

**INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH
TECHNOLOGY****EVALUATION OF POLYSORBATE 20 AS CORROSION INHIBITOR FOR MILD
STEEL IN ACIDIC MEDIUM AND THE SYNERGISTIC EFFECT OF A
CONDUCTING POLYMER, POLYETHOXYANILINE****Namita K.Johar¹, Amarpreet K Kalra², Kalpana Bhrara³ and Gurmeet Singh^{*4}**¹Applied Science Department, Maharaja Agrasen Institute of Technology, India²Department of Chemistry, S.G.T.B Khalsa College, University of Delhi, India³Department of Chemistry, Kirori Mal College, University of Delhi, India^{*4}Department of Chemistry, University of Delhi, India

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ABSTRACT

Polysorbate 20 (Poly-20) was evaluated as a corrosion inhibitor for mild steel (MS) in 0.5M sulfuric acid by weight loss and polarization techniques at different concentrations and temperatures. The inhibition efficiency increases with the decrease in concentration and was found to be maximum at 0.25% (v/v). The Nyquist plots were used to get the values of R_s (series resistance) and C_{dl} (double layer capacitance). The corrosion data has been analysed to determine the surface coverage at different temperatures. The effective activation energy values, E_{eff} has been reported. Poly-20 follows EI-Awady adsorption isotherm. The effect of Polyethoxyaniline (PEA), a conducting polymer mixed with equal volume of Poly-20 (0.25% (v/v)) on the corrosion inhibition of mild steel in 0.5M sulfuric acid has also been investigated. The inhibition efficiency was found to be higher with the conducting polymer solution than in Poly-20. The surface morphology of mild steel samples in absence and presence of PEA in Poly-20 were also examined using Scanning Electron Microscopy. Quantum chemical analysis of the inhibitors was carried out to supplement the results obtained from various other techniques.

Keywords: *Corrosion Inhibition, Mild Steel, Polysorbate 20, Polyethoxyaniline, Scanning Electron Microscopy (SEM), Nyquist plot, Quantum Chemical Analysis.*

I. INTRODUCTION

Polymers have a wide range of applications that far exceeds that of any other class of material available to man. Polymers have fascinated corrosion chemists due to following reasons:

- Larger molecular area and weight which allows better adsorption of the polymeric film on the surface of the metal.
- The polymeric chain can displace the water molecules from the metal surface thereby lowering the entropy of the system.
- The polymer has a multiple bonding sites which makes desorption of the polymer a slow process leading to a stronger and effective polymeric film on the surface of the metal.
- The polymers having cyclic rings and heteroatoms which have active centers of adsorption and hence show better inhibitive properties.

The conducting polymers like Polyacetylene, Polyaniline, Polypyrrole and Polythiophene have also shown anticorrosive properties by forming films on the metal surface and preventing them from getting corroded [1-15]. Many investigations on synergistic inhibition are also available in the literature. [16-22]. In the present study, Poly-20 is evaluated as corrosion inhibitor for MS in sulfuric acid by weight loss studies, electrochemical polarization studies, SEM and Quantum chemical studies. To further the study, a solution of poly-20 mixed with equal volumes of PEA is also evaluated for synergism.

II. EXPERIMENTAL

1. Preparation of Working Electrode and Polymer Solutions

The working electrode was soldered with insulated copper wire and was coated thoroughly with epoxy resin keeping some surface exposed for corrosion. The exposed mild steel surface (1cm x 1cm) was grinded using 150, 320, 400, 600 and finally polished with 1000 grade emery papers.

Poly-20 was prepared at three different concentrations (v/v) of 0.25%, 0.5% and 1%.

2. Preparation of Conducting Polymer: PEA

14.4 ml of 0.1M Phenacetidin and 89.0 ml of 0.1M HCl were mixed in a 1 litre flask, the mixture was stirred for about 4 hours with subsequent addition of Ammonium Peroxodisulphate for 2 hours, in water chiller at 0°C. Blackish red precipitates were obtained which were washed with acetone and were allowed to dry in the desiccator for 48 hours. Its saturated solution was prepared in 0.5M H₂SO₄, which was then mixed in equal volume with 0.25% of the polymer used [23].

3. Weight Loss Studies

Polished and washed mild steel coupons (1cm x 1cm x 1cm) were dried in a desiccator and finally weighed. The test specimens were dipped in 0.5M sulfuric acid, 0.25% Poly-20, 1% Poly-20 and 0.25% Poly 20 + PEA (equal volumes) under the thermostatic conditions of 308K and 338K. The weight losses of the coupons were evaluated after the immersion time of 6 hours.

