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Aqueous Extract of *Borassus flabellifer* Linn. (Palmyra Palm) Peduncle as a Green Inhibitor for Corrosion of Mild Steel in H_2SO_4 Medium

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Abstract

Inhibitive and absorptive nature of aqueous extract of palmyra palm (*Borassus flabellifer* Linn.) peduncle (BFP) for the corrosion of mild steel in 0.5 M H_2SO_4 was investigated using chemical and electrochemical techniques. Characterization of BFP was carried out using GC-MS and FT-IR spectroscopic studies. The aqueous extract of BFP was found to be a good adsorption inhibitor for the corrosion of mild steel in 0.5 M H_2SO_4 solution at all studied temperatures and period of immersion. Thermodynamic parameters revealed that there is a strong interaction between the metal surface and inhibitor. The adsorption characteristic of the inhibitor is consistent with the assumptions of El-Awady kinetic thermodynamic and Frumkin adsorption isotherm. A comprehensive adsorption model was proposed for the adsorption of aqueous peduncle extract of *Borassus flabellifer* Linn. on mild steel surface. The inhibition potentials of aqueous extract of BFP was enhanced by its phytochemical constituents. The electrochemical measurement reveal that the peduncle extract behaved like mixed type inhibitor. Surface analytical techniques such as SEM and FT-IR confirmed the adsorption of BFP on mild steel surface.

Key words: Corrosion inhibition, Palmyra palm peduncle extract, GC-MS and FT-IR.

Introduction

Acid solutions are often used in industry for cleaning, descaling and pickling of steel structures, processes which are normally accompanied by considerable dissolution of the metal. A useful method to protect metals and alloys deployed in service in aggressive environments against corrosion is addition of species to the solution in contact with the surface in order to inhibit the corrosion reaction and reduce the corrosion rate. Naturally occurring substances as inhibitors of acid cleaning process has continued to receive attention as replacement for synthesized organic inhibitors which are considered to be very toxic, expensive and environmentally unfriendly. The greatly expanded interest on naturally occurring substances, otherwise tagged 'green inhibitors' is attributed to the fact that they are cheap, ecologically friendly and posses no threat to the environment. In addition, they are readily available and renewable source of materials. Many plant leaves extracts such as *Phaseolus aureus* (Rajalakshmi et al., 2008), *Fenugreek* (Ehteram A.Noor, 2007), *opuntia* extract (El-Etre, 2003) and seeds extract such as *Strychnos Nuxvomica* (Ambrish Singh et al., 2011), *Areca catechu* (Vinod Kumar et al., 2011) and the aqueous destructive distillation extract obtained from *Cocos nucifera* petiole (Vijayalakshmi et al., 2011) *Mundulea Sericea* leaves (Leelavathi et al., 2013) and *Cocos nucifera* peduncle (Vijayalakshmi et al., 2013) have been reported to be promising corrosion inhibitors for mild steel in acid medium. In this context, the corrosion inhibition effect of aqueous extract of *Borassus flabellifer* Linn. peduncle on mild steel in 0.5 M H_2SO_4 medium is examined.

Borassus flabellifer L, colloquially known as "Palmyra" is a species of palm, belongs to the *Arecaceae* family and most of which are restricted to tropical, subtropical, and warm temperate climates. *Borassus flabellifer* L. peduncle (BFP) as a solid waste from palm tree after harvest of the fruit was converted into aqueous extract by destructive distillation process and can be utilized as corrosion inhibition for mild steel in 0.5 M H_2SO_4.

The present study investigates the inhibiting effect on the corrosion of mild steel in acidic medium by using corrosion monitoring techniques such as weight loss, potentiodynamic polarization and electrochemical impedance spectroscopy. GC-MS analysis was carried out to characterize the BFP extract. FT-IR investigation of the studied systems is envisaged in subsequent studies to ascertain the corrosion morphology and also determine the active components in the adsorbed layer. In addition to this, kinetic and activation parameters that govern metal corrosion and inhibition processes have also been evaluated.

