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RESEARCH ARTICLE

SULFONATED MELAMINE FORMALIN RESIN AS A CORROSION INHIBITOR FOR MILD STEEL IN ACIDIC MEDIA

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ABSTRACT

Inhibition of corrosion of mild steel by Sulfonated melamine-formalin resin was investigated in Sulfuric acid medium (0.5 M) at various temperatures (303,313,323,333K) with the conventional weight loss, electrochemical polarization and electrochemical impedance spectroscopy (EIS). Weight loss method reveals good inhibition efficiency of the Sulfonated melamine-formalin resin and supports the data of electrochemical measurements. Polarization techniques substantiate the adsorption of inhibitor as a mode of inhibition. EIS technique shows the change in impedance parameters with change in concentration of inhibitor which corroborates the adsorption phenomenon resulting in the formation of a protective layer on the surface of mild steel. Adsorption of the inhibitor follows the Langmuir adsorption isotherm. Thermodynamic parameters like activation energy, free energy, enthalpy and entropy of the present system were obtained and elaborated. Based on all these evidences a suitable mechanism of the inhibitive action of the resin is proposed.

INTRODUCTION

Corrosion is a spontaneous process which results in the wearing away of the metal. It occurs whenever the metal is exposed to aggressive conditions such as acids, bases, salts etc and is accelerated by the rise in temperature. Mineral acids such as sulfuric acid and hydrochloric acid have been frequently used in pickling baths, descaling processes and drilling operations (Schmitt, 1984). Mild steel, a major industrial material when subjected to pickling is extensively suffered by corrosion in acidic conditions which leads to catastrophe on many occasions. Science attempts to find solution to resist corrosion and many processes such as coating, painting, galvanizing or anodizing are the offshoots of extensive scientific research in this direction. Apart from these methods the use of inhibitors especially organic inhibitors play a vital role in controlling corrosion (Singh *et al*., 2012). Many organic compounds have proven inhibitive action and have been investigated by several workers (Ci *et al*., 2000; Bentiss *et al*., 2002; Bartos and Hakerman, 1992).

The mechanism of inhibition of corrosion by organic compounds vests with its ability to be adsorbed and to form a protective layer on the metal surface which can markedly change the corrosion resisting property of the metal (Ferreira *et al*., 2004; Atia and Saleh, 2003). In case of organic inhibitors the mode of action is controlled by many factors such as, nature of functional groups, availability of π-electron density, steric factor, molecular weight etc (Quraishi and Kumar, 2009; Popova *et al*., 2004, 2003; Fouda *et al*., 2005; Kobayashi *et al*., 1993; Ouali *et al*., 2010; Laarej *et al*., 2010; Achouri *et al*., 2001). Organic compounds containing the hetero atoms such as nitrogen, sulfur and oxygen having one or two lone pair of electrons are widely used as inhibitors and few examples are some Schiff bases, thiourea and amine melamine formaldehyde (Manivannan and rajendran, 2011; Al-luaibi and Seta azad, 2011; Brett *et al*., 1994). An earlier study Quraishi *et al*., 2009; has shown that the introduction of formaldehyde performs so much the inhibitive propeties of polyaniline on corrosion of iron in acid chloride solution due to the presence of π-electrons, quarternary nitrogen atom and large molecular size which ensures greater coverage of the metallic surface. So the adsorption of polymer molecules on the iron electrode surface

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is more, which leads to more inhibition efficiency. Apart from the structural aspects the other factors affect the goodness of organic inhibitor are the pH of the medium, temperature, time duration and composition of the metal (Sorkhabi *et al*., 2005; Bentiss *et al*., 2005). In this work Sulfonated melamine-formalin resin is used as the corrosion inhibitor in the corrosion of mild steel in H_2SO_4. Due to the presence of conjugation and the number of nitrogen atoms the Sulfonated melamine formalin resin could be a good inhibitor and the present study vindicate the above notion. An earlier study (Tizpar and Ghasemi, 2006) also proved that larger molecular size and more nitrogen atoms in the polymer molecules leads to better inhibition efficiency (Tang *et al*., 2003).

