficiency (%) Calculated from	
	$-E_{corr}$, mV/SCE	ba , mV/ dec	bc , mV/ dec	I_{corr} , mA/cm^2	R_p , Ω/cm^2	I_{corr} , mA/cm^2	R_p , Ω/cm^2
0	-434	140	232	6.488×10^{-3}	5.9	-	-
2	-496	107	256	1.147×10^{-3}	28.5	82.32	79.29
10	-492	116	119	7.588×10^{-4}	337	98.83	98.24

Impedance measurement:

The measurement of double layer capacitance gives the information about adsorption and desorption process and also the protective layer formation on the electrode surface. The double layer capacitance $\mathbf{C_{dl}}$, change transfer resistance $\mathbf{R_{ct}}$, observed for the mild steel corrosion in 1.0 M HCl for the addition of different concentrations of GSLE at room temperature determined from the Nyquist plots, shown **table-5 and figure-3.** The observed increase in the $\mathbf{R_{ct}}$ value and decrease in the $\mathbf{C_{dl}}$ value with the increasing addition of all the studied plant leaves extract show the components presents in the extract were adsorbed on the mild steel surface. The decrease in double layer capacitance with increasing addition of inhibitor is assigned to the increase in the thickness of the double layer [25,26].

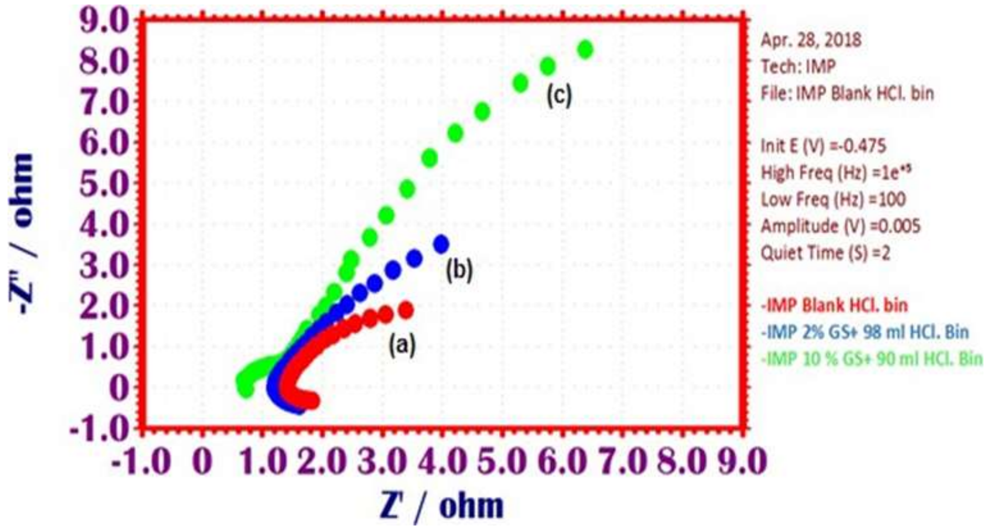

Figure-3: Nyquist plot for the corrosion of (a)mild steel in 1.0MHCl without inhibitor(b) with 2% and (c)with 10% aqueous leaf extract of GS

Table-5: Electrochemical impedance parameters from Nyquist plotsfor the corrosion of mild steel without and with the presence of various concentrations of GS leaves aqueous extract in 1.0 M HCl

	Nyquistplot		
Concentration of the aqueous leaves extract(%v/v)	**R_{ct},Ωcm^2**	**C_{dl} ,µF/cm^2**	**Inhibition efficiency(%)**
Blank	1.546	5.2006 10^{-6}	-
2	2.365	6.9642 10^{-7}	86.6
10	5.6192	2.9310 10^{-7}	94.4

FT-IR results for GSLE/Mildsteel/1.0M HCl:

The FT-IR spectral pattern of crude GSLE is shown **(Fig.4)** and the protective layer scratched from the surface of mild steel immersed in 1.0M HCl with (10% v/v) inhibitor concentration is shown **(Fig.5)**. The corresponding frequencies FT-IR peaks of crude GSLE and barrier layer are enlisted (**Table-5)**. The shift at 3347cm^{-1} to 3430cm^{-1} can be attributed to the presence N-H/O-H stretching[27,28].

