



Surface Assimilation and Corrosion Inhibition Characteristics of Water-Soluble Polyvinyl Alcohol on Mild Steel Surface in 0.5 M HCl Solution

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Abstract: As corrosion inhibitors for mild steel in acidic media, polymers are emerging materials due to its larger surface coverage area. This paper focuses on the characteristic of water-soluble solvent polyvinyl alcohol (PVA) at interface of mild steel under 0.5 M HCl, examined using electrochemical impedance spectroscopy, Tafel polarization, and weight reduction techniques. The findings of the study revealed that water-soluble polyvinyl alcohol as polymer inhibitor has restraint abilities, up to 93.7 percent for 3 hours in 0.5 M hydrochloric acid. An advanced form of high spatial resolution imaging of surface of mild steel at different stages was found by using scanning electron microscopy which shows that the sample immersed for 3 hrs in 0.5 M HCl with 100 ppm of water soluble PVA inhibitor provides good inhibition quality to corrosion on mild steel surface.

Keywords: Polymer inhibitor, Weight loss study, Langmuir adsorption isotherm, Polyvinyl Alcohol.

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INTRODUCTION

Corrosion is formed due to the weakening and debasement of metal because of natural conditions. It causes due to the natural dampness, which prompts the oxidation of metal surface. Unadulterated metals or blends are isolated from a short of oxides, sulfates, carbonates, etc and significantly unpredictable and react viably within the sight of O₂ and H₂O likewise to CO₂ which leads because of metal degradation. So it very well might be treated as "A surface marvel alluded to be the assault on metals or composites by the climate like air, water or soil in a synthetic or electrochemical response to make more steady mixes."(1-3)

To lessen the impact of the destructive condition on metals, corrosion inhibitors are found in various natural and synthetic form, which diminishes the consumption rate when present in the corrosion framework at a reasonable focus without altogether

change in the convergence of some other destructive operator are utilized(4,5).

Metals can be prevented from the corrosion problems, through following precautionary procedures like (6-8).

A. Cathodic Protection: It is a successful strategy to rheostat corrosion on arrangements either enclosed or inundated in an electrolyte; as designated by the action mode, anodes are named dazzled present and peacemaking.

B. Assurance with anticorrosive casing: It is primarily cast off to shape a somatic interference between the destructive situations to secure the arrangement. it's far utilized for maximum element with metallic components present to the climate.

C. Corrosion Inhibitors: Inhibitors are added to the damaging domain of materials (in ppm) to avoid the corrosion effect in metals. Pipelines, vessels, and

types of gear are its primary application where soluble inhibitors are used successfully.

According to NACE, inhibitors are “a substance, which retards corrosion when added to an environment in appropriate concentration.”

It was found from the Langmuir adsorption isotherm that soluble polymers are efficient corrosion inhibitors as compared to the components like drugs, natural products, heterocyclic organic compounds, organic dyes, and so on, widely used as corrosion inhibitors, which covered the metal surface to repress the contact to the surface of metal and dampness (9,10).

Nowadays, various industries are using acids for the purpose of pickling, descaling, cleaning etc., (11-14). HCl is exceptionally destructive to the metals or mild steel (amalgam of unalloyed metals). In this way, the proficiency of water soluble polyvinyl alcohol as corrosion inhibitor has been evaluated using HCl solution of 0.5 M, 0.1 M, 1.5 M, etc. Productivity can be determined by gravimetric investigation, potentiodynamic polarization and electrochemical impedance spectroscopy. This is likewise bolstered by the surface examinations done by atomic power microscopy (10,15,16).

Pieces of literature show the majority of polymers like polyaniline, polyanthranilic, poly-diphenyl amine, and so on, as insoluble in nature, which limits their properties in interaction with the metal surface through the pi-bond present in the moiety. These polymeric materials are less utilized as corrosion inhibitors. However, polymeric materials can be sulfonated, to expand the solvability of polymeric material in an acidic medium. (1,2,16,17).