MATERIALS AND METHODS

Resin preparation

Sodiumbisulphite is added to water to get a homogeneous solution. After getting homogeneous solution formalin is added to the solution and heated to 70 ° C, then melamine and formalin (37%) are added to the heated solution and maintained for 3 hrs at 90-95˚C. The pH of the system should be 7.0-9.0. The resultant solution obtained is converted into powder form (resin) which is readily soluble in water. The presence of various functional groups in the resin are conformed by FT-IR spectroscopy.

Preparation of electrode

Corrosion tests are carried out on electrodes cut from sheets of mild steel strips containing 0.01%Al, 0.05% Mn, 0.21% C, 0.05%S and the remaining of the iron is used for the measurement.

Mild steel 1cm × 1cm is abraded using different grades of emery paper and washed with distilled water and degreased with acetone. The specimens are prepared and dried at room temperature before used. Its surface area is 1 cm^2.

Weight loss method

The aggressive solution (0.5 M H_2SO_4) was prepared by dilution of analytical grade 98% H_2SO_4 with distilled water. Then the mild steel specimens are immersed in a 100ml beaker which contained 50ml of H_2SO_4 with and without addition of different concentrations of resin. After one hour the specimens are taken out washed, dried and weighed accurately. Then the tests are repeated at different temperatures, different concentrations of resin. In all cases duplicate experiments were performed and the mean values are reported. The inhibition efficiency is calculated as follows,

$$IE\,(\%) = ((CR^{\circ} - CR)/\,CR^{\circ}) \times 100 \qquad (1)$$

CR ° – average weight loss without inhibitor, CR- average weight loss with inhibitor

Molecular structure and IR spectra of the resin

The proposed structure of the resin is given as follows (Figure 1). IR spectrum of the resin was recorded using KBr pellet in the range of 500-4000cm^{-1}. From IR spectrum (Figure 2) we can get information regarding the molecular structure of melamine-formalin resin. The characteristic IR absorption bands of Sulfonated melamine-formalin resin are as follows: a broad band at 3452 cm^{-1} is attributed to N-H and O-H groups. The value at 2947 cm^{-1} is due to the stretching vibration of C-H groups.

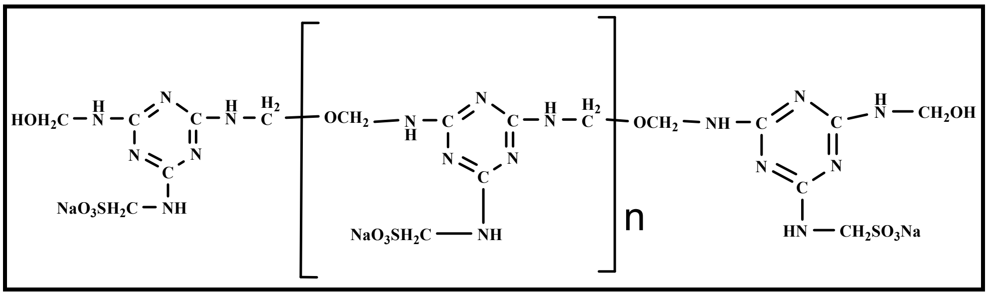

Figure 1. Molecular structure of Sulfonated Melamine-formalin resin (MF)