4. Potentiodynamic Polarisation Studies

The potential of the metal electrode Vs saturated calomel electrode (SCE) was measured with the help of a galvanostat having the range of 10mA to 100mA. A constant distance of approximately 1-2mm between the tip of luggin capillary and working electrode surface was maintained throughout the experiment. The solutions of Poly-20 at 0.25%, 0.5%, 1% concentration and 0.25% Poly 20+PEA (equal volumes) in 0.5M sulfuric acid were used for studies.

5. Potentiostatic Polarisation Studies

Steady state potentiostatic polarisation was done using the potentiostat by applying potentials of 40mV increments after every one minute and the corresponding current values were recorded one minute after the potential was applied. The anodic polarization experiments were repeated for each set and the reproducible data have been recorded.

6. Scanning Electron Microscopy

Polished specimen, which had a smooth pit free surface were subjected to corrosion exposure. After that these specimens were washed with acetone and kept in dessicator for 24 hours. MS coupons were dipped into the solution of sulfuric acid, 0.25%, 1% of Poly-20 and 0.25% Poly 20+PEA (equal volumes) in 0.5M sulfuric acid. These were then dried in the desiccator and subjected to scanning electron microscopic examination (SEM).

7. Impedance Measurements

Electrochemical Impedance is measured by electrochemical analyzer by applying an A.C potential to an electrochemical cell and measuring the current through the cell. The Nyquist plot is obtained. Various parameters like charge transfer resistance, R_{ct} and double layer capacitance C_{dl} have been calculated.

8. Temperature Kinetic Studies

Different inhibitors follow different adsorption patterns, which can be described by means of various adsorption isotherm models, eg. Langmuir, El-Awady, Temkin, Freundlich, Frumkin and Flory Huggins adsorption isotherm etc. Surface coverage (θ) was evaluated and different adsorption isotherms were tried.

Energy of activation is calculated by using the Arrhenius form, which is as follows:

$$k = Ae^{-E_a/RT}$$

where notations have their usual meanings. Since k is proportional to I_{corr}, from the plot of log I_{corr} vs 1/T, E_a is calculated.

9. Quantum chemical calculations

In the present study, Hyperchem Standard 7.5 for windows (Hypercube Inc. Florida) was used to create the initial geometries of Poly-20 and PEA. The geometries of these molecules were fully optimized by using MM⁺ (Molecular Mechanics) and then through AM1 (Austin Model 1). Information obtained from these calculations includes energy optimized molecular geometry, electron density and molecular orbital configurations, etc.

III. RESULTS AND DISCUSSION

1. Weight Loss Studies

Results of the effect of Poly-20 (0.25% and 1%) on the MS in 0.5M H₂SO₄ and 0.25% Poly-20 + PEA (equal volumes) in 0.5M sulfuric acid under the thermostatic conditions of 308K and 338K are shown in table 1. A linear variation was observed for the plot of logarithm of weight loss against time upto 6 hours (Fig.1). This confirms a first order reaction kinetics with respect to the mild steel in H₂SO₄ solution. The rate constant was calculated by using the formula :

$$k = 2.303 \log (w)/T \text{ in hours}$$

Where, w = Initial weight/Final weight

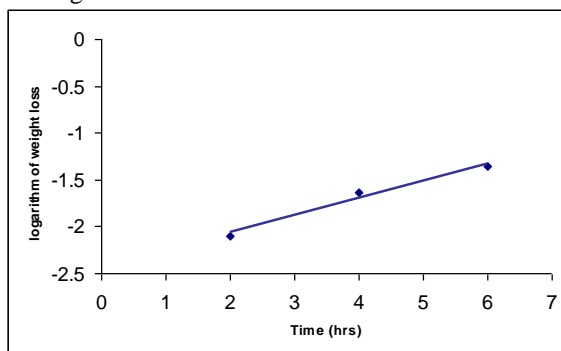


Figure 1. The plot of logarithm of weight loss against time (up to 6 hours) in 0.5M H₂SO₄

Table 1: Weight Loss and Kinetic parameters of MS in 0.5M H₂SO₄ in presence of Poly-20 and when mixed with PEA

Solutions	Weight loss / g (308 K)	Weight loss/g (338 K)	k/ hr ⁻¹ (308K)	k/ hr ⁻¹ (338K)	I% (308K)	I% (338K)
0.5MH ₂ SO ₄	0.10346	0.32568	0.0022683	0.071041		
0.25% Poly-20	0.01500	0.09504	0.0003322	0.002085	85.8	70.8
1% Poly-20	0.03001	0.11829	0.000668	0.002682	70.9	63.6
Poly-20+PEA	0.0020	0.02000	0.00008988	0.0009038	98	93.8

