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Study of Inhibition and Adsorption Properties of Mild Steel Corrosion by Expired Pharmaceutical *Gentamicin* Drug in Hydrochloric acid media

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ABSTRACT

This experiment avoids pollution and recycles of the drugs and also promotes economically benefited inhibitor. In this experiment expired Gentamicin drug was under studied for testing its inhibitive effect on mild steel in 1 M HCl acid medium with the weight loss and electrochemical techniques. The Various parameters were calculated such as corrosion rate, inhibition efficiency and surface coverage. The weight loss method shows that the inhibition efficiency increases when inhibitor concentration, immersion period and temperature increases.Obtained results in electrochemical Impedance and weight loss studies are very much excellent agreement with each other. Thermodynamic parameter like free energy value was negative, that shows spontaneous adsorption of inhibitor on the surface of the mild steel. The adsorption nature of the inhibitor on the surface of mild steel was under conformity with Langmuir adsorption isotherm. The mild steel surface morphology, with and without inhibitor was studied by using scanning electron microscopy

Keywords: Mild steel,acidic medium, economically benefitted corrosion inhibitor, expired-drug, electrochemical Impedance and weight loss methods, the mild steel surface morphology

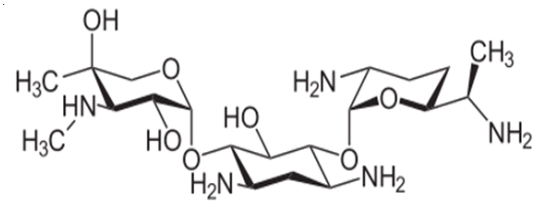
INTRODUCTION

Simple accessibility, minimal effort of mellow steel has numerous industrial applications. It is broadly utilized as a part of different ventures like sugar, cowhide, sustenance, petrochemical, paper and material businesses. In most mechanical procedures, the acidic arrangements are regularly utilized for the pickling,acid cleaning, de-scaling of acids etc. The decreasing corrosion rate of metals spares the current assets and thusly creates conservative advantages amid the mechanical applications. The lifetime of types of gear can be expanded and viable corrosion restraint will diminish the disintegration of harmful metals from the parts into nature[1-3]. Hydrochloric acid is most the most corrosive with most of the common metals and alloys, difficult of this acid to handle from corrosion and materials of constructions, due to its industrial applications the corrosion inhibitors are became an answer for corrosion attack witch lead to metal damage and also replacement of the metal. Many reviews on consumption inhibitors tell that a large portion of the inhibitors are organic with N, O and S atoms or with polar groups of N-hetero cyclic

compounds. They have fundamental properties with high electron thickness, making them the response focuses[4-10]. These block the dynamic corrosion locations by adsorption on the metallic surface and the majority of them are exceedingly dangerous to the people and also the earth. Thus replacement by these eco-friendly inhibitors is necessary. The use of pharmaceutical compounds which are contains hetero atoms in their structure, high solubility and high molecular weight offers interesting possibilities for corrosion inhibition[11-20]. Few drugs like azosulpha and antimalarial have been accounted for that they are great corrosion inhibitors on mild steel[21-23]. In thisreview Gentamicindrug (Eye/Ear Drops) that has been expired is chosen for the corrosion inhibitionon the mild steel in 1M hydrochloric acid medium by utilizing weight loss, electrochemical spectroscopy strategies. Gentamicinis a non-toxic pharmaceutical medication used to prevent or treat a wide variety of bacterial infections. It belongs to a class of drugs known as amino glycoside antibiotics, it contain N- atom having high electron density to block the active sites of corrosion.The inhibitor is available in the brand name of 'Gentamicin' fabricated in India by CIPLA LTD it goes about as an anticorrosive agent for mild steel in hydrochloric acid medium.