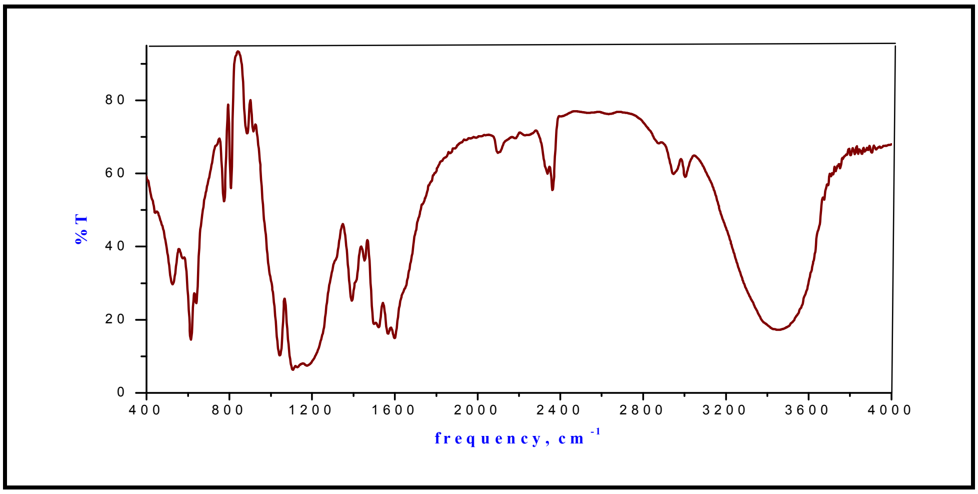

Figure 2. IR spectrum of Sulfonated melamine-formalin resin

Four absorption bands at 773, 808, 1392 and 1566cm^{-1} are related to melamine ring and R-NH_2-CH_2- group and the band at 1043 cm^{-1} is due to ether linkage group.

Electrochemical measurements

Electrochemical measurements were performed in a conventional three electrode cell consisting of mild steel as working electrode (WE), platinum as counter electrode (CE) and calomel as the reference electrode. By using emery paper, the mild steel (WE) were ground and degreased with acetone at room temperature before used. The area of the working electrode surface is 1 cm^2. The current density curves are obtained by linear extrapolation of the anodic and cathodic branches of the Tafel plots. Electrochemical Impedance Spectroscopy (EIS) studies were carried out in a frequency range of 1 MHz to 100 KHz with amplitude of 5 mV using AC signals at open circuit potential (OCP) with scan rate of 0.01 mVs^{-1}. Computer programs automatically controlled the measurements performed at rest potentials after 30 mts of exposure. Before recording the curves the test solution is de-aerated in magnetically stirred for few minutes in the cell with nitrogen. The charge transfer resistance (R_{ct}) values have been obtained from the diameter of semicircles of Nyquist plots. The inhibition efficiency has been calculated by using the following equation.

$$IE\ (\%) = ((R_{ct} - R^{o}_{ct}) / R_{ct}) \times 100 \quad (2)$$

R_{ct}- charge transfer resistance with inhibitor, R^{o}_{ct} - charge transfer resistance without inhibitor

RESULTS AND DISCUSSION

Weight loss method

Effect of concentration and temperature

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.

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).

$$\theta = (CR^{o} - CR) / CR^{o} \quad (3)$$

θ – degree of surface coverage, CR^{O}-average weight loss without inhibitor, CR- average weight loss with inhibitor

The plot of C/θ vs. C at different temperatures (303, 313, 323, 333K) is given in Figure 4.

Table 1. Inhibition efficiency (%) and surface coverage (θ) vs. concentration of inhibitor at different temperatures

Conc. of inhibitor (v/v) %	Inhibition efficiency (%)				θ				C/θ			
	303K	313K	323K	333K	303K	313K	323K	333K	303K	313K	323K	333K
2	50	41.7	36.6	35.7	0.50	0.41	0.36	0.35	4	4.8	5.5	5.7
4	57.9	48.2	39.1	40.2	0.57	0.48	0.39	0.40	7	8.3	10.2	10
6	61.1	45.7	37.7	36.6	0.61	0.45	0.37	0.36	9.8	13.3	15.9	16.6
8	67.5	43.1	35.2	35.7	0.67	0.43	0.35	0.36	11.9	18.6	22.7	22.8
10	83	40.2	34.9	33.9	0.83	0.40	0.35	0.34	12.04	25	28.6	29.4