The inhibition efficiencies decreased with increase in the concentration of the inhibitor solutions. The rate constants at 308K and 338K show the same trend. The rate constant value decreased considerably on addition of PEA to Poly-20. At 338K the rate constant was found to be higher than at 308 K

2. Potentiodynamic Polarisation Studies

The representative Tafel plots of MS in 0.5M H₂SO₄ in the presence of Poly-20 and Poly-20+ PEA are shown in Fig 2 and various corrosion parameters in table 2. The *i*_{corr} were determined from the plots and Inhibition Efficiencies were calculated using :

$$IE\% = \left[\frac{i_{corr}^{(a)} - i_{corr}^{(i)}}{i_{corr}^{(a)}} \right] \times 100$$

where, i_{corr}^a = the corrosion current density of uninhibited solution and i_{corr}^i = the corrosion current density of inhibited solution.

The inhibition efficiencies were found to decrease with increase in concentration and temperature. When mixed with equal volume of conducting polymer solution (PEA), the inhibition efficiency of Poly-20 increases from 85.8% to 96% at 308K. The weight loss results are in direct agreement with the electrochemical results. From table 2, this inhibitor shows no appreciable shift of E_{corr} towards any direction, even when mixed with PEA. It indicates that this inhibitor acts as a mixed type of inhibitor [24].

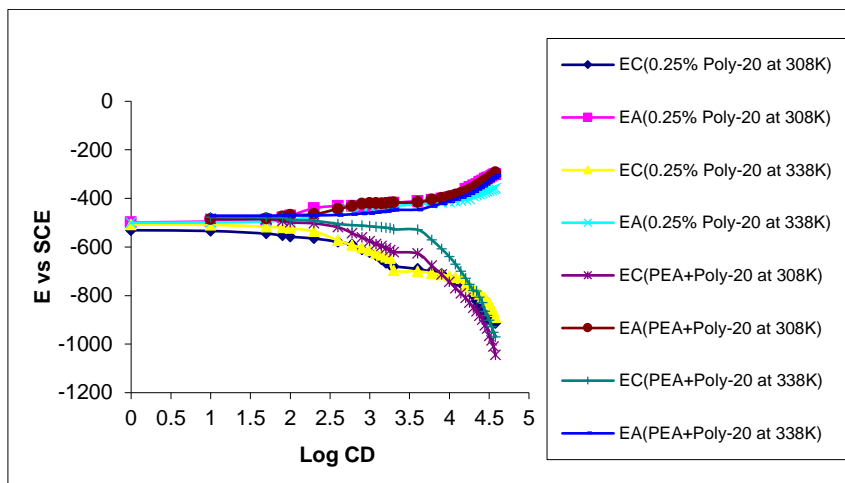


Figure. 2 Potentiodynamic Polarisation curves for MS in 0.5M H₂SO₄ in 0.25% of Poly-20 and Poly-20 +PEA at different temperatures (EA is anodic polarization and EC is cathodic polarization curves)

Table 2 Corrosion parameters of MS in 0.5M H₂SO₄ in various concentrations of Poly-20 and Poly-20 + PEA at different temperatures

Conc.(%)	Temp.	E _{corr} (mV)	Log I _{corr} (mAcm ⁻²)	bc (mV/dec)	ba (mV/dec)	I%
0.00	308K	-500	3.000	80	65	
0.25		-485	2.150	155	30	85.8
0.50		-510	2.300	160	50	80.0
1.00		-505	2.450	210	30	71.8
Poly-20 +PEA		-490	1.600	200	35	96.0
0.00	318K	-510	3.150	80	75	
0.25		-530	2.350	170	10	84.1
0.50		-540	2.500	170	60	77.6
1.00		-500	2.600	210	55	71.8
0.00	328K	-520	3.400	35	30	
0.25		-530	2.750	200	40	77.6
0.50		-550	2.800	170	50	74.8
1.00		-550	2.900	155	40	68.3
0.00	338K	-520	3.500	40	20	
0.25		-540	2.950	190	30	71.8
0.50		-500	3.000	220	75	68.3
1.00		-530	3.075	170	60	62.4
Poly-20+ PEA		-485	2.500	165	60	90.0

In presence of PEA the electrons of the pi-bonds are able to jump around the polymer chain and can easily interact with the d-electrons of the metal leading to good adsorption, thereby increasing the corrosion inhibition efficiency.

4. Surface Morphological (SEM) Studies

Scanning Electron micrographs of plain MS and MS exposed to various solutions are shown in Fig 4 (a)-Fig 4 (e). The Fig 4 (a) shows the micrographs of polished plain MS which is free from any pits and cracks whereas Fig 4(b) representing the corrosion products accumulated on the MS surface when exposed to 0.5M H₂SO₄. Fig 4(c) and (d) shows that the extent of corrosion is less in presence of Poly-20 and it is least on the MS surface in Poly-20 + PEA. The micrographs Fig 4(e) shows smoother surface which is free from pits and cracks.