Principally polymers were utilized as covering material rather than inhibitors but due to the exceptionally insoluble nature of the majority of the polymers in aqueous medium, limit their use as a corrosion inhibitor. As of late, a few endeavors have been made to solubilize the polymers for the utilization of corrosion inhibitor (18-21). Polymers are magnificent consumption inhibitors for metallic antiques in corrosive condition. Nearness of a little amount of polymers might be powerful in hindering the consumption of metals in fluid acidic condition (22-26). Time is a champion factor while describing the corrosion inhibitor. Drenching time is a crucial factor to be considered in identifying the soundness of the inhibitor film and the rate of inhibitor adsorption. Weight reduction estimations were additionally used to contemplate the impact of temperature on the corrosion of mild steel with the nearness of consumption inhibitor (27-29).

Polyvinyl alcohol (PVA), commonly called as the green polymer, is a synthetic polymer and has been

considered as most commonly used water-soluble synthetic polymer due to its biologically accepted nature and biomedical applications (11-13,30,31).

Mild steel samples of 10 cm² surface zone in 0.5 M hydrochloric acid solution was examined with the inhibitive behavior of water soluble PVA using different methods for finding inhibitor efficiency and surface coverage of inhibitor over mild steel surface in corrosive environment using Atomic force microscopic techniques in this article.

MATERIAL AND METHODS

The acid was prepared by using the dilution formula to find out the volume of the acid required to prepare a solution.

Dilution formula;

$$C_1V_1 = C_2V_2 \quad (1)$$

where C= concentration, V= volume, 1= original solution, 2= the new solution.

Usually HCl is available in 12 M solution so; 12 M x V₁ = 0.5 M x 1000; V₁ = 41.6 mL must be taken in 1000 mL (1 L) flask with enough water added to form 1000 mL of 0.5 M solution (10,17,32,33).

The weight reduction and electrochemical examinations were done on mild steel segments submerged with the interface of water-soluble PVA and HCl (AR grade) both acquired from Merck India and twofold refined water was utilized for the weakening of forceful medium (15,16).

Weight Loss Studies

A 10 cm² surface zone of mild steel was immersed at 35 °C in 0.5 M HCl for 3 hours of time, examined and embraced to analyze the consumption conduct of certain metals in various corrosive arrangements utilizing weight reduction method. The total examinations were done in the funnel-shaped carafe having glass plug with various centralization of PVA inhibitor to acquire the ideal inhibitor fixation. Such investigations were directed for ideal convergence of soluble PVA at different temperature ranges, time interim, and grouping of corrosive. Inhibition efficiency (%) and surface coverage (Θ) of the PVA on mild steel were obtained by using the relation: (9,34-37)

$$I. E. (\%) = \frac{W_0 - W_i}{W_0} \times 100 \quad (2)$$

$$\Theta = \frac{W_0 - W_i}{W_0} \quad (3)$$

W₀ and W_i represent the weight loss of metal in the absence of PVA inhibitor and in the presence of PVA inhibitor concentration respectively (10).

The corrosion rate is calculated by dilution formula as

$$CR = \frac{87.6 \times \text{weight loss (mg)}}{\text{Surface area of the specimen (cm}^2\text{)} \times \text{Time (h)} \times \text{Density of the specimen (g cm}^{-3}\text{)}} \quad (4)$$

It has been found that fixation increment leads to a decrease corrosion rate.

impedance spectroscopy experiments (3,10,16,41-43) have been used for further study.

Electrochemical Studies

Electrochemical systems are perfect for the investigation of the corrosion forms, as corrosion happens by means of electrochemical responses (34,36,38,39). In an electrochemical investigation process, a three-electrode assembly cell having a mild steel working electrode of 10 cm² in surface area is utilized to demonstrate the metal in a consuming framework of 1 cm² unpolished area facing the solution as working electrode. A saturated calomel electrode with large rectangular platinum foil was used as reference electrode and counter electrode respectively at 35 °C temperature (9,10,16,38,40). Acetone, water, and a variety of emery papers were employed on the working electrode preparation. An open circuit potential in circulated air through environment was directed in all electrochemical estimation using Potentiostat / galvanostat of Gamry instruments Inc. USA, (model G-300) and EIS programming. Tafel polarization, linear polarization resistance and electrochemical

