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Composite of melamine-formalin resin as a corrosion inhibitor in acid medium

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Abstract

Inhibition of corrosion of mild steel by composite of melamine-formalin resin was investigated in H_2SO_4 medium at various temperatures (303,313,323,333K) with weight loss method, potentio-dynamic polarization and electrochemical impedance spectroscopy (EIS) techniques. Weight loss method reveals good inhibition efficiency of the melamine-formalin composite and supports the data of electrochemical measurements. EIS technique shows the change in impedance parameters with change in concentration of inhibitor shows 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 calculated

KEYWORDS:

Composite of Melamine-formalin, Adsorption-isotherm, Potentiodynamic polarization and EIS study.

I. 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 acids have been frequently used in pickling baths, descaling processes and drilling operations [1]. 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 plays a vital role in controlling corrosion [2]. Many organic compounds have proven inhibitive action and have been investigated by several workers [3-5]. 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 [6-7]. In case of organic inhibitors the mode of action is controlled by many factors such as, nature of the functional groups, availability of π-electron density, steric factor, molecular weight etc [8-15].Organic compounds containing the hetero atoms such as nitrogen, sulfur and oxygen having one or two lone pairs of electrons are widely used as inhibitors and few examples are some Schiff bases, thiourea and amine melamine formaldehyde [16 - 18].Among the heteroatoms nitrogen, sulfur and oxygen the efficiency in corrosion inhibition is in the order S>N>O. 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 [19-20].In this work melamine-formalin (SMF) 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 composite melamine formalin resin could be a good inhibitor and the present study vindicate the above notion. An earlier study [21] also proved that larger molecular size and more nitrogen atoms in the polymer molecules leads to better inhibition efficiency [22].

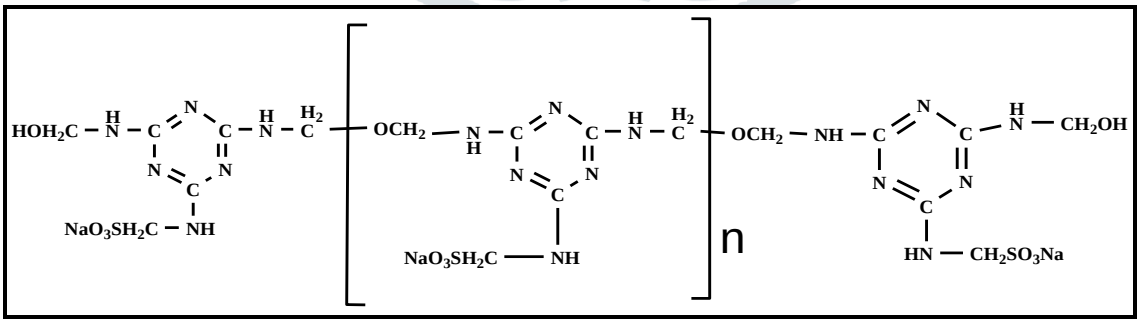
II. MATERIALS AND METHODS

2.1 Preparation of Composite melamine formalin(MF) resin

Sodiumbisulphite is added to water to get a homogeneous solution. After getting a homogeneous solution formalin is added to the solution and heated to 70˚ C, then melamine is added slowly after that formalin (37%) is again 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 is converted into powder form (resin) by putting in an oven at 150 ˚ C for half an hour. It is readily soluble in water. The presences of various functional groups in the melamine formalin resin are confirmed by FT-IR spectroscopy.

2.2 Molecular structure and IR spectra of the Composite (MF) resin

The proposed structure of (MF) resin is given as follows (**Figure 1)**.