Experimental

Corrosion tests were performed on mild steel sheets with weight percentage composition as follows; C, 0.11; Si, 0.061; Mn, 0.197; S, 0.023; P, 0.025; Ni, 0.010; Mo, 0.013; Cr, 0.037; Fe, 99.524. The mild steel sheet was mechanically pressed cut into 5 cm x 1 cm coupons with 2 mm thickness, mechanically polished, their edges were abraded with fine grade of emery papers, degreased with acetone, washed in double distilled water, dried, stored in a dessicator and used for weight loss method and 1 cm^2 with 5 cm long stem (isolated with Teflon tape) for electrochemical method. Appropriate concentrations of 0.5 M H_2SO_4 acid was prepared by using double-distilled water and AR grade sulphuric acid. The concentration range of BFP extract employed was varied from 0.5 to 3%v/v.

Inhibitor Material

In palmyra palm the flowers are borne on branching spikes, very generally protected by a spathe. When several flowers arise in a cluster on a common axis, the structure is referred to as an inflorescence. The common axis is the inflorescence axis which is called **peduncle.** The palmyra palm peduncles were collected from the tree, cleaned and chopped into small pieces, air dried and stored at room temperature prior to use. 250 gms of air dried palmyra palm peduncle pieces were transferred to a 2000 ml round bottomed flask, directly heated in a mantle with water condenser at about 80°C in the absence of air and without a carrier solvent (destructive distillation) (Akhter, et al., 2010)). The products obtained are i) A gaseous mixture or wood gas. ii) 90 ml of **aqueous dark brown liquid distillate obtained which was used for the corrosion inhibition studies**. iii) 15 ml of a thick black liquid or wood tar which was separated from the aqueous distillate using a separating funnel. iv) 85 gms of a solid residue or wood charcoal was left in the round bottle flask. Phytochemical composition of aqueous dark brown liquid distillate of BFP extract was analysed by Gas Chromatograph - Mass Spectrometry (GC - MS).

Characterization of peduncle extract by GC-MS and FT-IR spectroscopy

Characterization of BFP was done by GC-MS using Fisons Instrument. The FT-IR spectrum was recorded with a frequency ranging from 4000 to 400 cm^{-1} using BRUKER Optik GmbH FTIR spectrometer MODEL No - TENSOR 27 with the SOFTWARE - OPUS version 6.5 (CECRI – India) (Vinod Kumar, et al., 2011).

Mass loss method

The mild steel specimens in triplicate were immersed in 100 ml of electrolyte (0.5 M H_2SO_4) with and without the addition of different concentrations of BFP extract at room temperature and 40-70°C (Oguzie et al., 2004). The time duration of experiments were optimized and conducted for ½ , 1, 3, 6, 12 and 24 h at room temperature and ½ h in the temperature range of 40 - 70°C (error ± 2%) in stationary condition. At the end of exposure period, specimens were cleaned according to ASTM G31-72 (ANONIMO, 1999) and their weights were recorded using Denwar balance. The percentage inhibition efficiency (IE%) was calculated by the following equation

$$IE\ (\%) = (1 - W_i / W_o) \times 100 \qquad (1)$$

where W_0 and W_i are the weight loss in mg of mild steel coupons in the absence and presence of inhibited solutions.

Polarization and Impedance studies

Electrochemical studies were carried out using conventional three electrode cell with larger area platinum foil as counter electrode and saturated calomel electrode (SCE) as reference electrode. Solatron electrochemical analyzer (model 1280 B) interfaced with an IBM computer was used for measurements. The polarization studies were made after the specimen attained a steady state potential. The polarization was carried out using a Corrware software from a cathodic potential of -0.2 V to an anodic potential of +0.2 V with respect to the corrosion potential at a sweep rate of 2 mV/s. E versus log *I* curves were plotted. The linear TAFEL segments of the anodic and cathodic curves were extrapolated to corrosion potential to obtain the corrosion current densities. Z plot software was used for data acquisition and analysis of interfacial impedance. AC signals of 10 mV amplitude and a frequency spectrum from 100 KHz to 0.01 Hz was impressed and the Nyquist representations of the impedance data were analysed with Zview software.