RESULT AND DISCUSSION

Weight-Loss Technique

Variation of PVA concentration in solution: Metal degradation technique was applied to study the behavior of soluble PVA concentration in absent and present condition has been listed in Table 1. The effect of inhibitor efficiency on the mild steel surface under 0.5 M HCl solutions at varying inhibitor concentration has been plotted in Figure. 1. The inhibitor efficiency has been indicated to be 93.70% at 100 ppm concentration of inhibitor in acidic solution. It was clearly found from the Table 1 that increasing the inhibitor concentration gradually decreases the weight loss and corrosion rate of metal while increases the inhibition efficiency. To achieve more accuracy in results all the examinations of sample has been performed in triplicate. To avoid statistical errors in graphical representation, an error bar has also been reported in Figure 1 (4,5,11,44).

Table 1. Range of data showing inhibitor efficiency of PVA at 10 cm² surface area of mild steel for 3 hours at 35 °C in 0.5 M HCl solution.

Inhibitor concentration (ppm)	Weight loss (mg)	IE (%)	CR (mmpy)
N/A	81.05	-	30.15
15	46.29	42.89	17.22
25	22.50	72.24	8.37
50	18.93	76.64	7.04
75	9.97	87.70	3.71
100	5.11	93.70	1.90

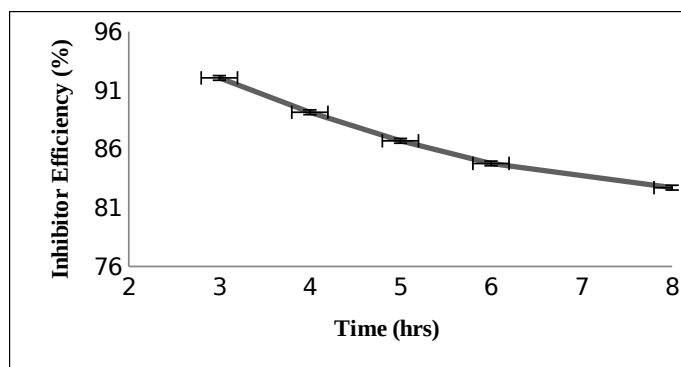


Figure 1. Change in PVA concentration lead to change in % inhibition efficiency at 35 °C for 3 hours of duration in 0.5 M HCl solution for exposed surface area of 10 cm² mild steel strip.

Variation of Immersion Time

In Figure 2, a graph with error bar represents the stability of inhibitor in the variable time interval. The data of weight loss of mild steel was recorded in 100 ppm concentration in 0.5 M HCl solution to the time range of 3 to 8 hrs at 35 °C. Figure 2 has been plotted to observe the inhibitor efficiency. The gradually increasing time of 3 to 8 hours of

immersion in mild steel specimen in 100 ppm inhibitor concentration with 0.5 M HCl solution decreases the inhibitor efficiency up to 82.71%. This plot discloses that the efficiency of inhibitor decreases with a slow rate due to the desorption of adsorbed layer of compound on mild steel surface (2,10).

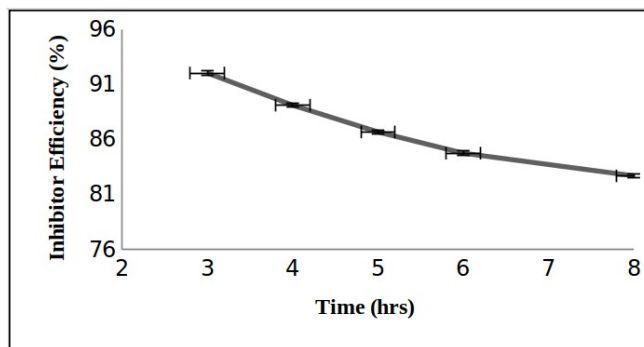


Figure 2. Change in immersion time of specimen leading to change in % inhibition efficiency at 35 °C for 3 hours duration in 0.5 M HCl solution for exposed surface area of 10 cm² mild steel strip.

