ANTICORROSIVE ACTIVITY OF SANTALUM ALBUM LEAVES EXTRACT AGAINST THE CORROSION OF MILD STEEL IN ACIDIC MEDIUM.

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Abstract
The inhibitive effect of Santalum Album (SA) plant extracts on the corrosion of mild steel in aqueous solution of 1N HCl was investigated by weight loss method, potentiodynamic polarization and electrochemical impedance spectroscopy techniques (EIS) at room temperature. Potentiodynamic polarization curves indicated that the studied plant extracts acts as mixed type inhibitor. EIS measurements showed that the corrosion process was under activation control. The inhibition efficiency increase with increase inhibitor concentration of plants extract at room temperature. The inhibitive effect of the santalum album plant could be attributed to the presence of corrosion protective compound in the plant which is adsorbedon the surface of the mild steel. Characterization of SA extract was carried out using FTIR spectroscopy. The SEM morphology of the absorbed protective film on the mild steel surface has confirmed the high performance of inhibitive effect of the plant extract.

Keywords: Santalum Album (SA)*; Mild Steel (MS); Corrosion Inhibition; Weight Loss Method; Electrochemical Techniques; Scanning Electron Microscopy (SEM).*

Introduction
Corrosion can be defined as the deterioration of a metal due to its reaction with its environment (Srikanth, 2006). Acidic solutions are used in many industrial areas. The most important applications are acid pickling, industrial acid cleaning, acid descaling, and oil well acidizing (Fouda, 2006). The use of inhibitor is one of the most practical methods for protection against corrosion and prevention of unexpected metal dissolution and acid consumption, especially in acid solution. Different organic and inorganic compounds have been studied as inhibitors to protect metals from corrosive attack. The efficiency of this organic corrosion inhibitor is related to the presence of polar function with S,O and N atoms in the molecule, heterocyclic compounds and pi electron (Benali, 2007; Merah, 2008; Benali, 2009; Benali, 2010;). Such compounds can adsorb onto the metal surface and block the active surface sites, thus reducing the corrosion rate. Although many synthetic compound show good anticorrosive activity, most of them are highly toxic to both human beings and the environment and they are often expensive and non – biodegradable (El-Etre, 2001;El-Etre, 2000; Saleh, 1983; Noor, 2007; Avwiri, 2003; Umoren 2003; Umoren 2009;). Thus, the use of natural products as corrosion inhibitors has become a key area of research because plant extracts are viewed as an incredibly rich source of naturally synthesized chemical compounds that are biodegradable in nature and can be extracted by simple procedures with low cost (Uromen, 2008; Bothi Raja, 2008; Shudan, 2012; Abiola, 2010; Ji, 2012; Oguzie, 2008; Lecante, 2011;). The extracts of their leaves,peels, seeds, fruits and roots(El-Etre, 2003; Sangeetha, 2011; Vijayalakshmi, 2011; Lahhit, 2011; Viondkumar, 2010; Bouyanzer, 2010; Odiongenyi, 2009; Obot, 2009; Aboia, 2010; and Janaina cardozo da Rocha,2010;)have been reported as effective corrosion inhibitors in different aggressive environments. The natural products extracted from leaves have been widely studied as corrosion inhibitors. A large number of scientific studies have been devoted to the inhibitive action of some plant extracts on the corrosion of mild steel in acidic media, showing that these extracts

could serve as good corrosion inhibitor.In this study, the medicinal plants leaves have been selected to study the inhibition effect on the corrosion of mild steel in 1N HCl media.

Materials and Methods

Preparation of Mild Steel Specimen

Mild steel strips were mechanically cut into strips of size 4 cm x 2 cm x 0.1cm containing the composition of C- 0.030%, Mn- 0.169%, Si- 0.015%, P- 0.031%, S - 0.029%, Cr- 0.029%, Ni- 0.030%, Mb- 0.016%, Cu- 0.017% and the remainder Fe and provided with a hole of uniform diameter to facilitate suspension of the strips in the test solution for weight loss method. For electrochemical studies, mild steel strips of the same composition but with an exposed area of $1cm^2$ were used. Mild steel strips were polished by using emery paper of various (400, 600, 800, 1000, and 1200) grade, subsequently degreased with acetone and finally washed with deionized water, and stored in the desiccators. Accurate weight of the metal was taken using four digital electronic balancemodel (shimadzu ay220).

