Corrosion inhibition study of bract extract of *Musa acuminata* inflorescence on mild steel in hydrochloric acid medium

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Abstract: *Inhibition efficiency of acid extract of dry Musa acuminata bract as corrosion inhibitor for mild steel in 1N hydrochloric acid was investigated in the present study. Experimental methods include weight loss, polarization and surface analysis studies. The inhibition efficiency increased with increase in inhibitor concentration and decreased with temperature suggesting the occurrence of physical adsorption. Potentiodynamic polarization curves reveal that inhibitor perform as a mixed type of inhibitor. The electrochemical impedance study showed a decrease in double layer capacitance as the adsorption of inhibitor lead to structural change at electrode-solution interface thereby controlling the mild steel dissolution by charge transfer mechanism. The mild steel surface in the absence and presence of the inhibitor was analysed by scanning electron microscopy (SEM). Adsorption isotherms were tested and the experimental data fit well with the Langmuir adsorption. Corrosion inhibitor has efficiency as high as 94.93% at 2% v/v inhibitor concentration. Inhibition mechanism can be attributed to the adsorption of phytochemical compounds of the bract extract on the surface of the mild steel.*
Keywords: *Bract extract, Hydrochloric acid, Langmuir adsorption, Mild steel, Musa acuminata.*

I. Introduction

Metals are most commonly unstable in the atmosphere. Corrosion is the process of a metal returning to the material's stable thermodynamic state. Primary reasons for concern about corrosion are safety, economics and conservation [1]. Corrosion is both costly and dangerous. Billions of dollars are spent annually for the replacement of corroded structures, machinery and components. Premature failure of these can result in human injury, loss of life and collateral damage. Corrosion of metallic surfaces can be controlled or reduced by the addition of chemical compounds to the corrodent [2].

Most practical methods for protection against successive dissolution of metal by corrosion are use of inhibitors. The use of organic compounds containing O, S and N to reduce corrosion of mild steel has been studied, but pose environmental crisis, as they are toxic and expensive [3]. The extracts of plant however contain mixtures of compounds having O, S and N element and are ecofriendly. These compounds having nitrogen and sulphur as constituent atoms were studied as corrosion inhibitor in various acid media. Literature reveals that not much work has been done on the inhibition of mild steel in 1N hydrochloric acid using bract extract.

Thus attempt has been made to investigate the inhibition action of *Musa acuminata* bract extract as corrosion inhibitor in hydrochloric acid medium for various concentrations over a range of temperature (303 K – 353 K).

II. Materials and methods

2.1 Properties and Applications of *Musa acuminata* bract

Banana plant is the largest herbaceous flowering plants of the genus *Musa*. When a banana plant is mature, the corm stops producing new leaves and begins to form a flower spike or Inflorescence. At first, it is a large long-oval, tapering, and purple-clad bud. As it opens it is seen that the slim, nectar rich, tubular, toothed, white flowers are clustered in whorled double rows along the floral stack (Fig. 1). Female flowers occupy the lower 5 to 15 rows; above them may be some rows of hermaphrodite or nectar flower. Male flowers are borne in the upper rows. Shortly after opening, the inflorescence begins to bend downward and the male flowers and bracts shed out leaving most of the upper stalk naked except at the very tip which remains unopened and contain last formed male flowers. Each cluster of flower is covered by a thick, waxy, hood like bract, purple outside, deep red within (Fig. 2). Bracts lift from the first hand in 3 to 10 days. The bracts are soon shed and fully grown fruits in each cluster become a hand of bananas. Ethno medicinal survey revealed that the flowers of *musa* species have been used to treat many illnesses. *Musa* flowers have been traditionally used to treat menorrhagia, bronchitis, dysentery, diabetes, heart pain, diarrhea, stomach cramps, ulcers etc. Reduces painful and excess menstrual bleeding, enhance milk supply in lactating mothers [4, 5].

