

# Inhibitive Action of Calcium Lignosulfonate on the Corrosion of Mild Steel in Sulfuric Acid Medium

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**Abstract-** The functioning of calcium lignosulfonate (CLS) as the potential inhibitor in the corrosion of mild steel in 0.5 M H<sub>2</sub>SO<sub>4</sub> medium has been studied. The various parameters in connection with the corrosion behaviour of the metal and the inhibitive action of CLS were obtained by weight loss, potentiodynamic polarisation, electrochemical impedance spectroscopy (EIS) and scanning electron microscopy (SEM) methods. The effect of temperature on the mild steel surface has been reported. The results of the present investigation show that CLS acts as a good inhibitor and it is of mixed type. The mode of action is by the process of adsorption at anodic and cathodic sites. The scanning electron microscopic studies gives the picture of surface morphology of mild steel in the presence and absence of inhibitor.

**Index Terms-** Calcium Lignosulfonate, Corrosion inhibitor, Mild steel, Sulfuric acid, Langmuir adsorption isotherm.

to their multiple active sites adsorb more strongly on the metal surface compared with their monomer analogues. Therefore, it is expected that the polymers will be the better corrosion inhibitors [3-13]. In the case of polymers by increasing the hydrocarbon chain length, solubility decreases. Thus, the presence of hydrophilic functional groups which increase the solubility are required. Thus efforts are now directed towards the formulation of modern, environmentally safe inhibitors. These inhibitors are chosen because they are eco-friendly, economical, readily available and obtained from renewable sources. The green inhibitors are more significant in the sense that they do not contain any heavy metal in their composition [14-17]. Several authors also reported the use of natural products as the potential corrosion inhibitors for various metals and alloys under different environments [18-31]. In the present investigation we used calcium lignosulfonate as the inhibitor which is a green inhibitor with potential inhibitive property and eco friendly.

## I. INTRODUCTION

Materials of iron especially mild steel, are largely used in acidic media in most industries including oil/gas exploration and ancillary activities. During such activities, hydrochloric acid is widely used in pickling, descaling and stimulation of oil wells in order to increase oil and gas flow. Normally inhibitors are used to control corrosion in such a corrosion conditions the protection of metal from corrosion phenomena is of paramount importance and some have been found to be hazardous to health and the environment at large [1]. The unique advantage of the possibility of adding inhibitors is that this can be done without disruption of the industrial process. Specific chemical compounds are often used as inhibitors in these processes mainly to control the metal dissolution reaction and thereby increasing the service life of steel materials. The inhibition efficiency has been described as primarily due to their adsorption at corroding metal surfaces. A protective film formed due to adsorption of these inhibitor molecules restricts either the movement of ions away from the corroding surface or the consumption of electrons.

Organic corrosion inhibitors are useful when their addition in small amounts prevents corrosion. When higher concentrations of organic compounds are added additional testing for environmental impact is required. Compounds containing nitrogen, sulphur and oxygen are being used as inhibitors [2]. The most efficient inhibitors are organic compounds having pi bonds in their structures. However polymeric compounds owing

## II. EXPERIMENTAL

The aggressive solution (0.5M H<sub>2</sub>SO<sub>4</sub>) was prepared by dilution of analytical Grade 98% H<sub>2</sub>SO<sub>4</sub> 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 CLS. The mild steel specimens used had a rectangular form (1 cm x 1 cm). The immersion time for the weight loss was 1 h 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.

### 2.1 Weight loss Studies

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

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

Where W is the weight loss (mg),  $\rho$  is the density of the specimen (cm<sup>3</sup>), A is the area of the specimen (cm<sup>2</sup>), and t is the exposure time (h).

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

CR<sup>o</sup>- Average weight loss without inhibitor, CR- Average weight loss with inhibitor

## 2.2 Preparation of the CLS

Calcium lignosulfonate is an amorphous material obtained from the sulfite pulping of softwood. The lignin framework is a sulfonated random polymer of three aromatic alcohols: coniferylalcohol, *p*-coumaryl alcohol, and sinapyl alcohol, of which coniferylalcohol is the principal unit. After completion of the pulping, the water-soluble calcium lignosulfonate is separated from the cellulose, purified (ultrafiltration), and acidified. The recovered material is evaporated and spray dried. The commercial product has a weight average molecular weight range of 40,000 to 65,000. It is purchased from Lanxess india private limited. It is obtained as a powder form and it is soluble in water.