Preparation of the Plant Extract

The medicinal plants of Santalum album leaves were taken and cut into small pieces, and dried in room temperature and ground well in to powder. 10g of the powder was refluxed in 200 ml distilled water and kept overnight. The refluxed solution was then filtered carefully, the filtrate volume was made up to 500ml using double distilled water which was the stock solution, and the concentration of the stock solution was expressed in term of ppm.

From the stock solution, 5 - 30 ppm concentration of the extract was prepared using 1N hydrochloric acid. Similar kind of preparation has been reported in studies using aqueous plant extract in the recent years.

Weight Loss Method

Mild steel specimens were immersed in 200ml of 1N HCl solution of various concentration of the inhibitor in the absence and presence of mild steel for 24 hours at room temperature. The weights of the specimens before and after immersion containing were determined using four digit electronic balance (shimadzu ay220).

From the mass loss measurements, the corrosion rate was calculated using the following relationship.

$$CR\ (mmpy) = \frac{K \times Weight\ Loss}{D \times A \times t\ (in\ hours)} \qquad (1)$$

Where, K = 8.76×10^4 (constant), D is density in gm/cm^3 (7.86), W is weight loss in grams and A is area in cm^2.

The inhibition efficiency (%) was calculated using equation (2) respectively,

$$IE\ \% = \frac{W_0 - W_i}{W_0} \times 100 \qquad (2)$$

Where, W_0 and $W_i$are the weight loss in the absence and presence of the inhibitor

FTIR Measurement

FTIR spectra were recorded in a BRUKER ALPHA 8400S spectrometer. The film was carefully removed, mixed thoroughly with KBr made into pellets and FTIR spectra were recorded.

Pontetiodynamic Polarization Methods

Potentiodynamic polarization measurements were carried out using CHI608D electrochemical analyzer. Experiment were carried out in a conventional three electrode cell assembly with mild steel specimen of $1cm^2$ area which was exposed and the rest being covered with red lacquer, was used as working electrode, a rectangular Pt foil as the counter electrode, and a saturated calomel electrode as standard reference electrode. A time interval of 15 minutes was given for each experiment to attain the steady state open circuit potential.The polarization was carried from a cathodic potential of -800mV (vs. SCE) to an anodic potential of -200mV (vs. SCE) at a sweep rate of 1 mV per second. From the polarization curves, Tafel slopes, corrosion potential, and corrosion current were calculated. The inhibitor efficiency was calculated using the formula:

$$IE\% = \frac{I_{Corr} - I^*_{Corr}}{I_{Corr}} \times 100 \qquad (3)$$

Where I_{corr} and I^*_{corr} are corrosion current in the absence and present of inhibitors.

Electrochemical Impedance Method

The electrochemical AC-Impedance measurements were also performed using CHI608D electrochemical analyzer. Experiments were carried out in a conventional three electrode cell assembly as that used for potentiodynamic polarization studies. A sine wave with amplitude of 10 mV was superimposed on the steady state open circuit potential. The real part Z' and the imaginary part Z" were measured at various frequencies in the range of 100 KHz to 10 MHz. A plot of Z' versus Z" was made. From the plot, the charge transfer resistance (R_{ct}) was calculated, and the double layer capacitance (C_{dl}) was then calculated using formula:

$C_{dl} = 1/2\pi f_{max} Rct (4)$

Where R_{ct} is charge transfer resistance, and C_{dl} is double layer capacitance. The experiments were carried out in the absence and presence of different concentration of inhibitor.

$$IE\% = \frac{R_{ct} - R^0_{ct}}{R_{ct}} X100 \qquad (5)$$

Where R_{ct} and R^0_{ct} are the charge resistance values in the inhibited and uninhibited solution respectively.