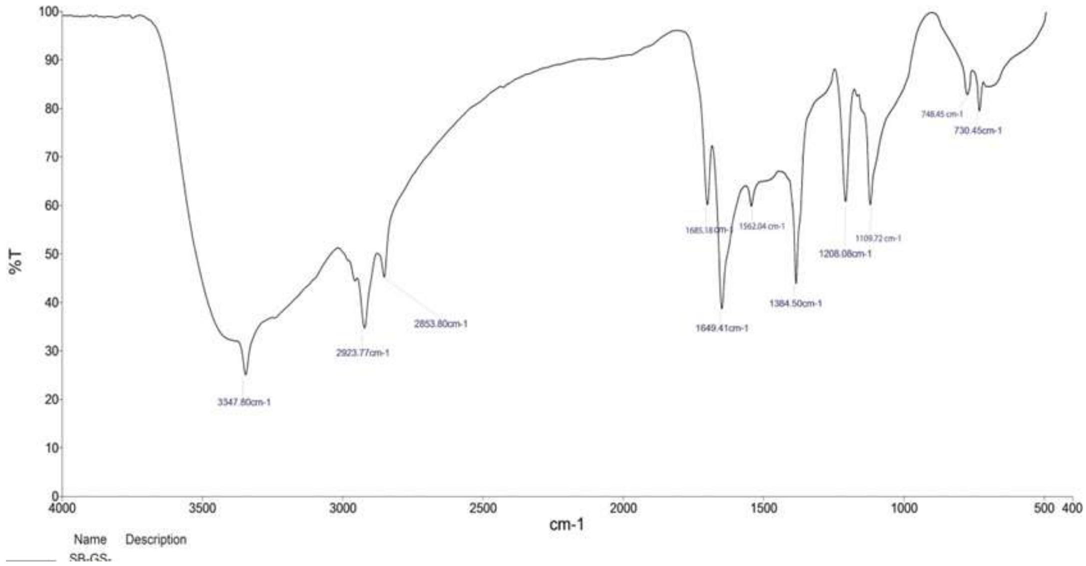

Figure-4: FT-IR spectra of aqueous leaf extract of GS

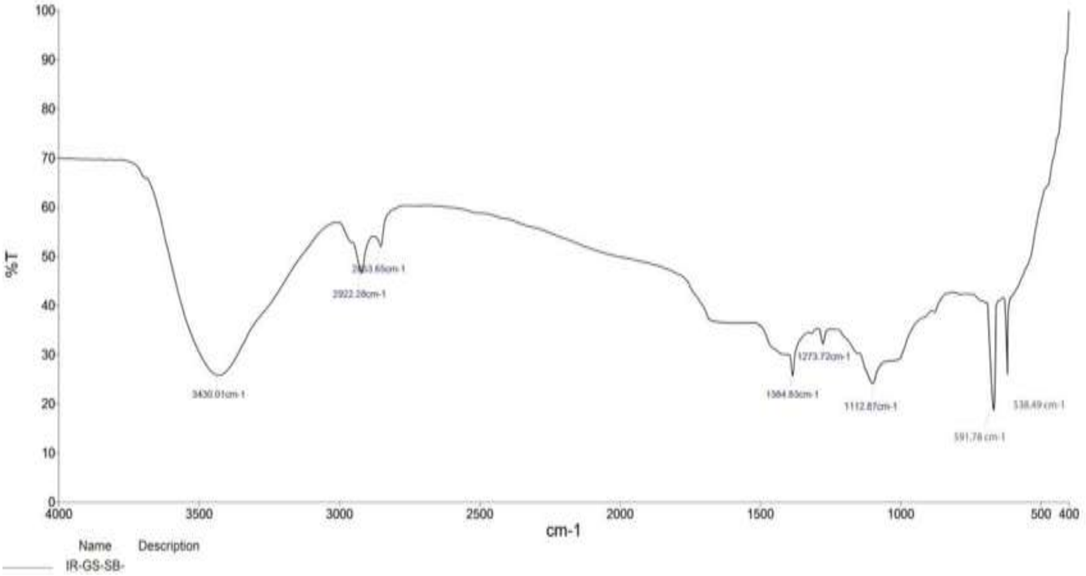

Figure-5:FT-IR spectra of scratched film from the mild steel surface after immersion in 1.0 MHCl in the presence of10% aqueous leaf extract of GS

The peak at 2923cm^{-1} and 2853cm^{-1} indicates the presence of CH bond in both systems. The peaks for 1649, 1685, 1562cm-1shows the presence C=C, C=O and C-C groups in aromatic ring. But these are not found in the spectra of adsorbed layer, it represents the formation of complex may be formed through these functional groups.

The shifts from 1208 cm-1, 1109cm-1 to 1273cm-1 and 1112.87cm-1 indicate the presence of C-O bond [29]. The peaks at 730cm-1, 745cm-1 are assigned to the presence of substituted benzene in crude GSLE. The new adsorption band frequencies are observed at 538cm-1 and 591cm-1for the adsorbed layer. It may be due to the formation of Fe-complex.

V. Summary and Conclusion

- Aqueous extract of Gloriosa superba leaves effectively protects the corrosion on mild Steel in 1.0 M HCl medium.
- Inhibition efficiency on corrosion of mild steel increased with increase in concentration of the Gloriosa superba extract. The maximum efficiency 94.78% was found for 10% at 303K for 2hours of an immersion time.
- The extract was found to be more efficient at lower temperatures.
- The leaf of Gloriosa superba protects the corrosion on mild steel because of the phytochemical constituents present in the inhibitor converting the corrosion into the noble.

- Tafel slopes of potentiodynamic polarization curves shows that the ba and bc curves turned towards the low Icorr values with the presence of inhibitors. It shows that the inhibitors were efficient to suppress the anodic dissolution and cathodic hydrogen evolution. The reasonable reduction in the Icorr values indicates corrosion inhibition by investigated inhibitors.
- The Nyquist plots of electrochemical impedance obtained for the mild steel in the presence and in the absence of the evaluated inhibitors, at room temperature show the increase in Rct and decrease in Cdl with increase in concentration of the inhibitor. It indicates the adsorption of the phytochemical components of leaf extract on the surface of mild steel and the formation of film at the interface of metal / solution.
- The evaluated plant leaf aqueous extract are characterized by the FT-IR and UV-Visible spectroscopic techniques. The reports confirmed the presence of hetero atom centres in the plant leaves extracts to form complex with metal surface and act as an inhibitors on metal corrosion.
- FI-IR spectroscopic study implied that the presence of heteroatoms in the functional groups of the phytochemical constituents of the plant leaf extract, they are adsorbed on the metal surface. It also indicates that there may be a formation of Fe-complex on the surface of the metal and it prevents the corrosion on the surface of the mild steel in acid medium.
- The observed results are suggests that the leaf extract of Gloriosa superba is an excellent corrosion inhibitor for the mild steel in 1.0 M HCl solution and it can be preferably used to replace toxic, non-decomposable and non- biodegradable inhibitor.

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