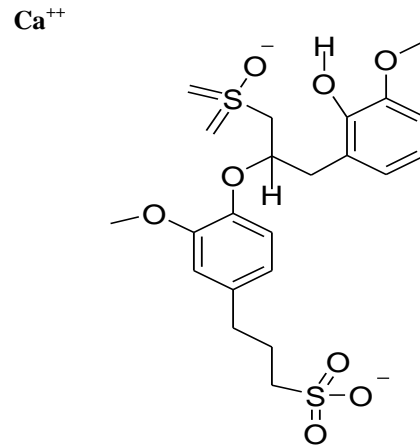


Figure 1 Molecular structure of Calcium Lignosulfonate

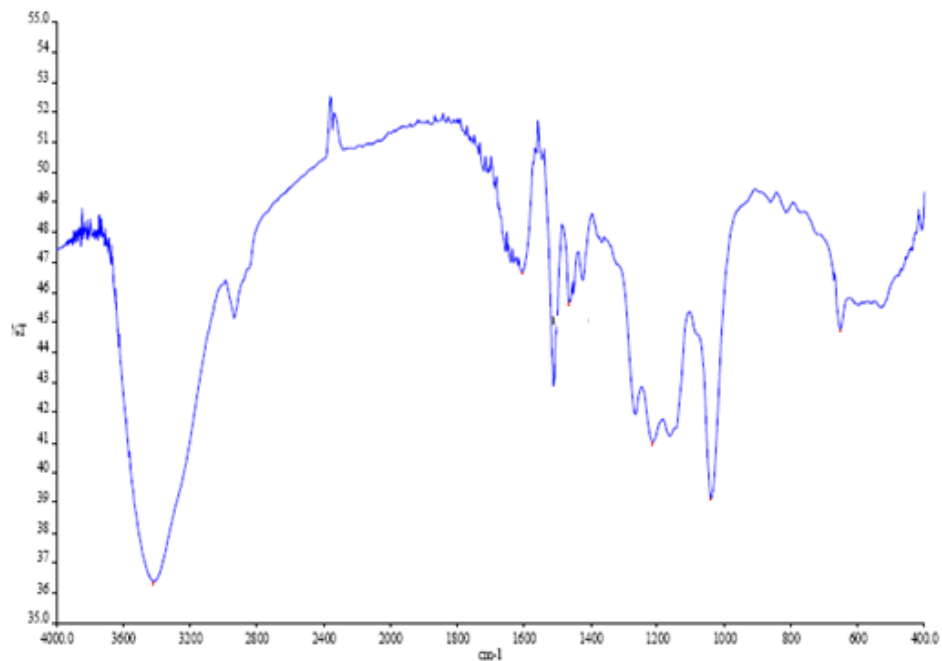


Figure 2 IR Spectrum of Calcium Lignosulfonate

Infrared spectrum of SLS was taken in KBr pellet form in the region 400- 4000 cm<sup>-1</sup>. The characteristic IR absorption bands of SLS are as follows: a broad band at 3423 cm<sup>-1</sup> is attributed to stretching vibrations of O-H groups, the band at 1043 cm<sup>-1</sup> is due to ether linkage group, the band at 1385 cm<sup>-1</sup> is

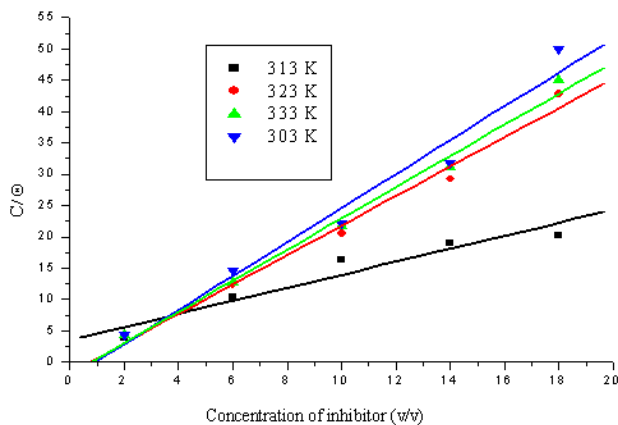
due to (- CH<sub>3</sub>) linkage group, the band at 1214 cm<sup>-1</sup> corresponds to plane bending vibrations of O-H groups.