Phytochemical Analysis

Phytochemical screening were performed to assess the qualitative chemical composition of the different samples of plants extract using commonly employed precipitation and coloration reactions to identify the major secondary metabolites like alkaloids, flavonoids, glycosides, proteins, phenolic compounds, saponins, starch, steroids, tannins and terpenoids.

Scanning Electron Microscopy

The mild steel specimen immersed in blank and in the inhibitor solution for a period of one day was removed, rinsed with double distilled water, dried and observed in a scanning electron microscope to examine the surface morphology. The surface morphology measurements of mild steel were examined using (JEOL) computer controlled scanning electron microscope.

Result and Discussion

Weight Loss Methods

Weight loss method was done for mild steel in 1 N HCl with various concentrations of santalum album leaves extract ranging from 5 to 30 ppm, and the corresponding values of inhibition efficiency and corrosion rate are given in Table 1. It was observed from the table that the corrosion rate decreased and thus the inhibition efficiency increases with increasing concentration of santalum album leaves extract (5 ppm to 30 ppm,). The maximum inhibition efficiency of about 84.40% was achieved at 25 ppm of santalum album leaves extract. This result indicated that santalum album leaves extract could act as an excellent corrosion inhibitor.

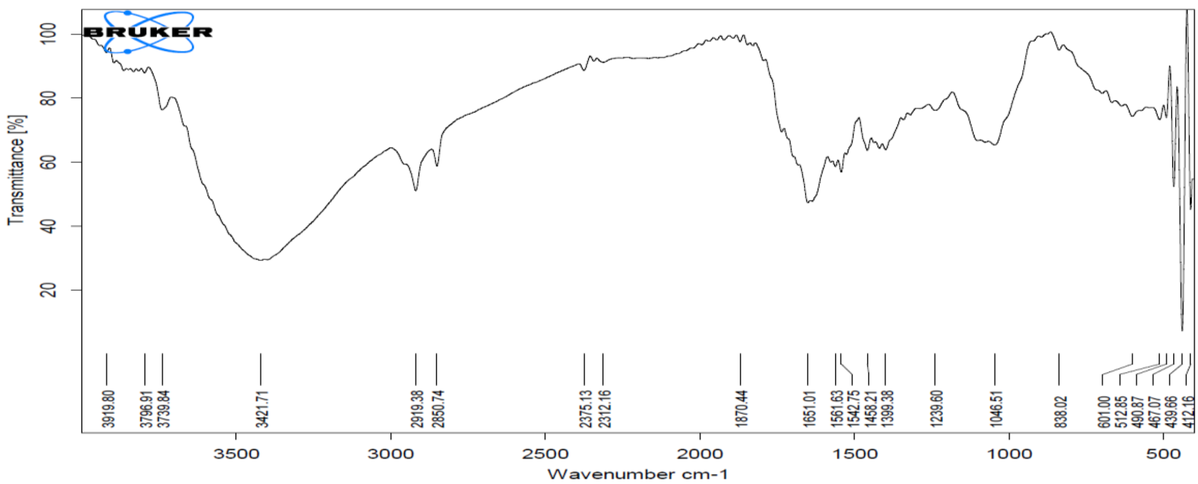
Table 1- Data from Weight Loss Method for MS corroding in 1 N HCl solutions at various concentrations of SA leaves extract.

Conc. of SA leaves Extract (ppm)	Corrosion Rate (mmpy)	Inhibition Efficiency (%)
0	0.2440	*
5	0.0611	72.50
10	0.0480	77.98
15	0.0475	78.85
20	0.0337	80.96
25	0.0314	84.40
30	0.0326	83.14

FTIR Measurement

FTIR spectra have been used to analyze the protective film formed on metal surface. Lalitha, (2005) have confirmed that FTIR spectrometer is a powerful instrument that can be used to determine the type of bonding for organic inhibitor adsorbed on the metal surface. Although various compounds present in the SA leaves extract which contributed in effective working in the inhibitor, it was very difficult to identify each compound separately to know the group present in the SA leaves extracts.