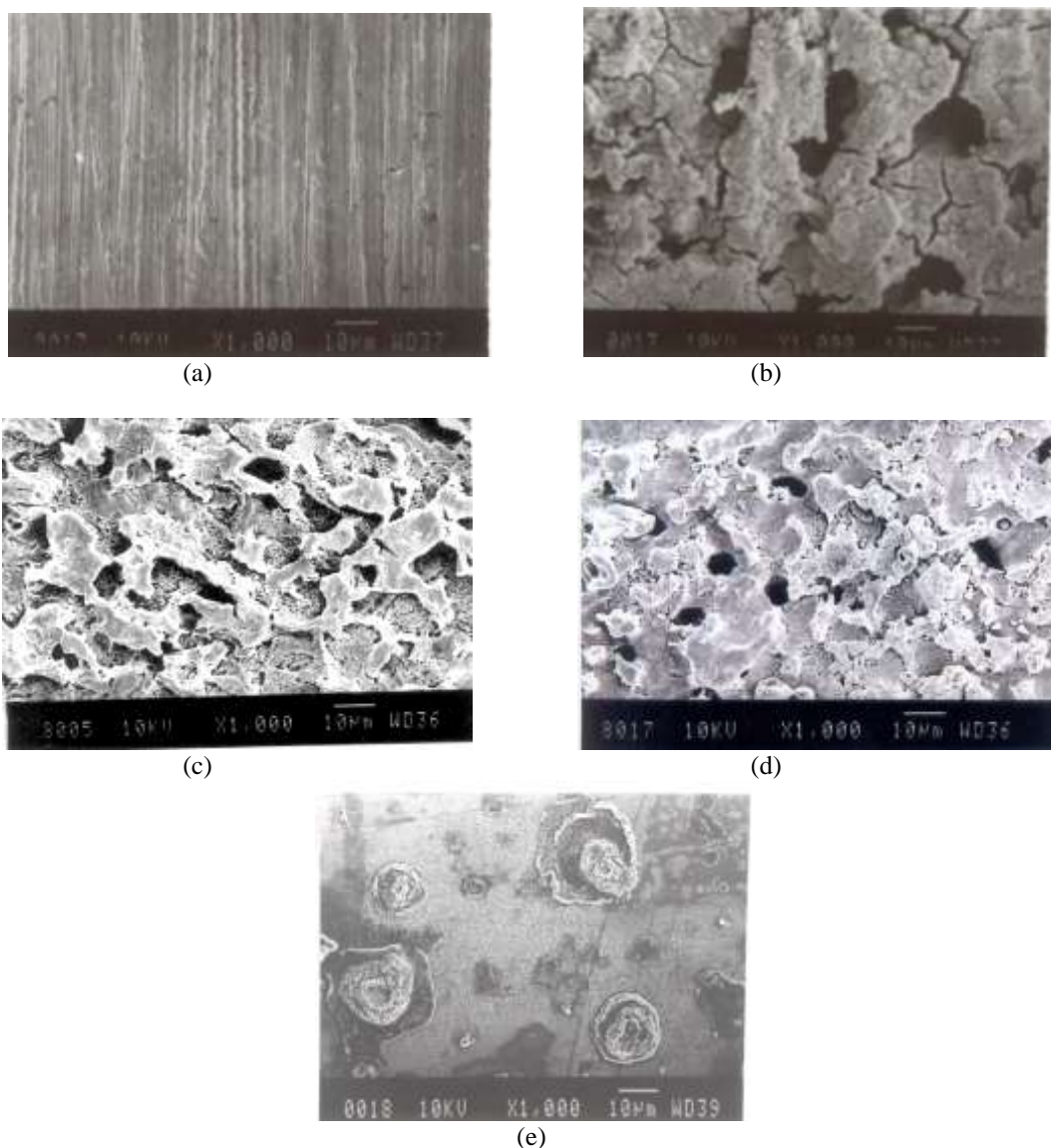


Fig. 4 SEM image of surface of (a) Plain MS (b) MS in 0.5 M H₂SO₄ (c) MS in 0.25% Poly-20 in 0.5 M H₂SO₄ (d) MS in 1% Poly-20 in 0.5 M H₂SO₄ (e) MS in Poly-20+ PEA at 1000 magnifications

5. Impedance Measurements

Electrochemical impedance measurements were carried out in the frequency range of 0.01mHz to 1mHz with 0.01V peak to peak amplitude at the open circuit potential. Fig 5 shows the Nyquist plot for the corrosion of MS in 0.5M H₂SO₄ and in the presence of Poly-20.