Fig. 1 Inflorescence with floret and bract

Fig. 2 Thick, waxy, hood like bract, purple outside, deep red out side

Previous research on preliminary phytochemcial screening of dried leaves and fruit peels of *Musa paradisiaca* revealed the presence of some glycosides, anthocyanins, tannins, flavanoids and carbohydrates [6]. No research has been reported on the phytochemical screening of banana flowers except a quantitative study on saponin and flavanoid [7] and later phenolic content by Loganayaki et al [8]. The bract part of *Musa paradisiaca* was reported to contain anthocyanins such as delphinidin, pelaragonidin, peonidin and malvidin [9, 10]. Total phenolic content in bract was reported as the lowest compared to other parts such as rhizome, fruit peel, ovary, petiole, pseudo stem and leaves [11].

2.2 Preparation of plant extract
Musa acuminata inflorescence was collected from the farm in Thirumalayampalayam, Coimbatore, Tamilnadu, India. The bracts were separated from the florets, shade dried and blended to powder. 25 g of the powder was refluxed with 1N HCl acid for 3h and kept overnight. The extract was filtered and the filtrate volume was made up to 500 ml using the same acid and stored. This 5% stock solution was diluted with appropriate quantity of 1N HCl to obtain test solution ranging from 0.05% to 2% v/v concentration.

2.3 Preparation of specimen
The mild steel specimens of size 1x5x0.5 cm^3 were polished with 400 and 600 grade emery paper, degreased with acetone, washed with distilled water, dried and kept in a desiccator for experimental study.

2.4 Weight loss method
Weight loss measurement illustrates the process of rusting. Preweighed mild steel specimens were suspended with the help of glass hook in 100 ml of 1N HCl acid solution in the absence and presence of inhibitor at various concentrations under an immersion time of 1h, 3h, 5h, 7h, 12h, and 24h at room temperature. The specimens were removed after specified time intervals, washed with distilled water, dried and reweighed. The experiment was also conducted at high temperatures as done at room temperature for a period of 1h in the absence and presence of inhibitor in 1N HCl acid using the thermostat, set at higher temperatures of 313 K to 353 K.

2.5 Electrochemical study
2.5.1 Potentiodynamic polarisation
Potentiodynamic polarization studies were carried out using frequency response analyzer PARSTAT 2273 (Princeton Applied Research USA) and IBM personal computer to record Tafel polarisation curve and Nyquist impedance curve. The polarisation measurements were carried out to evaluate the corrosion current, corrosion potential and Tafel slopes. Experiments were carried out in a conventional three-electrode cell assembly. Mild steel rod of 15 cm and 5 mm diameter mounted on a Teflon leaving 0.19625 cm^2 of surface area exposed to solution was used as working electrode. It was polished with 120, 200, 400, 600, 800, 1000 grade emery papers, cleaned with acetone, washed with distilled water and dried at room temperature, before it is immersed in test solution. A rectangular platinum foil and calomel electrode served as auxiliary and reference electrodes respectively. A time interval of 10 min was allowed for each experiment to attain the steady state open circuit potential. The measurements were carried out in the frequency range of $10^6 - 10^{-2}$ Hz at the open circuit potential by superimposing a sinusoidal AC signal of small amplitude 10 mV. The data acquisition was performed using the Power Suite software and analysed using Zsimpwin software (version 3.21) to evaluate the corrosion kinetics parameters, such as I_{corr}, E_{corr}, Tafel slopes b_a and b_c [12].

From the polarisation curves, Tafel slopes, corrosion potential and corrosion current were analysed using computer software [13]. The inhibitor efficiency by Tafel method was calculated using the equation (1):

$$\text{Inhibitor Efficiency (\%)} = \frac{I_{corr}\text{ (blank)} - I_{corr}\text{ (Inh)}}{I_{corr}\text{ (blank)}} \times 100 \qquad \rightarrow (1)$$

where,

I_{corr} (blank)	=	Corrosion current without inhibitor
I_{corr} (Inh)	=	Corrosion current with inhibitor.