Figure 1 - FT-IR spectra of SA leaves extract.



FTIR spectra of the SA leaves extract is shown in Figure 1. It was observed from the figure, the peak obtained at 3421 cm^{-1}can be assigned to N-H stretching was observed. Absorption band at 2919 cm^{-1} and 2850 cm^{-1} can be assigned to C-H stretching vibration. Medium absorption band at 2375 cm^{-1} and 2312cm^{-1} are observed due to CN (nitrile) or alkyne stretching vibration. Other strong peak obtained at 1870 cm^{-1} corresponds to C=O (may be aldehyde or ketone). Strong peaks obtained at 1651cm^{-1} and 1561cm^{-1} are due to N-O stretching or N-H bending vibration. Absorption band at 1458cm^{-1} can be assigned to C-H bending in CH_3 or O-H bending vibration. Peaks observed at 1399cm^{-1}, 1239cm^{-1} and 1046cm^{-1} are due to C-N and C-O stretching vibration. Few weak peaks can also observed at 1542cm^{-1}, 1530.41cm^{-1}, which correspond to C=C stretching vibration of aromatic ring. On the basis of the result, it can be said that SA leaves extract contain Nitrogen and Oxygen

(N-H, C=N, C-N, O-H, C=O, C-O) in various functional group and aromatic ring, which make this extract attractive for being used as inhibitor.

Pontetiodynamic Polarization Methods

The potentiodynamic polarization curves for mild steel in 1 N HCl with and without inhibitor (extract) are shown in Fig.2. It was evident from the figure that the anodic and cathodic curves for mild steel inhibited with extract were shifted towards positive potential region compared to the blank metal immersed in 1 N HCl. The corrosion parameters such as corrosion potential (E_{corr}) and corrosion current density (I_{corr}), obtained from Tafel plots are given in Table 2. From the table, it is observed that the I_{corr}values are found to decrease with increase in the inhibitor concentrations, ranging from 5 to 30 ppm.

Figure 2 -Potentiodynamic polarization (Tafel) curve for mild steel in 1N HCl solution in the absence and presence of different concentration of SA extracts.

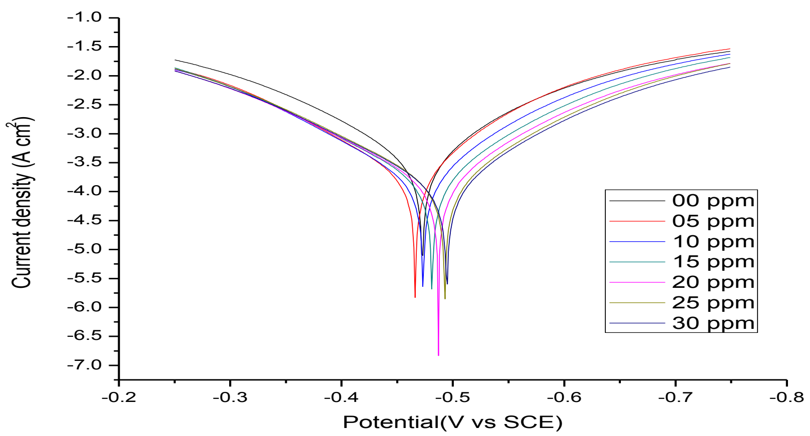


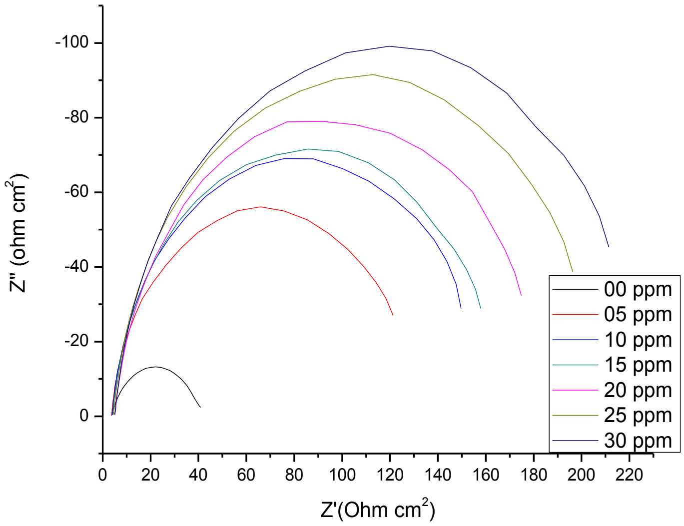
Table 2 - Electrochemical parameter from polarization measurement and calculated values of inhibitor efficiency.

