



## Study of Inhibitive Action of Bio-degradable Crab Shell Extract on Mild Steel in Acidic Medium

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### Abstract

The inhibition efficiency of crab shell (CS) extract was investigated on mild steel (MS) in 1M H<sub>2</sub>SO<sub>4</sub>. The corrosion resistant behaviour was assessed by weight-loss method, thermodynamic and polarization studies. The maximum inhibition efficiency of 93.20% was recorded at 12 hours of immersion time at optimum concentration. The inhibition efficiency diminishes with increasing temperature. The results of the Langmuir adsorption study support the nature of monolayer adsorption. The activation and thermodynamic analysis demonstrated that the reaction is natural and spontaneous. The mixed type character of the inhibitor was shown by potentiodynamic polarisation studies. The values of R<sub>ct</sub> and C<sub>dl</sub> obtained from EIS indicated that the CS extract acts as an effective inhibitor. SEM examination revealed the creation of the protective layer on the surface of the mild steel.

**Keywords:** Crab shell; Corrosioninhibition; Mild Steel; H<sub>2</sub>SO<sub>4</sub> solution

### 1. Introduction

The serious consequences of corrosion lead to plant shutdowns, waste of valuable resources, reduction in efficiency of the materials and economic loss. Mild steel is one of the most popularly used material as it has various advantageous features and quite affordable. In acidic environment, mild steel corrodes quickly. Based on the literature survey, 1M sulphuric acid was selected as corrosive medium. Sulphuric acid is a common industrial chemical that is used in the production of a variety of products as well as a pickling agent, either alone or in combination with other acids [1]. This acid is aggressive, easily attacks the surface of the materials to which it is in contact. The storage of acid in tanks or other stagnant containers, in particular, leads to the rapid corrosion process [2]. In the current study, measurements were performed in a stagnant solution based on this view.

There are several methods includes in corrosion protection such as design modification, protective coatings, corrosion inhibitors etc., Among those methods, corrosion inhibition is the convenient and easy method as it has high solubility character and forms a uniform layer on metal surface to create a water-based or solvent-based coating. In particular, recent studies focused on green inhibitors as they are

biodegradable and do not contain heavy metals and other toxic compounds, whereas many synthetic compounds may cause damage to both environment and human health. [3-4].

Literature surveys showed that many plant extracts were being utilised as corrosion inhibitor. Generation and availability of seafood waste is enormous when compared to plant derivatives. A large quantity of seafood waste is not properly disposed and it causes an ill effect to the environment [5]. The present study aims to recycle the seafood waste for corrosion prevention in order to minimize the disposal issues of seafood waste. It mainly consists of calcium carbonate and protein. The mixture of amino acids present in protein may be used as a safe corrosion inhibitor.

### 2. Experimental

#### 2.1 Material and methods

Mild steel specimens with the dimension of 5x1cm<sup>2</sup> were cut into pieces and polished mechanically with different grades of emery sheets followed by the process of degreasing and washing with deionized water. Dried and weighed samples were used for further experimental procedures.

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Crab shells were collected from a nearby fish market. It is then washed, dried at shadow and crushed well using mortar. About 25g of the powdered crab shell was weighed and refluxed in 500ml of 0.5M H<sub>2</sub>SO<sub>4</sub> for 3 hrs and kept overnight for cooling. It is then filtered and made upto 500ml to get 5% extract. Various concentrations of crab shell extracts were prepared from the stock solution.

## 2.2. Corrosion Study

### 2.2.1. Weight Loss Measurements

Weight loss of MS specimens were monitored at different intervals of time (3,6,12 and 24 hrs.). In a glass beaker, the pre-weighed specimens were immersed in 100ml of 1M H<sub>2</sub>SO<sub>4</sub> solution in the absence and presence of inhibitor of different concentrations (0.1 to 0.5%). After the elapsed time, the specimens were taken out from the beaker, washed with deionized water and then with acetone. The air-dried specimens were reweighed accurately. For better result, experiments were carried out in triplicate.