Variation of Solution Temperature

A temperature variation at the range of 298 K to 328 K in 0.5 M HCl in 100 ppm inhibitor concentration has been plotted in Figure 3 and found that at increasing temperature, aqueous acid solution lowers the inhibitor efficiency up to 75.25%, which

represents that the adsorption process predominates over desorption up to 328 K where it takes place at 298 K and desorption of molecules of PVA on mild steel surface decreases due to high rate of heat of diluted HCl (45-47).

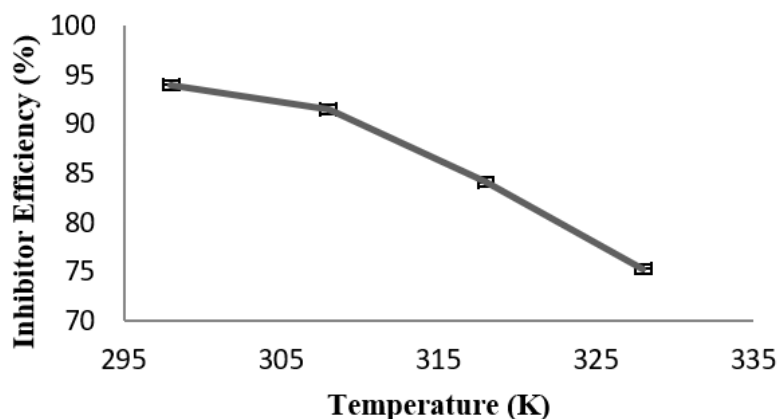


Figure 3. Temperature variation of acidic solution lead to change in % inhibition efficiency at 100 ppm inhibitor diluted for 3 hours duration in 0.5 M HCl solution for exposed surface area of 10 cm² mild steel strip.

Variation of Acid Concentration

An acidic concentration of 100 ppm PVA has been varied from 0.5 M to 2 M HCl for 3 hours at 35 °C on immersed mild steel structure and plotted in Figure 4. It has been observed that the increasing

concentration of acid in the solution decreases the inhibitor efficiency from 95.28% to 73.49%. Figure 4 has shown that the concentration level of 0.5 M HCl is very effective with a marginal decrease in inhibition efficiency (48).

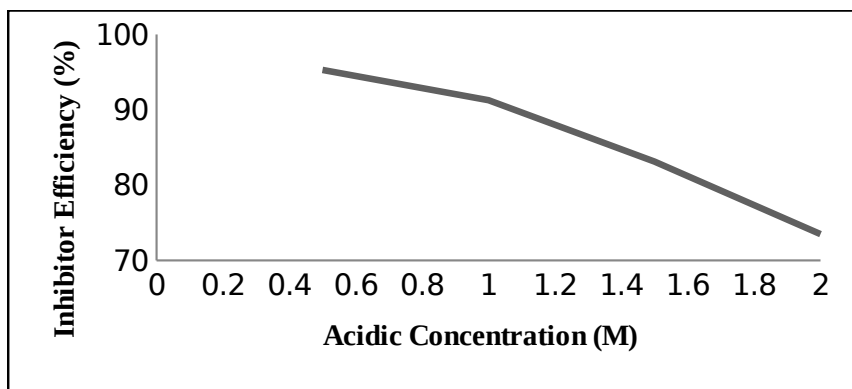


Figure 4. Variation of acidic solution lead to change in % inhibition efficiency at 100 ppm inhibitor diluted for 3 hours duration in 0.5 M HCl solution for exposed surface area of 10 cm² mild steel strip.

Adsorption Isotherm

Adsorption is the adherence of atoms, ions or molecules from a gas, liquid or dissolved solid to a surface (33). To support the adsorption isotherm study of the PVA molecule on the metal surface, Langmuir, Temkin, and Frumkin isotherms were employed. The Langmuir adsorption isotherm was found to be the best tool to explain the behavior of PVA molecule adsorption on the surface of the metal. This study defined the charge and the nature, electronic characteristics, adsorption of solvent of metal surface and other ionic species, on the electrochemical potential at solution interface (11). Langmuir adsorption isotherm expression (Formula 5) was used to calculate the adsorption isotherm by

fetching the value of surface coverage values (θ) from Table 1.