2.5.2 Electrochemical Impedance spectroscopy (EIS)

The electrochemical AC-impedance measurement was carried using frequency response analyzer PARSTAT 2273 (Princeton Applied Research USA) and IBM personal computer. Experiments were carried out in the three-electrode cell assembly as that used for potentiodynamic polarisation studies. The impedance measurements were made at corrosion potentials 1.66 mVs^{-1}. The results are presented in the form of Nyquist plot. The real part (Z') and the imaginary part (Z") were measured at various frequencies and a plot Z' against Z" were made. From the plot, the charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) were calculated using the "Z" view software. Impedance measurements were carried out for mild steel in 1N HCl without and with inhibitors for the selected concentration [14].

The inhibitor efficiency by linear polarization method was calculated using the equation (2):

$$\text{Inhibitor Efficiency (\%)} = \frac{R_{ct}\text{ (Inh)} - R_{ct}\text{ (blank)}}{R_{ct}\text{ (Inh)}} \times 100 \qquad \rightarrow (2)$$

where,

R_{ct} (inh)	=	Charge transfer resistance with inhibitor
R_{ct} (blank)	=	Charge transfer resistance without inhibitor

2.6 Surface examination studies

Surface analysis of mild steel specimens was done to study the changes that occur during the corrosion of mild steel in the absence and presence of the inhibitor. The inhibitive action of plant extracts on mild steel corrosion was investigated by scanning electron microscope (SEM) technique. The mild steel specimens were immersed in 1N HCl solution in the absence and presence of 2% v/v concentration of the inhibitor for a period of 3h at room temperature. After the specified time the specimen was removed, washed carefully with distilled water without disturbing the surface and dried. SEM micrograph of the uninhibited and inhibited samples were taken to examine the nature of corrosion product formed on the surface of mild steel specimens using JOEL SEM model JSM 6360 at Metallurgy department, PSG College of technology, Coimbatore, India.

III. Results and Discussion

3.1 Weight loss method

3.1.1 Effect of inhibitor concentration

The weight loss method of monitoring corrosion is useful because of its simple application and high reliability. Fig. 3 reflects the inhibition efficiency of various concentration of inhibitor on mild steel in hydrochloric acid. It is observed that there was marked reduction in the corrosion rate of mild steel even for a concentration of 0.05% v/v. Maximum inhibition efficiency was shown at 2% v/v concentration. Further increase in inhibitor concentration did not show significant increase in efficiency. Increase in IE with increase in concentration indicates strong adsorption of constituents present in the bract extract on the surface of mild steel giving a protective layer [14].
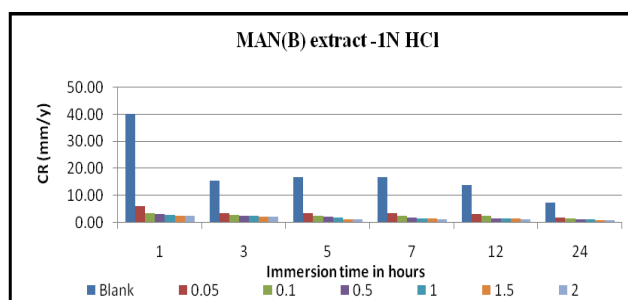

Fig. 3 Effect of immersion time on CR of mild steel in 1N HCl without and with MAN (B) extract

3.1.2 Effect of immersion time

Table 1 gives the values of corrosion rate and percentage inhibition efficiency obtained in 1N HCl in absence and presence of MAN (B) extract at 303K for immersion period from 1h to 24h. The inhibition efficiency increases with increase in concentration of the inhibitor irrespective of the time of immersion. Maximum IE of 94.93% was observed for 2% v/v concentration at 5h of immersion period. Immersion for a longer period leads to desorption of the adsorbed phytochemical constituents [15]. It is clear that bract extract of *Musa acuminata* acts as a good corrosion inhibitor for mild steel in 1N HCl solution.

