



ISSN: 0975-833X

RESEARCH ARTICLE

SODIUM LIGNOSULPHONATE (SLS) AS CORROSION INHIBITOR FOR MILD STEEL IN SULFURIC ACID MEDIUM

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ARTICLE INFO

Article History:

Received 10th May, 2014
Received in revised form
28th June, 2014
Accepted 07th July, 2014
Published online 06th August, 2014

Key words:

Sodiumlignosulphonate,
Corrosion inhibitor, Mild steel,
Sulfuric acid.

ABSTRACT

The corrosion and inhibition behaviour of mild steel in 0.5M H₂SO₄ in the presence of Sodiumlignosulphonate (SLS) has been investigated. The inhibitor effect of this naturally occurring SLS on the corrosion of mild steel in 0.5M H₂SO₄ was studied by weight loss, potentiodynamic polarization and electrochemical impedance spectroscopic methods. The inhibition efficiency of SLS on corrosion of mild steel in 0.5M H₂SO₄ is very good. The result of the different techniques confirmed the adsorption of SLS on the mild steel surface as the cause for the inhibition effect. Maximum inhibition efficiency of 90.9% at 18% (v/v) of SLS suggests that the presence of SLS in solution enhances the surface coverage. Based on the experimental evidences a mechanism is proposed to explain the inhibitory action of SLS.

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INTRODUCTION

Mild steel has found wide applications in a broad spectrum of industries and machinery. In the industrial atmosphere it is much affected by corrosion process. To overcome this problem, the efforts are deployed to stop or delay the maximum attack of metals in various corrosive media. The isolation of a metal from corrosive agents is the most effective way to prevent electrochemical corrosion. Among the different control methods available (Blustein et al., 2005; Amin et al., 2010) the use of corrosion inhibitors is usually the most appropriate way to achieve this objective. Most corrosion inhibitors are synthetic organic compounds having hetero atoms in their aromatic or long carbon chain (Nataraja et al., 2011). Most of the well-known acid inhibitors are organic compounds containing nitrogen, sulphur, and oxygen atoms. Compounds with π -electrons and functional groups containing hetero atoms which can donate lone pair of electrons are found to be particularly useful as inhibitors for corrosion of metals (Caliskan et al., 2011; Rafiquee et al., 2008; Popova et al., 2004). These organic compounds can adsorb on the metal surface, block the active sites on the surface and thereby reduce the corrosion rate. Structural effect of organic

compounds as organic inhibitors was also studied (Popova et al., 2004; Fouda et al., 2005; Kobayashi, et al., 1993; Skryler et al., 1991; Fouda et al., 1986). The results are in agreement with the fact that the inhibition efficiency of organic compounds depends on charge density, mode of interaction with the metal surface and adsorption on mild steel surface.

However, the toxic effects of most synthetic corrosion inhibitors, the obligations of health and human security have led to the search of green alternatives as the corrosion inhibitors that are environmentally friendly and harmless (Trabanelli et al., 1991). These green inhibitors are obtained as extract of common plants. These materials are preferred because of the excellent bio-degradability, eco-friendliness, low cost, easy availability from renewable sources of materials. Compounds showing good inhibition efficiency with low environmental risk among the so-called "green corrosion inhibitors" are mainly organic molecules that act by adsorption on the metal surface. Some examples are ascorbic acid (Ferreira et al., 2004; Gonclaves et al., 2001) succinic acid (Giacomelli et al., 2004; Amin et al., 2007) acidtryptamine (Moretti et al. (2004) caffeine Fallavena et al. (2006) and other extracts of natural substances (Bouyanzer et al., 2006; Raja et al., 2008; Rahim et al., 2008; Chauhan et al., 2007; Abdel-Gaber et al., 2006).

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The green inhibitors are significant that they do not contain any heavy metal in their composition (Al-Luaibi Salah *et al.*, 2011). Several authors also reported that the use of natural products as the potential corrosion inhibitors for various metals and alloys under different environments (Chauhan *et al.*, 2007; Olusegun *et al.*, 2009; Quraishi *et al.*, 2010; Okafor *et al.*, 2008; Abdel-Gaber *et al.*, 2008; . Emeka *et al.*, 2006; Abdel-Gaber *et al.*, 2006; El-Etre *et al.*, 2005; Ramananda Singh *et al.*, 2012; Ramananda Singh *et al.*, 2011). The present work is aimed to study the inhibitive and adsorption properties of SLS for the corrosion of mild steel in sulfuric acid.