In these plots, the curves showing the black points represent the corrosion of MS in 0.5M H₂SO₄ in the absence of the inhibitor and the blue points represent the corrosion in the presence of the inhibitor.

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ICTM Value: 3.00

The impedance parameters namely the double layer capacitance (C_{dl}), charge transfer resistance (R_{ct}) and the inhibition efficiencies were calculated from the graphs and are given in table 5.

The inhibition efficiency of the inhibitors was calculated using the following relation,
 $I\% = [R_{ct}(i) - R_{ct}(a)]/R_{ct}(i) \times 100$

The double layer capacitance was calculated using the following relation,
 $C_{dl} = 1/2\pi f R_{ct}$

Where, f is the frequency at the maximum height of the semicircle on the imaginary axis.

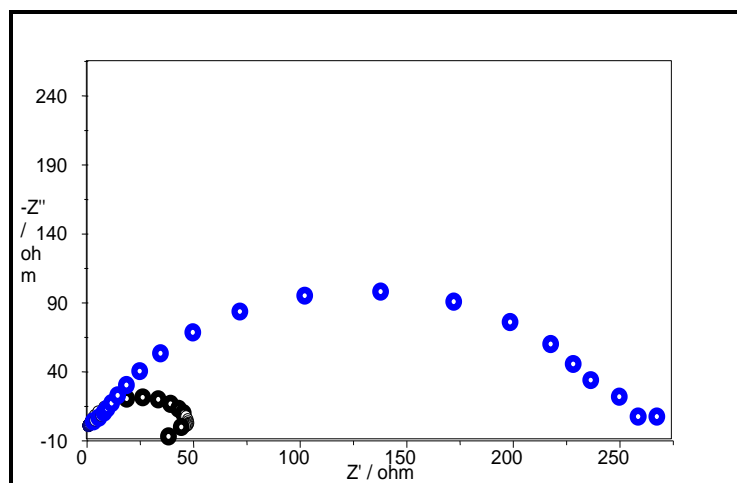


Figure 5: Nyquist Plot for the Corrosion of MS in 0.5M H₂SO₄ and in the presence of Poly-20

Table 5: Impedance Parameters for the Corrosion of MS in 0.5 M H₂SO₄ in the presence of Poly-20

Solution	$R_s(\Omega\text{cm}^2)$	$R_{ct}(\Omega\text{cm}^2)$	F (Hz)	$C_{dl}(\text{Fcm}^{-2})$	I%
0.5M H ₂ SO ₄	1.96549	47.3900	7.44	4.55×10^{-4}	
0.25% Poly-20	5.26794	250.100	3.10	2.0×10^{-4}	81.0

The impedance response of mild steel in 0.5M H₂SO₄ changes significantly on inhibitor addition. In presence of Poly-20, the diameter of the capacitive loop increases as compared to the acid, which is an indication of the inhibition of the corrosion process. The value of charge transfer resistance increases that in turn leads to a decrease in the i_{corr} for mild steel in the acidic solution. As the impedance diagrams for solutions examined have almost a semi-circular appearance, it indicates that the corrosion of steel is of course controlled by a charge transfer process. In the present case, the double layer capacitance value decreases on addition of Poly-20 revealing that a complete film is formed on the metal surface when dipped in the lowest concentration of the inhibitor solution, which further indicates the adsorption of the inhibitor on the metal surface.

6. Temperature Kinetic Studies

Corrosion and surface coverage parameters of MS in 0.5M H₂SO₄ in the presence of Poly-20 are shown in table 6. Different adsorption isotherms like El-Awady, Temkin, Freundlich, Frumkin and Flory Huggins were tried. The best fit adsorption isotherm was found to be El-Awady adsorption isotherm, the plot of $\log \theta/1 - \theta$ against $\log \text{conc.}$ For the MS in 0.5M H₂SO₄ in the presence of Poly-20 at different temperatures is shown in Fig 6 and the slope and correlation coefficient values calculated from El-Awady isotherm for Poly-20 is shown in Table 7. The mean R^2 value was found to be 0.9840 proving that the adsorption of Poly-20 in 0.5M H₂SO₄ follows El-Awady isotherm.

The logarithm of I_{corr} against $1/T$ was plotted for MS in 0.5M H₂SO₄ in the presence of Poly-20 at different concentrations as shown in Fig 7. The slope of this curve gives the value of effective activation energy and was found to be 7.809 kJ/mol. Table 8 gives the value of E_{eff} for the corrosion of MS in 0.5M H₂SO₄ in presence of Poly-20.