Weight loss measurements were also performed at different temperatures of 303K, 313K, 323K and 333K in the absence and presence of inhibitor (0.1% to 0.5%) for 3hrs. immersion time.

From the weight loss of specimens, the percentage of inhibition efficiency ( $\eta\%$ ), surface coverage ( $\theta$ ) and corrosion rate (CR) were determined by using the following equations (1-3) [6].

$$\eta(\%) = (w_0 - w_i)/w_0 \times 100 \quad (1)$$

$$\theta = (w_0 - w_i)/w_0 \quad (2)$$

where,  $w_0$  and  $w_i$  are the weight loss value in the absence and in the presence of inhibitor respectively and  $\theta$  is the surface coverage of specimen by the inhibitor.

$$CR = (87.6 \times w)/DAT \quad (3)$$

where, CR is the corrosion rate in millimetre per year,  $w$  is the weight loss of specimen,  $D$  is the density of specimen,  $A$  is the area of specimen exposed to corrosive media and  $T$  is the immersion time in hours.

### 2.2.2. Potentiodynamic Polarization (Tafel slope) and Electrochemical Impedance Spectroscopy (EIS) studies

Potentiodynamic Polarization (Tafel slope) and Electrochemical Impedance Spectroscopy (EIS) were employed in addition to weight loss methods. For each run, cleaned electrodes and freshly prepared solution were used to get accurate results. The working electrode was immersed in test solutions for

one hour at room temperature before taking measurements.

Polarization studies were conducted using platinum electrode and saturated calomel electrode as counter electrode and reference electrode. MS was set as a working electrode and it was dipped in 1M H<sub>2</sub>SO<sub>4</sub>. The polarization curves were recorded by changing the potential from -200mV to +200mV with respect to OCP at a scan rate of 1mVsec<sup>-1</sup>. EIS measurements were carried out with a 10MHz to 100 KHz frequency range at amplitude of 5mV.

The inhibition efficiency ( $\eta\%$ ) was evaluated from the values obtained from polarization and EIS measurements using the following equations (4) and (5) respectively [7-8].

$$\eta(\%) = (i_{corr}^0 - i'_{corr})/i_{corr}^0 \times 100 \quad (4)$$

where,  $I_{corr}^0$  and  $I'_{corr}$  are the corrosion current densities in the absence and in the presence of various concentrations of the inhibitor.

$$\eta(\%) = R_{ct}^0 - R'_{ct}/R_{ct}^0 \times 100 \quad (5)$$

where,  $R_{ct}^0$  and  $R'_{ct}$  are the charge transfer resistance in the presence and in the absence of inhibitor respectively.

### 2.2.3. Surface Morphology

MS specimens were immersed in 1M H<sub>2</sub>SO<sub>4</sub> as well as in acid containing an optimum concentration of inhibitor for 24 hours. Then, the specimens were removed, washed, dried and surface characteristics were studied using scanning electron microscope to compare the surface morphology of fresh, polished specimen with specimens immersed in the blank and the inhibitor solution.

## 3. Results and Discussion

### 3.1. Weight Loss Measurements

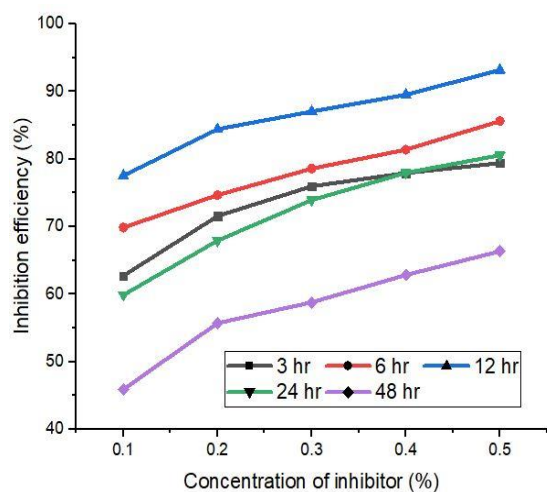
#### 3.1.1. Effect of immersion period

The efficiency of the inhibitor at different concentrations with specific intervals of the immersion period were analysed using weight loss method is represented in Fig.1. The figure inferred that increasing level of concentration of inhibitor likely reduces the corrosion rate of MS with the increasing immersion time.