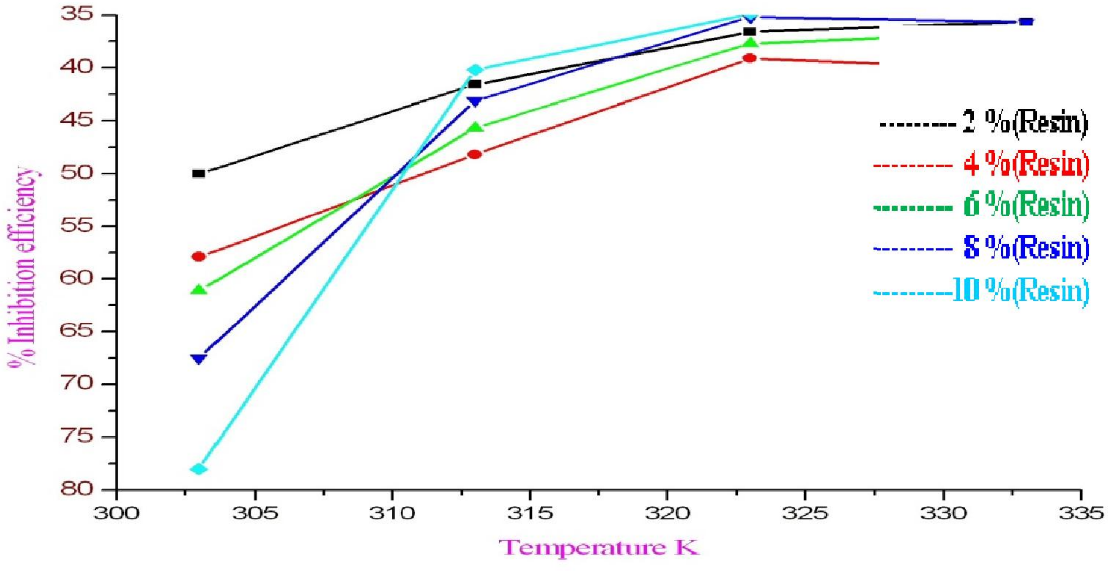

Figure 3. Variation of Inhibition efficiency (%) with temperature at various concentrations of inhibitor

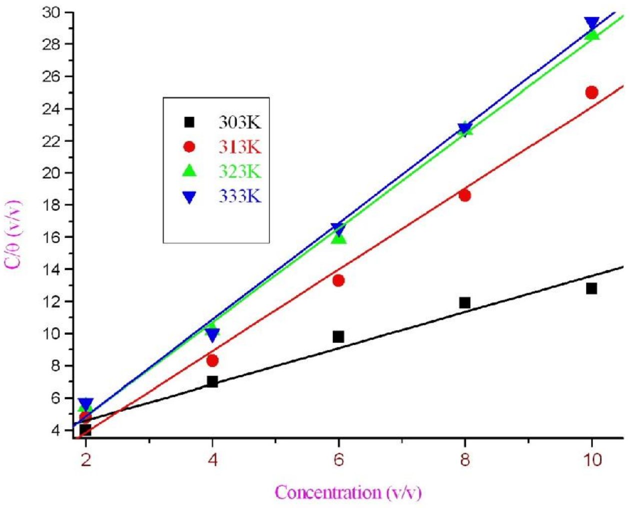

Figure 4. The relationship between C/θ and C at different temperatures in 0.5M H_2SO_4

The system obeys Langmuir adsorption isotherm which implies that the adsorption of the inhibitor obeys monolayer adsorption on the surface of the mild steel.

Thermodynamic aspects

Thermodynamic parameters like ΔE, ΔH, ΔG and ΔS are calculated by using the following equations. These values also help to understand the corrosion mechanism. The values of ΔE are obtained by the equation (3a).