Table 1 CR and IE of MAN (B) extract in 1N HCl acid at various concentration and immersion period

Conc. (% v/v)	1h		3h		5h		7h		12h		24h	
	CR mm/y	IE %	CR mm/y	IE %	CR mm/y	IE %	CR mm/y	IE %	CR mm/y	IE %	CR mm/y	IE %
Blank	40.23	-	15.34	-	16.72	-	16.72	-	13.83	-	7.20	-
0.05	6.02	85.04	3.12	79.66	3.19	80.93	3.42	79.52	2.80	79.79	1.49	79.37
0.10	3.34	91.69	2.64	82.81	2.23	86.67	2.40	85.62	2.17	84.29	1.21	83.17
0.50	2.79	93.08	2.34	84.75	1.81	89.20	1.48	91.14	1.37	90.13	1.04	85.62
1.00	2.67	93.35	2.15	85.96	1.60	90.40	1.35	91.91	1.30	90.60	0.85	88.14
1.50	2.34	94.18	2.01	86.93	0.89	94.67	1.27	92.38	1.22	91.20	0.76	89.49
2.00	2.23	94.46	1.82	88.14	0.85	94.93	1.13	93.24	1.13	91.80	0.70	90.32

3.1.3 Effect of temperature

Weight loss measurement was carried out over a range of 303 K – 353 K in presence and absence of the inhibitor for an immersion period of 1h, to evaluate the stability of the adsorbed film on the mild steel. The results obtained are listed in Table 2. The IE increases up to 313 K and then decreases thereafter. At elevated temperatures the rate of dissolution of mild steel increases as time lag between adsorption and desorption decreases and hence the inhibition efficiency decreases. Also with increased desorption of inhibitor molecules at higher temperature, more surface area of mild steel comes in contact with the acid environment, resulting in decrease in IE with increase in temperature [16].

Table 2 CR of mild steel and IE of MAN (B) extract in 1N HCl at different temperature

Conc. % v/v	303 K		313 K		323 K		333 K		343 K		353 K	
	CR mm/y	IE %	CR mm/y	IE %	CR mm/y	IE %	CR mm/y	IE %	CR mm/y	IE %	CR mm/y	IE %
Blank	52.49	-	111.78	-	237.50	-	230.48	-	448.36	-	328.33	-
0.05	11.15	78.77	17.94	83.95	39.01	83.58	49.15	78.68	97.41	78.28	128.61	60.83
0.10	7.69	85.35	13.26	88.14	27.75	88.32	35.55	84.58	90.39	79.84	95.74	70.84
0.50	4.68	91.08	5.80	94.82	18.06	92.40	17.83	92.26	41.24	90.80	65.42	80.08
1.00	3.57	93.21	4.90	95.61	13.37	94.37	12.26	94.68	38.90	91.32	47.14	85.64
1.50	3.45	93.42	4.46	96.01	11.26	95.26	12.15	94.73	27.97	93.76	50.15	84.73
2.00	2.23	95.75	3.46	96.91	10.03	95.78	11.15	95.16	24.19	94.61	44.58	86.42

3.2 Adsorption isotherm

The surface coverage (θ) values for various concentrations of the inhibitor in HCl medium was evaluated from the weight loss data. The data were tested graphically to find a suitable adsorption isotherm. A plot of C/θ against C generated a straight line (Fig. 4) with slope close to unity and regression coefficient greater than 0.9 indicating strong adherence to Langmuir adsorption isotherm. The application of Langmuir isotherm to the adsorption of MAN(B) extract on the mild steel surface indicates that there is no interaction between the adsorbate and adsorbent [13,17].
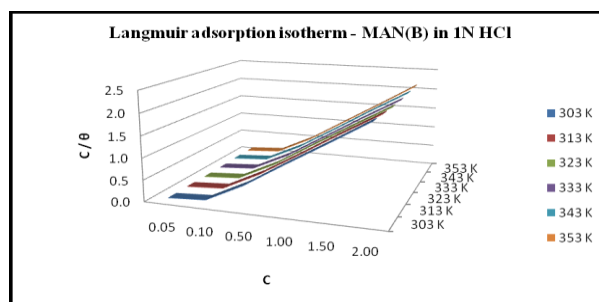