Results and discussions

Characterization of BFP extract

Gas chromatography (GC)-mass spectroscopy (MS) spectra of aqueous dark brown liquid distillate of BFP extract contains 11 major peaks along with many small peaks indicating presence of more than 11 major compounds. The identification of the isolated extract compounds was achieved by comparing obtained mass spectra of unknown peaks with those stored in the NIST.02 (US National Institute of Standards and Technology) and Wiley.7n. mass spectral electronic libraries.

FT-IR spectrum of BFP extract is shown in fig.2. In the FT-IR spectra of BFP extract contains bands corresponding to phenolic hydroxyl groups (3452.62 cm^{-1}), C-H stretching (2943.76 cm^{-1}), carbonyl groups (1733.09 cm^{-1}), aromatic C=C bending (1639.54 cm^{-1}), two bands appear for the C-O stretching vibrations in esters in 1274.53 and 1055.18 cm^{-1}, aromatic ring (664.39 cm^{-1}) (Goh Meng Seng, et al., 2006).

Mass loss studies

In order to assess the stability of inhibitive behavior of BFP extract on a time scale, weight loss measurements were performed in 0.5 M H_2SO_4 solution in absence and presence of BFP extract concentration varied from 0.5 %v/v to 3 %v/v for the immersion time from ½ h to 24 h at room temperature. Inhibition efficiency was found to increase with increase in concentration of BFP extract, probably due to an increase in the metal surface area covered by the inhibitor. The inhibition efficiency of the extract was increased with increasing immersion time from ½ h to 24 h (85% IE at ½ h to 97% IE at 24 h in 0.5 M H_2SO_4 solution). The increase in inhibition efficiency up to 24 h reflects the strong adsorption of constituents present in the extract on the mild steel surface, resulting in a more protective layer formed at mild steel and acid medium interfaces (Rajalakshmi et al., 2010). Thus, BFP extract effectively inhibit the mild steel corrosion in 0.5 M H_2SO_4 medium and the adsorbed layer was found to be stable at longer time of immersion.

Effect of temperature on inhibition efficiency [IE(%)]

The influence of temperature on the corrosion rate in absence and presence of BFP extract was investigated by weight loss trends in the temperature range 303 K – 343 K during ½ h of immersion. The obtained data reveal, the inhibition efficiency increased with an increase in the inhibitor concentration indicating that adsorption of inhibitor species on mild steel surface. This suggests that the inhibitor species are adsorbed on the mild steel / solution interface where the adsorbed species mechanically screen the coated part of the metal surface from the action of the corrosive medium. In the present investigation, maximum IE was found to be 94.23% at 3%v/v concentration at 333 K temperature in 0.5 M H_2SO_4.

Adsorption considerations

The values of degree of surface coverage (θ) are quite useful in determining inhibitor adsorption characteristics. Surface coverage data are applied in construction of adsorption isotherm, which give detailed information on adsorption mechanisms. Attempts were made to fit (θ) values to the Frumkin, Freundlich, Temkin, and Langmuir isotherms and correlation coefficient (R^2) values were used to determine the best-fit isotherm. By far, best result was obtained for El-Awady kinetic thermodynamic and Frumkin adsorption isotherm model, which has the form (Bouklah, et al, 2006):

$$\log [\theta/(1-\theta)] = \log K' + y \log C \quad \text{[El-Awady kinetic thermodynamic]} \qquad (2)$$

$$\frac{\theta}{1-\theta} \exp(-f\,\theta) = KC \quad \text{[Frumkin adsorption isotherm]} \qquad (3)$$

where θ, is the degree of surface coverage, 'K' is the equilibrium constant of adsorption process and 'C' is the concentration of the inhibitor. From the plot of log (θ/1-θ) versus log C straight lines were obtained with $R^2 > 0.9$ for all the systems studied, indicating that the experimental data fit well into El-Awady kinetic thermodynamic adsorption isotherm. The deviation in the values of the slopes of El-Awady kinetic thermodynamic plots from unity may be advocated to be due to the mutual interaction between adsorbed molecules in close vicinity. 1/y is the number of inhibitor molecules occupying one active site (or the number of water molecules replaced by one molecule of BFP extract) (Table 1). The values of 1/y obtained are more than unity, indicating that each molecule of the phytochemical compound from the aqueous extract involved in the adsorption process is attached to more than one active site on the metal surface (Oguzie, et al, 2004).