## III. WEIGHT LOSS METHOD

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 ( $\theta$ )				C/ $\theta$			
	303K	313K	323K	333K	303K	313K	323K	333K	303K	313K	323K	333K
2	52.3	48.2	47.0	46.0	0.52	0.48	0.47	0.46	3.80	4.20	4.30	4.34
6	58	49.8	47.1	41.7	0.58	0.49	0.47	0.41	10.34	12.24	12.76	14.63
10	61.6	49.1	46.1	45.0	0.61	0.49	0.46	0.45	16.4	20.50	21.7	22.2
14	73.8	48.3	45.2	44.0	0.72	0.48	0.45	0.44	18.97	29.20	31.1	31.8
18	88.4	42	40.9	36	0.88	0.42	0.40	0.36	20.3	42.85	45	50.00

The data obtained from the conventional weight loss method is given in **Table 1**. It is inferred from the data that with the increasing concentration of the inhibitor the inhibition efficiency increases regularly at 303K. However at higher temperatures (313, 323, 333K) inhibition efficiency increases initially with increasing concentration of inhibitor and at later stage there is a decreasing trend (**Table 1, Figure 3**). This result shows that at higher temperatures the phenomenon of desorption predominates rather than adsorption and so there is decreasing inhibition efficiency.



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

#### IV. ADSORPTION ISOTHERM

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

$$\theta = (CR^0 - CR) / CR^0 \quad (3)$$

$\theta$  – degree of surface coverage,  $CR^0$ -average weight loss without inhibitor,  $CR$ - average weight loss with inhibitor.

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  $C/\theta$  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.

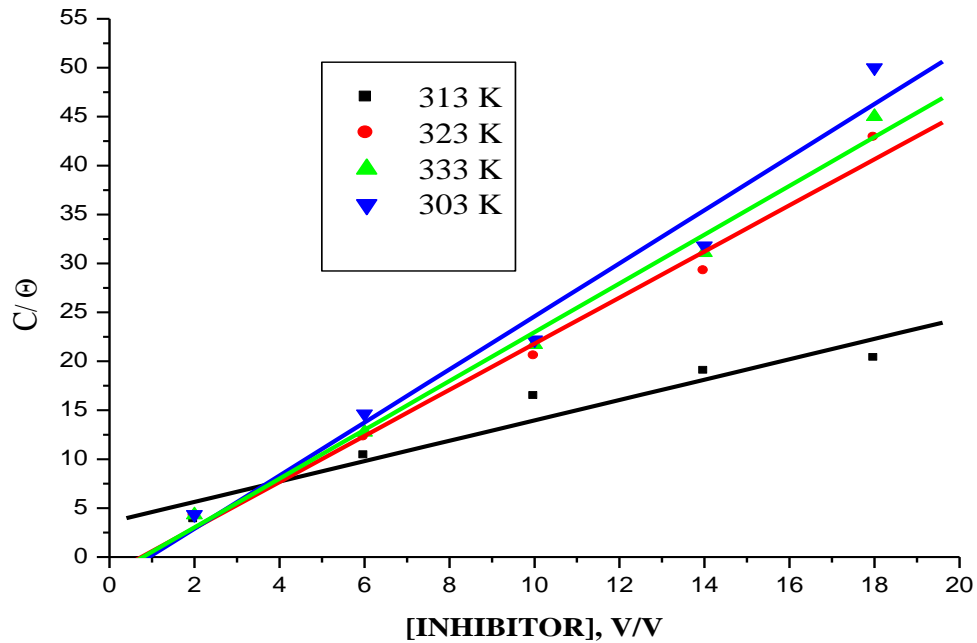


Figure 4 Variation of  $C/\theta$  vs.  $C$  at different temperatures

#### V. ELECTROCHEMICAL STUDIES

Table 2 Potentiodynamic parameters in different concentrations of CLS at 303 K

$C$	$E_{corr}$	$I_{corr}$	$\beta_c$	$\beta_a$	IE
% (v/v)	(mv/SCE)	( $10^{-3}$ mA/cm <sup>2</sup> )	(mv/dec)	(mv/dec)	%
0	-740	7.78	5.73	6.54	
2	-734	3.87	5.93	7.77	50.25
6	-723	3.04	6.43	7.48	60.92
10	-740	1.36	6.91	8.08	82.51
14	-756	1.10	7.23	8.59	85.86
18	-763	0.89	7.28	8.81	88.56