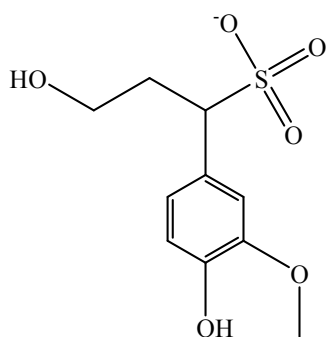


Figure 1. Typical structure of lignosulfonate

MATERIALS AND METHODS

Weight loss measurements

The aggressive solution (0.5M H₂SO₄) was prepared by dilution of analytical Grade 98% H₂SO₄ with double-distilled water. Prior to all measurements, the mild steel samples (0.09% P; 0.38% Si; 0.01% Al; 0.05% Mn; 0.21% C; 0.05% S and the remainder iron) were polished with different emery paper up to 1200 grade, washed thoroughly with double-distilled water, degreased with AR grade ethanol, acetone and drying at room temperature. The solution volume was 100 mL with and without the addition of different concentrations of SLS. The mild steel specimens used had a rectangular form (1 cm x 1 cm). The immersion time for the weight loss was 1 hr at 30 °C. After the corrosion experiments, the specimens of steel were carefully washed in double-distilled water, dried and then weighed. The rinse removed the loose segments of the film of the corroded samples. Duplicate experiments were performed in each case and the mean value of the weight loss is reported. Weight loss allowed us to calculate the mean corrosion rate as expressed in mpy.

The corrosion rate and inhibition efficiency IE(%) were calculated according to the Eqs. (1) and (2) respectively.

$$\text{Corrosion rate (mpy)} = 534 W/\rho A t \quad \dots\dots\dots(1)$$

Where *W* is the weight loss (mg), ρ is the density of the specimen (gcm⁻³), *A* is the area of the specimen (cm²), and *t* is the exposure time (h).

$$\text{IE\%} = \frac{\text{CR}^{\circ} - \text{CR}}{\text{CR}^{\circ}} \times 100 \quad \dots\dots\dots(2)$$

CR^o- Average weight loss without inhibitor, CR- Average weight loss with inhibitor

Preparation of SLS

SLS is a green inhibitor and normally obtained from the pulp of eucalyptus wood. In the present work we purchased the SLS from Laxness India private limited. It is obtained as powder form and soluble in water. It is anticipated that SLS may be used as a multifunctional water treatment agent through chemical modification based on its structural analysis. Infrared spectrum of SLS was taken in KBr pellet form in the region 500- 4000 cm⁻¹. The characteristic IR absorption bands of SLS are as follows: a broad band at 3452 cm⁻¹ is attributed to stretching vibrations of O-H groups; at 1629 cm⁻¹ is for stretching vibration of C=O groups, the band at 1385 cm⁻¹ is due to (-CH₃) linkage group, the band at 1030 cm⁻¹ corresponds to plane bending vibrations of O-H groups.

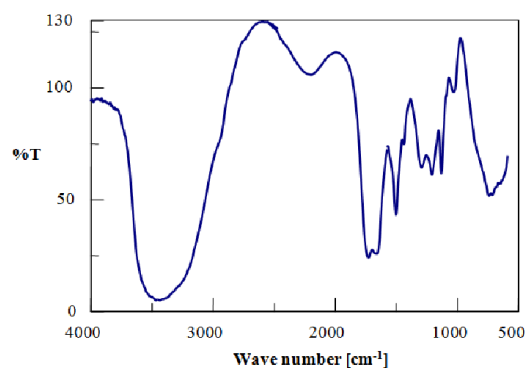


Figure 2. Infrared spectrum of SLS

RESULTS

The results obtained from the conventional weight loss method at different concentrations of the inhibitor and at different temperatures is given in (Table 1, Figure 3). The results indicate that i) but with the increasing concentration of the inhibitor the inhibition efficiency increases regularly at 303 K. ii) at the same concentration of the inhibitor the inhibition efficiency decreases with rise in temperature iii) in the case of higher temperatures (313 K, 323 K, 333 K) there is a slight increase in inhibition efficiency initially with rising concentration of the inhibitor and later stage there is a decreasing trend. It indicates desorption of the inhibitor predominates rather than adsorption and so there is decreasing inhibition efficiency at elevated temperatures.