This value suggests that Poly-20 adsorbs on the MS surface through Physio-sorption. The value of activation energy was found to be higher in presence of lowest concentration of the polymer suggesting that the rate of formation of corrosion products is slow in 0.25% concentration of Poly-20.

Table 6. Corrosion Parameters and Surface Coverage of MS in 0.5M H₂SO₄ in the presence of Poly-20

Conc. (%)	Temperature	<i>i</i> _{corr} (mA.cm ⁻²)	I%	θ	θ/1-θ
0.00	308K	3.000			
0.25		2.150	85.8	0.858	6.042
0.50		2.300	80.0	0.800	4.000
1.00		2.450	71.8	0.718	2.540
0.00	318K	3.150			
0.25		2.000	84.1	0.841	5.280
0.50		2.500	77.6	0.776	3.460
1.00		2.600	71.8	0.718	2.540
0.00	328K	3.400			
0.25		2.450	77.6	0.776	3.464
0.50		2.800	74.8	0.748	2.960
1.00		2.900	68.3	0.683	2.150
0.00	338K	3.500			
0.25		2.600	71.8	0.718	2.546
0.50		3.000	68.3	0.683	2.150
1.00		3.075	62.4	0.624	1.650

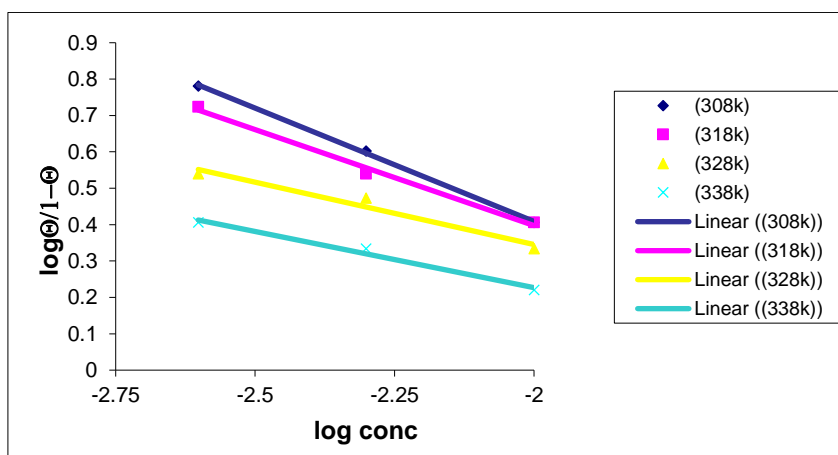


Fig 6. Curve Fitting of the Plot log θ/1-θ against log Conc. for MS in 0.5M H₂SO₄ in the presence of Poly-20 at Different Temperatures (To check El-Awady isotherm)

Table 7. Slope and Correlation Coefficient Values Calculated from El-Awady Isotherm for Poly-20

Temp.	Slope (y)	R ²
338K	-0.3088	0.9841
328K	-0.3427	0.9609
318K	-0.5274	0.9918
308K	-0.6233	0.9993

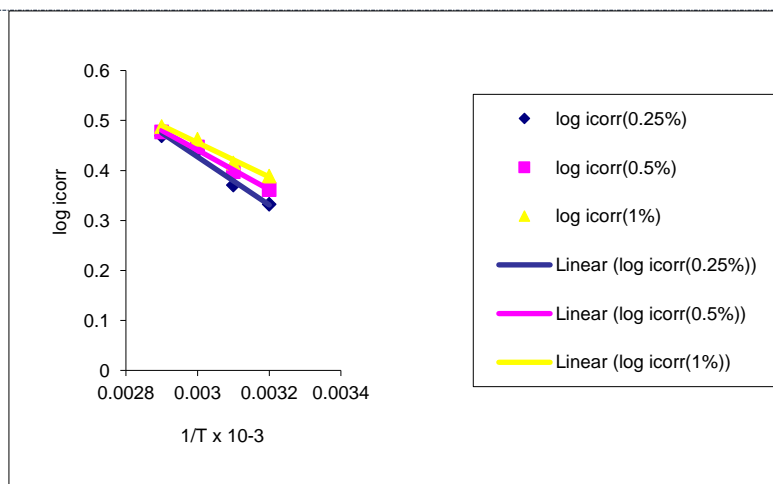


Figure 7: Plot of $\log i_{corr}$ against $1/T$ for MS in $0.5M H_2SO_4$ in the presence of Poly-20 at Different Concentrations

Table 8 Calculated Values of E_{eff} for Corrosion of MS in $0.5M H_2SO_4$ in the presence of Poly-20

Conc. (%)	E_{eff} (kJ/mole)
0.25	9.2800
0.50	7.5700
1.00	6.5770

7. Quantum Chemical Calculations

Figures 8(a) & 9 (a) shows the optimized geometries of the Poly-20 and PEA as ball and stick models and the various optimized parameters calculated are given in table9

Red colour denotes oxygen, blue denotes nitrogen, cyan denotes carbon and yellow denotes hydrogen.