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 (Figure 5) and from the slope of the straight line ΔE is obtained. The heat of adsorption (ΔH_{ads}) could be calculated from equation (4).

$$\Delta H_{ads} = 2.303R\ [\ (\log\theta_2/1-\theta_2)-(\log\theta_1/1-\theta_1)]\ T_2T_1/\ T_2-T_1\ \ \text{J/mol} \qquad (4)$$

θ_1,θ_2 – degree of surface coverage at any two temperatures T_1 and T_2. The heat of adsorption (ΔH_{ads}) can be approximately regarded as the standard heat of adsorption (ΔH˚) equation (4). The equilibrium constant K is given by the equation (5).

$$K = \theta / C(1-\theta) \qquad (5)$$

θ – degree of surface coverage, C- concentration of the inhibitor

The standard adsorption free energy (ΔG˚) was obtained according to equation (6). The equilibrium constant K can be related to the standard free energy of adsorption (ΔG˚) (Al-Lohedan *et al*., 1996) (equation 7). Here K is equilibrium constant for the adsorption process, 55.5 is the concentration of water mol/L, R is gas constant (k.cal.mol^{-1}) and T is the absolute temperature.

$$\Delta G^0\ (\text{ads}) = -RT\ \ln(55.5\ K) \qquad (6)$$

$$\ln K = -\ln 55.5 - (\Delta G^\circ/RT) \qquad (7)$$

The standard adsorption (ΔS˚) can be obtained by using the equation (8)

$$\Delta S^o = \Delta H^o - \Delta G^o / T \qquad (8)$$

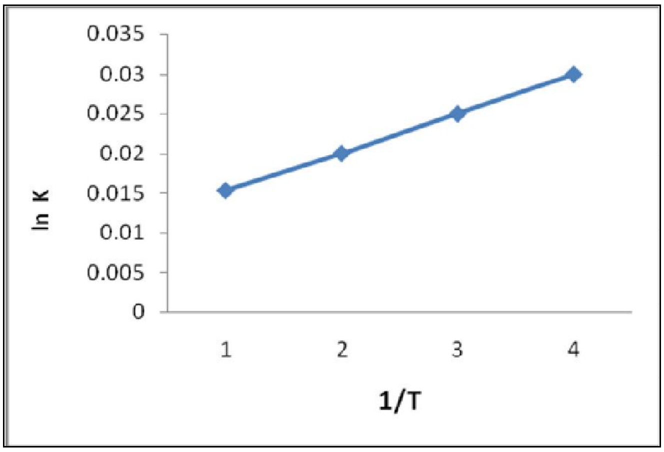

Figure 5. The relationship between ln k vs. 1/T

We observed that for the increasing concentration of inhibitor (2, 4, 6, 8, 10 % (v/v) activation energy (ΔE) increases (1.50, 2.49, 2.58, 3.48, 4.46 kJ/mol) at 303K. These results are in good agreement with reported studies (Bastidas *et al*., 2000; Elachouri *et al*., 1995; Migahed *et al*., 2010; Ahmed and Musa, 2008; Umoren, 2006). It indicates that there is strong inhibitive action on the mild steel surface (Al-Luaibi and Seta azad, 2011). The negative values of ΔH˚ show that the adsorption of inhibitor is an exothermic process (Nataraja *et al*., 2011). The negative values of ΔG˚ suggest that the adsorption of inhibitor molecule on the mild steel surface is a spontaneous process. The values of ΔS˚ are though positive their magnitude is small which supports a near uniform orientation of adsorbed molecules in inhibitive mechanism. The values of ΔG˚ are helpful to understand the nature of adsorption of inhibitor on the metal surface. Basically there are two types of interactions of the inhibitor with the metal surface, either physisorption or chemisorptions (Okafor *et al*., 2007; Edukok *et al*., 2012; Larabi *et al*., 2006; Larabi *et al*., 2004; Benali and Ouazene, 2011; Larabi *et al*., 2007). In general the values of ΔG˚ up to -40 kJ/mol implies the nature of adsorption as physical adsorption, while the values of ΔG˚ higher than - 40 kJ/mol infers chemisorption which means a co-ordinate type of bond formation between the inhibitor and metal surface. In the present study the values of ΔG˚ in the range of -24.77 to -25.55 are indicative of physical adsorption which supports the other experimental evidences in this work (Stoyanava *et al*., 1997).

