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Inhibition Effect of *Parthenium Hystophrous L* Extracts on the Corrosion of Mild Steel in Sulphuric Acid

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ABSTRACT: The inhibition effect of Parthenium hystophrous L extract on the corrosion of mild steel in 1N sulphuric acid has been studied at different concentrations as well as different temperatures of inhibitor by mass loss studies, adsorption isotherms, Tafel polarization and electrochemical impedance spectroscopy (EIS) between 308 and 338K. The exposed mild steel surface in the presence of inhibitor using Scanning Electron Microscope (SEM) is also studied. The corrosion rate increased with increase in temperature and decrease with increase in concentration of inhibitor compared to blank. The adsorption of this extracts has been found to obey Tempkin and Langmuir adsorption isotherm. Polarization results reveal that the extract is a mixed type inhibitor. The maximum inhibition efficiency of this extract observed at room temperature is 95.12%. @ JASEM

Iron and its alloys are extensively used in many engineering applications in various environments especially in inorganic and organic environments (Snavely 1970, Pultivova, L A 1960 and Desai et al 1984) because of their excellent combination of properties. Various types of inhibitors have been used for mild steel corrosion in acid medium (Sazon et al 1993). Concentrated mineral acids are used extensively in pickling, cleaning, desiccating and oil well activities. Acidification of such metallic materials cause corrosion damage to metals (Sazon et al 1993). It has been speculated that organic inhibitors are most effective with iron and that polar organic compounds containing sulphur and nitrogen are good corrosion inhibitors for the acidic dissolution of metals (Hackeman et al 1955 and Ebenso et al 1965).

Many organic inhibitors with hetero atoms have been studied (Rajanarayana 1992). High electron density of the sulphur and nitrogen atoms in these heteroatoms, help the organic molecules to get chemisorbed onto the metal surface. Due to the aggressiveness of sulphuric acid and sulphuric acid solutions against structural materials such as carbon steel, the use of corrosion inhibitors are usually required to minimize the corrosion effect (Shalby et al 2001). Despite the effectiveness of these inhibitors, the chemicals may be harmful to the environment. Hence, it is important to produce eco-friendly natural inhibitors. Parthenium hystophrous L is used externally for skin disorders and also possesses anti-tumor properties (Uphof 1996). The root is useful to arrest dysentery (Singh et al 1959). Detection of the plant is often taken internally as a remedy for a wide variety of ailment (Dominguez et al 1970) and its leaf yields an essential oil (Rao1956). Extensive analysis about the characteristics and inhibition behaviour of parthenium hystophrous L has not been carried out due to fear of its allergic character. In this investigation, the corrosion of mild steel in 1 N sulphuric acid in the absence and presence of parthenium hystophrous *L* extract as corrosion inhibitor at 308K, 318K, 328K and 338K has been studied by weight loss method, polarization techniques, electrochemical impedance spectroscopy (EIS) and adsorption isotherms.

It is aimed to reduce the corrosion rate and to increase the inhibition efficiency and surface coverage for mild steel corrosion by adsorbed inhibitors at various temperatures. The adsorption characteristics of Parthenium hystophrous *L* extracts were studied in order to access the adsorption isotherms. Various adsorption isotherms were used to find out the nature and type of inhibitor. The adsorption isotherm fitted well with experimental data can only explain the nature inhibitor. In this investigation, Langmuir and Tempkin adsorption isotherm was made to explain the nature and type of adsorption takes place on the metal surface.

EXPERIMENTAL

Preparation of Extract: Extracts of parthenium hystophrous L was prepared by using the Soxhlet apparatus using water as a solvent for 21 hours. 3 kg of plant material in natural condition was air dried for 8 to 10 days in shade. Then ground to powdered form. 300g of finely powdered dried material was taken in a 1000mL round bottom flask and sufficient quantity of triply distilled water was added to cover the powder completely. The RBF was covered with stopper and left for 48h. Then the resulting paste was refluxed for 48h. Then it was filtered. The reaction procedure for extraction was repeated for maximum extraction. To clean the stuff obtained, it was boiled with activated charcoal (2g) to remove gung, in order to obtain pure plant extract.. From this extract 0.1%,

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0.2%, 0.3%, 0.4% and 0.5% solutions were prepared by mixing 0.1 ml, 0.2 ml , 0.3 ml, 0.4 ml and 0.5 ml with 1 N sulphuric acid and made up to 100 ml.