Materials

Mild steel strips that are mechanically cut into segments (strips) of dimensions 5 cm x1 cm x 0.2cm, with a hole (2mm) which has a uniform diameter length at one of its end for hooking. The composition of strip is as follows: 0.256 % Mn, 0.034% C, 0.004% P, 0.023 % Si and the remainder Fe. For studies in electrochemistry, these specimens of the same composition were made by fixing the mild steel of size 1 cm^2 to a mild steel rod with a diameter 1mm utilizing araldite. Every piece was polished with various grades of emery paper and was degreased with acetone and was washed with some distilled water, dried and was stored in the desiccators. Weight of these specimens (strips) was given utilizing electronicbalance. Analar grade HCL acid and double time distilled water were utilized to make all the solutions. ExpiredGentamicin (Eye/Ear Drops)drug obtained from medical shop and used for this study without any further purification.



Gentamicin

RESULTS AND DISCUSSION

Weight loss method

Initial weights ofthe pretreated example's are noted triplicate was inundated in the 100 ml (1M HCl) solution in nearnessandnonappearance of the inhibitors at various concentrations (0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, and 1.0%) for the period of 0.5, 2, 4, 6, 8 and 24 hourswith the help of glass hooks after the experiment period the final weights were also noted and also studiedimpact of temperature on mild steel corrosionat fivedistinct temperatures ranging from 303K to 343K. From this the inhibition efficiency (IE %), rate of corrosion (mils Penetration per year), surface coverage (θ) were estimated by utilizing the following formulae.

$$IE(\%) = \frac{W_U - W_I}{W_U} \times 100$$

Where, $W_I$and W_U are weight losses in acids with and without inhibitors respectively.

$$\text{Corrosion rate}\,(\text{mmpy}) = \frac{87.6 \text{ x weight loss}\,(\text{mg})}{\text{density}\,(\text{gm/cc}) \text{ x area}\,(\text{cm}^2) \text{ x time}\,(\text{hours})}$$

Corrosion parameters obtained from this method are given in Tables 1 & 2. It uncovers that, the inhibition efficiency incremented with the increase in temperature,concentration and immersion period of inhibitor asit is shown in the Figs 1 & 2**.** The maximum inhibition efficiency 92.36% was noticed in 6 hours immersion period at room temperature (303 K) with concentration of 0.9% .This outcome shows that the inhibitor could act as effective corrosion inhibitor for mild steel in 1M HCl medium.

Potentiodynamic Polarization method

Electrochemical analyzer was used for Potentiodynamic polarization measurements. These estimations were made to assess the corrosion potential, corrosion current and Tafel slopes. A conventional three

Table.1: Influence of concentration of Expired Gentamicin inhibitor on the corrosion of 1M HCl at room temperature (303 K) with different time periods

Time Inhibit. or Concentration (%V/V)	1/2hours CR (mpy)	IE (%)	2 hours CR (mpy)	IE (%)	4 hours CR (mpy)	IE (%)	6 hours CR (mpy)	IE (%)	8 hours CR (mpy)	IE (%)	24 hours CR (mpy)	IE (%)
Blank	**514.52**		**590.34**		**633.18**		**647.62**		**699.58**		**714.34**	
0.1	472.69	40.22	500.21	44.36	506.64	49.28	514.68	55.23	546.24	52.36	564.38	43.26
0.2	408.64	47.65	395.64	53.26	391.12	57.23	386.98	60.21	402.82	58.65	445.67	49.68
0.3	321.76	55.36	314.06	60.25	306.16	64.65	293.19	67.64	308.19	65.23	348.68	58.31
0.4	249.08	63.21	238.18	66.21	229.06	69.36	221.98	72.63	240.68	70.26	279.98	62.35
0.5	187.23	65.23	180.64	68.65	174.33	72.25	166.82	75.34	184.96	73.65	223.14	67.32
0.6	146.72	67.54	138.21	71.26	130.28	74.65	123.04	78.26	142.08	76.31	180.86	69.11
0.7	138.66	69.28	127.82	74.63	124.67	78.56	102.98	83.69	126.82	79.98	139.68	73.01
0.8	124.28	73.02	121.36	77.65	115.29	83.99	98.89	88.69	111.21	85.02	125.68	76.99
0.9	119.66	76.65	112.99	80.36	106.28	86.23	95.29	92.36	107.08	88.55	116.69	80.64
1.0	126.78	74.36	118.87	78.21	116.72	83.26	110.88	87.66	116.66	85.06	146.95	74.66