Potentiodynamic polarization measurements

Potentiodynamic polarization measurements were carried out for this system and the details are given (Table 3). The kinetic parameters obtained by this method (Figure 6) such as E_{corr}, I_{corr}, anodic and cathodic tafel slopes (β_a & β_c) are given in Table 3. It is observed that with addition of increasing concentration of inhibitor the corrosion potential E_{corr}, β_a & β_c values do not change appreciably with the addition of the inhibitor indicator indicating that the inhibitor is not interfering with the anodic dissolution or cathodic hydrogen

Table 2. Thermodynamic parameters: ΔG°, ΔH° and ΔS° with inhibitor in the mild steel corrosion at different temperatures (303 – 333 K)

Concentration of the inhibitor % (v/v)	Free energy of adsorption - ΔG° = KJ/mol				Change in enthalpy ΔH° = KJ/mol	Change in entropy ΔS° = J/mole
	303 K	313 K	323 K	333 K		
2	25.77	25.67	25.92	26.61	-17.76	26.41
4	24.73	24.71	24.41	24.93	-11.51	43.60
6	24.13	23.76	23.09	23.71	-19.42	15.50
8	24.06	22.31	22.08	22.89	-3.91	60.51
10	25.55	21.38	21.49	22.03	-17.96	25.06

Table 3. Potentiodynamic Polarization parameters for the corrosion of mild steel in 0.5 M H_2SO_4 containing different concentrations of inhibitor at 303K

Concentration % (M)	E_{corr} (mV/ sec)	I_{corr} (Amp/cm^2)	β_a (mV/decade)	β_c (mV/decade)	IE %
0	-0.485	2.626	7.723	5.706	
0.002	-0.439	9.836	12.853	7.975	62.5
0.004	-0.432	7.505	12.597	8.48	71.4
0.006	-0.433	7.098	12.035	8.001	73.0
0.008	-0.493	5.539	13.161	7.919	78.9
0.010	-0.452	4.305	14.701	8.180	83.6

evolution reactions . Hence the inhibitor could acts as a mixed type inhibitor. From the Potentiodynamic polarization measurements the inhibition efficiency is attained using the equation.

$$\text{INHIBITION EFFCIENCY(\%)} = \frac{I_{corr} - I_{corr(inhibitor)}}{I_{corr}} \times 100 \qquad (9)$$

where I_{corr} and $I_{corr\ (inhibitor)}$ are the values of corrosion current densities of mild steel without and with the inhibitor respectively. It is observed that the inhibition efficiency increase with increase in concentration of the inhibitor.

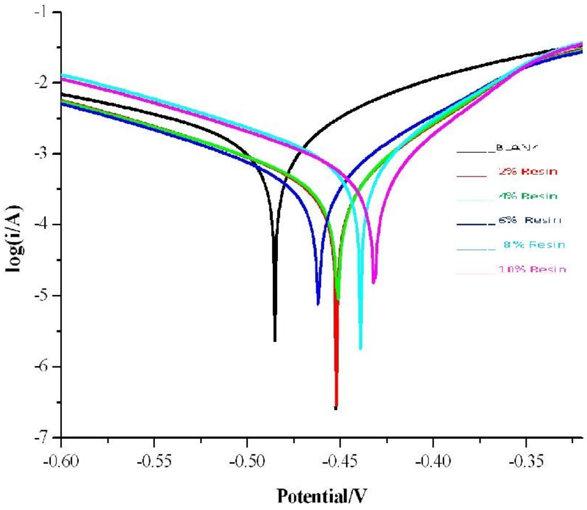

Figure 6. Potentiodynamic polarization curves of mild steel in 0.5 M H_2SO_4 with and without inhibitor at different concentrations