Molecular structure of melamine-formalin resin composite

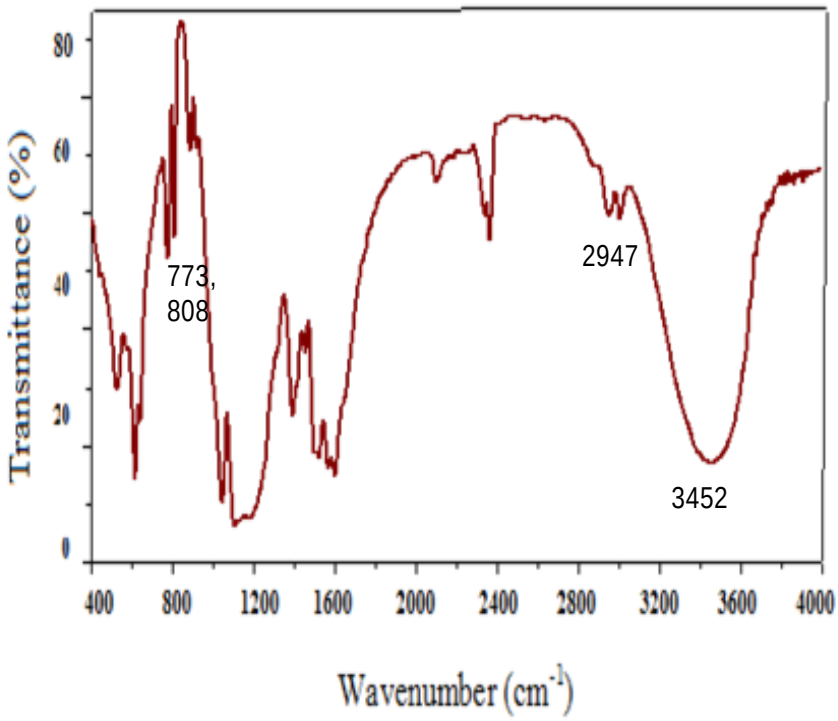


Figure 1 IR spectrum of Composite MF resin.

IR spectrum of composite (MF) resin was recorded using KBr pellet in the range of 400-4000 cm^{-1}. From IR spectrum (**Figure 1**) we can get information regarding the molecular structure of melamine-formalin resin. The characteristic IR absorption bands of MF 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.

Four absorption bands at 773, 808, 1392 and 1566 cm^{-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.

III. RESULTS AND DISCUSSION

3.1 Weight loss method

The weight loss method of obtaining corrosion rate and inhibition efficiency is useful because of its simple experimental set up and reliability. Weight loss data of mild steel in 0.5 M H_2SO_4 in the absence and presence of various concentrations of the inhibitor are listed in Table 1. The data shows that corrosion rate decreases and inhibition efficiency increases with the increase in the inhibitor concentration (from 2 mM to 10 mM) ie.the corrosion inhibition is enhanced with the increase in inhibitor concentration.

The corrosion rate is given as follows

Corrosion rate = (534 × weight loss) / (Density (D) × surface area (A) × Time (T)) (2)

T - Time in hours

A- Surface area of the mild steel specimen

D- Density of the mild steel specimen

With the values of the weight loss of the specimen without the inhibitor and with the inhibitor inhibition efficiency is calculated.

Inhibition efficiency is defined as follows

$$IE\ (\%) = [(CR^o - CR)/ CR^o)] \times 100 \quad (3)$$

CR^o – average weight loss without inhibitor, CR- average weight loss with inhibitor

Table 1 Inhibition efficiency (%), Surface coverage (θ) and C/θ values at different temperatures for the different concentrations of the inhibitor (MF) of mild steel in 0.5 M H_2SO_4

Conc. of inhibitor mM	Inhibition efficiency (%)				Surface coverage (θ)				C/θ			
	303K	313K	323K	333K	303K	313K	323K	333K	303K	313K	323K	333K
2	50.0	41.70	36.6	35.7	0.50	0.41	0.36	0.35	4.00	4.80	5.50	5.70
4	57.9	48.20	39.1	40.2	0.57	0.48	0.39	0.40	7.00	8.30	10.20	10.0
6	61.1	45.70	37.7	36.6	0.61	0.45	0.37	0.36	9.80	13.30	15.90	16.60
8	67.5	43.10	35.2	35.7	0.67	0.43	0.35	0.36	11.90	18.60	22.70	22.80
10	83.0	40.20	34.9	33.9	0.83	0.40	0.35	0.34	12.80	25.00	28.60	29.40

Corrosion data (inhibition efficiency (%), surface coverage (θ) and C/θ values) obtained from the conventional weight loss method at different concentrations of the inhibitor and at different temperatures is given in **Table 1.** These results show that at higher temperatures the phenomenon of desorption predominates rather than adsorption and so there is decreasing inhibition efficiency. This may be explained by the fact that the time lag between the process of adsorption and desorption of inhibitor molecules over the metal surface is becoming shorter with increase in temperature. It is observed that for almost all the concentrations there is a marked decrease in inhibition efficiency with increase in temperature. It is reported that the decrease in the stability of the adsorbed film at high temperature is the reason for this behavior. The increase in temperature may also weaken the forces, the adsorbed molecule will have with the metallic surface. These factors will result in the desorption of the inhibitor

film from the surface of the metal. This observation will support the physisorptive type of interaction between the inhibitor and the metal surface [23-25].