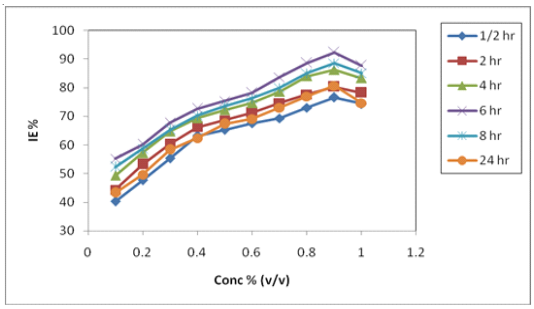


Fig.1. Gentamicin inhibitor Inhibition efficiency with various concentrations and time periods at the room temperature (303 K)

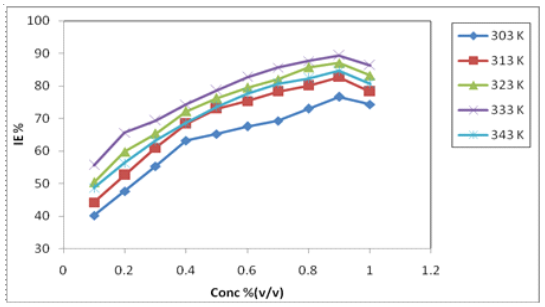


Fig. 2. Gentamicin inhibitor Inhibition efficiency with various concentrations and temperatures

Table. 2: Effect of temperature on the corrosion of mild steel in the presence of difference concentration of Expired *Gentamicin* inhibitor in 1M HCL

Temperature Inhibit or concentration (%V/V)	303K3 CR (mpy)	IE (%)	13K CR (mpy)	IE (%)	323K CR (mpy)	IE (%)	333K CR (mpy)	IE (%)	CR (mpy)	343K IE (%)
Blank	**514.52**		**1539.18**		**3519.25**		**8109.48**		**16527.78**	
0.1	472.69	40.22	1006.36	44.28	2432.69	50.36	5257.26	55.65	11063.23	48.65
0.2	408.64	47.65	821.63	52.64	1802.69	59.82	3806.23	65.63	7706.21	56.32
0.3	321.76	55.36	563.21	60.98	1698.31	65.32	2722.61	69.36	5423.65	63.21
0.4	249.08	63.21	390.23	68.36	1230.65	72.16	1960.23	74.32	3994.25	68.65
0.5	187.23	65.23	269.32	72.98	893.22	76.21	1426.33	78.61	2904.66	73.63
0.6	146.72	67.54	188.78	75.36	647.23	79.45	999.69	82.65	1725.36	77.65
0.7	138.66	69.28	169.08	78.22	499.29	82.01	785.25	85.65	1529.99	80.65
0.8	124.28	73.02	162.55	80.11	466.21	85.75	726.32	87.65	1389.45	82.31
0.9	119.66	76.65	158.36	82.65	438.62	87.06	665.32	89.31	1198.32	84.65
1.0	126.78	74.36	166.32	78.23	528.95	83.22	869.66	86.32	1456.01	80.65

electrode cell assembly which contains working electrode as the mild steel specimen with area 1 cm^2 which is exposed and rest covered with araldite, the rectangular Pt foil was used as counter electrode and theSCE as reference electrodes. To get the steady state open circuit potential for each test, ten to thirty of minutes time interval was given. Polarization measurements were given out from the cathode with potential of -450mV (vs. SCE) to an anode with potential of -250mV (vs. SCE)at a sweep rate of 10 mV per second. From the polarization curves corrosion potential, Tafel slopes and corrosion rates were calculated. The inhibitor efficiency was given by utilizing the following formulas.