Mass loss measurement: Mild steel specimens were cut to size 5x1 cm from mild steel having the following percentage composition: Fe = 99.686, Ni = 0.0013, Mo = 0.015, Cr = 0.043, S = 0.014, P = 0.009, Si = 0.007, Mn = 0.0196, C = 0.017. Mass loss measurements were performed as per ASTM method described previously (ASTM G 1990 and Ajmal et al 1994). The surface of the specimens were polished with emery papers ranging from 110 to 410 grades and degreased with trichloroethylene. Specimens were dried and stored in vaccum dessicator containing silica gel. These specimens were initially weighed in an electronic balance. After that the specimens were suspended with the help of PTFE threads in conical flask containing 1 N sulphuric acid (in presence and absence of inhibitors). The experiments were carried out at 308K - 338K. The specimens were removed after a definite exposure period, washed with distilled water to remove any corrosion products and finally washed with acetone. After that they were dried in an oven and re-weighed. All the solutions were prepared using AR grade chemicals with double distilled water. .

Adsorption Isotherm: An adsorption isotherm gives the relationship between the coverage of an interface with an absorbed surface (the amount adsorbed) and the concentration of the species in solution. Fitting the data to one of the adsorption isotherm can give evidence to inhibitors obeying Longmuir adsorption isotherm can be deduced from the plot of log (θ/1- θ) vs. log C. For Tempkin adsorption isotherm, plot of θ vs. log C will be linear. The adsorption isotherm was found to obey tempkin (exp $^{(a-\theta)-1}$/1-exp $^{(-ac-\theta)}$) and Langmuir (θ /1- θ) isotherm.

Surface Morphology: The surface morphology of the exposed surface in the presence of inhibitor was studied using Scanning Electron Microscope. The sample tested with SEM was sputtered with thin gold film to prevent surface charging effects.

Potentiodynamic Polarization Measurements: For potentiostatic polarization measurements, mild steel electrode of 1 cm x 1cm dimensions with stem was cut. The stem and the face of the electrode were marked with araldite, so as to expose an area of 1 cm^2. The electrodes are polished using 1/0, 2/0 and 3/0 and 4/0 emery papers and degreased with trichloroethylene. Potentiodynamic polarization measurements were carried out in conventional three-electrode cell using BAS-100A model instrument. The potential of the test electrode was measured with respect to SCE and platinum was used as auxiliary electrode and the experiments were carried out at 308K - 338K.

Electrochemical Impedance Measurements: Electrochemical Impedance measurements were carried out in a conventional three-electrode cell. Mild steel strips coated with lacquer except for an exposed area of 1 cm^2 were used as working electrode. The experiments were carried out using different concentrations of extracts of partheinium between 0.1- 0.5%. The saturated calomel electrode and the platinum were used as reference and counter electrodes respectively and the experiments were carried out at 308K - 338K. The Electrochemical Impedance was carried out using CHI 760 Electrochemical workstation.

RESULTS AND DISCUSSIONS

Mass Loss Studies: From the mass values, the inhibition efficiency (IE %) and surface coverage (θ) and corrosion rate were calculated using the following equations (Al-Shamma et al 1987). The results obtained from mass loss measurements are presented in Fig 1and 2 which show the effect of concentration of inhibitor with inhibition efficiency (IE %) and corrosion rate in 1 N sulphuric acid for an immersion period of 1 to 5 hours.

$$\text{Inhibitor Efficiency} = \frac{W_u - W_i}{W_u} \times 100$$

$$\text{Surface Coverage} = \frac{W_u - W_i}{W_u}$$

$$\text{Corrosion Rate (mmpy)} = \frac{87.6 \times W}{A \times T \times D}$$

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Where W_u and W_i are the weight loss of the mild steel in the absence and presence of inhibitor respectively at the same temperature. W = loss in weight (mg), A=area of the specimen in cm^2, T=Time in hrs, D = Density of the specimen in g/m^3.

The data show that, addition of the extract of the inhibitor has reduced the corrosion rate. The inhibition efficiency and surface coverage increased with increase in concentration of the inhibitor and decreased with rise in temperature from 308K-338K. The values of rate of corrosion are found to be decreased up to 4 hours in all the concentration of inhibitor.