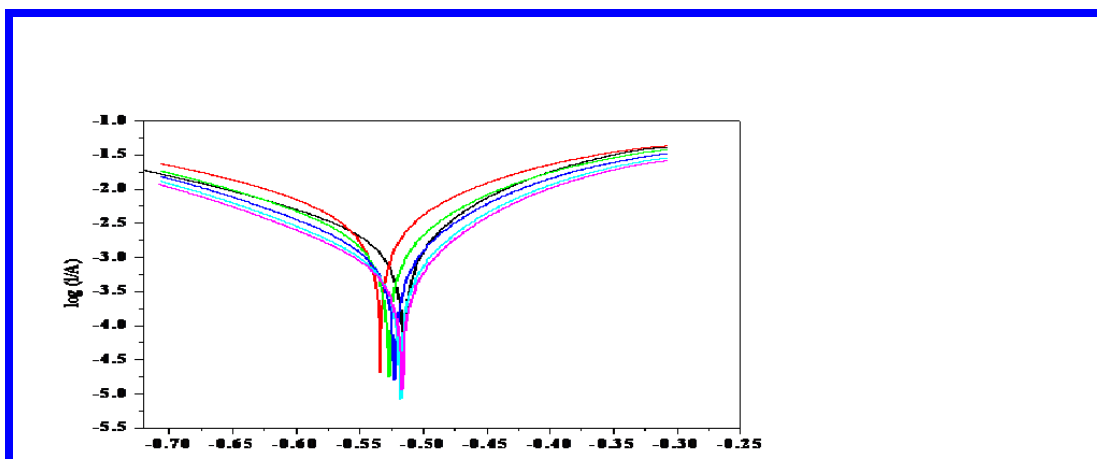
Electrochemical measurements are performed in a conventional three electrode cell consisting of a mild steel working electrode (WE), platinum counter electrode (CE) and silver as the reference electrode. By using emery paper, these electrodes are degreased and dried at room temperature before used. The area of the working electrode is 1 cm<sup>2</sup>. The current density (I<sub>corr</sub>) curves obtained by extrapolation of the Tafel lines.

Potentiodynamic polarization measurements were carried out for this system and the details are given **Table 2**. The kinetic parameters obtained by this method **Figure 5** such as E<sub>corr</sub>, I<sub>corr</sub>, anodic and cathodic Tafel slopes (β<sub>a</sub> & β<sub>c</sub>) are given in **Table 2**. It is observed that with addition of increasing concentration of inhibitor the corrosion potential (E<sub>corr</sub>) shifts slightly towards the cathodic direction. For the blank, E<sub>corr</sub> is -740mV and at higher concentration of inhibitor 18 % (v/v) its value is -763 mV. These values indicate that there is a dismal effect on corrosion potential with increase in the concentration of the inhibitor. However there

is a progressive change in the values of anodic and cathodic Tafel slopes. This clearly shows that the mechanism of anodic dissolution of metal and cathodic hydrogen evolution steps are affected by the inhibitor. This shows that the inhibition is due to the adsorption of the inhibitor on the mild steel surface. The values of β<sub>c</sub> and β<sub>a</sub> show increasing trend indicating the inhibitive action is of mixed type. From the Potentiodynamic polarization measurements the inhibition efficiency is attained using the equation.

$$IE\% = \frac{I_{corr} - I_{corr}(\text{inhibitor})}{I_{corr}} \times 100 \quad (4)$$

Where I<sub>corr</sub> and I<sub>corr (inhibitor)</sub> are the values of corrosion current densities of mild steel without and with the inhibitor respectively. The inhibition efficiency increases with the increase in concentration of the inhibitor.



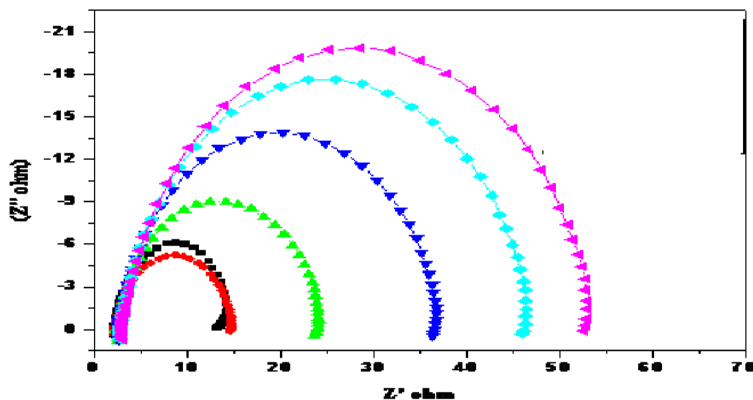
**Figure 5** Polarization curves of mild steel in 0.5 M H<sub>2</sub>SO<sub>4</sub> with and without CLS at various concentration.