Effect of surface coverage (θ)

In the present investigation the values of θ , the degree of surface coverage is calculated at different concentrations by using the equation (3).

$$\theta = (\text{CR}^{\circ} - \text{CR}) / \text{CR}^{\circ} \quad \dots\dots\dots(3)$$

θ – degree of surface coverage, CR^o-average weight loss without inhibitor, CR- average weight loss with inhibitor.

Table 1. Variation of inhibition efficiency and surface coverage (θ) with different concentration of the inhibitor and at various temperatures

Inhibitor Concentration (V/V)	Inhibition efficiency (%)				Surface coverage (θ)				C/ θ			
	303K	313K	323K	333K	303K	313K	323K	333K	303K	313K	323K	333K
2	50.00	47.90	45.20	39.00	0.50	0.47	0.45	0.39	4.00	4.25	4.44	5.12
6	58.00	49.80	47.10	41.70	0.58	0.49	0.47	0.41	10.34	12.24	12.76	14.63
10	66.70	48.10	46.00	40.00	0.66	0.48	0.46	0.40	15.15	20.83	21.73	25.00
14	80.00	45.10	43.70	38.70	0.80	0.45	0.43	0.38	17.50	32.11	32.55	36.80
18	90.00	42.00	40.90	33.00	0.90	0.42	0.40	0.33	20.00	42.85	45.00	54.54

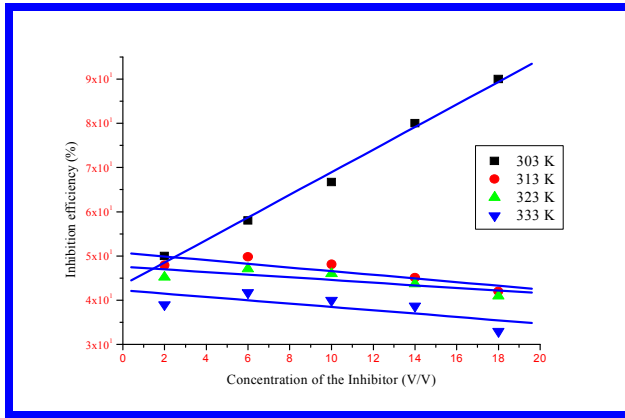


Figure 3. Variation of Inhibition efficiency (%) with various concentrations of inhibitor at different temperatures 303K, 313K, 323K and 333K

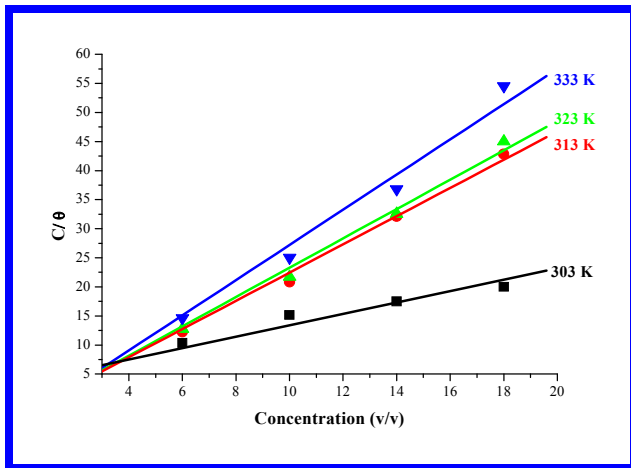


Figure 4. Variation of C/θ vs. C at different temperatures

At lower temperature the surface coverage increases with rise in concentration of the inhibitor due to adsorption of the inhibitor on the metal surface. Nevertheless, at higher temperature the surface coverage decreases with reference to a particular concentration. To understand the nature of adsorption of the inhibitor on the metal surface, several adsorption isotherms are in handy to fit the experimental data.