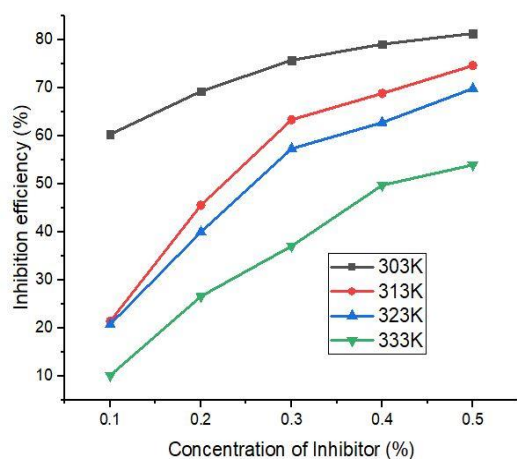
#### 3.1.2. Effect of Temperature

Temperature is one of the parameters that influence the inhibition process of corrosion. Increase in temperature is associated with increase in corrosion rate thereby reducing inhibition efficiency. Fig. 2 is plotted for temperature study and from the data, it was observed that desorption taking place at

higher temperatures [9-11]. 81.37% inhibition efficiency was attained at 303K with 0.5% concentration of CS extract.



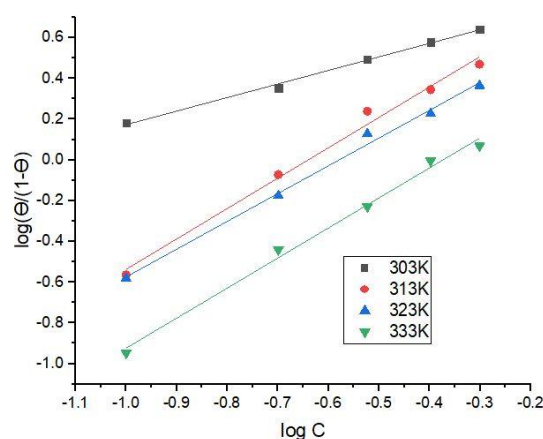
**Fig. 1.** Effect of different concentrations of CS extract on MS with different immersion period in 1M H<sub>2</sub>SO<sub>4</sub> solution



**Fig. 2.** Effect of temperature on MS with different concentrations of CS extract in 1M H<sub>2</sub>SO<sub>4</sub>

### 3.1.3. Adsorption Isotherm

The nature of interaction between MS and inhibitor is being confirmed by adsorption isotherms. The plot of  $\log C$  with  $\log(\theta/1-\theta)$  provides the information about the nature of adsorption. Weight loss measurements at different concentrations of inhibitor in the temperature range of 303K-333K after 3 hours immersion time were used to identify the nature of adsorption process. Monolayer adsorption of inhibitor on MS is confirmed from the values of  $R^2$  which is close to unity (0.99608, 0.9903, 0.99392, 0.99309) which is obtained from the plot of  $\log C$  vs  $\log(\theta/1-\theta)$  (Fig.3) [12].



**Fig. 3.** Langmuir Adsorption isotherm for the dissolution of MS in 1M H<sub>2</sub>SO<sub>4</sub> in the absence and in the presence of CS extract

### 3.1.4. Activation Parameters

#### 3.1.4.1. Energy of activation

The activation energy of the corrosion process on MS can be analyzed in the absence and presence of various concentrations of inhibitors in 1M H<sub>2</sub>SO<sub>4</sub>. Kinetic parameters obtained from the Arrhenius plot ( $1000/T$  versus  $\ln CR$ ) and transition state plot ( $1000/T$  versus  $\ln CR/T$ ) are indicated in Fig.4 and Fig.5 respectively. The values of various kinetic and thermodynamic parameters of the corrosion process were analyzed using Arrhenius equation (6) and transition state equation (7) and it is listed in Table 1. [13-14].

$$\ln CR = \ln A - E_a/RT(6)$$

$$\ln CR/T = [\ln(R/Nh) + (\Delta S^*/R)] - \Delta H^*/RT(7)$$

where, CR is the corrosion rate, T is the temperature, h is the Planck's constant, N is the Avogadro number, R is the universal gas constant,  $\Delta S^*$  is entropy of activation and  $\Delta H^*$  is enthalpy of activation.