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \tag{5}$$

where, C_{inh} is the concentration of PVA corrosion inhibitor employed and K_{ads} represents the equilibrium constant of adsorption in acid medium. Figure 5 represents the C_{inh} vs C_{inh}/θ graph having straight line with the migration coefficient (R) to be almost unity. Unit value of R represents that the adsorption of the inhibitor on mild steel surface follows the Langmuir adsorption isotherm (9,49,50).

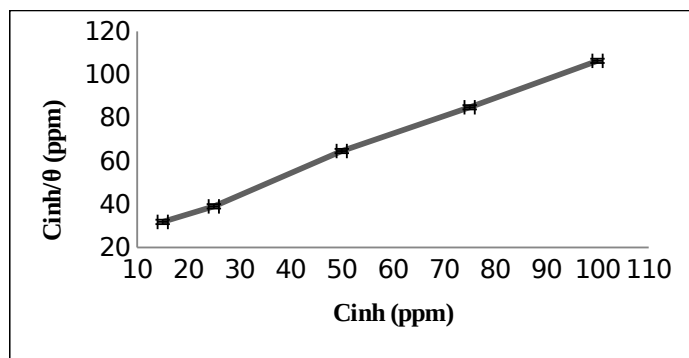


Figure 5. Adsorption of PVA inhibitor molecules on the mild steel surface in 0.5 M HCl solution using Langmuir’s adsorption graph with error bar.

Tafel and Linear Polarization Study

To find the corrosion current density, Table 2 represents a free corroding potential system, corrosion potential (E_{corr}), anodic (ba) and cathodic (bc) Tafel slopes and corrosion current density (I_{corr}). These parameters are used to evaluate the efficiency of PVA polymer inhibitor in 0.5 M HCl solution at mild steel surface in presence and absence of PVA in the concentration. The variation in the range of corrosion potential (E_{corr}) while increasing the inhibitor concentration in the solution has been shown in Table 2. This indicates that the high rate of inhibitor may increase the current

density. The small amount of change between Tafel plots indicates the mix characteristic of inhibitor property (51). This corrosion current density (I_{corr}) shown in Table 2 indicates that increasing the inhibitor concentration will lead to a higher voltage than 85 mV with respect to zero solution concentration and resultant mixed type of inhibitor property. The calculated inhibition efficiency using tafel polarization study supports the Table 1 results stated above.

Calculation of Polarization resistance (R_p) from the slope of the potential current lines can be found by

the relation between working electrode surface area (A), and potential change dE and di, using Equation 6 as:

$$R_p = A \frac{dE}{di} \quad (6)$$

The results has been plotted in Figure 6 and states that PVA conquers the H₂ progression reaction on

cathode and found more polarized than the anodic reaction suspension.

The value of R_p increases while increasing the concentration of inhibitor to 100 ppm in 0.5 M HCl solution at same temperature and the good inhibitor efficiency was found to be 91.6%. From the value, as shown in Table 2, it was indicated that higher the concentration of the inhibitor may result in an increase in the efficiency of the inhibitor.

Table 2. Potentiodynamic polarization parameters for mild steel without and with PVA in 0.5 M hydrochloric acid

Inhibitor Conc.	Tafel data					Linear Polarization data	
	-E _{corr} (mV vs SCE)	b _a (mV dec ⁻¹)	b _c (mV dec ⁻¹)	I _{corr} (μA cm ⁻²)	IE (%)	R _p (Ω cm ²)	IE (%)
N/A	469	75	150	2140	-	20.1	-
25	472	74	152	410	80.8	120	83.2
50	470	70	157	265	87.6	180	88.8
75	466	73	149	225	89.5	206	90.2
100	471	74	161	190	91.1	240	91.6