Table. 3: Electrochemical polarization (Tafel) parameters for the corrosion of mild steel in 1M HCL without and with various concentrations of inhibitor at room(303 K) temperature

conc. (%)	$-E_{corr}$ (mV)	I_{corr} (mA/cm^2)	I_{corr} IE (%)	b_a (mV/dec)	b_c (mV/dec)	$R_p\Omega cm^2$)	R_p IE (%)
Blank	346.8	0.996	-	70.0	114	18.92	-
0.4	306.0	0.355	64.16	77.0	107	54.86	65.51
0.7	304.5	0.329	66.90	70.0	110	56.27	66.38
0.9	305.1	0.249	75.06	68.0	109	73.15	74.13

$$\text{Inhibition efficiency} = \frac{I_{corr} - I_{corr\,(i)}}{I_{corr}} \times 100$$

$$\text{Inhibition efficiency} = \frac{R_{p\,(i)} - R_p}{R_{p(i)}} \times 100$$

Where, I_{corr} and $I_{corr(i)}$ are the corrosion current in absence and presence of the inhibitor, similarly R_p and $R_{p\,(i)}$ arethe polarization resistance in the absence and presence of the inhibitor.

Polarization studies observed that when concentration of inhibitor increased from 0.4% to 0.9% the I_{corr} values decreased 0.996 mA/cm^2 to 0.249 mA/cm^2 with inhibition value increase from 64.16% to 75.06% and also (R_p) values increased from 18.92Ùcm^2 to 73.15Ùcm^2 with increase of inhibition efficiency from 65.51% to 74.13%. Tafel polarization behaviors of the mild steel 1M HCL shows in the Fig 3. From the graph we can observe that the inhibitor behaves like mixed type [24-28]. In the Table 3. All the calculated data of Polarization studies was given.

Electrochemical Impedance methods

In the estimations of electrochemical impendence, cell same as utilized for potentiodynamic polarization. An AC potential 50 mV was super forced on the consistent open circuit potential. The genuine part (Z') and the fanciful part (Z") were measured at different frequencies in the scope of 10 kHz to 10MHz. The genuine and imaginary parts of the impendence were plotted in Nyquist plots. From the plots of Z' versus Z," the charge transfer resistance (R_{ct}) quantities were acquired. The estimation of (R_t + R_s) compares to the point where the plot cuts z' and at a higher frequency the distinction amongst Rt and Rs gives the charge exchange resistance R_{ct} values. The dowble layer capacitance C_{dl} quantities were acquired from the condition

1A=BLANK, 1B=0.4%, 1C=0.7%, 1D=0.9%

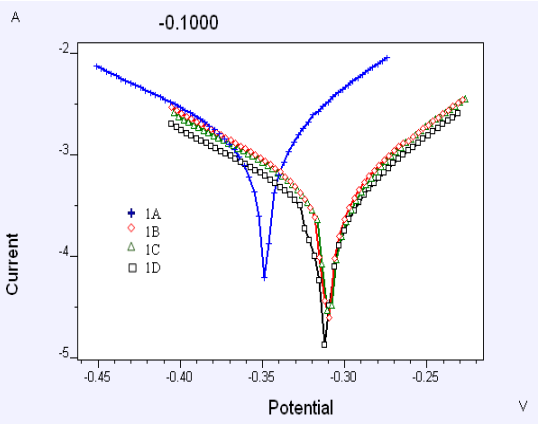


Fig. 3. Potentiodynamic polarization technique diagram for mild steel in 1M HCl in the absence and presence of different concentrations of the inhibitor

$$C_{dl} = \frac{1}{2\pi f_{max} R_{ct}}$$

Where, C_{dl} means double layer capacitance
R_{ct} means charge transfer resistance
f_{max} means frequency at Z" value maximum.

The inhibition efficiencies were got from R_p and R_{ct} quantities as follows

$$\text{Inhibition efficiency} = \frac{R_{P\ (i)} - R_p}{R_{p\ (i)}} \times 100$$

Where, $R_{ct}$and $R_{ct\ (i)}$are charge transfer resistance in the absenceand presence of the inhibitor Nyquist plots shows in the Fig. 4 for the measurements of the genuine part (Z') and imaginary one (Z"). We can conclude the corrosion is mostly controlled by the process of charge transfer from semi-circle curves of the impedance. In Table.4, parameters for impedance of mild steel in 1M HCL in the presence and absence of the inhibitor are shown. The values of R_{ct} had increased from 3.43 Ùcm² to 15.91 Ùcm² and simultaneously the values of C_{dl} had decreased 438ìF/Cm² to 88 ìF/Cm² with the increase in inhibitor concentrations.