Fig. 4 Langmuir isotherm for adsorption of MAN (B) extract in 1N HCl on mild steel surface

3.3 Electrochemical study
3.3.1 Potentiodynamic polarisation

The cathodic and anodic polarisation curves recorded for mild steel in 1N HCl solutions without and with various concentration of MAN (B) extract is illustrated in Fig. 5. Electrochemical corrosion kinetic parameters obtained by Tafel extrapolation method are given in Table 3. No definite shift in the corrosion potential (E_{corr}) is detected, although there was no specific relation between E_{corr} and inhibitor concentration.

It can be seen from Fig. 5, the anodic and cathodic reactions are affected by the inhibitor, indicating that MAN (B) acts as mixed-type inhibitors. The addition of MAN (B) extract to HCl solution therefore reduces the anodic dissolution of iron and also retards the cathodic evolution reaction. It can be seen from the polarisation results that the corrosion current density (I_{corr}) decreased with the increase in inhibitor concentration, due to the increase in the blocked fraction of the metal surface by adsorption. The polarisation resistance (R_p) values were determined from the corrosion potential. It was found that R_p values increase with increase in MAN (B) extract concentration [18]. The inhibition efficiencies calculated from the corrosion current density and the polarisation resistance increased with the inhibitor concentration reaching a maximum value at 2% v/v.

Table 3 Potentiodynamic polarization parameters for mild steel in IN HCl in absence and presence of MAN (B)

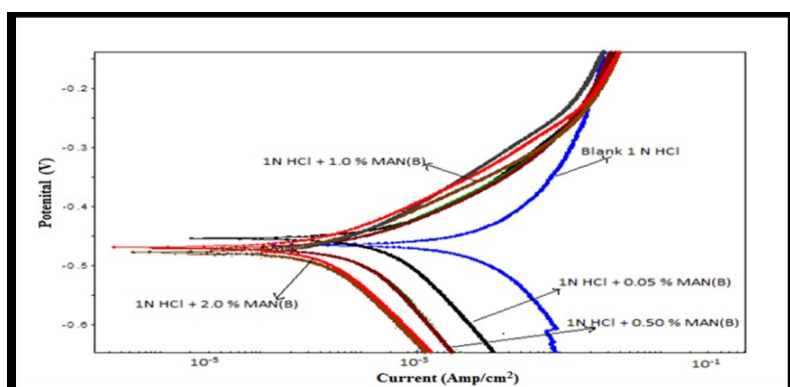| Conc. % v/v	$-E_{corr}$ (V)	Tafel slope		I_{corr} mAmpcm^2	IE % Tafel	R_p Ohmcm^2	IE % Linear
		b_a mV/dec	b_c mV/dec				
Blank	0.466	206.34	258.78	2887.00	-	6.72	-
0.05	0.454	111.32	221.12	272.50	90.56	8.27	18.74
0.50	0.471	83.62	202.22	111.90	96.12	12.07	44.32
1.00	0.478	81.63	178.40	50.78	98.24	12.85	47.70
2.00	0.469	85.27	176.06	49.08	98.30	22.36	69.95

Fig. 5 Potentiodynamic polarization curves for mild steel in 1N HCl without and with various concentration of MAN (B) extract