Electrochemical impedance spectroscopy (EIS)

The effect of the inhibitor concentration on the impedance behavior of mild steel in 0.5 M H_2SO_4 has been studied by electrochemical impedance spectroscopy (EIS) after 30 mts of immersion and the Nyquist plots are given in Figure 7. The impedance spectra show a single semicircle and as the concentration of inhibitor increases diameter of the semicircle increases. Deviations of perfect shape are often referred to the frequency dispersion of interfacial impedance which arises due to the roughness and other in homogeneity of the surface. It is evident from the results that melamine formation resin inhibited the corrosion of mild steel in 0.5 M H_2SO_4 at all the concentrations used and the IE % increased continuously with increasing concentration at 303K and the maximum IE% observed at the concentration of the inhibitor 10% (v/v) (Table 4). 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 probably 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.

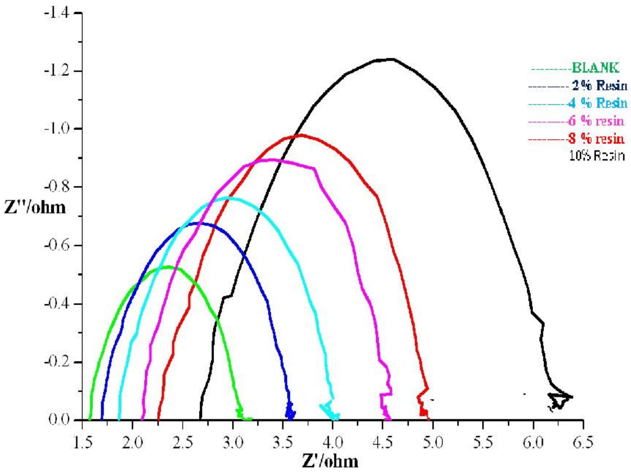

Figure 7. Nyquist plots for mild steel immersed in 0.5 M H_2SO_4 solution without and with inhibitor at various concentration

Table 4. Electrochemical impedance parameters at different concentrations of inhibitor

S.No	Conc. % (M)	R_{ct} Ω cm^2	C_{dl} μF cm^{-2}	IE %
1	Blank	0.642	2.48	0
2	0.002	1.528	1.04	57.98
3	0.004	2.131	0.74	69.87
4	0.006	2.683	0.59	76.07
5	0.008	3.101	0.51	79.29
6	0.010	3.843	0.41	83.29

The increase in R_{ct} values is attributed to the formation of protective film at the metal-solution interface. These observations suggest that the inhibitor functions by adsorption at the metal surface thereby causing decrease in C_{dl} values and increase in R_{ct} values. The details pertaining to all EIS parameters are given below.

$$C_{dl} = \frac{1}{2 \text{ x } 3.14 \text{ x } f_{max} \text{ x } R_{ct}} \qquad (10)$$

C_{dl} – double layer capacitance, f_{max}- frequency maximum

$$IE(\%) = \frac{R_{ct} - R^{o}_{ct}}{R_{ct}} \text{ x } 100 \qquad (11)$$

R_{ct}- charge transfer resistance with inhibitor, R°_{ct}- charge transfer resistance without inhibitor

The inhibitor has been found to be give good inhibition due to the electon density on the N-H group. This leads to the strong electrostatic attraction of inhibitor over the mild steel surface thereby resulting in the high inhibition efficiency when compared to the other resin.

Conclusions

Sulfonated melamine formalin resin acts as a good inhibitor in the corrosion of mild steel in H_2SO_4 medium. Weight loss, Potentiodynamic polarization and Electrochemical impedance spectroscopy (EIS) studies establish that the inhibition efficiency (IE %) increases with increasing inhibitor concentration of Sulfonated melamine-formalin resin. The adsorption obeys Langmuir adsorption isotherm and the negative value of the Gibbs free energy (ΔG°) indicates that there is strong interaction between inhibitor molecules and the mild steel surface.

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