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Adsorption and Thermodynamic Study of Corrosion Inhibition Properties of *Mimosa pudica* on Mild Steel in 2M H₂SO₄

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Abstract : Controlling the corrosion of metals and alloys are of technical, economical and environmental importance. The use of inhibitors are the best methods to control corrosion. Introduction of green corrosion inhibitor can stop the use of expensive and toxic organic inhibitor. This study investigates the corrosion inhibition of mild steel in 2M sulphuric acid media by using ethanolic extract of Mimosa pudica by weight loss method. The potentiality of Mimosa pudica to inhibit corrosion was studied based on concentration of inhibitors in the range of 1g/l -4g/l and temperature range of 30°C-50 °C. Calculation from weight loss method revealed that inhibition efficiencies (IE %) increases with increasing inhibitor concentration. The thermodynamic and adsorption parameters of this process such as Activation Energy (Ea), Enthalpy (Δ H°), Entropy (Δ S°) and Free energy (Δ G°) were calculated. Increase in Ea values with increase in inhibitor concentration proved a physical adsorption mechanism. The Δ H°calculated proved that this is an exothermic process. The experimental data fit into the Frendulich adsorption isotherm. Surface investigation of the mild steel coupons were done by SEM analysis which showed the formation of a protective film of the inhibitor molecule on the mild steel surface.

Keywords : *Mimosa pudica*, mild steel, corrosion inhibition, green inhibitors, weight loss method.

Introduction:

Corrosion is the deterioration or destruction of metals and alloys in the presence of an environment by chemical or electrochemical means. It is an irreversible interfacial reaction of a material (metal, ceramic, and polymer) with its environment which results in its consumption or dissolution into the material of a component of the environment. Often, but not necessarily, corrosion results in effects detrimental to the usage of the material considered. Exclusively physical or mechanical processes such as melting and evaporation, abrasion or mechanical fracture are not included in the term corrosion. It is a natural process, which converts a refined metal to a more chemically-stable form, such as its oxide, hydroxide or sulphide. Due to corrosion many useful properties of metal such as malleability, ductility and electrical conductivity are lost ^{1, 2, 3}. Corrosion is also known as the atmospheric oxidation of metals where oxygen combines with the metal and forms a new layer that can either be bad or good.

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Mild steel is the most common form of steel. It is extensively used as a constructional material in many industries because of its excellent mechanical properties, duct ability, weld ability and low cost ⁴. Mild steel have good strength, hard and can be bent, worked or can be welded into an endless variety of shapes for uses from vehicles (like cars and ships) to building materials, it has wide range of applications in nut bolt, chains, knives, armour, pipes, military equipment etc. However, this metal undergoes deterioration when it is exposed to acidic medium like sulphuric acid and hydrochloric acid, which are normally used in industry for pickling and de-scaling of metals ⁵. Mild steel has diverse applications found but it suffers severe corrosion in acidic environment ^{6, 7, 8, 9}.

There are numerous methods for controlling the corrosion of metals¹⁰,^{11, 12,13}. One of the best methods to reduce the rate of metallic corrosion is by the addition of inhibitors; even small concentrations can result in the decrease of the corrosion rate of the metal surface ¹⁴⁻²³. These compounds are adsorbed on the metallic surface and block the active corrosion sites; most of them are highly toxic to both human beings and the environment. Organic compounds containing heteroatoms with high electron density such as phosphorus, nitrogen, sulphur, oxygen, with double or triple bonds in their structures are effective corrosion inhibitors due to their high tendency for adsorption ²⁴⁻²⁷. It has been confirmed that the compounds having both nitrogen and sulphur in their molecular structure²⁸⁻³² have excellent corrosion inhibition ability.

The uses of non-toxic and natural products as corrosion inhibitors have become important because of the advantages of their environmentally friendly and biodegradable³³⁻³⁷in nature, readily availability, renewable sources, and ecological aspects and can be synthesized by simple procedure with low cost ³⁸⁻⁴³. Hence the use of natural medicinal plants as corrosion inhibitors has gained popularity ⁴⁴⁻⁴⁶. Plant extracts are an incredibly rich source of naturally occurring organic compounds like proteins, polycarboxylic acids, polysaccharides, tannin, alkaloids and pigments etc. These compounds work as potential inhibitors for many metals in acidic environment⁴⁷⁻⁵³.