Here the plot of C/θ vs. C (Langmuir adsorption isotherm) at different temperatures (303, 313, 323, 333K) is given in Figure 4. The linear relationship indicates the present system obeys

Langmuir adsorption isotherm which implies that the adsorption of the inhibitor results monolayer adsorption on the surface of the mild steel.

Thermodynamic parameters

Thermodynamic parameters like ΔE, ΔH, ΔG and ΔS are calculated by using the following equations. These values are useful to understand the corrosion mechanism.

$$\ln k = -\Delta E/RT + \ln A \tag{4}$$

A-Pre-exponential factor, k-corrosion rate, R-gas constant, ΔE-activation energy. The plot of ln k vs. 1/T gives a straight line and from the slope of the straight line ΔE is obtained. We observed that for the increasing concentration of inhibitor (2, 6, 10, 14, 18 % (v/v)) activation energy (ΔE) increases (15, 24.9, 33.4, 43.3, 55.8 J/mol) at 303K. This result shows that there is strong inhibitive action on the mild steel surface with rise in concentration of the inhibitor.

The heat of adsorption (ΔH_{ads}) could be calculated from equation (5).

$$\Delta H_{ads} = 2.303R [(\log \theta_2 / (1-\theta_2)) - (\log \theta_1 / (1-\theta_1))] T_2 T_1 / (T_2 - T_1) \tag{5}$$

Where θ₁, θ₂ are the degree of surface coverage at any two temperatures T₁ and T₂. The heat of adsorption (ΔH_{ads}) can be approximately regarded as the standard heat of adsorption (ΔH°) equation (5).

The equilibrium constant K is given by the equation (6).

$$K = \theta / C(1 - \theta) \tag{6}$$

θ – Degree of surface coverage, C- concentration of the inhibitor The standard adsorption free energy (ΔG°) was obtained according to equation (7). The equilibrium constant K can be related to the standard free energy of adsorption (ΔG°) (equation 7). Here K is equilibrium constant for the adsorption process, 55.5 is the concentration of water mol/L, R is gas constant and T is the absolute temperature.

$$\Delta G_{ads} = -RT \ln(55.5K) \tag{7}$$

$$\ln K = -\ln 55.5 - (\Delta G^\circ/RT) \tag{8}$$

The standard entropy of adsorption (ΔS°) can be obtained by using the equation (9)

$$\Delta S^0 = \frac{\Delta H^0 - \Delta G^0}{T} \dots\dots\dots(9)$$

The values of ΔH₀, ΔG₀ and ΔS₀ obtained by using the above equations for the present system is given in Table 2. These thermodynamic quantities represent the algebraic sum of the values for adsorption and desorption. The negative value of ΔG₀ indicates the spontaneous adsorption of inhibitor on the surface of the mild steel. It is generally accepted that low values of ΔG₀ (< 40 KJ mol⁻¹) indicate that the mode of adsorption is physisorption where the interaction between the charged metal surface and organic molecules is of electrostatic in nature. The negative values of ΔH₀ also show that the adsorption of an inhibitor is an exothermic process. The positive values of ΔS₀ mean that the process of adsorption is accompanied by an increase in entropy. This small values of entropy is indicative of the fact that the adsorbed molecules are not chaotic and there is approach of uniform orientation on the metal surface.

Table 2. The thermodynamic parameters of adsorption of SLS on the mild steel surface at various temperatures

Temp. K	ΔG° (KJ/mol)	ΔH° (KJ/mol)	ΔS° (KJ/mol. K)
303	-2918	-4.903	9.61
313	-2905	-4.922	9.29
323	-2896	-2.466	8.97
333	-2893	-13.265	8.64

Electrochemical Measurements

Potentiodynamic Polarization

The electrochemical experiments were carried out using a corrosion cell Ag/AgCl electrode (saturated KCl) as reference electrode, Pt wire as counter electrode and mild steel coupons as working electrode. All the measurements were carried out at room temperature (30 ± 1 oC). Before starting the measurements the specimen was left to attain a steady state indicated by a constant potential.

