Research Article

Chemical Review and Letters

Synthesized Schiff base acted as eco-friendly inhibitor for mild steel in

1N H₂SO₄

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ARTICLE INFO

ABSTRACT

2,2'-((1Z,1'Z)-(((propane-1,3-diylbis(oxy))bis(2,1-phenylene))bis(methanylyliden-e))bis(azanyly lidene))diethanol (2-PPM) was synthesized. This compound evaluated as novel corrosion inhibitor for mild steel in 1N H₂SO₄ solution. Polarization study showed that the compound was mixed type inhibitor. Electrochemical impedance study showed that the presence of this compound decreases the double layer capacitance and increases the charge transfer resistance.Weight loss study showed that, the corrosion inhibition property of 2-PPM on the external area of mild steel samples in the solution is mainly depends on the inhibitor concentration and immersion period. Further, the maximum protection efficiency obtained by weight loss technique was 65 %. SEM and AFM results fully support the chemical and electrochemical results.

1. Introduction

Mild steel has very low carbon amount that makes it appropriate for multiple utilizations like steel marketing, pipelines, construction, sheets, container, and metallic vessels. It corrodes hardly when subjected to an acidic media and made a great economic loss [1]. H₂SO₄ was generally exploited to liquidate scales and rust in steel equipments. Organic ligands having functional groups or heteroatoms like O, S, N, or connected multiple bonds can be adsorbed quickly on the mild steel [2-5]. Some researchers have studied the derivatives of Schiff-base as a good corrosion inhibitor [6-9]. Sorkhabi et al. had investigated different physical and chemical properties of three Schiff base in HCl [10]. Emregület al. has reported LOH and LACOH as a corrosion inhibitor in HCl medium on mild steel [11]. Shokry et al. have found out the concentration of Schiff bases that obtained from o-hydroxy of diamines and aromatic aldehydes of o-methoxy by electrochemical methods and surface examination method [12]. Daoud et al. have analyzed the effects of L on mild steel X52 in 1 M Sulfuric Acid acid [13]. Sliem et al. have investigated the corrosion inhibition properties of a synthesized aminothiazole Schiff base on mild steel in Sulfuric Acid acid [14]. Current research in the study of corrosion science is focused on the exploration of new nontoxic corrosion inhibitor. The organic compound possessing N, S, P and O atoms, functional groups, phenyl rings and pi-electrons adsorb on the mild steel surface via chemical or physical adsorption phenomena [15-19]. The organic compounds with high molecular weight having high surface coverage, which prove to be robust corrosion inhibition property [20-22]. A number of organic compounds exhibit good corrosion inhibition property but their application as corrosion inhibitor for different metals in industries is banned due to the toxic and expensive nature [23,24]. Generally, Schiff base compounds have heteroatoms in their ligand that is expected to show the good corrosion inhibition property. This property can be examined with electrochemical, which contain some kind sensors [25-30]. Herein reporting the systematic and detailed examination of application of 2,2'-((1Z,1'Z)-(((propane-1,3-diylbis(oxy))bis(2,1 phenylene))bis(methanylylidene))bis(azanylylidene))diethanol on the mild steel in 1N H₂SO₄ solution. The weight loss, Tafel curves, impedance spectroscopy, and SEM results are presented.

2. Materials and methods

Ligand 2,2'-((1Z,1'Z)-(((propane-1,3-diylbis(oxy)) bis(2,1-phenylene))bis(methanylylidene))bis(azanyly lidene))diethanol (Fig.1) was prepared according to literature method [31, 32]. 2-[3-(2-formyl

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phenoxy)propoxy] benzaldehyde (0.284 g, 1 mmol) and ethanol amine (0.122 g, 2 mmol) were mixed and heated under reflux for 3 hour in ethanol (30 ml). The solution was refined and the filtrate was reduced to ca 10 cm3. Yield: 85%. Anal. Calc. for $C_{21}H_{26}N_2O_4$: C, 68.09; H, 7.07; N, 17.28. Found: C, 67.74; H, 7.17; N, 17.39. IR (KBr, cm^{-1}): 1638 (υ C=N Schiff base), 1490 (υ C=C), 3365 (υ OH). ^{1}H NMR (DMSO-d$_6$, ppm, 300 MHz): δ 2.36 (m, 2H), 3.71 (t, 4H), 3.82 (t, 4H), 4.25 (t, 4H), 6.99 (m, 2H), 7.40 (m, 2H) 7.96 (m, 2H), 7.99 (m, 2 H), 875 (s, 2H). ^{13}C NMR (DMSO-d$_6$, ppm, 300 MHz): δ 62.48, 63.57, 65.03, 76.73, 77.05, 112.20, 121,09, 125.62,125,74, 12684, 127.40, 132.16 (aromatic rings), 158.88 (Schiff base). The chemical structure of the is shown in the Figure 1.
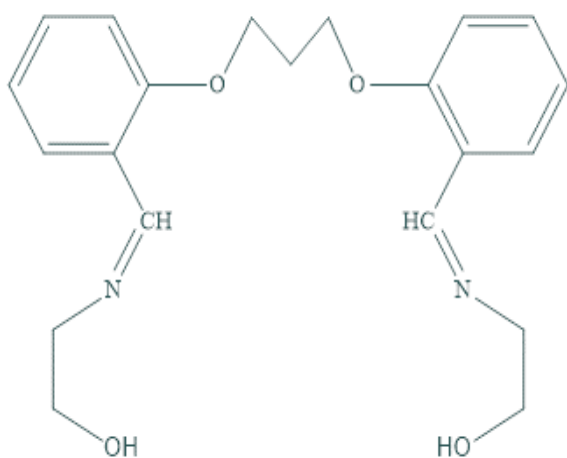