3.3.2 Electrochemical impedance spectroscopy (EIS)

The corrosion behaviour of mild steel in 1N HCl solution in the presence of MAN (B) was investigated by the EIS method at 303 K (Fig. 6). It is apparent that the impedance response of mild steel in uninhibited HCl solution has significantly changed after addition of MAN (B) in the corrosive solution. As a result, the semicircle in the presence of inhibitor is bigger than that in the absence of inhibitor (Blank) and increases as the inhibitor concentration increases. This confirms that the impedance of inhibited substrate increases with the concentration of MAN (B) in 1N HCl. The parameters deduced from the fit of Nyquist diagram for 1N HCl medium containing various concentrations of MAN (B) are given in Table 4. Addition of MAN (B) increases C_{dl} value indicating reduction of surface inhomogeneity due to the adsorption of plant extract molecules. Capacitance values decreases on increasing the MAN (B) concentration indicates reduction of charges accumulated in the double layer due to formation of adsorbed plant extract layer. The charge transfer resistances of double layer (R_{ct}) increases on increasing the MAN(B) concentration indicating that increase in concentration of bract extract decreases corrosion and increases inhibition [19]. The maximum of corrosion inhibition efficiency of 93.55% (C_{dl}) and 95.60% (R_{ct}) was noticed for 2% v/v bract extract.

Table 4 Impedance parameters for mild steel in the absence and presence of MAN (B) in 1N HCl

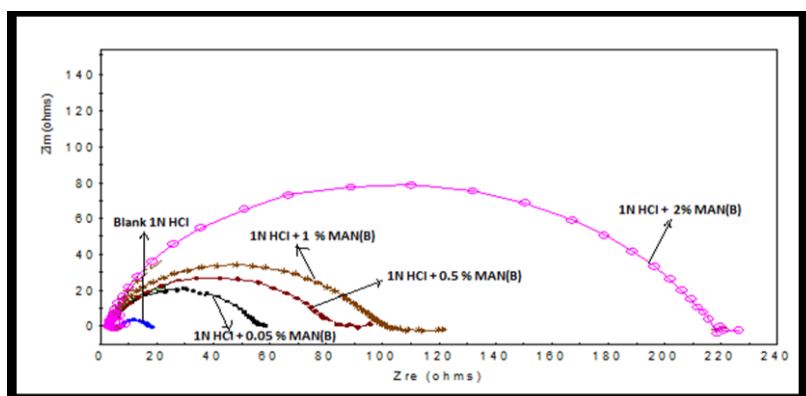| Conc. (% v/v)	C_{dl} (μFcm^{-2})	R_{ct} Ohmcm^2	IE %	
			C_{dl}	R_{ct}
Blank	1.986×10^{-4}	8.63	-	-
0.05	1.860×10^{-5}	48.86	90.63	82.34
0.50	1.627×10^{-5}	73.61	91.81	88.28
1.00	1.328×10^{-5}	92.34	93.31	90.65
2.00	1.282×10^{-5}	196.20	93.55	95.60

Fig. 6 Nyquist plots of mild steel in 1N HCl without and with various concentration of MAN (B)