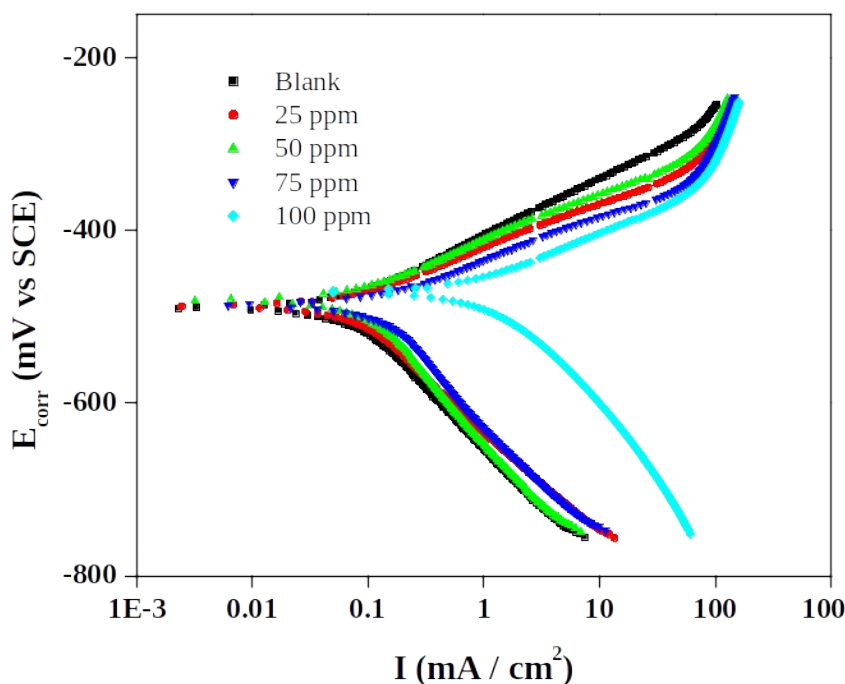


Figure 6. Tafel polarization curve in absence of inhibitor and presence of optimum concentration PAA inhibitor molecule on the surface of mild steel in 0.5 M HCl solution.

Electrochemical impedance spectroscopy and Surface morphology study

To study the experimental condition such as scan rate, voltage, frequency and the ionic species and concentration of electrolyte, the three regions of double layer i.e. space charge, compact layer, and diffuse layer is studied by modifying model of equation circuit reported by McDonald J.R. (9,20,50) for constant phase angle. Mansfeld’s A F. theoretical circuit is used to explain the interface model of HCl to mild steel phase angle (2,20). A Nyquist graph in

Figure 7 has been plotted to represent the behavior of corrosion on the surface of mild steel configuration in the presence and absence of inhibitor. All the experiment has been undertaken on mild steel surface at 0.5 M HCl solution at 35 °C, while varying the inhibitor concentrations. It has been observed from the electrochemical impedance spectroscopy plot that the semicircle diameter increases with increase in the concentration of inhibitor up to 100 ppm.

The basic electrochemical impedance parameter like solution resistance (R_s), admittance parameter (Y_0), total number of parts (n), resistance to charges transfers (R_t), and double electric layer capacitance (C_{dl}) was found to calculate the adsorption of metal and acid molecules interface due to the potential difference between solution and electrode at corrosion state using the equation 7.

$$C_{dl} = (Y_0 R_t^{1-n})^{1/n} \quad (7)$$

In the absence and presence of a PVA inhibitor at 0.5 M HCl concentration, Table 3 shows the values of R_s , Y_0 , n , and C_{dl} decreases with the addition of an inhibitor in concentrations, while R_t increased with the addition of an inhibitor. A protective film formed on the mixture and metal interface is due to the addition of more inhibitor molecules to the concentration, which resulted in an increase in the R_t value. The surface heterogeneity may increase and decrease as per the adsorption of inhibitor molecules on the adsorption of metal surface, which will affect the value of n . Equation 8 was used to plot the relation between protective layers thicknesses (d) and double layer capacitance (C_{dl}) by using the value of dielectric constant (ϵ) of protective layer and free space permittivity (ϵ_0) (52):

$$C_{dl} = \frac{\epsilon \epsilon_0}{d} \quad (8)$$

The result from the above plots revealed the percentage inhibitor efficiency to be 96.68% on mild steel surface in the presence of 100 ppm PVA in 0.5 M HCl, and this is in accordance with the data in the above studies. Surface study of all the samples was done to examine the impedance spectra, which were shown as depressed semicircle under real axis and appeared like a depressed capacitive loop using Figure 7 and Figure 8. The raising surface roughness and dislocation on metals property may lead to the heterogeneity on the mild steel surface due to dislocations or adsorption of the inhibitor molecules(14,53).