The sigmoidal shape shows that the adsorption of the inhibitor on mild steel surface follows Frumkin isotherm. In Frumkin's adsorption isotherm, one adsorption molecules of BFP extract displaces one of adsorption water. The slope of the lines given by equation

$$\ln \frac{f(\theta,x)}{C} = \ln K + 2a\theta \qquad (4)$$

Table 1. Adsorption parameters for El-Awady kinetic thermodynamic isotherm for adsorption of BFP extract in 0.5 M H_2SO_4 on mild steel at different temperatures.

Acid Medium	Adsoption parameters	Temperature (K)				
		303	313	323	333	343
0.5 M H_2SO_4	R^2	0.965	0.980	0.984	0.990	0.990
	1/y	1.672	1.425	1.309	1.284	0.985

Energy of activation and thermodynamic parameters

The activation energies (E_a) calculated from the plot of log CR versus 1/T. Linear plots was obtained. The values of E_a were computed from the slope of the straight lines. The value of E_a decreases from 58 to 41 kJ/mol with the increase in inhibitor concentration of 3%v/v in 0.5 M H_2SO_4 (Martinez, et al., 2002). The decrease in the E_a value at higher level of inhibitor efficiency was reported in the literature (Tang, et al., 2006). Senthil Kumar et al., 2009, explained that at higher levels of inhibition the net corrosion reaction shifts from that on the uncovered part of the metal surface to the covered one. As a result of which a surface film is formed and surface area of the metal covered by inhibitor species increases as temperatures rises. These observations further support the occurrence of both physical and chemical adsorption for BFP extract species on mild steel surface from 0.5 M H_2SO_4 medium (Noor, 2007).

Adsorption of any component in the interphase is attended by a change in Gibbs energy and in enthalpy of the system. The enthalpy and entropy for the adsorption of BFP extract on mild steel was deduced from the thermodynamics basic equation

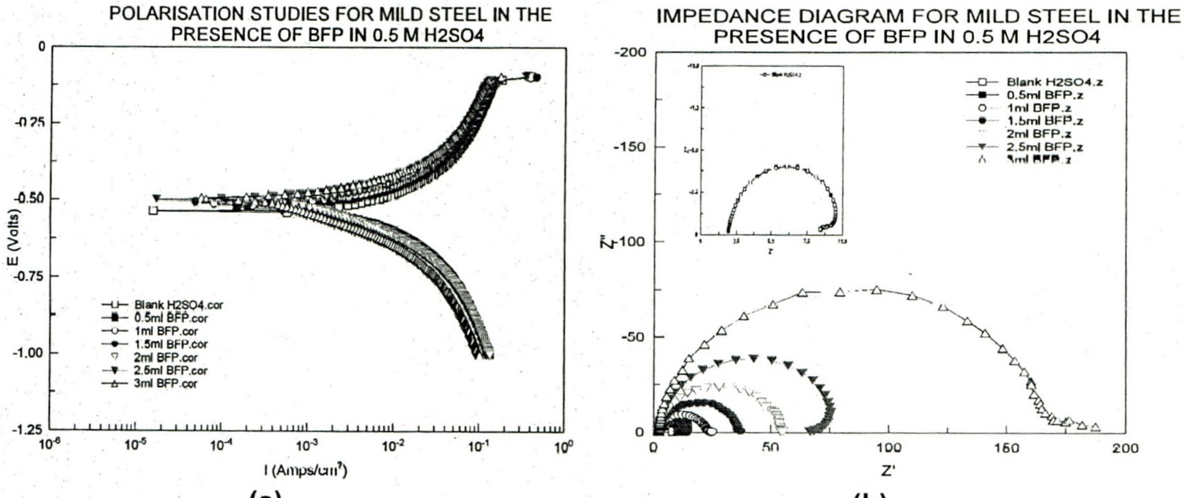
$$\Delta G^\circ_{ads} = \Delta H^\circ_{ads} - T\Delta S^\circ_{ads} \qquad (5)$$