Figures 8 (b) & 9(b) show the electrostatic potential mapped on the 3-D charged density isosurfaces for Poly-20 and PEA.. These figures give us the pattern in which the electron density is spread on the molecule. **Green colour denotes the positive charge density and pink colour negative charge density.** The negative clouds on the molecules contribute to donation to the metal surface. From these figures it can be easily traced out that the donation is mainly through the heteroatoms i.e. nitrogen and oxygen. There is an extensive charge distribution on the PEA molecule, which may help in improving the inhibition efficiency to a great extent.

Figures 8(c) &9(c) gives the numbering of the atoms in the molecule and figures 8(d) &9(d) gives the respective charges on the numbered atoms. The adsorption would take place from that atom which has the highest negative charge density.

Dipole moment values also give us an idea about the inhibition efficiency. Higher the value of dipole moment, higher is the extent of polarization, and greater is the tendency of donation of electrons. Therefore, we can say that greater is the dipole moment, greater is the inhibition efficiency. However, in the present case, there is a contradiction in the results because there are other factors like behaviour of molecules in the solvent and mechanism of adsorption, which play an important role in the mechanism of corrosion inhibition.

Positive heat of formation of PEA suggests that the formation of the inhibitor molecule is not spontaneous, as it requires energy for its formation. Therefore, it can split apart into its components on adsorption leading to lesser stability. Similarly, negative binding energy suggests that Poly-20 and PEA molecules are stable. PEA was found to be more stable than Poly-20.

Table 9 Optimized AMI parameters for inhibitors using Hyperchem7.5

Solutions → Parameters↓	Poly-20 N=1, but (OCH ₂ CH ₂)=8	PEA (n=4)
No. of electrons	160	160
E_{HOMO} (eV)	-10.2598	-8.6976
E_{LUMO} (eV)	1.1950	-1.2198

Binding Energy(kcal/mol)	-5631.21	-9133.8
Heat of formation (kcal/mol)	-433.22	180.04
Dipole Moment(Debye)	4.113	4.654
$E_{LUMO}-E_{HOMO}$	11.4548	7.4778
Inhibition Efficiency (at 308K)	85.8	