The inhibition efficiency was calculated using the relationship [10]

$$IE\% = \frac{I_{corr} - I_{corr}(inhibitor)}{I_{corr}} \times 100 \dots\dots(10)$$

The values of corrosion current density (I_{corr}), corrosion potential (E_{corr}), the cathodic Tafel slope (β_c), anodic Tafel slope (β_a), and the IE as functions of surfactant concentration, were calculated from the polarization curves and the data is given in Table 3. The values of IE% are obtained using equation [10] where I_{corr} and I_{corr(inh)} are the uninhibited and inhibited corrosion current density values, respectively, determined by extrapolation of Tafel lines to the corrosion potential. It is clear from the data given in Table 3 that the presence of inhibitor has resulted a marked decrease in corrosion current value (I_{corr}) from 7.69 mA/cm² of the blank to 4.70 mA/cm² by the addition of 2%(v/v) of the inhibitor and further decreased regularly with increasing concentration of

inhibitor and it reaches 0.69 mA/cm² at 18% (v/v) of the inhibitor.

Table 3. Potentiodynamic polarization parameters in different concentrations of SLS at 303 K for the corrosion of mildsteel in H₂SO₄ medium

C % (v/v)	E _{corr} (mv/SCE)	I _{corr} (10 ⁻³ mA/cm ²)	β _c (mv/dec)	β _a (mv/dec)	IE %
0	-742	7.699	5.121	7.882	
2	-734	4.700	6.033	7.286	38.9
6	-725	4.170	6.422	7.198	45.8
10	-756	4.070	6.284	7.272	47.1
14	-743	2.190	6.358	7.141	71.5
18	-763	0.690	2.862	5.989	90.9

These results indicate its ability to inhibit the corrosion of mildsteel in 0.5M H₂SO₄ solution. Both the anodic and cathodic current densities were decreased, indicates that the inhibitor protected through adsorption on the mildsteel surface. The anodic Tafel slopes of the present system without the inhibitor is 7.882. In the presence of the inhibitor the values are in the range 7.286 – 5.989mV/dec. This change clearly demonstrates the role of the inhibitor in the anodic dissolution of the metal (corrosion). In the same way the cathodic Tafel slopes are in the range 6.033-2.862 mV/dec. This result shows the influence of the inhibitor in the cathodic hydrogen evolution process. Hence the Tafel polarization study reveals that the inhibitor acts as mixed type (anodic- cathodic) inhibitor. However, there is no remarkable change in (E_{corr}) values and such inhibitive action is due to the adsorption of the inhibitor on the mildsteel surface.

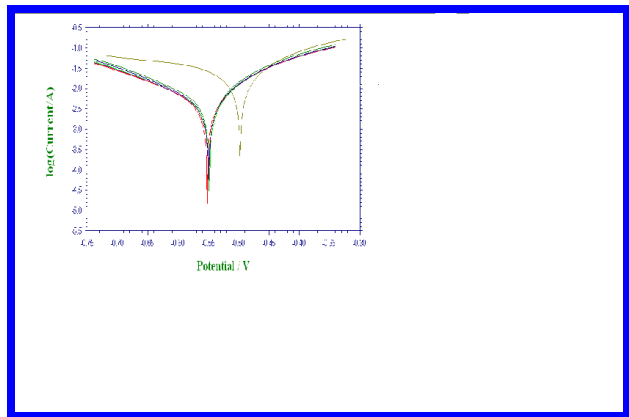


Figure 5. Polarization curves of mild steel in 0.5 M H₂SO₄ with and without SLS at various concentration

Electrochemical Impedance Spectroscopy

Electrochemical impedance spectroscopy (EIS) study was carried out in a frequency range of 100KHz to 10 MHz with amplitude of 10 mV using AC signals at open circuit potential (OCP). The charge transfer resistance (R_{ct}) values have been obtained from the diameter of semicircles of Nyquist plots. Figure 6 shows the Nyquist plots (curves obtained by the plot of real vs imaginary components of impedance as a function of the impressed frequency) of a-c impedance measurements of

mildsteel in 0.5 M H₂SO₄ in the presence of different concentrations of the inhibitor (SLS). Normally Nyquist plots are obtained as semicircles indicating the corrosion of mildsteel is an activating controlled single charge-transfer process. However the deviations of the semicircle indicate frequency dispersion of interfacial impedance due to the roughness and other inhomogeneity of the surface.