3.1 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 (4).

$$\theta = (CR^{o} - CR) / CR^{o} \qquad (4)$$

θ – Degree of surface coverage, CR^{o}-average weight loss without inhibitor, CR- average weight loss with inhibitor.Attempts were made to fit θ values to various isotherms including Langmuir, Frumkin and Temkin isotherms. Plot of log θ/c vs θ will give Temkin Isotherm and the plot of log θ/ (1- θ) c vs θ results in Frumkin Isotherm. The fitting of the values with respect to these two isotherms was not good. By far the best fit was obtained with Langmuir isotherm. The calculated values of log θ/c and log θ/ (1- θ) c are given in **Table 2** for various concentration of the inhibitors and at different temeperatures.

Table 2 Calculated values of θ, log θ/c and log θ/ (1- θ) c at different temperature with various concentrations of inhibitor (MF) for mild steel in 0.5 M H_2SO_4

Conc.of inhibitor (mM)	Θ				log θ/c				log θ/(1- θ) c			
	303K	313K	323K	333K	303K	313K	323K	333K	303K	313K	323K	333K
2	0.50	0.41	0.36	0.35	-0.60	-0.68	-0.74	-0.75	-0.30	-0.53	-0.55	-0.56
4	0.57	0.48	0.39	0.40	-0.84	-0.92	-1.01	-1.0	-0.47	-0.63	-0.79	-0.77
6	0.61	0.45	0.37	0.36	-0.99	-1.12	-1.20	-1.22	-0.76	-0.86	-0.99	-1.02
8	0.67	0.43	0.35	0.36	-1.07	-1.26	-1.35	-1.35	-0.59	-1.02	-1.17	-1.17
10	0.83	0.40	0.35	0.34	-1.10	-1.39	-1.46	-1.48	-0.45	-1.17	-1.28	-1.30

Langmuir adsorption isotherm was tested to fit the experimental values. Langmuir isotherm is given by the equation

$$C/\theta = 1/K_{ads} + C \quad (5)$$

Where θ = degree of surface coverage, C = inhibitor concentration, K_{ads}= equilibrium constant of the process of adsorption.

The plot of C/θ vs. C at different temperatures (303, 313, 323, and 333K) is given in **Figure 3**. The linear plot indicates that the present system obeys Langmuir adsorption isotherm. It presumes that

i. The adsorption of the inhibitor obeys monolayer adsorption on the surface of the mild steel.