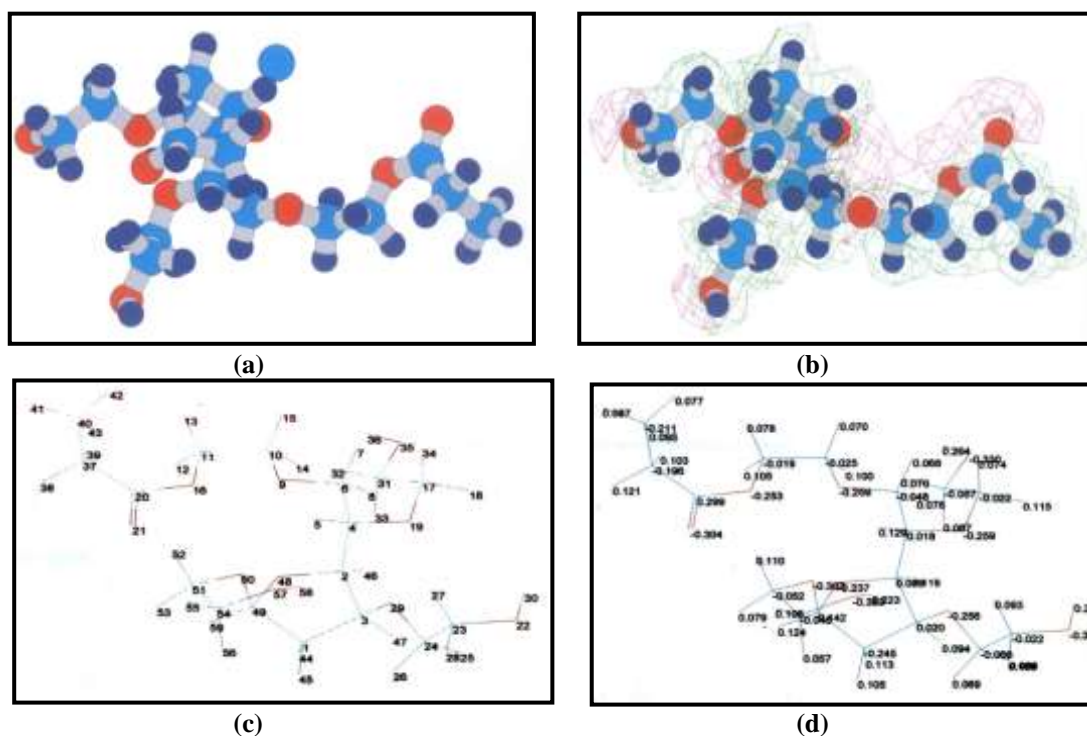
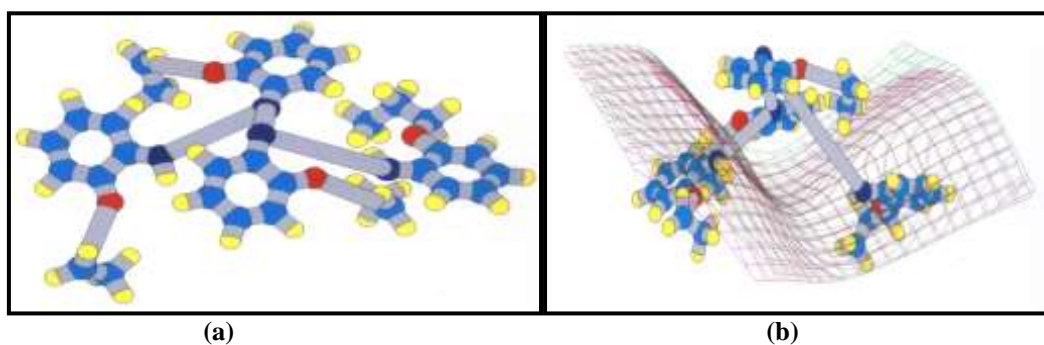
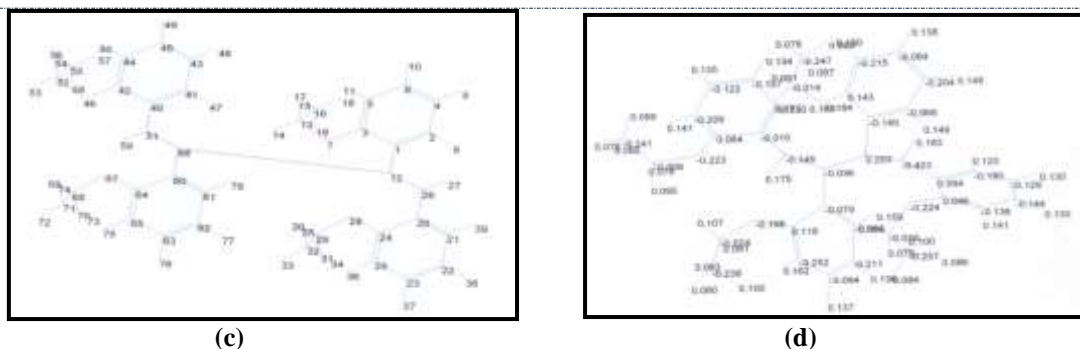


Figure 8: (a) Ball and Stick Model of the Optimized Geometry of Polysorbate20 (Red→Oxygen, Blue→Hydrogen, Cyan→carbon); (b) Electrostatic Potential Mapped on to 3-D Charge Density Isosurface of Polysorbate20; (c) Numbering of Atoms in Polysorbate20 (d) Respective Charges on the atoms in Polysorbate20





(c) (d)
Figure 9: (a) Ball and Stick Model of the Optimized Geometry of PEA (Red→Oxygen, Blue→Nitrogen, Cyan→carbon, Yellow→Hydrogen); (b) Electrostatic Potential Mapped on to 3-D Charge Density Isosurface of PEA; (c) Numbering of Atoms in PEA; (d) Respective Charges on the Atoms in PEA

IV.CONCLUSIONS

From the various studies, it can be concluded that:

1. From the weight loss studies, the rate constant was found to be lower when MS was present in the lowest concentration of the inhibitor and when mixed with the conducting polymer solution (PEA).
2. Poly- 20 is a mixed inhibitor inhibiting both cathodic and anodic processes to an equal extent, even on addition of PEA.
3. The inhibition efficiency decreased with the increase in the concentration of Poly-20 with increase in temperature.
4. The inhibition efficiency increases when PEA is added to Poly-20 from 85.8 to 96%.
5. Poly-20 and Poly-20 + PEA are non-passivating type of inhibitors.
6. The values of E_{eff} suggest that the inhibitors are physically adsorbed on the metal surface.
7. SEM reveals the least corrosion on the MS surface at 0.25% Poly-20 + PEA.
8. The impedance results supplement the results obtained from potentiodynamic polarisation studies. The results suggests strong adsorption of Poly-20 on the MS surface at 0.25%.
9. Quantum Chemical Studies further supplement the results obtained from various other techniques

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