Table. 4: Electrochemical impedance parameters for the corrosion of mild steel in 1M HCl without and with various concentrations of inhibitor at room(303 K) temperature

Conc.(%)	R_{ct}(Ωcm²)	C_{dl}µF/cm²	IE(%)
blank	3.43	438	-
0.4	9.21	132	62.76
0.7	12.95	164	73.51
0.9	15.91	106	78.44

1A=BLANK, 1B=0.4%, 1C=0.7%, 1D=0.9%

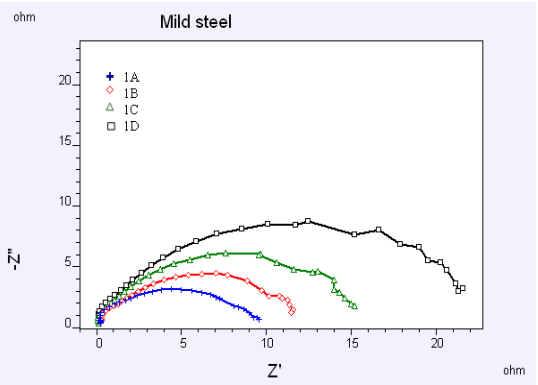


Fig. 4.Impedance technique for mild steel diagram in 1M HCl in the absence and presence of different concentrations of the inhibitor

Thermodynamic Consideration

From the following Arrhenius equation the activation energy with different concentrations at various temperatures was calculated.

$$\text{Log}\frac{\rho_2}{\rho_1} = \frac{E_a}{2.303 \times R}\left[\frac{1}{T_1} - \frac{1}{T_2}\right]$$

Where, ñ$_1$corrosion rate at T_1 and ñ$_2$ at T2 temperature. 'R' is a real gas constant.

The change in free energy at different temperatures comparison with room temperature for various concentration of inhibitor was calculated using the following equation

$$\Delta G_{ads} = -2.303 \times 8.314 \times T \times \text{Log}\left(K \times 55.5\right)$$

$$K = \frac{\theta}{(1-\theta)C}$$

Where, Surface coverage (è), Concentration (C), Temperature (T) & Equilibrium constant (K).The change in enthalpy of adsorption ("H) and change in entropy of adsorption ("S) can be calculated by using the following equations.

$$^{\Delta}H = E_a - RT$$

$$\Delta S = \frac{\Delta H - \Delta G}{T}$$

The values for mild steel in 1M HCl acid with and without the inhibitor of the activation energy (Ea), free energy ("Gads), the entropy ("S) and the enthalpy ("H) are calculated and are shown in Table 5. Value of activation energy (Ea) of blank is 73.83 kJ mol^{-1} and 51.66 kJ mol^{-1} for 0.9% inhibitor concentration, it's now clear that the addition of the inhibitor leads to decrease in the obvious activation energy (Ea) with some value lesser than the value of uninhibited solution which is trailed by the dull decline with the incrementation in the concentration of the inhibitor which points out the action upon mild steel in the 1M HCl acid takes place through via the chemical adsorption. Negative value of free energy of the adsorption ("Gads) points out adsorption which is spontaneous. +ve values of

enthalpy recommend that high temperatures will favor the complexation process and same in fantastic agreement with the incrementing stability with the temperature. The negative estimations of ÄSact indicated a greater order which is generated during the activation process. This can be accomplished by the creation of initiated complex and it shows the affiliation or fixation with the resulting loss in the degrees of the freedom of amework(system) in this procedure. This also supports suppositionof the chemical adsorption. Figure 5 illustrates the Arrhenius plot for the mild steel dissolution in the1M HCl acid without and with the inhibitor at various temperatures.