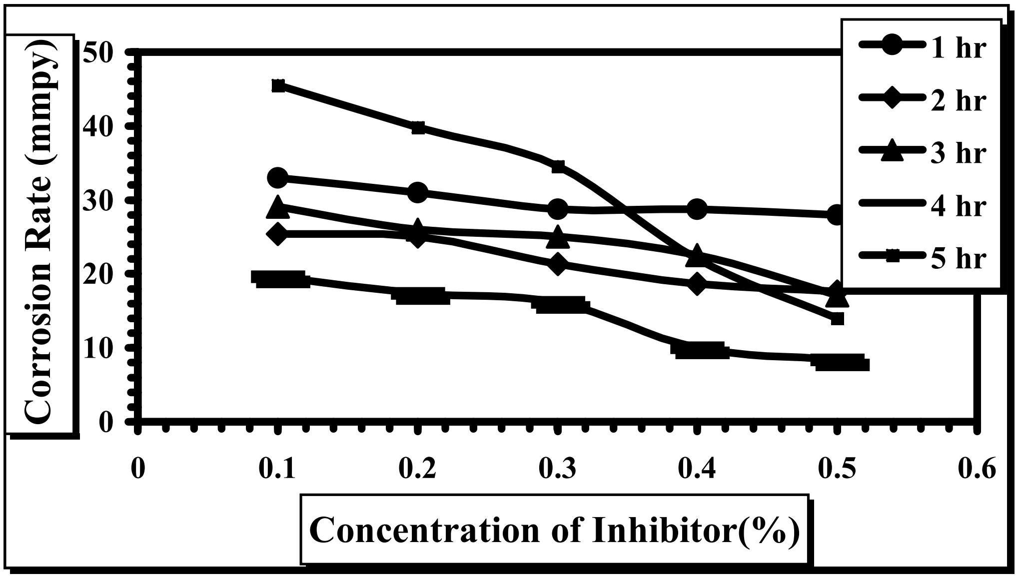

Figure 1: Effect of concentration of inhibitor with corrosion rate (mmpy) of Mild Steel in 1 N Sulphuric acid with Parthenium extract (1-5 hour)

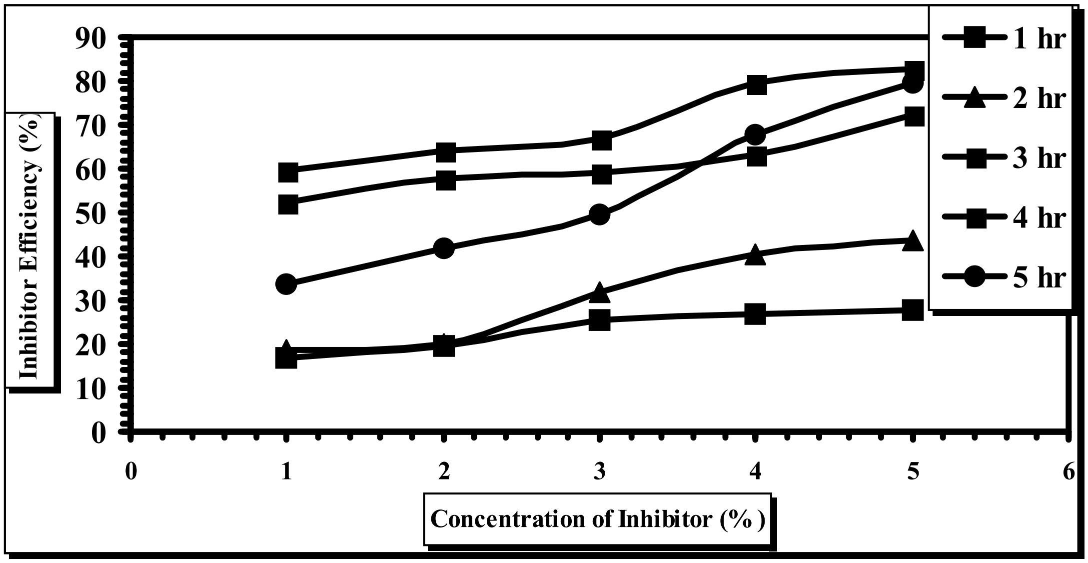

Figure 2: Effect of concentration of inhibitor with inhibitor efficiency (Parthenium extract) in 1 N Sulphuric acid (1-5 Hours)

Adsorption Isotherms: The electrochemical process on the metal surface is likely to be closely related to the adsorption of the inhibitor (Hackerman et al 1962) and the adsorption is known to depend on the chemical structure of the inhibitor (Cheng et al 1998). The adsorption of the inhibitor molecules from aqueous solution can be regarded as quasi substitution process (Atya et al 1984) between the organic compound present in the extract which is in the aqeous phase, org (aq) and water molecules at the electrode surface, $H_2O_{(S)}$.