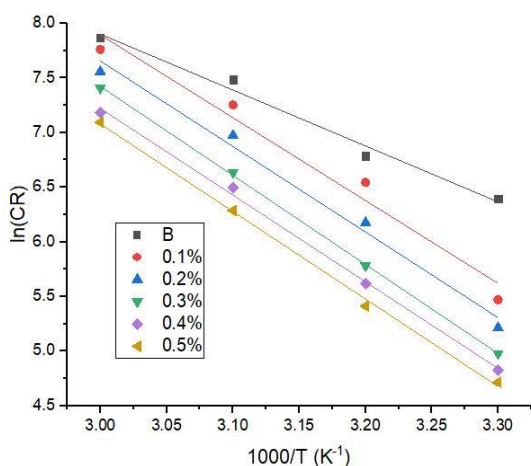
The kinetic parameters were enlisted in Table 1. The activation energy ( $E_a$ ) of inhibited solutions was found to be higher when compared to uninhibited solution, which suggests that there was a hindrance in metal dissolution by the formation of metal complex layer on the surface of MS and retardation of corrosion rate [15]. Positive values of  $\Delta H^*$  reflect the endothermic process of metal dissolution.  $\Delta H^*$  values less than 40KJ/mol are represented as physical adsorption and around 100KJ/mol is represented as chemical adsorption [16]. From Table 1, enthalpy values were observed in between 40 to 100KJ/mol which ensures the nature of mixed-type adsorption. The large negative values of entropy in the absence

and in the presence of inhibitor inferred that the activated complex in the rate determining step represents an association step rather than dissociation

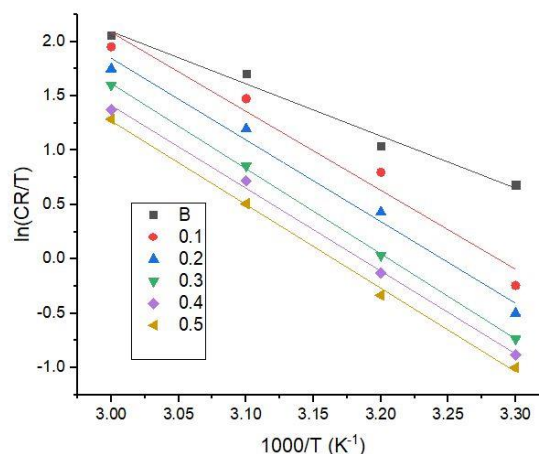
step which means that a decrease in disordering takes place on going from reactants to the activated complex [17].

**Table 1. Kinetic parameters of adsorption of various concentrations of CS extract for the dissolution of MS in 1M H<sub>2</sub>SO<sub>4</sub>**

Conc. (%)	E <sub>a</sub> (kJ/mol)	-ΔG (KJ/mol)				ΔH* (kJ/mol)	ΔS* (J/mol)	R <sup>2</sup>
		303	313	323	333			
Blank	98.03	-	-	-	-	39.95	-181.03	0.98512
0.1	145.22	16.96	13.04	13.35	11.43	60.44	-173.64	0.97248
0.2	150.00	16.20	14.16	13.99	12.74	62.52	-173.13	0.98843
0.3	156.09	16.00	14.99	14.79	12.93	65.16	-172.41	0.9997
0.4	151.99	15.76	14.88	14.63	13.60	63.38	-173.26	0.99786
0.5	153.44	15.56	15.05	14.87	13.44	64.01	-173.17	0.9981



**Fig. 4. Arrhenius plot for the MS dissolution in 1M H<sub>2</sub>SO<sub>4</sub> in the absence and in the presence of various concentrations of CS extract at different temperature**



**Fig. 5. Transition state plot for the MS dissolution in 1M H<sub>2</sub>SO<sub>4</sub> in the absence and in the presence of various concentrations of CS extract at different temperature**

### 3.1.4.2. Thermodynamic parameters of adsorption

Adsorption free energy ( $\Delta G_{ads}$ ), the most significant thermodynamic adsorption parameter was assessed by the following equation (8) [18].