Conc. ppm	E_{corr}/ (mV/ SCE)	I_{corr}/ (mA/cm^2)	b_c (mV/dec.)	b_a(mV/dec.)	LPR Ohm*cm^2	IE (%)
Blank	-0.473	3.972x10^{-4}	110.44	105.57	62	*
05	-0.466	2.102x10^{-4}	96.50	096.46	100	47.08
10	-0.473	1.794x10^{-4}	97.53	097.81	118	54.83
15	-0.481	1.672x10^{-4}	97.62	101.17	130	57.90
20	-0.487	1.541x10^{-4}	99.51	104.31	143	61.20
25	-0.495	1.360x10^{-4}	99.83	105.19	164	65.76
30	-0.493	1.434x10^{-4}	98.15	104.75	154	63.90

It was noted from the table that the addition of green inhibitor decreases the dissolution rate of mild steel in 1N HCl acid media. The corrosion current density values decreased considerably for green inhibitor in the acid media. As can be seen, the mild steel with 25 ppm concentration of the inhibitor showed excellent resistance than when compared to other concentrations of the inhibitor. However, the shift in the values of corrosion potential (E_{corr}) for SA leaves extract was not significant (Lakshmi prabha, 2012). This observation clearly showed that the inhibition of mild steel in the presence of the extract control both cathodic and anodic reactions and thus the inhibitor acts like mixed type inhibitors.

Electrochemical Impedance studies

Figure 3 - Nyquist plots for mild steel in 1N HCl acid solution without and with presence of different concentration of SA extract.



The surface resistances of blank and mild steel specimens with inhibitor in 1N HCl solutions were investigated using EIS techniques. The Nyquist plot of mild steel in 1N HCl in the absence and the presence of various concentration of green inhibitor was shown in Figure 3. The presence of a single semi-circle in the blank and for different concentrations of the inhibitor systems corresponds to the single charge transfer mechanism during dissolution of mild steel, which is unaltered by the presence of inhibitor components. The impedance parameters were calculated for mild steel in 1N HCl with and without inhibitors are given in Table 3.

As noticed in Figure 3. the obtained impedance diagrams are almost in a semi-circular appearance, indicating that the charge transfer process mainly controls the corrosion of mild steel. Deviations of perfect circular shape are often referred to the frequency dispersion of interfacial impedance. This anomalous phenomenon may be attributed to the inhomogeneity of the electrode surface arising from roughness or interfacial phenomena. In fact, the presence of SA leaves extract enhanced the values of R_{ct} in acidic solution. Values of double layer capacitance are also brought down to the maximum extent in the presence of inhibitor and the decrease in values of C_{dl} follows the order similar to that obtained for I_{corr} in this study. The decrease in C_{dl} shows that the adsorption of this inhibitor takes place on the metal surface in acidic solution.

Moreover, the increase in the value of R_{ct} with the inhibitor concentration leads to the increase in inhibitor efficiency. The maximum R_{ct}value of 199.2Ω$cm^2$and minimum C_{dl} value of 1.292 µF/$cm^2$are obtained at an optimum concentration of 25 ppm with maximum inhibition efficiency of 84.40%.

Table.3 Impedance parameter for mild steel in 1N HCl acid solution in the absence and presence of varied concentration of inhibitor.