A number of adsorption phenomenon such as, Tempkin (Bc=exp$^{(a-\ \Theta)-1}$/1-exp$^{(-ac-\ \Theta)}$ –(1), Langmuir (Bc= Θ/1- Θ) - (2), Bokris (Bc=(Θ/1- Θ)exp$^{(p1\ \Theta3/2-q\ \Theta3)}$ – (3) and Frumkin (Bc= Θ/1- Θ)exp$^{(-2a\ \Theta)}$- (4) has also been reported to explain the adsorption process. In our investigation, we have verified only two adsorption isotherm (Tempkin - (1) and langmuir –

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(2)) to explain the adsorption process during forming the protective film in presence of inhibitors.

Adsorption isotherms are very important in determining the mechanism of organo electrochemical reactions. The most frequently used isotherms are those of Langmuir, Frumkin, Parsons, Tempkin, Flory-Huggins and Bockris - Sinkles (Frumkin1925, Ikeda et al 1982, Parsons 1964 and Bockris et al 1964). The mechanism of inhibition of corrosion is generally believed to be due to the formation and maintance of a protective film on the metal surface. The plot of surface coverage (θ) obtained by mass loss method versus log C for different concentrations of the compound show a straight line indicating that the adsorption of the compounds from acids on mild steel surface follows Tempkin adsorption isotherm (Quraishi et al 2002). This also pointed to corrosion inhibition by these compounds being a result of their adsorption on the metal surface. The Langmuir and Tempkin adsorption isotherms for mild steel in 1N sulphuric acid with Parthenium hystophrous *L* were studied for the time duration (1 to 5 hrs). The Langmuir and Tempkin adsorption isotherm for Parthenium hystophorous L (308K) are shown in the Fig 3 and Fig 4. The experimental data fits the Langmuir and Tempkin adsorption isotherm.

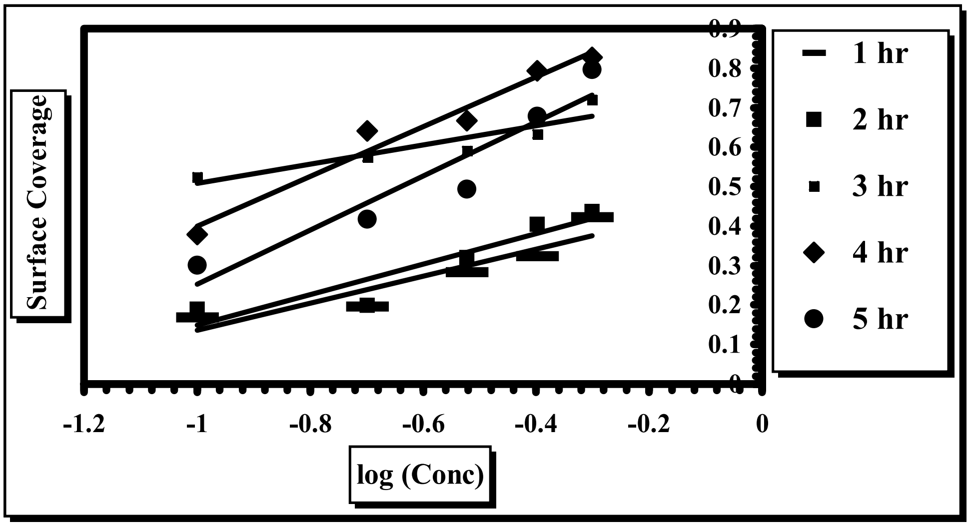

Figure 3: Tempkin adsorption isotherm for mild steel corrosion in 1 N sulphuric acid

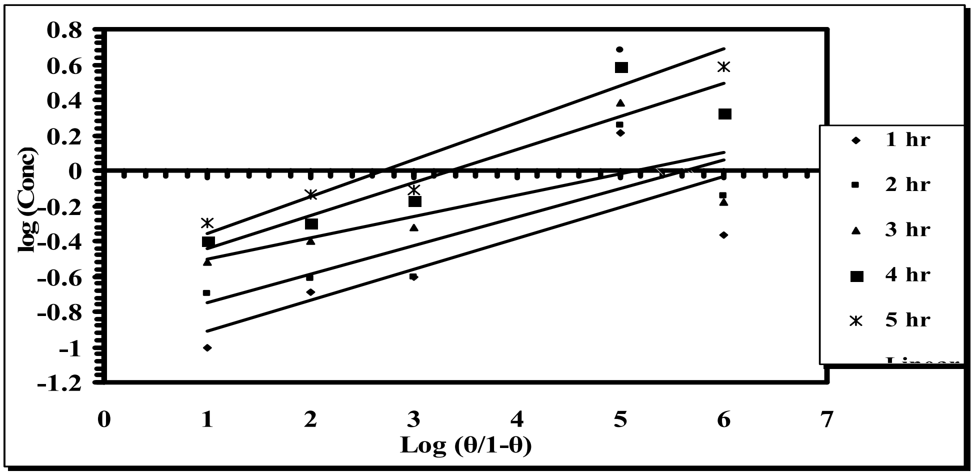

Figure 4: Langmuir adsorption isotherm for mild steel in 1N sulphuric acid with inhibitor