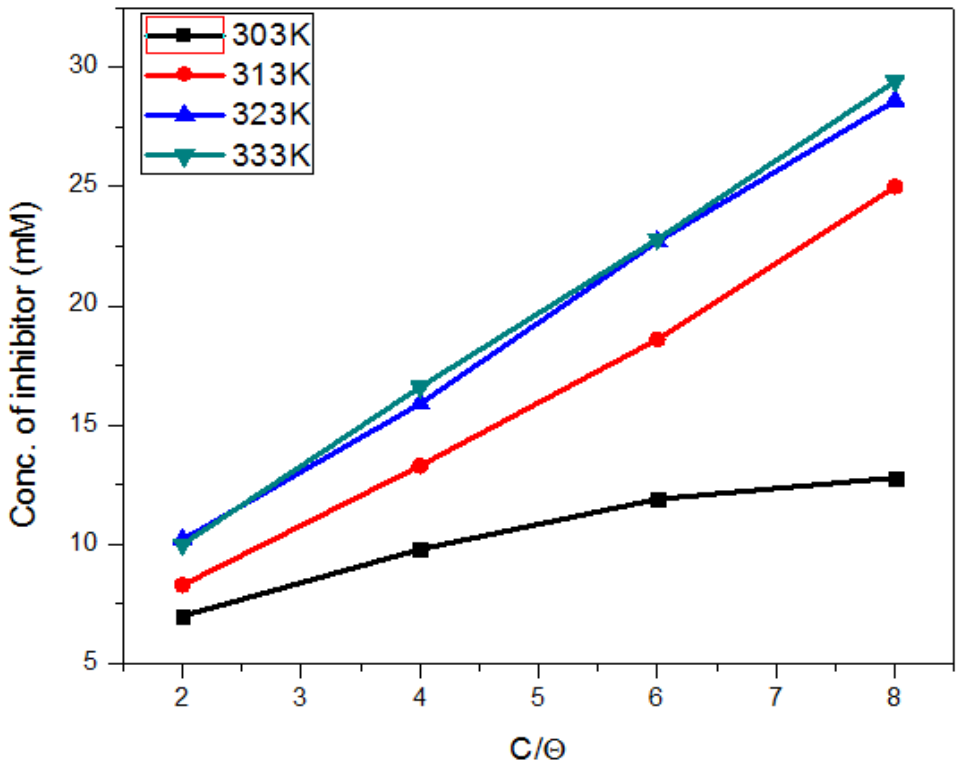


Figure 3 The relationship between C/θ Vs Concentration of composite MF at different temperatures in the inhibition of mild steel in 0.5 M H_2SO_4

3.2 Thermodynamic aspects

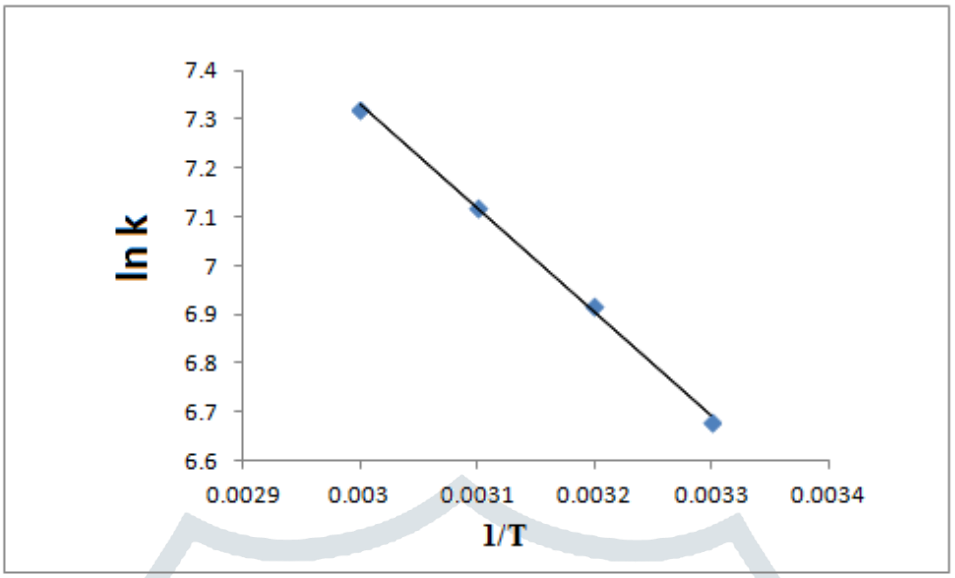


Figure 4 The relationship between ln k vs 1/T of mild steel in 0.5 M H_2SO_4

The plot of ln k vs 1/T gives a straight line (**Figure 4**) and from the slope of the straight line the value of ΔE is obtained

Table 3 Thermodynamic parameters: ΔG°, ΔH° and ΔS° with inhibitor SMF in the mild steel corrosion at different temperatures (303,313,323 and 333 K) in 0.5 M H_2SO_4

Concentration of the inhibitor (mM)	Free energy of adsorption - ΔG^o = KJ/mol				Change in enthalpy at 303 K ΔH^o = KJ/mol	Change in entropy at 303 K ΔS^o = J/mole
	303 K	313 K	323 K	333 K		
2	24.77	25.67	25.92	26.61	-17.76	23.13
4	24.73	24.71	24.41	24.93	-11.51	43.63
6	24.13	23.76	23.09	23.71	-19.42	15.54
8	24.06	22.31	22.08	22.89	-3.91	66.50
10	25.55	21.38	21.49	22.03	-17.96	25.04

We observed that for the increasing concentration of inhibitor (2, 4, 6, 8, 10 mM) 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 [27-31] which support that there is strong inhibitive action of the inhibitor on the mild steel surface [32].