Several researches have been done on the use of extract of plant as inhibitor for metals against corrosion in different aggressive media ^{54, 55}. However, not much has been reported on the use of the extracts of Mimosa Pudica (Sensitive plant/Touch-me-not) plant as inhibitors against corrosion of mild steel in Sulphuric acid

Methodology

Preparation of plant extract:

Fresh and healthy leaves of Mimosa Pudica were collected, washed thoroughly for 3-4 times in running tap water to remove soil particles and finely clean with distilled water. The leaves were dried in shade. After it was completely dried, grinded into fine powder and stored in an air tight polyethylene bags. Stock solutions of these plant extract were prepared by soaking known amount of the powdered plants in solution of ethanol. The sample was filtered after 48 hours and the filtrate was heated so as to remove ethanol from the sample. From this stock solution, test concentrations of 0.1, 0.2, 0.3, and 0.4 g/ 100ml were prepared by diluting with 2M Sulphuric acid.

Preparation of mild steel coupons:

Mild steel test coupons were prepared by cutting mild steel plate into pieces with dimension of 5.5cm x 2.5cm. Those specimens were then polished with emery paper. After polishing the specimens, rinsed them with distilled water, dried and sealed in zipper bag.

Weight loss method

Mild steel coupon was weighed and immersed in 100 ml solution of 2M Sulphuric acid containing 0.1, 0.2, 0.3 and 0.4 g of Mimosa Pudica extract separately in an open beaker. The beaker was placed into a thermostat maintained at 303 kelvin. After the duration of 3 hours, the specimens were removed from thermostat and cleaned with distilled water, dried and finally weighed. The experiment was repeated at various temperature of 313K and 323K.

The average weight loss of the mild steel coupon was taken as the difference between the Initial Weight (W_0) of the mild steel and Final Weight (W) after the immersion. From the weight loss data, the corrosion rates (CR) were calculated from equation (1):⁵⁶

$$CR = \frac{W_L}{At}$$
 (1)

Where W_L is weight loss of mild steel in mg, A is the specimen surface area and t the immersion period in hours.

From the corrosion rate, inhibition efficiencies of the plant extracts (IE%) can be determined by using the equation (2) 56

$$\mathbf{IE\%} = \left[1 - \frac{W_1}{W_2}\right] \times 100 \tag{2}$$

Degree of surface coverage (θ) is important in order to know interaction of inhibitor with mild steel surface that can be determined through Equation (3):⁵⁶

$$\theta = \left[1 - \frac{W_1}{W_2}\right] \times 100 \,(3)$$

Where W_1 and W_2 are the weight losses (g/dm³) for mild steel in the presence and absence of inhibitor.

Result and Discussion

Weight loss method:

The corrosion behaviour of mild steel in 2M Sulphuric acid at various temperatures (ranges from 303-323 K) were studied by weight loss method. The results are tabulated in (**Table.1**). From the results, it can be seen that the corrosion rate of mild steel in 2M sulphuric acid increases with increase in temperature from 303K-323 K. The same is evident from the straight line behaviour obtained by plotting a graph between corrosion rate and temperature, which is shown in(**Figure I**).