The values of free energy of adsorption, ΔG°_{ads}, for the BFP extract in the temperature range 303 K - 343 K are obtained. The negative value of ΔG°_{ads} ensures the spontaneous adsorption and the stability of the inhibitor on the metal surface (Bouklah, et al, 2006). It is usually accepted that the values of ΔG°_{ads} around -20 kJ/mol or lower Indicate the electrostatic interaction between charged metal surface and charged organic molecules in the bulk of the solution while those around -40 kJ/mol or higher in value charge sharing or charge transfer between the metal surface and organic molecules (Moretti, et al, 1996). From the values of -ΔG°_{ads} the adsorption of the studied inhibitor species on mild steel surface from 0.5 M H_2SO_4 solution depending on the applied temperature range, indicating the BFP extract adsorb physically at low temperature and at high temperature, chemisorption is favoured.

The positive sign of ΔS°_{ads} arises from substitutional process, which can be attributed to the increase in the solvent entropy and more positive water desorption entropy (Solmaz et al., 2008). This leads to an increase in disorder due to the fact that more water molecules can be desorbed from the metal surface by one inhibitor molecule (Avci, 2008). The negative sign of ΔH°_{ads} for the system indicates that the adsorption of inhibitor molecules is an exothermic process.

Electrochemical measurements

Potentiodynamic studies were carried out and the corrosion kinetic parameters such as corrosion potential (E_{corr}), corrosion current density (I_{corr}), anodic Tafel slope (b_a) and cathodic Tafel slope (b_c) deduced from the curves are given in Table 2. There is no regular trend in the values of b_a and b_c which indicates the mixed type behaviour of the BFP extract. The corrosion current density values decrease from 672 μA/cm^2 (blank 0.5 M H_2SO_4) to 52 μA/cm^2 after the addition of 3%v/v of BFP extract resulting in 92% of inhibition efficiencies in 0.5 M H_2SO_4. E_{corr} values do not change appreciably with the addition of the inhibitor indicating that the inhibitor affects the anodic dissolution and cathodic hydrogen evolution reactions and acts as mixed type of inhibitor. Polarization resistance value (R_p) obtained from the LPR method (Table 2) showed a steep increase in value from 6.5 Ωcm^2 (blank 0.5 M H_2SO_4) to 26.5 Ωcm^2 after the addition of the highest concentration of BFP extract. The Impedance parameters of mild steel by addition of different concentrations of BFP extracts in 0.5 M H_2SO_4 indicate that increasing charge transfer resistance is associated with a decrease in the double layer capacitance. The decrease in the C_{dl} values could be attributed to the adsorption of the chemical constituents of BFP extracts, respectively, at the mild steel surface. It has been reported that the adsorption process on the metal surface is characterized by a decrease in C_{dl} [Abdel-Gaber, et al., 2006].



(a) (b)

Figure 1. (a) Potentiodynamic polarization and (b) Nyquist plots for mild steel in 0.5 M H_2SO_4 in the presence and absence of BFP extract

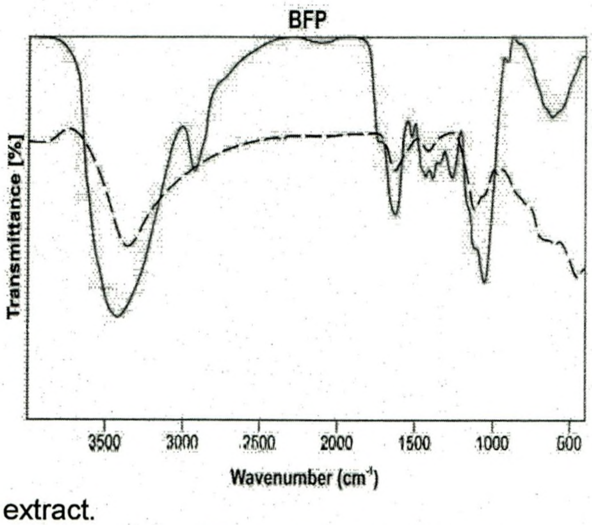
Table 2. Results of potentiodynamic polarization and impedance studies for the corrosion of MS in the presence of BFP extract in 0.5 M H_2SO_4