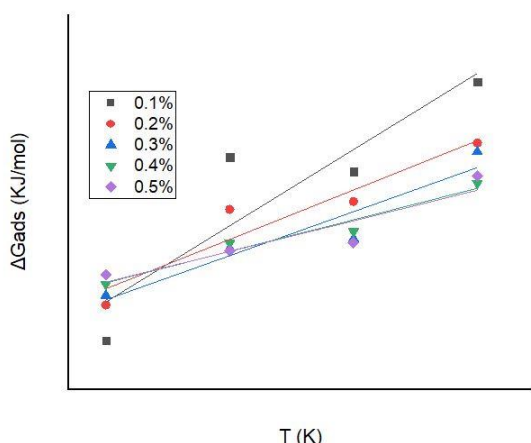
$$\Delta G_{ads} = -RT \ln(55.5 K_{ads}) \quad (8)$$

where, K is the adsorption equilibrium constant, R is the gas constant, T is the absolute temperature and 55.5 is the concentration of water in solution (molL<sup>-1</sup>) [19].

Thermodynamic parameters were recorded in Table 2. In general,  $\Delta G$  values up to -20KJ/mol and less than -40KJ/mol is attributed to physical adsorption and chemical adsorption respectively [20].  $\Delta G$  values were found below -20KJ/mol which support that the process of physical adsorption of inhibitor on MS. Negative enthalpy values implies that the exothermic process and positive entropy values decreases with increase in concentration of inhibitor indicates that the system goes from more ordered arrangement to less ordered arrangement.

**Table 2. Thermodynamic parameters of adsorption of various concentration of CS extract for the dissolution of MS in 1M H<sub>2</sub>SO<sub>4</sub>**

Conc. (%)	-ΔG (KJ/mol)				-ΔH* (kJ/mol)	ΔS* (J/mol)
	303	313	323	333		
0.1	16.96	13.04	13.35	11.43	65.47	162.8
0.2	16.20	14.16	13.99	12.74	47.82	105.5
0.3	16.00	14.99	14.79	12.93	44.60	94.1
0.4	15.76	14.88	14.63	13.60	36.12	67.3
0.5	15.56	15.05	14.87	13.44	35.53	65.4



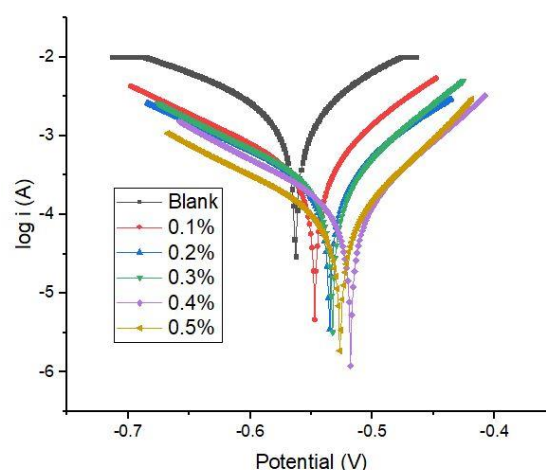
**Fig. 6.** Arrhenius plot for the MS dissolution in 1M H<sub>2</sub>SO<sub>4</sub> in the absence and in the presence of various concentrations of CS extract at different temperature

### 3.2. Electrochemical Studies

#### 3.2.1. Potentiodynamic polarization studies

Polarization curves were plotted for MS in 1M H<sub>2</sub>SO<sub>4</sub> in the presence and in the absence of CS extract and represented in Fig.7. The results observed from polarization curve is shown in Table 3. The corrosion current density (*I*<sub>corr</sub>) was dropped with the increased concentration of CS extract and thus the corrosion rate is getting reduced which means that the inhibitor formed a barrier layer over the surface of MS. No significant change was

observed in corrosion potential (*E*<sub>corr</sub>) and the maximum displacement in *E*<sub>corr</sub> value was about -46mV, which means that the inhibitor might be mixed-type. Both *b*<sub>a</sub> and *b*<sub>c</sub> values showed the lower current density with the addition of CS extract, signifying the dropping level of both hydrogen evolution and metal dissolution reactions, in specific, hydrogen evolution was inhibited markedly while comparing the metal dissolution process [21].