Langmuir And Tempkin Adsorption Isotherm at Various Temperatures*:* The effect of temperature on surface coverage of mild steel in 1 N sulphuric acid with 0.1 to 0.5 % of Inhibitor between the temperature ranges from 308K to 338K was also studied. The data were calculated and shown in the

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table 4 and 5. From the table it can be observed that the surface coverage values of mild steel in 1 N sulphuric acid with inhibitor are found to be decreased with increase in concentration of inhibitor at all the temperature when compared to blank.

Table 1 and 2: Surface behavior of mild steel in 1N sulphuric acid with inhibitor for Longmuir and Tompkins adsorption isotherm at various temperatures

Concentration of Inhibitor (%)	Surface Coverage (θ) 3 08 k	318 k	328 k	338 k
0.1	0.5142	0.4142	0.2517	0.1420
0.2	0.5513	0.4225	0.3498	0.1535
0.3	0.5601	0.4908	0.4648	0.1594
0.4	0.5800	0.4967	0.4738	0.2001
0.5	0.6801	0.5140	0.6119	0.3408

Concentration of Inhibitor (%)	$\log(\theta/1-\theta)$ 308 k	318 k	328 k	338 k
0.1	0.0244	-0.1505	-0.4732	-0.7812
0.2	0.0894	-0.1357	-0.2692	-0.7415
0.3	0.1048	-0.0160	-0.0612	-0.7221
0.4	0.1402	-0.0057	-0.0455	-0.6018
0.5	0.3275	0.0243	0.1977	-0.2865

Surface morphology: In the present work SEM was operated at 20 KV. Fig. 5 shows the micrograph of the exposed surface in the presence of inhibitor. There is a much less damage on the surface with inhibitor observed by SEM studies. The surface of mild steel which_was found to be free of any noticeable defect such as cracks and pits at a magnification of 2000 in which the inhibitor coating is clearly observed. Flakes showing corrosion products are observed in the micrographs. This is attributed to the formation of a protective film on the mild steel surface. It is quite apparent from the micrographs that the uniform products like metal hydrides and their oxides are also visible. From that it can be inferred that the extract acts as **a** good inhibitor at higher concentrations.

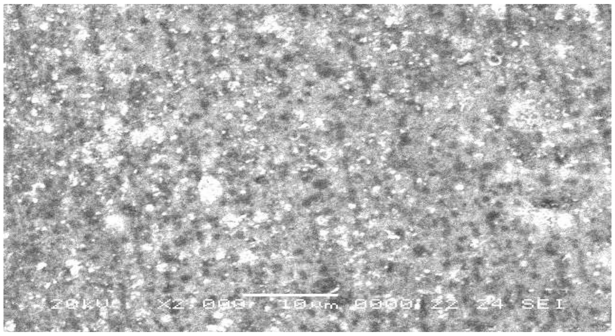

Figure 5: The SEM micrograph of the exposed surface in the presence of inhibitor with the magnification of 2000

Potentiodynamic Polarisation Studies: The measurements of corrosion current by polarization method for mild steel in 1N H_2SO_4 containing various concentrations of inhibitor was studied. The polarization behaviour of mild steel functioning as a working electrode in 1N H_2SO_4 was studied at various temperatures (308 – 338K) is shown in Fig.6. It is evident that Parthenium extract inhibitor brings about considerable polarization of the cathode as well as anode. It was therefore, inferred that the inhibitive action is mixed type. The cathodic and anodic Tafel slopes increased with increasing inhibitor concentrations and the increase was predominant in the case of the former indicating that the cathodic inhibition was dominating through the inhibitive active is of mixed nature. The non- constancy of Tafel slopes for different inhibitor concentration reveals that the inhibitor act through their interference in the mechanism of the corrosion processes at the cathode as well as anode. According to Butler and Volmer, the reaction type rate is proportional to current. The current potential dependence characteristics, is similar to kinetics of electrode processes. The dependence of over voltage on the current density for hydrogen evolution reaction has been shown to η.

$\eta = a + b \log i$ by Tafel (where a and b are constants) The corrosion parameters deduced from Tafel polarization such as corrosion current i_{corr}, corrosion potential E_{corr}, Tafel slopes b_a, b_c and inhibition efficiency are given in table 3. The i_{corr} values decrease with the increasing concentration of inhibitors. The inhibition efficiencies were determined from the values of corrosion current and the inhibition efficiency values were found to show good agreement, with those obtained from mass loss measurements.