Adsorption Consideration

Corrosion Inhibition is identified with the adsorption of inhibitor mol on metal surface. Surface coverage (θ) = IE/100. In order to find out the adsorption isotherm for present review, a plot C/θ and C ought to be straight. This is demonstrative the adsorption takes after Langmuir adsorption isotherm. These isotherms give the best fit with the correlation co efficient almost near to (0.9989) unity. In Fig. 6 shows a straight line showing that Langmuir adsorption isotherm. Organic molecules contain polar atoms which are adsorbed on metal surface and interact with mutual attraction or repulsion. Because this plot is straight, the angles are never solidarity, Langmuir adsorption isotherm shown as:

$$C/\theta = 1/k + C$$

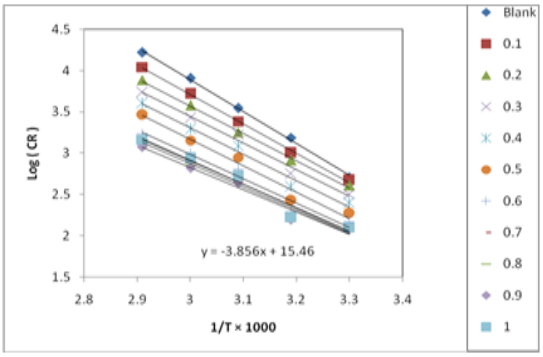


Fig. 5. Arrhenius plot for mild steel dissolution in 1M HCl from 303 K to 343 K temperature with and without inhibitor

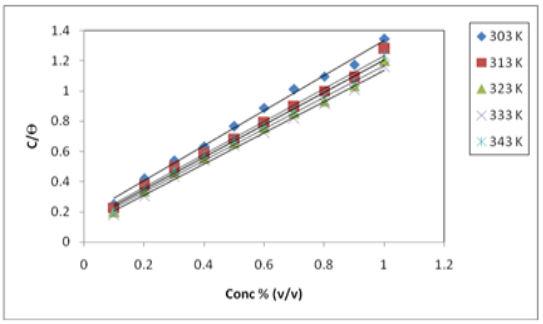


Fig. 6. Langmuir isotherm plot on mild steel surface in 1 M HCl solution for the adsorption of inhibitor

Table. 5: Thermodynamic data for mild steel corrosion in 1M HCl in the presence and absence of Expired *Gentamicin* inhibitor in 1M HCl for the period of ½ hour.

inhibit. or Conc. (%V/V)	Ea (KJ/mol)	- ΔG (KJ/ mol)										-ΔS (KJ/mol)	ΔH (KJ/mol)
temp		θ	303K	θ	313K	θ	323K	θ	333K	θ	343K	303K	303K
Blank	73.83												71.31
0.1	67.97	0.402	14.92	0.442	15.34	0.503	15.95	0.556	16.49	0.486	15.78	0.265	65.45
0.2	63.36	0.476	13.93	0.526	14.44	0.598	15.17	0.656	15.80	0.563	14.81	0.246	60.84
0.3	61.92	0.553	13.69	0.609	14.27	0.653	14.74	0.693	15.21	0.632	14.51	0.241	59.4
0.4	61.33	0.632	13.79	0.683	14.36	0.721	14.82	0.743	15.10	0.686	14.40	0.239	58.81
0.5	61.16	0.652	13.45	0.729	14.36	0.762	14.79	0.786	15.14	0.736	14.45	0.237	58.64
0.6	56.45	0.675	13.25	0.753	14.22	0.794	14.81	0.826	15.33	0.776	14.54	0.221	53.93
0.7	54.09	0.692	13.06	0.782	14.23	0.820	14.84	0.856	15.51	0.806	14.61	0.213	51.57
0.8	54.03	0.730	13.19	0.801	14.19	0.857	15.20	0.876	15.61	0.823	14.55	0.213	51.51
0.9	51.66	0.766	13.38	0.826	14.31	0.870	15.18	0.893	15.73	0.846	14.68	0.206	49.14
1.0	55.91	0.743	12.80	0.782	13.34	0.832	14.15	0.863	14.76	0.806	13.71	0.218	53.39