Figure 1. Chemical structure of 2-PPM.

The inhibitor concentration utilized in this work is 0.5 mg/L, 1 mg/L, 1.5 mg/L and 2.0 mg/L. The steel chemical composition is is C: 0.17, Si: 0.35, P: 0.2, S: 0.05, Cu: 0.01, N: 0.01 Mn: 0.42, Cr: 0.01%w, Fe:Rest. The mild steel metal wiped with different grade emery paper, washed with acetone and eventually dried. The 1N H_2SO_4 solution is created by dissolving the Merck product volume of concentrated H_2SO_4 solution in the triple distilled water. Weight loss (mass loss) of the mild steel in the 1N H_2SO_4 solution without and with 2-PPM at 333 K with different immersion period (2, 4, 6, 8 and 10 hours). After the specified time, the mild steel samples are submerged from the 1N H_2SO_4 solution and weight loss of steel sample was recorded. The protection efficiency of the corrosion inhibitor is calculated from the below equation [32],

$$\text{Corrosion inhibition efficiency (\%)} = \frac{(W_1 - W_2)}{W_1} \times 100 \quad (1)$$

Where, W_1= Unprotected steel weight loss, and W_2=Protected steel weight loss of the system.

The corrosion protection efficiency of 2-PPM was calculated from the below relations [26]:

$$\text{Corrosion protection efficiency} = [1 - \frac{i^{'}_{corr}}{i_{corr}}] \times 100, \quad (2)$$

$$\text{Corrosion protection efficiency} = \frac{R_{ct(inh)} - R_{ct}}{R_{ct(inh)}} \times 100, \quad (3)$$

Where, $i^{'}_{corr}$ = Protected (steel metal) corrosion current density, i_{corr} = Unprotected (steel) corrosion current density, R_{ct} = Unprotected (steel) charge transfer resistance, $R_{ct\,(inh)}$= Protected (steel metal) charge transfer resistance.

In these experiments, rectangular coupons were cut into 5 ×3 ×0.5 cm dimensions used for weight loss measurements. The surface areas of the coupons were mechanically abraded with 220 up to 2000 grades of emery papers degreased with acetone and rinsed by distilled water just before immersion. Standard weight loss tests were carried out under the procedure according to ASTM G1 and G31 [21].

Polarization experiments were performed using a Compact-stat from IVIUM Technologies controlled by a Windows-based PC running dedicated software. A three-electrode cylindrical Pyrex glass cell configuration was used consisting of mild steel as working electrode, a platinum electrode as counter, and a saturated calomel electrode (SCE) as a reference. Before each experiment, the working electrode was immersed in the test cell at open circuit potential (E_{OCP}) for 30 min until steady state, condition was reached. The polarization curve was acquired by scanning the potential at constant sweep rate of 1 mV s-1. The electrochemical cells were placed in a Faradaic cage to avoid electromagnetic disturbance from external sources. All tests were carried out at constant temperatures by controlling the cell temperature using a water bath. All the experiments were performed in quiescent conditions and solutions were open to the atmosphere under unstirred conditions. Fresh solution and fresh steel samples were used for each sweep. For each experimental condition, at least three measurements were performed to ensure the reliability and reproducibility of the data.

The EIS measurements were carried out at 298 ± 1 K in a conventional three electrode glass cell with the same electrodes and electrolyte used in polarization measurements. Experiments were conducted using computer controlled IVIUM potentiostat/galvanostat system. All experiments were performed after an exposure period of 30 minutes until a steady state situation. Signals were applied in the frequency range from 100 KHz to 10 MHz with peak-to-peak A.C. amplitude of 10 mV.

The impedance diagrams were plotted in the Nyquist representation. Fitting of experimental impedance spectroscopy data to the proposed equivalent circuit was done by means of IVIUM potentiostat/galvanostat software.

The Steel specimens of size 1 cm×1 cm×0.43 cm were abraded with emery paper (up to 2000) to give a homogeneous surface, then washed with distilled water and acetone. The specimens were immersed in 1N H$_2$SO$_4$ prepared with and without addition of 2 mg/l at 25°C for 6 h, cleaned with distilled water.