Table 3: Potentiostatic polarization values for mild steel in 1 N sulphuric acid with p*arthenium hystophrous L* extract.

Concentration of Pantheism %	OCP mV	Ecorr Vs SCE mV	Icorr Ma/cm^2	Tafel Constant		Inhibition Efficiency (%)
				b_a (mV)	b_c (mV)	
Blank	-0.552	520	820	40	70	---
0.1	-0.547	500	500	40	70	39.02
0.2	-0.540	570	400	50	75	51.21
0.3	-0.539	530	100	60	80	89.80
0.4	-0.538	510	70	65	90	91.46
0.5	-0.537	500	40	70	100	95.12

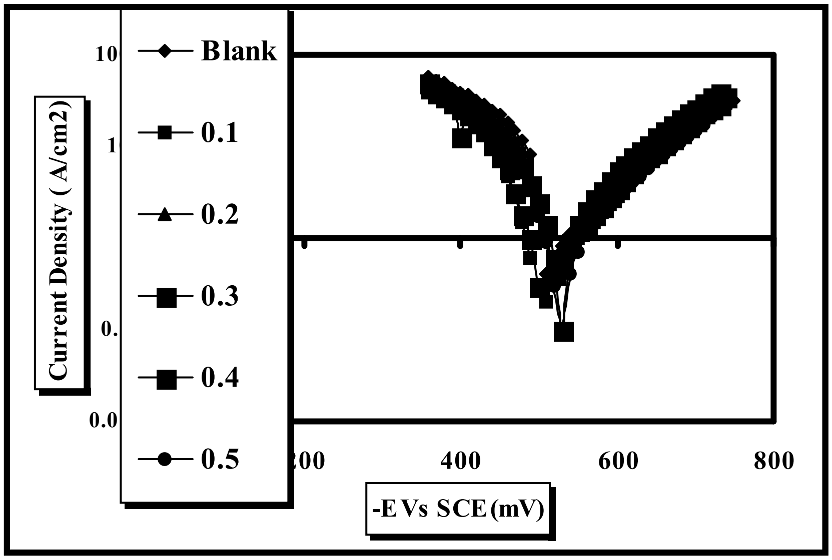

Figure 6: Comparative potentiodynamic polarization curves for mild steel in 1N sulphuric acid medium at various concentrations of the inhibitor

Electrochemical Impedance Measurements: Figures 7 and 8 are the impedance diagrams (Bode impedance and Bode phase angle plot) for various concentrations of the inhibitor at 308 K. It is, clear that the corrosion resistance of the mild steel increases with increasing concentration of the inhibitor. Corrosion resistance and capacitance values calculated from the diagrams have been presented in table 4. From the table, it is observed that the capacitance values were found to increase with decreasing concentration of the inhibitor indicates the formation of pits with intergranular corrosion. The capacitance in 0.1% (inhibitor concentration) was 1.056E-04 F/cm^2 whereas in the case of 0.5% inhibitor concentration the capacitance was 5.816E-05 F/cm^2. It can be seen from the table that 0.5% concentration has the maximum R_p value and the least C_{dl} value. This clearly indicates that 0.5% concentration of the inhibitor has more corrosion resistant when compared to the other inhibitor concentrations. This indicates that a marked difference in corrosion resistance of the inhibited and uninhibited mild steel. The values of charge-transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) can be calculated using Nyquist and Bode plots (Mansfeld et al 1993). The value of R_{ct} in blank 1N H_2SO_4 is 412 Ohm while in the presence of 0.5% inhibitor is 12460 Ohm. The C_{dl} value in the presence of inhibitor decreases suggesting that the inhibition can be attributed to surface adsorption of inhibitor. (Macdonald 1987).

The equivalent circuit most frequently used in the literature for bare metals is the Randles cell which is represented in fig 9. This is a simple circuit which includes the charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) in parallel and the solution resistance (R_s) in series and can be applied to the fitting one time constant data. For a bare metal, the polarization resistance, R_p may be defined as the intercept of the experimental impedance data with the real axis of a Nyquist plot as a frequency approaches zero. For such a simple circuit, the magnitude of R_{ct} is the diameter of the Nyquist semicircle along the real axis. In this simplest case, these two values, R_{ct} and R_p may be considered to be the same. The decrease in the R_p confirms the behaviour of decrease in R_p and is generally attributed to an increase in the metal area in contact with the test solution, i.e., the initiation of the corrosion at the interface. The impedance response indicates that the corrosion processes are controlled by mass transport of either reactants (such as dissolved oxygen) or alternatively the formation of corrosion products.