Temp.	Mimosa pudica					
(K)	Weight loss of mild steel					
	Initial(g)	Final(g)	Weight oss(g)	Corrosion rate (mg cm ⁻² h^{-2})		
303	11.147	11.055	0.092	2.23		
313	11.092	11.725	0.177	4.29		
323	10.943	10.651	0.292	7.07		

Table 1: Temperature Vs Corrosion Rate



Figure 1: Corrosion rate verses temperature

Sl.No	Concentration of Mimosa	Initial weight	Final weight	Weight loss (g)	corrosion rate	Inhibitor efficiency	Surface
	pudica	(g)	(g)	1055 (5)	(ing cm 2 m 1)	(%)	(g)
	(g/100ml)						
Room	Temperature 303H	K					
1	0	11.147	11.055	0.092	2.23	_	_
2	0.1	10.946	10.877	0.069	1.67	25	0.25
3	0.2	11.046	10.977	0.069	1.67	25	0.25
4	0.3	11.094	11.04	0.054	1.3	41.3	0.41
5	0.4	11.126	11.089	0.037	0.89	59.78	0.59
Room Temperature 313K							
1	0	11.902	11.725	0.177	4.29	_	_
2	0.1	11.152	11.035	0.117	2.83	33.9	0.339
3	0.2	11.189	11.102	0.087	2.1	50.85	0.5085
4	0.3	11.207	11.145	0.062	1.5	64.98	0.6498
5	0.4	11.466	11.418	0.048	1.16	72.89	0.7289
Room Temperature 323K							
1	0	10.943	10.651	0.292	7.07	_	_
2	0.1	10.995	10.785	0.21	5.09	28.09	0.2809
3	0.2	11.185	11.05	0.135	3.27	53.77	0.5377
4	0.3	11.738	11.623	0.115	2.78	60.62	0.6062
5	0.4	11.003	10.915	0.088	2.13	69.87	0.6987

Table 2: Effect of temperature in the presence of inhibitor

Effect of Temperature

The effect of temperature on the corrosion inhibitive action of Mimosa pudica extract was determined at the above mentioned temperature range(303K to 323K). From the(**Table 2**) it is observed that inhibition efficiency increased with the increase in inhibitor concentration.

The inhibition efficiency was found to increase when the temperature increased from 303k to 313K but with further increase in temperature to 323k it is found to decrease which can be attributed to desorption of plant extract molecules from the metal surface at higher temperature. At higher temperature, more desorption of inhibitor takes place and greater surface area of mild steel comes in contact with acid environment, resulting in an increase in corrosion rate. This decrease in inhibition efficiency with rise in temperature is actually suggestive of physical adsorption mechanism⁵⁶



Figure 2:Corrosion rate Vs Inhibitor concentration

It was observed that for a given temperature corrosion rate of mild steel decreased with increasing percentage of Mimosa pudica (up to 0.4g/100ml). It is evident from the graph obtained by plotting the corrosion rate of mild steel against inhibitor concentrations, which is shown in (Figure 2)

Effect of Inhibition concentration

Datas in (**Table 2**) also showed that inhibition efficiency increased with the increase in inhibitor concentration. This is proved by the plot of inhibition efficiency verses inhibitor concentration as shown in(**Figure 3**).



Figure 3: Inhibition efficiency Vs Inhibitor concentration



Figure 4: The Arrhenius plots of log CR versus 1/T

Thermodynamic and Adsorption Parameters

Thermodynamic properties such as Activation Energy (Ea), Enthalpy (Δ H°) and Entropy of Activation (Δ S°) were studied in order to understand the mechanism of inhibition process involved. The thermodynamic functions for dissolution of mildsteel without and with the addition of optimum concentration of inhibitor at various temperatures were calculated using the Arrhenius equation ⁵⁷

$Log CR = E_a/2.303RT + log A$

Where CR is the corrosion rate, E_a is the activation energy, A is the pre exponential factor. The thermodynamic parameters are tabulated in(**Table 3**). The data showed that the Ea values in the presence of different concentrations of the extract are higher than that in the free acid. The increase in the apparent activation energy

in the presence of the extract denotes physical adsorption mechanism ⁵⁸. Physical adsorption happens due to electrostatic force between negatively charged metal surface and positively charged organic species (inhibitor).