Inhibit or conc. (%v/v)	Electrochemical					Impedance method				LRP	
	I_{corr} (μA /cm²)	$-E_{corr}$ (mV/S CE)	b_a (mV/dec)	b_c (mV/ dec)	IE (%)	R_{ct} (Ω cm²)	IE (%)	C_{dl} (μF/c m²)	θ	R_p (Ω cm²)	IE (%)
Blank	672	536	222	186	-	7.8	-	349	-	6.5	-
0.5	333	522	179	135	50	12.6	38	306	0.123	9.9	34
1.0	213	512	149	112	68	20.6	61	284	0.186	11.1	41
1.5	184	516	144	108	72	34.0	76	255	0.269	13.7	52
2.0	174	513	149	98	74	51.9	84	141	0.595	15.1	56
2.5	67	514	128	76	90	75.7	89	106	0.696	24.9	73
3.0	52	521	135	81	**92**	164.5	95	82	0.765	26.5	**75**

Surface Analytical Techniques

FT-IR study

FT-IR spectra of the pure extract and corrosion product were presented in Figure 2. From the spectra, it is observed that the polymeric O-H stretching frequency of carboxylic acid is downshifted from 3452.62 to 3368.58 cm^{-1}. The carbonyl (C=O) group frequency has vanished from 1712.09 cm^{-1} which is due to the interaction between the functional group and the metal. The C=C stretching frequency is also changed from 1639.54 to 1637.75 cm^{-1}. Tertiary amine group (C-N) frequency has also vanished from 1274.53 cm^{-1}. These group frequency changes for the functional groups of organic molecules of BFP extract confirm the adsorption on the metal surface, which is responsible for preventing corrosion (Kawser, et al., 2000)



______ FT-IR spectra of BF extract.

--------- FT-IR spectra of corrosion product of BF extract on MS surface.

Figure 2: FT-IR spectrum of the corrosion product of mild steel in the presence of BFP extract as an inhibitor in 0.5 M H_2SO_4.

SEM

Surface examination using SEM has carried to investigate the effect of inhibitor on the surface morphology of the mild steel. The surface damage has diminished in the presence of inhibitor. It is revealed that the metal surface was highly covered with the protective layer formed by BFP extract which prevents the metal from further attack of acid media thus inhibiting corrosion (Pandian Bothi Raja, 2009).

Mechanism of corrosion inhibition

The inhibitor molecules inhibit the corrosion of mild steel by adsorption on the mild steel-solution surface. The mechanism of corrosion inhibition of mild steel by the phytochemical compounds present in the extract of BFP can be attributed to substitutional adsorption involving Fe in mild steel and the inhibitor (inh) as follows,

$$Fe + Inh \leftrightarrow Fe^{-}(Inh)_{ads} \qquad (6)$$

The adsorption of the inhibitor molecules on the mild steel surface is due to the lone pairs of electrons on O atom as well as an electron donating group such as $-CH_3$ and $-OCH_3$ groups, on the phenyl ring which may cause an increase in the π-electron density and as a consequence the surface coverage is expected to increase. This indicates that the inhibition efficiency of the extract is due to synergistic effect of the phytochemical constituents present in the BFP extract (Okafor et al., 2007).

Conclusion

From the experimental results obtained in the present study, the following conclusions could be drawn:

- BFP extract inhibits mild steel corrosion in 0.5 M H_2SO_4 medium.
- In 0.5 M H_2SO_4 medium, the inhibition efficiency of BFP increases with increasing concentration and temperature.
- Thermodynamic data for adsorption of BFP extract on mild steel suggest the spontaneous adsorption of the studied extract and the occurrence of comprehensive adsorption (physical and chemical adsorption) for the inhibitor species on mild steel from 0.5 M H_2SO_4 medium.
- The adsorption of BFP extract on the mild steel surface in 0.5 M H_2SO_4 obeys the El-Awady kinetic thermodynamic and Frumkin adsorption isotherm model.
- BFP extract acts as a mixed type inhibitor for the corrosion of mild steel in 0.5 M H_2SO_4 without modifying the mechanism of hydrogen evolution reaction and metal dissolution.
- Surface analytical techniques – FT-IR and SEM confirmed the adsorption of BFP on mild steel surface.

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