Table 4: Electrochemical impedance values for mild stell in 1 N sulphuric acid with *parthenium hystophrous L*

Concentration of the Inhibitor(%)	R_p (Ohm)	C_{dl} (Farads)
Blank	412	1.0412E-01
0.1	1417	1.056E-04
0.2	1422	9.01E-05
0.3	1847	5.816E-05
0.4	7315	5.954E-05
0.5	12460	8.298E-05

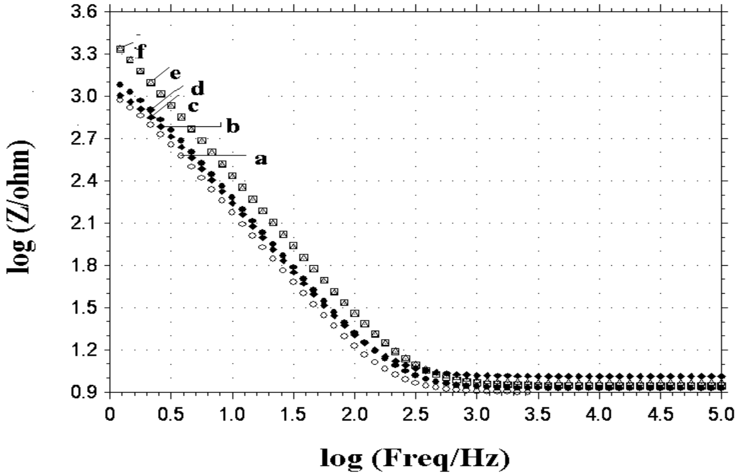

Figure 7: Comparative Bode impedance plots for mild steel in 1N sulphuric acid medium at various concentrations of the inhibitor (a) Blank (b) 0.1% Inhibitor (c) 0.2% Inhibitor (d) 0.3% Inhibitor (e) 0.4% Inhibitor (f) 0.5% Inhibitor

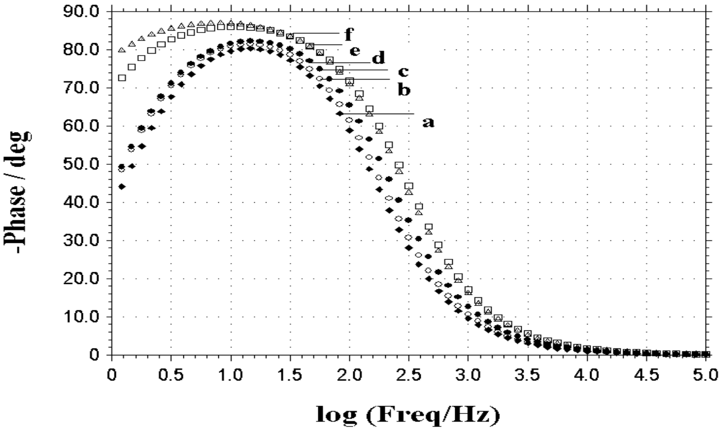

Figure 8: Comparative Bode phase angle plot for mild steel in 1N sulphuric acid medium at various concentrations of the Inhibitor (a) blank (b) 0.1% inhibitor (c) 0.2% inhibitor (d) 0.3% inhibitor (e) 0.4% inhibitor (f) 0.5% inhibitor

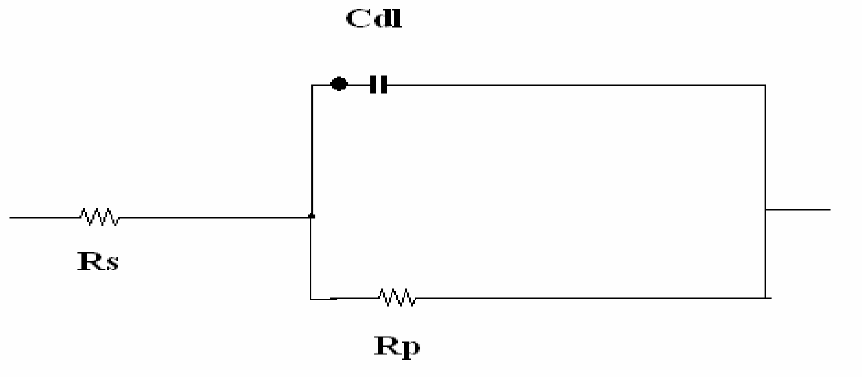

Figure 9: Equivalent Circuit for the corrosion behaviour Corrosion Behaviour of Mild Steel in 1 N Sulphuric acid with Parthenium extract