The negative values of ΔH˚ show that the adsorption of inhibitor is an exothermic process [33]. 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 positive as listed in **Table 3**. The positive value is attributed to the fact that the adsorptions of the inhibitor molecules are regarded as a quasi-substitution process between them in the aqueous phase and water molecules in the interface of the metal and solution. The values of ΔS˚ are positive and 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 namely physisorption and chemisorption.

In general the values of ΔG˚ up to 20 kJ/mol implies that the nature of adsorption as physical adsorption, which means that the inhibitive action is due to the electrostatic interaction between the charged molecules and the charged metal. The inhibitor molecule is having the electron rich oxygen, sulfur and nitrogen atoms in the moiety. In the highly acidic medium the molecule becomes charged due to these atoms and hence electrostatic interactions. If the values of ΔG˚ are higher than - 40 KJ/mol infers chemisorption which means a co-ordinate type of bond formation between the inhibitor and metal surface [34-39]. In the present study the values of ΔG˚ in the range of -24.77 to -25.55 KJ/mol are indicative of physical adsorption which supports the other experimental evidences in this work [40-41].

3.3 Potentiodynamic polarization measurements

Potentiodynamic polarization measurements were carried out for mild steel specimens in 0.5 M H_2SO_4 at 303K in the presence of different concentrations of the inhibitor (SMF). Electrochemical polarization parameters like corrosion current densities (I_{corr}), corrosion potential (E_{corr}) cathodic Tafel slopes (β_c), anodic Tafel slopes (β_a) and inhibition efficiency (IE %) have been listed in **Table 4.** There is a notable decrease in the corrosion current density I_{corr} (8.30 -5.10 mA/ cm^2) in the presence of inhibitor. Lowest value of (I_{corr}= 1.36 mA/cm^2) was observed when the inhibitor concentration is 10 mM. It is also interesting to see an increase in the inhibition efficiency as the concentration of the inhibitor is increased. The data show that inhibitor molecules are in acidic media and affect both the anodic and cathodic sites uniformly. When the value of E_{corr} is having a shift >85 with respect to the E_{corr} of uninhibited solution, the inhibitor is supposed as either anodic (or) cathodic type [42-43]. However in the present study the maximum shift is small and is < 85 which suggests that inhibitor is a mixed type for mild steel specimens in H_2SO_4. The inhibition efficiency is obtained using the equation. 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 increases with increase in concentration of the inhibitor.

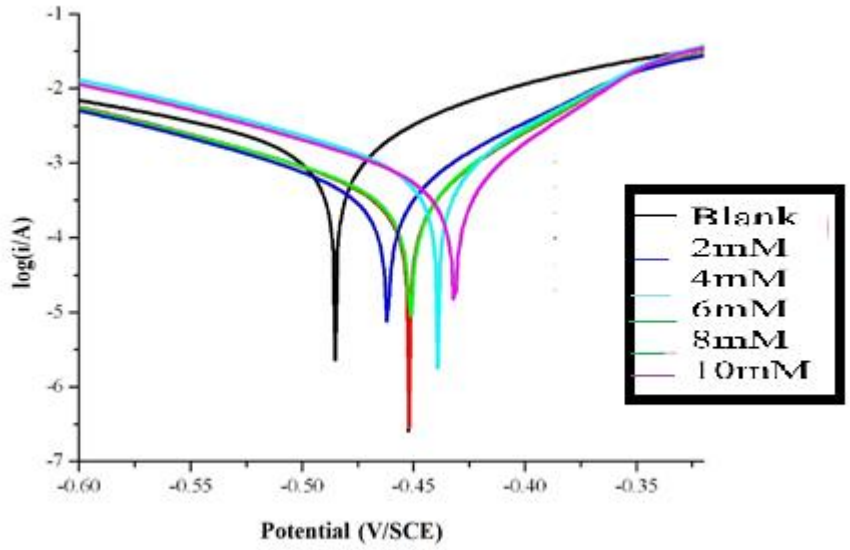


Figure 4 Potentiodynamic polarization curves of mild steel in 0.5 M H_2SO_4 with and without inhibitor composite (MF) at different concentrations at 303 K