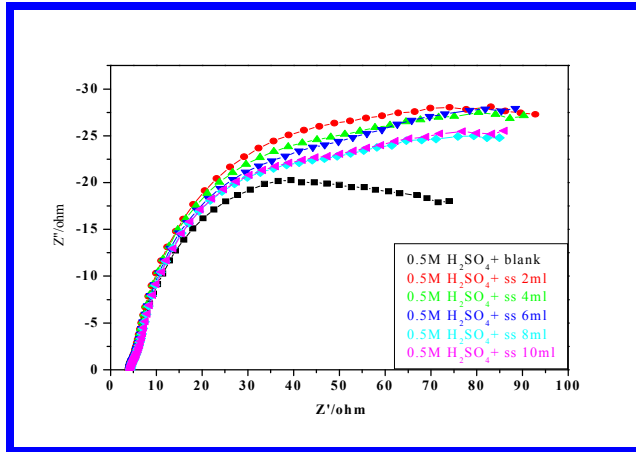


Figure 6. Nyquist plots for mild steel immersed in 0.5 M H₂SO₄ solution without and with inhibitor at various concentrations

The inhibition efficiency has been calculated by using the equation (12).

$$C_{dl} = \frac{1}{2 \times 3.14 \times f_{max} \times R_{ct}} \quad \dots\dots\dots(11)$$

C_{dl} – double layer capacitance, f_{max} – frequency maximum, R_{ct} - charge transfer resistance

$$IE \% = \frac{R_{ct} - R_{ct}^0}{R_{ct}} \times 100 \quad \dots\dots\dots(12)$$

Table 4. Electrochemical impedance parameters at different concentrations of SLS

conc. % (v/v)	R_{ct} $\Omega \text{ cm}^2$	C_{dl} $\mu\text{F cm}^{-2}$ 10^{-6}	IE %
0	2.50	5.68	0
2	4.77	3.31	38.87
6	5.32	2.98	45.1
10	5.52	2.88	47.06
14	10.31	1.55	71.66
18	29.96	0.05	90.25

R_{ct} - charge transfer resistance with inhibitor, R_{ct}^0 - charge transfer resistance without inhibitor Inspection of results from Table 4, that with increase in concentrations of the inhibitor the R_{ct} increased and C_{dl} value decreased. Increase in the value of charge transfer resistance (R_{ct}) is indicative of the fact that the inhibitor has the corrosion protection effect. Actually the R_{ct} values increased from 2.50 $\Omega \text{ cm}^2$ to 29.96 $\Omega \text{ cm}^2$ at the highest concentration of the inhibitor 18% (v/v) which is attributed to the formation of and steady increase in the

compactness of the adsorbed layer of the inhibitor molecules. On the other hand the decrease in the C_{dl} values show that the role of inhibitor molecules in corrosion protection is due to the adsorption of the metal solution. The reasonable decrease in the values C_{dl} have inferred that the roughness on the metal surface has decreased owing to the adsorption of the inhibitor molecules. The highest inhibition efficiency of 90.25% is achieved at the concentration of the inhibitor (18% v/v). This value validates the value (IE= 90.9%) as obtained by the potentiodynamic polarization and weight loss method. Hence SLS is acting as a good inhibitor in the corrosion of mild steel in H₂SO₄ medium.

Mechanism

Lignosulfonate contains both hydrophilic groups (sulfonic, phenylic, hydroxyl and alcoholic) and hydrophobic groups (carbon chain), as shown in Figure 1. It is an anionic surfactant, possessing a certain degree of surface activity, which may promote surface adsorption and further particle dispersion, form a thin film on the metal, and hence have a potential application in corrosion and scale inhibition.

Conclusion

- 1.SLS has been found to be a good corrosion inhibitor for mild steel in 0.5M H₂SO₄ medium.
- 2.The Inhibition efficiency (%) and corrosion resistance of the mild steel increased with inhibitor concentrations.
- 3.The Tafel polarization curves indicate that it is a mixed type inhibitor.
- 4.Electrochemical impedance study corroborates the Tafel polarization and weight loss measurements in the excellent behavior of inhibitor in corrosion protection of mildsteel.

Acknowledgement

The corresponding author thanks to the Management, Principal and Head of the Department of Chemistry, Tiruvedakam West for research facilities.

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