**Table 3** Electrochemical impedance parameters at different concentrations of inhibitor

conc. % (v/v)	R <sub>ct</sub> Ω cm <sup>2</sup>	C <sub>dl</sub> , μF cm <sup>-2</sup> 10 <sup>-7</sup>	IE %
0	2.92	5.45	
2	4.77	3.33	38.87
6	5.32	2.98	45.16
10	5.52	2.88	47.09
14	10.31	1.54	71.66
18	24.90	0.53	88.27

Electrochemical impedance spectroscopy (EIS) studies were 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 Nyquist plots for mild steel immersed in 0.5 M H<sub>2</sub>SO<sub>4</sub> solution without and with inhibitor at various concentrations**

The effect of the inhibitor concentration on the impedance behaviour of mild steel in 0.5 M H<sub>2</sub>SO<sub>4</sub> has been determined and the Nyquist plots are given in **Figure 6**. The impedance spectra show a single semicircle and as the concentration of inhibitor increases diameter of the semicircle increases. The results indicate that the  $R_{ct}$  significantly increases with increase in concentration of inhibitor and  $C_{dl}$  tends to decrease. This decrease in  $C_{dl}$  may be due to decrease in local dielectric constant and/or an increase in the thickness of protective layer at electrode surface which enhances the corrosion resistance of the mild steel. The increase in  $R_{ct}$  values is attributed to the formation of protective film at the metal-solution interface.

The double layer capacitance ( $C_{dl}$ ) and the inhibition efficiency has been calculated by using the equations (5) and (6).

$$C_{dl} = \frac{1}{2 \times 3.14 \times f_{max} \times R_{ct}} \quad (5)$$

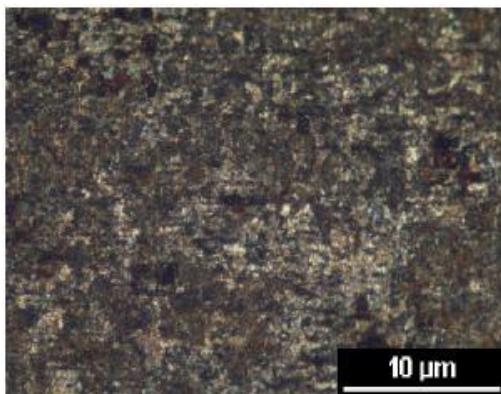
$C_{dl}$  – double layer capacitance,  $f_{max}$ - frequency maximum,  $R_{ct}$ - charge transfer resistance

$$IE \% = \frac{R_{ct} - R_{ct}^0}{R_{ct}} \times 100 \quad (6)$$

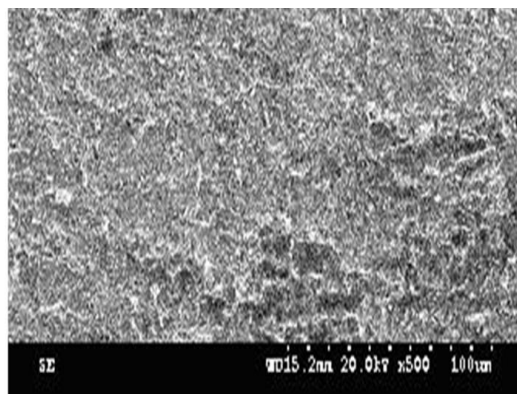
$R_{ct}$  - charge transfer resistance with inhibitor,  $R_{ct}^0$  - charge transfer resistance without inhibitor

#### VI. SCANNING ELECTRON MICROSCOPY (SEM)

The mild steel specimen immersed in 0.5M H<sub>2</sub>SO<sub>4</sub> for 3 hrs was taken out and washed with distilled water, dried and taken SEM micrograph. Similarly the mild steel specimen immersed in resin solution containing acid for 3 hrs was taken out, rinsed with double distilled water, dried and subjected to surface examination by scanning electron microscopy (SEM) using HITACHI S-3000H instrument.



**Figure 7 SEM micrograph without inhibitor**



**Figure 8 SEM micrograph with inhibitor**

Scanning Electron Microscopy (SEM) is a wonderful technique to examine the surface features with better understanding. In the present work the SEM micrograph of mild steel in 0.5 M H<sub>2</sub>SO<sub>4</sub> solution in the absence and presence of inhibitor after 3hours exposure is given in **Figure 7 and Figure 8**.

The SEM image shows that the mild steel was affected by uninhibited solution of sulfuric acid and holes are formed due to pitting corrosion, however in the presence of inhibitor no holes are found in the SEM image **Figure 8**. This is due to the adsorption of inhibitor and shielding the mild steel surface from corrosion.

## VII. CONCLUSIONS

1. CLS has been found to be a good corrosion inhibitor for mild steel in 0.5M H<sub>2</sub>SO<sub>4</sub> medium.
2. The Inhibition efficiency (%) and corrosion resistance of the mild steel increased with increase in inhibitor concentrations.
3. The Tafel polarization curves indicate that it is a mixed type inhibitor.
4. The present system obeys Langmuir Adsorption Isotherm.

SEM images show that the corrosion of the mild steel is due to pitting and the addition of inhibitor protects the mild steel surface from the corrosion.

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