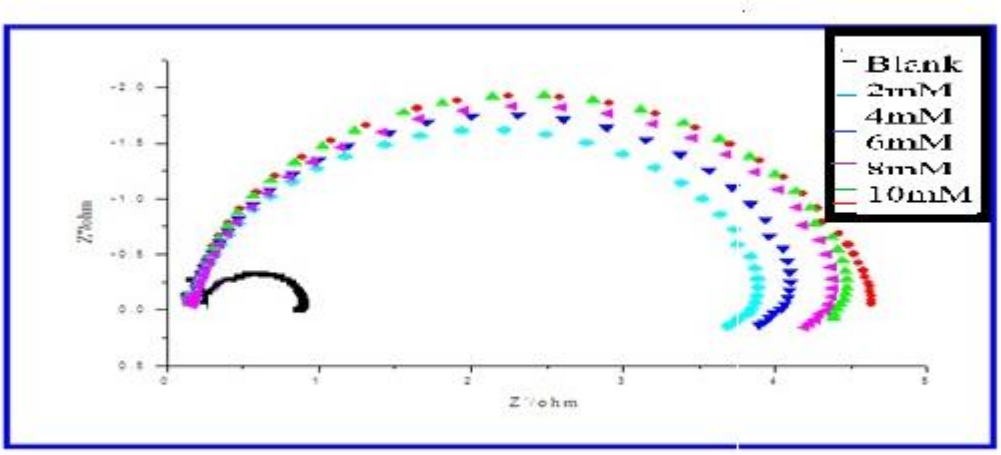
Table 4. Potentiodynamic Polarization parameters for the corrosion of mild steel in 0.5 M H_2SO_4 containing different concentrations of inhibitor at 303K

Concentration (mM)	E_{corr} (mV/SCE)	I_{corr} (mA/ cm^2)	β_a (mV/decade)	β_c (mV/decade)	IE %
0	-485	8.30	7.72	5.70	
2	-439	5.10	12.85	7.97	62.50
4	-432	4.10	12.59	8.48	71.40
6	-433	3.60	12.03	8.00	73.00
8	-493	3.30	13.16	7.91	78.90
10	-452	1.36	14.70	8.18	83.60

3.4 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 and the Nyquist plots are given in **Figure 5.** The impedance spectra show a single semicircle and as the concentration of inhibitor increases diameter of the semicircle increases indicating a charge transfer process which mainly controls the corrosion of mild steel in 0.5 M H_2SO_4 medium. Deviations of perfect shape are often referred to the frequency dispersion of interfacial impedance which arises due to the roughness and other inhomogeneity of the surface.

Figure 5 Nyquist plots for mild steel immersed in 0.5 M H_2SO_4 solution without and with inhibitor composite (MF) at various concentrations.



It is evident from the results that sulfonated melamine formalin resin (SMF) 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 (mM) (**Table 5**). 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. 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.

$$C_{dl} = \frac{1}{2 \times 3.14 \times f_{max} \times R_{ct}} \quad (6)$$

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

$$IE\ \% = \frac{R_{ct} - R^{0}_{ct}}{R_{ct}} \times 100 \quad (7)$$

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

Table 5. Electrochemical impedance parameters for the corrosion of mild steel in 0.5 M H_2SO_4 at different concentrations of inhibitor composite (MF) at 303 K

S.No	Conc. (m M)	R_{ct} Ω cm^2	C_{dl} µF cm^{-2} 10^{-7}	IE %
1	0	0.64	2.48	0
2	2	1.52	1.04	57.98
3	4	2.13	0.74	69.87
4	6	2.68	0.59	76.07
5	8	3.10	0.51	79.29
6	10	3.84	0.41	83.29

It is clear that with increase in surface coverage the C_{dl} values decreases. These observations vindicate the phenomenon of adsorption of inhibitor on the metal surface which is responsible for corrosion control. The high performance of the composite resin in the present investigation is attributed to the presence of rich π-electrons, quaternary nitrogen atom and its large molecular size [43 – 47].

Conclusion

Composite of Melamine formalin (MF) 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) measurements establish that the inhibition efficiency (IE %) increases with increasing concentration of SMF. 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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