Table. 6: Inhibition efficiency competition of inhibitor for mild steel in 1M HCl from weight loss, polarization and impedance techniques for the period of ½ hour studies

	Inhibition efficiency (%)			
		Polarization		
Conc.(%)	Weight loss	I_{corr}	R_p	Impedance
0.4%	63.21	64.16	65.51	62.76
0.7%	69.28	66.90	66.38	73.51
0.9%	76.65	75.06	74.13	78.44

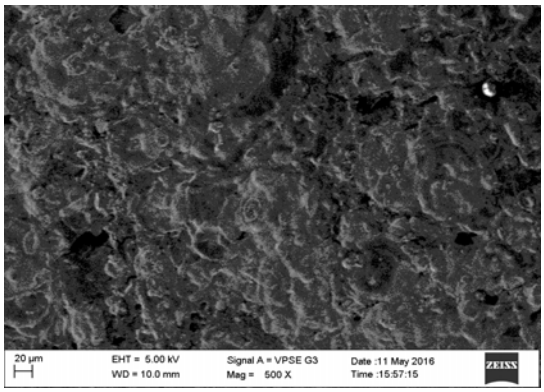


Fig. 7. The SEM photo of mild steel when immersed in 1M HCl without inhibitor

Morphology examination

The photos of mild steel in acid medium containing without and with the inhibitor are shown in Fig. 7& 8. It is watched that the assault of mild steel within the existence of inhibitor in 1 M HCl is less compared to the nonappearance of inhibitor, because of the presence of adsorbed layer of the inhibitor which blocks corrosion rate of metal evidently. This is ascribed to the contribution of the compound of the interaction of the inhibitor with active sites on surface of metal, thus results in enhanced surface coverage of the metal so that there is decrease in contact amongst aggressive medium and the metal.

CONCLUSION

The expired Gentamicin(Eye/Ear drops) pharmaceutical drug acts as excellent, efficient and economically benefitted inhibitor on the mild steel corrosion in the 1M HCl acid medium. The efficiency of the inhibitor is found to increase with period of immersion, temperatures and concentration.The Inhibitor with concentration (0.9%) showed

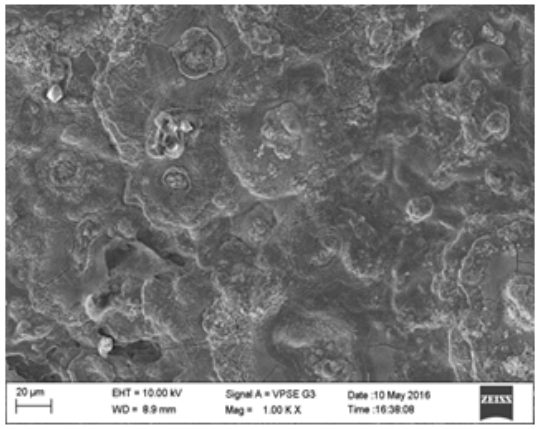


Fig. 8. The SEM photo of mild steel when immersed in 1M HCl containing 0.9% concentration of inhibitor.

maximum efficiency (92.36%) in 6h immersion period at room temperature (303K) and found sufficient for pickling. The chemical adsorption mechanism of the inhibition process was conformation with the data obtained; Langmuir adsorptions were best fitted into the obtained results. Thermodynamic parameters like activation energy (Ea) values also conformed to a chemical adsorption mechanism. Free energy (Gads) of absorptionvalues are negative which pointed out spontaneous adsorption of the inhibitor on the metal surface. The positive values of Enthalpy are proposing high temperature favors inhibition efficiency. The semi circle curves of impedance points that corrosion is mainly controlled by phenomena of charge transfer. High performance of the inhibitive effect on the surface of mild steel has confirmed by SEM morphology of protective film. Obtained results in electrochemical and weight loss studies are very much excellent agreement with each other. This experiment promotes eco-friendly and economically benefitted inhibitor and also avoids pollution and recycles the drugs. Inhibition efficiency competition of inhibitor for mild steel in 1M HCl for three difference methods is given in the Table 6.

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