An alternative formula of Arrhenius equation is:⁵⁷

$$\ln \frac{CR}{T} = \ln \left(\frac{R}{Nh}\right) + \frac{\Box S^0}{R} - \frac{\Box H^0}{RT}$$

Where **h** is plank's constant, **N** the Avogadro's number, ΔH° is the enthalpy of activation and ΔS° the entropy of activation. From the plot of lnCR/T verses 1/T(**Figure 4**) values of ΔH° and ΔS° were calculated and are listed in (**Table 3**). The negative signs of enthalpies (ΔH°) reflect the exothermic nature of dissolution process⁵⁷. This indicates that IE % decreases when the temperature increases. Such behaviour can be interpreted on the basis that increasing the temperature resulted in desorption of the adsorbed inhibitor molecules from the metal surface. The negative values of ΔS° might be explained in the following way: before the adsorption of inhibitors onto the metal surface, inhibitor molecules might freely move in the bulk solution, but with the progress in the adsorption, inhibitor molecules were orderly adsorbed onto the metal surface, and as a result, there was a decrease in entropy⁵⁷. In addition, small negative values of ΔS° for inhibited system showed a number of water molecules on mild steel surface being displaced by inhibitor molecules.

Table (3):Activation parameters for the dissolution of mild steel coupon in the absence and presence of mimosa pudica extract at optimum temperature.

Concentration of the inhibitor	Free energy of adsorption(ΔG°) (kJ/mol)	Activation energy (Ea) (kJ/mol)	Enthalpy (ΔH°) (KJ/mol)	Entropy of activation(∆S°) (KJ/mol)
Mimosa Pudica				
Blank	-	10.97	-0.0174	-0.1024
0.1	-14.58	12.54	-0.0199	-0.104
0.2	-14.71	14.11	-0.0224	-0.1049
0.3	-15.16	13.06	-0.0207	-0.1074
0.4	-15.36	12.54	-0.0199	-0.1082

Adsorption equilibrium constant and free energy of adsorption were calculated using the relationship ⁵⁷

$$\Delta G^{\circ}_{ads}$$
 = - RT ln(55.5k_{ads})

where 55.5 is the concentration of water in solution in mol/L and is the universal gas constant. The values of K were obtained through the equation: 5^{7}

$$K = \frac{\theta}{(1-\theta)C}$$

Where C is the concentration of the extract. The negative values of ΔG°_{ads} ensure that the adsorption of the inhibitor molecule on to the steel surface is a spontaneous process. Generally, values of ΔG°_{ads} up to -20 kJ/ mol are consistent with physisorption, while those around -40 kJ/molor higher values are associated with chemisorption as a result of the sharing or transfer of electrons from organic molecules to the metal surface to form a coordinate type of metal bonds⁵⁸. In the present study ΔG°_{ads} value was found to be between -14.5 kJ/mol to -15.3kJ/mol which indicated the process to be physiosorption.

Adsorption Isotherm

The inhibitive action of organic components in plant extracts is attributed to their adsorption on the metal surface. The adsorbed layer acts as a barrier for mass and charge transfer leading to a reduction in the rate of corrosion of the metal. Adsorption isotherms that describe the behaviour of adsorbed species examine the variation of θ with concentration of the organic species. The adsorption isotherms provide important clues regarding the nature of the metal-inhibitor interaction ⁵⁸. In order to obtain the adsorption isotherm, the degrees of surface coverage, for various concentrations was calculated. The obtained θ values were applied to different

adsorption isotherm equations and the Freundlich adsorption isotherm fitted best. The Freundlich adsorption isotherm is given as; ⁵⁸

$$\frac{\theta}{1-\theta} = C + B$$

Where C is the inhibitor concentration and B is the adsorption coefficient.

Surface Investigation

SEM Analysis:

The formation of an adsorbed protective film of the inhibitor molecule on the mild steel surface is also confirmed by SEM studies. (Figure 5) represent the morphology of the mild steel specimens immersed in 2M sulphuric acid in absence and presence of optimum concentration of Mimosa pudica for 3 h immersion time.