Surface morphology of the specimen represents the formation of adsorbed polymer film over the sample. The polished Figure 8(a) represents the smoother image of the surface while Figure 8(b) shows high roughness on the surface of the sample due to the effect of acid concentration. As compared to Figure 8(b), Figure 8(c) shows more perfect graph due to the impact of PVA inhibitors addition to the interface of mild steel strip.

Table 3. Electrochemical impedance parameters values of with and without PVA for mild steel corrosion in 0.5M HCl solution

Inhibitor concentration (ppm)	R_s ($\Omega \text{ cm}^2$)	R_p ($\Omega \text{ cm}^2$)	Y_0 ($\mu\text{F cm}^{-2}$)	n	C_{dl} ($\mu\text{F cm}^{-2}$)	IE (%)
N/A	1.23	8.3	1181.5	0.858	55	-
25	1.17	136.9	205.7	0.836	48	93.90
50	1.19	157.1	112.3	0.831	42	94.71
75	1.20	202.2	87.5	0.825	38	95.89
100	1.19	250.1	74.3	0.822	35	96.68

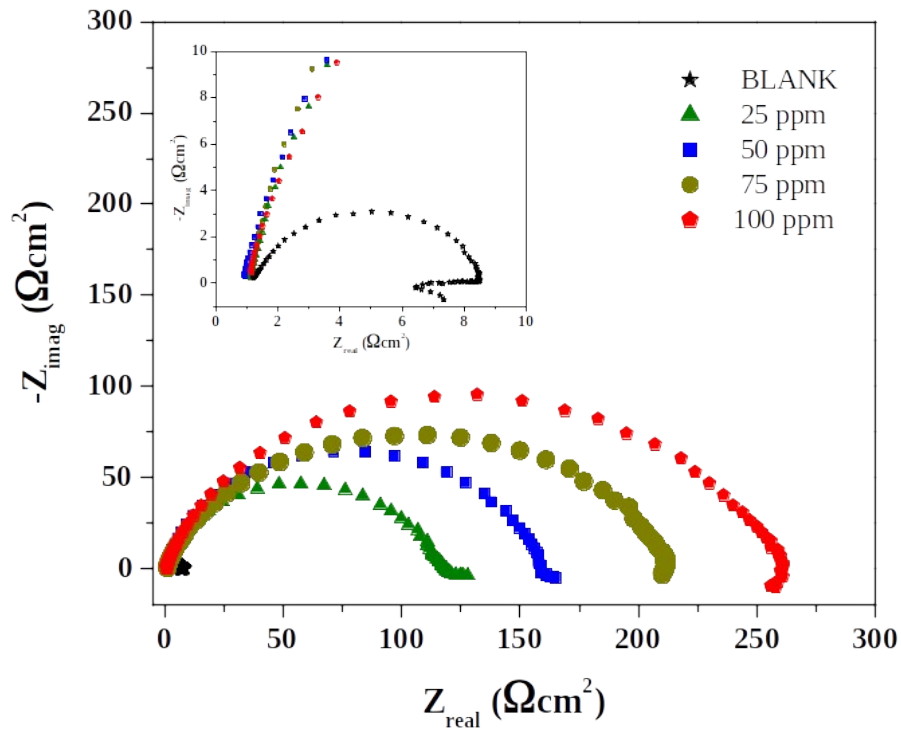


Figure 7. Nyquist plot of corrosion of mild steel in absence and presence of optimum concentration of PVA inhibitor in 0.5M hydrochloric acid solution