3.4 Surface examination studies

Surface examination studies through scanning electron microscopy for the mild steel specimens exposed to 1N HCl at 303 K for 3h in the absence and presence of 2% v/v concentration of the MAN (B) extract are shown in Fig. 7 a, b, c. The parallel features on the polished mild steel surface (Fig. 7a) before exposure to the corrosive acid medium, are associated with polishing scratches. Examination of Fig.7b revealed that the specimen immersed in 1N HCl was rough and highly damaged due to the direct attack of aggressive acids. Fig.7c clearly showed that the mild steel surface was covered with the protective layer formed by the inhibitor which prevents the metal from further attack of acid medium thus inhibiting corrosion [20].
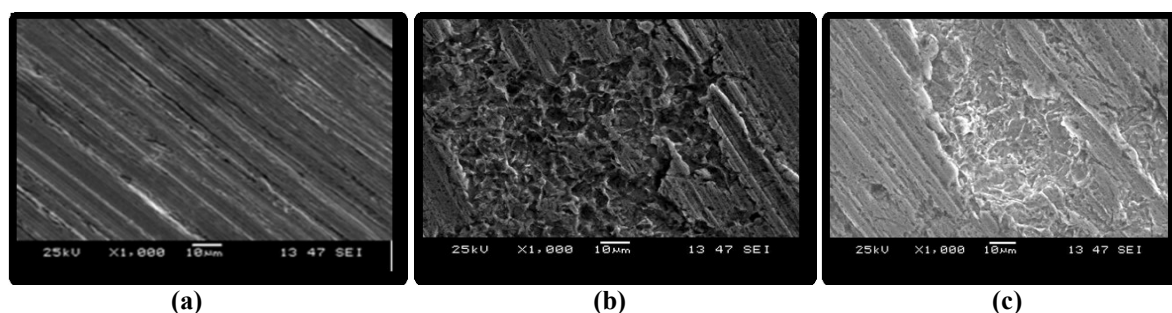

(a)   **(b)**   **(c)**

Fig. 7 (a) Polished mild steel surface (b) Mild steel surface exposed to 1N HCl (c) Mild steel surface exposed to 1N HCl + MAN(B) extract

3.5 Proposed mechanism for inhibition by *Musa acuminata* bract extract

The mechanism of inhibition can be understood by knowing the mode of interaction of the inhibitor molecules with mild steel surface. Inhibitors function by adsorption and/ or hydrogen bonding to the metal. This depends on the chemical composition and structure of the inhibitor, the nature of the metal surface, and the properties of the medium. Structural and electronic parameters like functional group, steric and electronic effects may also be responsible for the inhibition efficiency of any inhibitor, that is, the adsorption mechanism. The compounds have to block the active corrosion sites on the metal surface and hence the adsorption occurs by the bonding of the free electrons of inhibitor with the metal.

The bract extract may constitute organic compounds containing (i) lone pair of electrons present on a hetero-atom (eg. N, S, P, O) (ii) pi-bond (iii) triple bonds (eg. cyano groups) and (iv) heterocyclic compounds such as pyridine ring pyrrole, imidazole etc. Phytochemical analysis of *Musa acumianta* bract showed the presence of alkaloids, flavonoids, saponins, tannins, terpenoids, coumarins, glycosides, total phenols and

steroids. These compounds possess hetero atoms such as –O and –N which strengthen their adsorptive property over mild steel surface. The inhibiting influence of these molecules may be attributed to their adsorption through the –NH, C=O, OH, COOH etc. groups and also may be due to the presence of pi electrons in the rings. These organic molecules get physisorbed on the metal surface forming a protective film and hence the anti-corrosive behaviour [21].

IV.   Conclusion

Musa acuminata bract extract acts as efficient corrosion inhibitor on mild steel in 1N HCl acid solution. Temperature effect shows that *Musa acuminata* exhibits efficiency up to 313 K. Potentiodynamic polarisation studies indicated that the studied bract extract affected both anodic and cathodic reactions by blocking the active sites of mild steel surface and are of mixed type inhibitor. Results obtained in weight loss methods (non-electrochemical methods) have good agreement with potentiodynamic polarisation and impedance method (electrochemical methods). The corrosion inhibition action may be due to the presence of the various phytochemicals in the bract extract. The nature of the adsorption of MAN (B) extract on the mild steel surface was evaluated as physisorption. Inhibitor can be tested in the actual work spot of manufacturing and storehouse of electronic components and to other types of industrial materials and standardised as commercial inhibitor. In industrial environment, pickling of metals in HCl in the presence of bract extract can reduce largely the hydrogen evolution and acid mist formation. A chemical analysis of the extract solution can be done to get an idea of the active compounds present which are actually responsible for corrosion inhibition.

Acknowledgements

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