SI.NO	Concentration (ppm)	R_{ct}(ohm cm^2)	C_{dl} ($\mu F/cm^2$)	IE (%)
1	Blank	31.07	7.36	-
2	5	113	2.377	72.50
3	10	141.1	1.739	77.98
4	15	146.9	1.525	78.84
5	20	163.2	1.443	80.96
6	25	199.2	1.292	84.40
7	30	184.3	1.342	83.14

Phytochemical Analysis

Phytochemical screening was carried out on the aqueous extracts freshly prepared to the common phytochemical methods described by Harborne(1998). The findings of the phytochemical screening of the aerial parts aqueous extract are shown in Table 4. The phytochemical analysis showed that the aqueous extract contain secondary metabolites included terpenoids, saponin, phenol, tannins and anthocyanin(Misra, 2012).

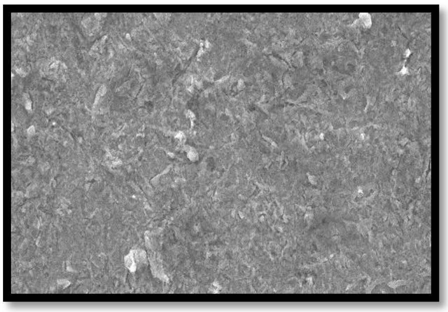
Table 4 - Phytochemical screening test of extract of SA leaves.

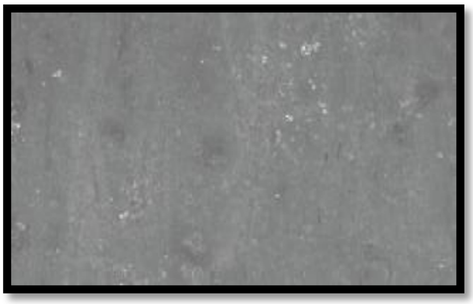
Phytochemical test	Aqueous extract
Alkaloids	-
Carbohydrates	-
Diterpens	+
Saponins	+
Phytosterols	-
Tannins	+
Flavanoids	+
Phenol	+
Glycosides	-
Amino acids	+
anthocyanine	+

(+).. Presence (-)... Absence

Scanning Electron Microscopy (SEM) Studies

Figure 4 - SEM image of the surface of mild steel after immersion for 24 hours in 1N HCl solution in the presence and absence of optimum concentration of the SA plants leaves extract.





(4a) (4b)

Surface examination of the mild steel specimens was made using JEOL scanning electron microscope (SEM). The mild steel specimens after immersion in 1N HCl solution for 24 hours at room temperature in the absence and presence of optimum concentration of the plant extract were taken out, dried and kept in a dessicator and the SEM image areshown in Figure .4(a) and 4(b). The protective film formed on the surface of the mild steel was confirmed by SEM studies. From the SEM image, it was found that more grains were found in SEM image of mild steel immersed in 1N HCl solution in the absence of the inhibitor. Whereas no grain were found in the SEM image of mild steel immersed in 1N HCl solution in the presence of the plant extract. Fig.4(b) indicates that in the presence of inhibitor SA leaves extract consisting of 25 ppm respectively, corrosion is suppressed as can be seen from the decrease of corroded areas. The metal surface is almost free from corrosion due to formation of insoluble complex on the surface of the metal. In the presence of inhibitor, the surface is covered by a thin layer of inhibitor which effectively controls the dissolution of mild steel (Benita Sherine, 2010).

Concusion

The results obtained show that SA leaves extract is a good corrosion inhibitor for mild steel under acidic condition. The maximum inhibition efficiency was 84.40%. Good agreement between the inhibition efficiencies calculated using different techniques was obtained. The adsorption of the green inhibitor onto the mild steel surface was characterized by the decrease in (i) the cathodic and anodic current densities observed in the potentiodynamic polarization curves carried out in the presence of SA leaves extract, (ii) the polarization resistance in the solution containing the inhibitor, (iii) the double capitance computed from electrochemical impedance spectroscopy experiments. It was also found that inhibitor worked as a mixed type inhibitor retarding both anodic and cathodic reactions. Surface images of the mild steel surface clearly showed that SA leaves extract inhibited corrosion of mild steel by getting adsorbed on the metal surface.

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