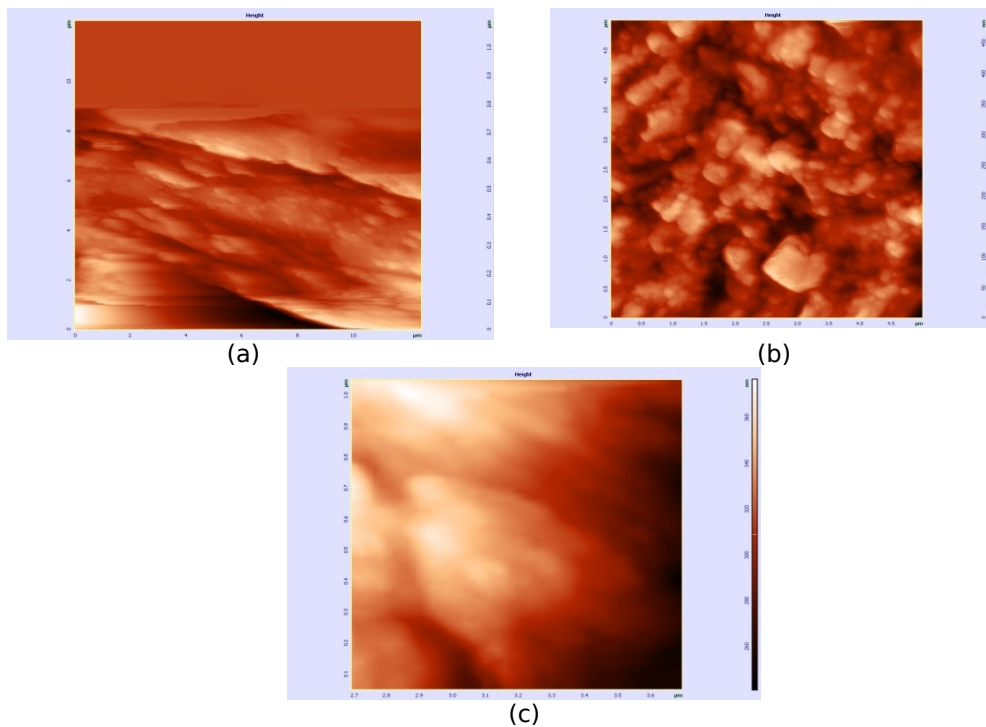


Figure 8. Surface morphological analysis through atomic force microscopy of mild steel sample. (a) Polished sample, (b) sample immersed for 3 hrs in 0.5 M HCl without inhibitor, (c) sample immersed for 3 hrs in 0.5 M HCl with 100 ppm PVA inhibitor.

CONCLUSIONS

It has been indicated that polymers, particularly the water soluble ones, are productive corrosion inhibitors in various aqueous media. (48-50). Tested water-soluble PVA exhibits outstanding corrosion inhibition properties against the surface of mild steel in a 0.5 M HCl solution. Impedance, polarization and gravimetric methodology findings suggest better effective water-soluble properties of PVA. The above study shows that the effectiveness of PVA as a corrosion inhibitor depends not only on the good results of the environmental conditions, but also on the steel surface flora and electrochemical potential at the tip, and also on the erection of the inhibitor itself. Polarization studies have shown that at 0.5 M HCl concentration at 100 ppm, PVA acts on both anodic and cathodic type reactions and shows that it is a mixed-type inhibitor. The PVA inhibitor efficiency was observed to be higher when the inhibitor concentration increased up to 100 ppm, with a dramatic change in the corrosion rate compared to the time of solution immersion.

The initiation boundaries mirror the endothermic metal disintegration process. The adsorption of PVA follows the Langmuir isotherm model and the estimations of the free vitality of adsorption show its unconstrained physical nature. The consequences of the arrangement of examinations have uncovered that the procedures engaged with corrosion inhibitors gives a uniform result as for all classes of mixes so far explored with one inhibitor in a given framework. The statement was supported by the results studied using characteristic atomic force microscopy.

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