**Fig. 7.** Potentiodynamic polarization curves for the MS dissolution in 1M H<sub>2</sub>SO<sub>4</sub> in the absence and in the presence of various concentrations of CS extract

**Table 3.** Electrochemical kinetic parameters calculated by using polarization measurements for dissolution of MS in 1M H<sub>2</sub>SO<sub>4</sub> in the absence and presence of various concentration of CS extract

Conc. %	- <i>E</i> <sub>corr</sub> mV	<i>I</i> <sub>corr</sub> μA	<i>b</i> <sub>a</sub> mVdec <sup>-1</sup>	<i>b</i> <sub>c</sub> mVdec <sup>-1</sup>	C.R. mmy <sup>-1</sup>	η%
Blank	563.15	2838.8	220.59	139.27	32.99	-
0.1	547.33	408.23	151.37	83.45	4.74	85.62
0.2	535.28	284.26	160.38	92.05	3.30	89.99
0.3	532.96	253.68	146.39	82.26	2.95	91.06
0.4	526.51	129.33	135.37	81.19	1.50	95.44
0.5	517.84	85.89	131	73.22	1.00	96.97

#### 3.2.2. Electrochemical impedance spectroscopy

Fig.8 shows the Nyquist plots for MS dipped in the absence and in the presence of CS extract. An increase in the diameter of semicircle with increasing concentration of inhibitor can be observed from the Fig.8. and the charge-transfer resistance (*R*<sub>ct</sub>) increased as the concentration of inhibitor increases. From the increasing values of charge-transfer resistance (*R*<sub>ct</sub>) listed in Table 4., it is evident that the corrosion rate is getting slow down by forming a surface film on the specimen. Double layer capacitance decreases with increase in inhibitor

concentration indicating the retardation of metal dissolution and thereby decreases in local dielectric constant of the electrical double layer [22-23]. Further, it showed the maximum inhibition efficiency as 90.92% at the level of 0.5% concentration of the inhibitor.

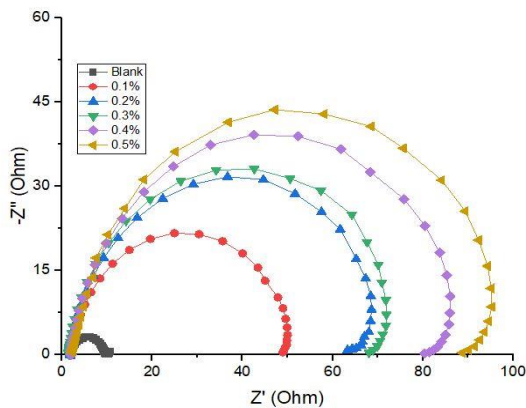
### 3.3. SEM Analysis:

Scanning Electron Microscope photographs of plain MS, specimen dipped in 1M H<sub>2</sub>SO<sub>4</sub> and dipped in the mixture of 1M H<sub>2</sub>SO<sub>4</sub> with 0.5% inhibitor for 24 hours were shown in Fig.9a, 9b and 9c respectively.

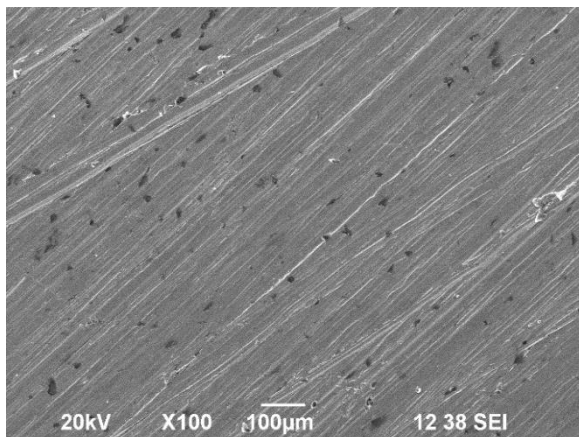
Smooth surface of a fresh and polished specimen (Fig.9a) confirms that there was no corrosion takes place. In Fig.9b, rough surface was noticed which represents the strong corrosion on the surface. A protective layer was observed on the surface of the specimen in the presence of inhibitor (Fig.9c) support the process of inhibition.