Fig 5:Morphology of mild steel in sulphuric acid in the presence and absence of inhibitor

However, the SEM obtained for inhibited mild steel specimen indicates that the metal surface is fully covered with inhibitor molecules giving it high degree of protection. It can be concluded that the mild steel surface morphology is remarkably improved in presence of inhibitors. The inhibitor molecules reduces the corrosion by decreasing the active surface area of the substrate and inactivate only the available part of surface imposed into corrosive medium, which is termed as simple blocking the surface area. This finding further suggests the formation of protective film of inhibitors on mild steel surface

EDX Analysis

Energy-dispersive X ray spectroscopy (EDX,EDS,EDXS or XEDS) is an analytical tool used for the elemental analysis or chemical characterization of a sample. The aim of this study was to conform the finding of weight loss that inhibitors form a protective film on mild steel surface. To achieve this target the EDX spectra of mild steel was recorded in the absence and presence of optimum concentration of inhibitor extract. However the EDX spectra of mild steel in presence of plant extract shows characteristic peak for oxygen and nitrogen which suggest that plant extract forms a protective film on mild steel.



Figure 6: EDX spectra for mild steel in absence of inhibitor

Quantitative Results for: Base (103)

Element	Net Counts	Weight %	Atom %
С	78	0.35	1.49
N	641	1.77	6.51
Si	1268	0.99	1.81
S	0	0.00	0.00
S	0		
Cr	10352	17.97	17.77
Cr	4895		
Mn	2493	6.13	5.74
Mn	2526		
Fe	22564	65.71	60.50
Fe	15901		
Со	1750	6.46	5.64
Со	0		
Ni	131	0.62	0.54
Ni	2526		
Total		100.00	100.00



Figure 7: EDX spectra of mild steel in the presence of inhibitor

Element	Net	Weight %	Atom %
	Counts		
С	75	0.28	1.09
N	545	1.24	4.22
0	4965	4.22	12.57
Si	1580	1.02	1.74
S	0	0.00	0.00
S	0		
Cr	11603	16.88	15.47
Cr	2702		
Mn	3214	6.59	5.72
Mn	1670		
Fe	25194	61.30	52.33
Fe	15753		
Со	2515	7.77	6.29
Со	0		
Ni	180	0.71	0.57
Ni	2495		
Total		100.00	100.00

Quantitative Results for: Base (105)

Conclusion

It is important to minimize or control metal corrosion technically, economically and environmentally which is a major industrial problem. Generally green inhibitors are excellent inhibitors under a variety of corrosion environments for metals as they are non-toxic and bio degradable. In this study, ethanolic extracts of Mimosa Pudica (touch-me-not) was studied for its role as corrosion inhibitor due to the presence of certain compounds which attribute to its inhibition properties. The CR was reported to be maximum in blank solution when there is no inhibitor to retard the deterioration process.With the increasing concentration of inhibitor concentration the inhibition efficiency also increased and corrosion rate decreased. The maximum Inhibitor efficiency for Mimosa Pudica was found at 313K temperature at 4gm/litre.

Adsorption mechanism of molecules on mild steel surface can be explained from thermodynamics analysis of activation energy (Ea), free energy (ΔG^0), enthalpy (ΔH^0) and entropy of activation (ΔS^0). For Mimosa Pudica the activation energy of the corrosion inhibition process increased with the inhibitor concentration. The data shows that the Ea values in the presence of different concentrations of the extractare higher than that in the free acid. The increase in apparent activation energy in the presence of the extract denotes physical adsorption mechanism. Similarly the activation energy which< 40 kJ /mol and the negative values of enthalpy proved the mechanism tobe physical adsorption.

The entropy parameter for adsorption of inhibitors molecule on mild steel is negative for the extracts indicating that the entropy of inhibitor molecules in solution phase is higher than in solid phase indicating a spontaneous process. The negative values of ΔG^0 shows that adsorption of inhibitors on mild steel is a spontaneous process and also suggest the strong interaction of the inhibitor molecules on to the mild steel surface. For Mimosa Pudica ΔG^0 values are greater than the threshold values of -40 kJ /mol, which again emphasis physical adsorption.

From this study it can be concluded that, the herb Mimosa Pudica can be used as green inhibitor for mild steel at room temperature and temperature slightly higher than room temperature.

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