Temperature Effect On Corrosion Behaviour Of Mild Steel In 1 N Sulphuric Acid With Inhibitor: The effect of temperature on corrosion inhibition of mild steel in 1 N sulphuric acid with 0.1 to 0.5 % of inhibitor between the temperature ranges from 308K to 338 K has been studied. The data were calculated and shown in the table 3. From the table it can be observed that the rates of corrosion of mild steel in 1N sulphuric acid with inhibitor are found to be decreased with increase in concentration of inhibitor at all the temperature when compared to blank. The highest inhibition efficiency (68.01%) was attaining at 308 K for 0.5% concentration of inhibitor, further increase in the temperature, inhibition efficiency was decreased. This is may be due to the desorption of metal surface (Zhang et al 2002). Surface coverage is also calculated from the mass measurements.

Table 3: Temperature effect on corrosion behaviour of mild steel in 1N sulphuric acid with inhibitor

Concentration of inhibitor(%)	Weight loss in mg	Corrosion Rate(mmpy)	Surface Coverage (θ)	Inhibiton Efficiency (%)
		308 k Temperature		
Blank	92.413	68.6630	-----	----
0.1	44.89	33.2865	0.5142	51.42
0.2	41.46	30.8046	0.5513	55.13
0.3	40.6	30.1956	0.5601	56.01
0.4	38.81	28.8359	0.5800	58.00
0.5	29.55	21.9557	0.6801	68.01
		318 k Temperature		
Blank	288.7	214.5048	-------	------
0.1	199.1	125.6410	0.4142	41.42
0.2	166.7	123.8580	0.4225	42.25
0.3	147.0	109.2210	0.4908	49.08
0.4	145.3	107.9582	0.4967	49.67
0.5	140.3	104.2432	0.5140	51.40
		328 k Temperature		
Blank	365.6	420.2472	-----	----
0.1	367.7	314.4386	0.2517	25.17
0.2	302.7	273.2020	0.3498	34.98
0.3	297.6	224.9068	0.4648	46.48
0.4	219.5	221.1175	0.4738	47.38
0.5	215.5	160.1300	0.4907	49.07
		338 k Temperature		
Blank	304.2	226.0213	-------	------
0.1	261.0	193.9236	0.1420	14.20
0.2	257.5	191.3231	0.1535	15.35
0.3	255.7	189.9887	0.1594	15.94
0.4	243.3	180.7725	0.2001	20.01
0.5	200.5	148.9720	0.3408	34.08

Reasons for Inhibition: It has already been reported by many workers (Hackerman et al 1962 and Meakins1971, 1963) that most of the organic inhibitors work by way of adsorption on the metal surface as has been established in the present work. Adsorption bond strength depends upon the electron density available at the point of adsorption. Point of adsorption may be any electron-donating element such as N, O, S, P etc. present in the molecular structure of the inhibitor. If there is more than one electron donating elements present then which atom will have a higher tendency to become point of adsorption depends upon the distribution of electrons on the overall structure of the inhibitor molecule. In the presence of inhibitor, a thin greenish black film has always been observed on the surface of the specimens. This shows that the inhibition is due to the formation of some complex film with metal ions.

Conclusions: The corrosion rates of mild steel in 1N sulphuric acid are found to decrease with increasing time duration and concentration of inhibitor used. The corrosion rate decreases with increase of immersion time for blank at 308K -338K. Polarization measurements were made and the inhibitor efficiency was calculated from corrosion current densities. Extract of parthenium hystophrous L act as mixed type inhibitor. Inhibition efficiency of parthenium hystophrous L in 1 N sulphuric acid decreased with raise in temperature. From the impedance studies we can conclude that both the extract of parthenium hystophrous L can act as good inhibitors in preventing corrosion of mild steel in 1N sulphuric acidThe maximum inhibition efficiency of Inhibitor was found to be 83.54 %, 95.12% and 95.02% by weight loss studies, polarization study and impedance studies respectively. The inhibitor efficiency and surface coverage has been found to increase with increase in concentrations of inhibitor. The rate of corrosion of mild steel in 1N sulphuric acid with and without inhibitor is found to increase with increase in temperature. The surface coverage and inhibition efficiency of mild steel on addition of inhibitor are found to decrease with increase in temperature. Tempkin and langmuir adsorption isotherms were verified for extract of parthenium hystophrous L inhibitor adsorb on metal surface to prevent corrosion.. It was also found that the cheapest extract of parthenium hystophrous L have a comparable inhibitive property to organic and inorganic inhibitors.

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