**Table4. Electrochemical impedance parameters for dissolution of MS in 1M H<sub>2</sub>SO<sub>4</sub> in the absence and presence of various concentration of CS extract**

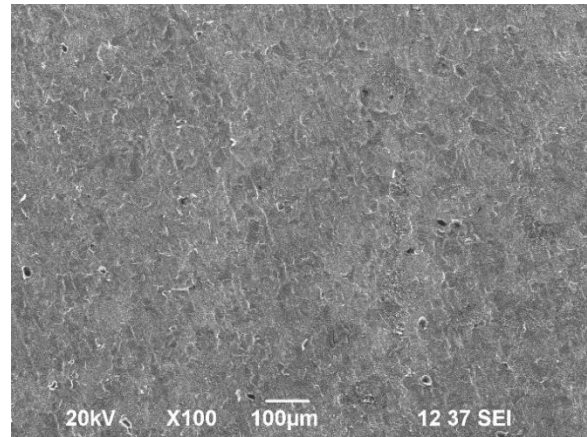
Conc. %	R <sub>s</sub> Ωcm <sup>2</sup>	R <sub>ct</sub> Ωcm <sup>2</sup>	Cdl μFcm <sup>-2</sup>	η%
Blank	2.05	7.49	212.60	-
0.1	2.21	45.1	35.31	83.39
0.2	1.82	59.3	26.85	87.37
0.3	1.58	63.1	25.24	88.13
0.4	2.07	77.8	20.47	90.37
0.5	3.53	82.5	19.30	90.92



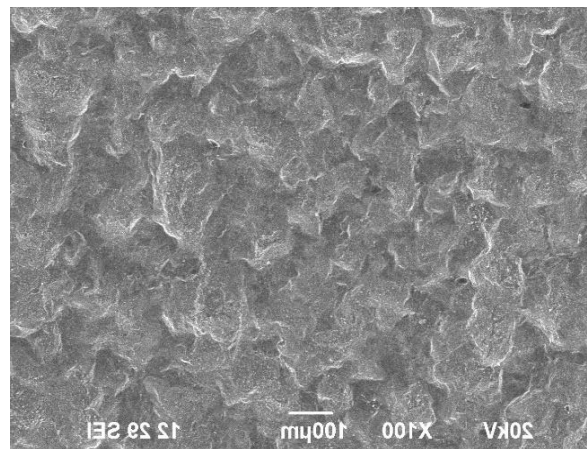
**Fig. 8. Nyquist plot for the MS dissolution in 1M H<sub>2</sub>SO<sub>4</sub> in the absence and in the presence of various concentrations of CS extract**



**Fig. 9a. SEM for Pure MS surface**



**Fig. 9b. SEM for MS in 1M H<sub>2</sub>SO<sub>4</sub>**



**Fig. 9c. SEM for mild steel in 1M H<sub>2</sub>SO<sub>4</sub> with 0.5% of CS extract**

#### 4. Conclusion

According to the results obtained from the present study, the following conclusions can be drawn:

- The inhibitor prepared from CS extract was found to be good with increasing immersion time and showed the maximum efficiency as 93.20% at 12 hours. Increase in temperature leads to a decrease in inhibition efficiency.
- The adsorption of CS extract obeys with the Langmuir adsorption isotherm and from the activation parameters, it is concluded that it follows physical adsorption.
- The negative values of  $\Delta G_{ads}$  implies that the process is spontaneous
- From the  $E_{corr}$  values observed from polarization study, it is revealed that the inhibitor is mixed-type.
- The results obtained from EIS were in good agreement.

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