The surface morphology of the electrode surface was evaluated by atomic force microscopy (AFM) NanoSurf easyscan2. The mild steel topography in protected and unprotected systems was visualized by scanning electron microscopy (VEGA) and atomic force microscopy (NanoSurf Easyscan 2) technique.

3. Results and Discussion
3.1. Weight Loss Technique
The influence of different concentration of 2-PPM on mild steel's corrosion in the 1N H$_2$SO$_4$ solution was depicted in the table 1. From the table 1, it is clear that, the rise in the concentration of the inhibitor enhances the protection efficiency of the corrosion inhibitor. This indicating that, the 2-PPM adsorbed on the mild steel in 1N H$_2$SO$_4$ solution and forms strong protective layer on the steel surface in 1N H$_2$SO$_4$ solution. Hence, high protection efficiency is observed at 2 mg/L of 2-PPM. Further, the protection efficiency decreases with increase in the immersion time from 2 to 10 hours. The percentage protection efficiency decreases with rise in the immersion time is due to desorption of protective layer generated on the surface of mild steel in 1N H$_2$SO$_4$ solution [33].

Moreover, the high immersion time leads to enhance the steel corrosion rate and decrease the corrosion inhibition efficiency. The outcomes of weight loss method clearly show that, the corrosion inhibition property of Schiff base ligand is mainly concentration and immersion period (contact time) dependent mode[34].

3.2. Tafel curves Studies
The Tafel plot curves recorded for the steel metal in the nonattendance and attendance of four different amounts 2-PPM in 1N H$_2$SO$_4$ solution is shown in the Figure 2. The calculated electrochemical parameters are shown in the Table 2. It is observed that, the corrosion current density values reduces with rise in the concentration of the Schiff base compound. It is indicating that, 2-PPM inhibiting the dissolution or disintegration of mild steel metal by adsorption phenomena.

This shows that, the rate of steel reaction in 1N H$_2$SO$_4$ solution is reduced due to formation of thick invisible film on the external area of mild steel in 1N H$_2$SO$_4$ solution [35]. It is also observed that, presence of Schiff base ligand suppresses the both cathodic and anodic dissolution process equally. This shows that, mixed steel corrosion inhibition behavior of 2-PPM (controls both anodic and cathodic mild steel corrosion reaction) on steel surface in 1N H$_2$SO$_4$ solution [36].

Table 1. Weight loss (gravimetric) results

Concentration (mg/L)	Contact time (hours)	Protection efficiency in percentage
Blank	2	
0.5		30.000
1.0		51.000
1.5		54.500
2.0		65.000
Blank	4	
0.5		28.253
1.0		47.428
1.5		51.603
2.0		63.777
Blank	6	
0.5		27.627
1.0		45.116
1.5		49.767
2.0	8	61.744
Blank		
0.5		27.368
1.0		39.223
1.5		47.106
2.0	10	58.932
Blank		
0.5		26.755
1.0		37.055
1.5		46.992
2.0		57.503

The presence of inhibitor shifts both anodic and cathodic branches to the lower values of cor-rosion

current densities and thus causes a remarkable decrease in the corrosion rate (figures 2). As was expected both anodic and cathodic reactions of steel electrode corrosion were inhibited with the increase of the inhibitor concentration. This result suggests that the addition of the inhibitor reduces anodic dissolution and also retards the hydrogen evolution reaction [17]. The electrochemical processes on the metal surface are related to the adsorption of the inhibitor and the adsorption is known to depend on the chemical structure of the inhibitor. The parallel cathodic Tafel curves in Figure 2 show that the hydrogen evolution is activation-controlled and the reduction mechanism is not affected by the presence of the inhibitor.

Table 2. Tafel curves results

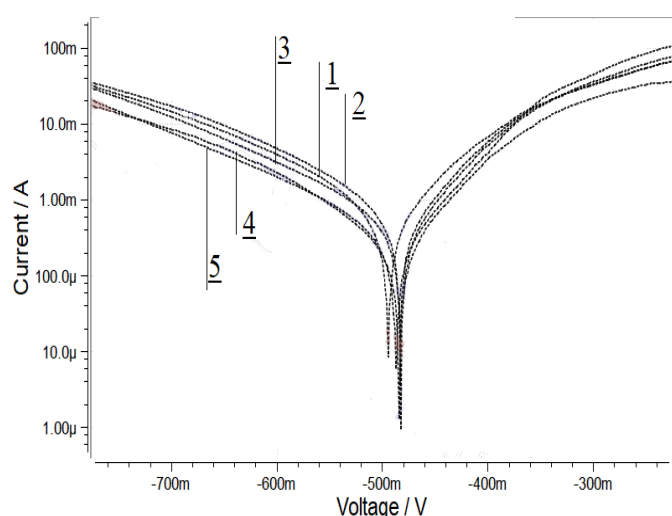| Concentration mg/L	$-E_{corr}$ mV	I_{corr} µAcm^{-2}	β_a mV/dec	$-\beta_c$ mV/dec	θ	IE %
blank	489	22۶	۷۷	۴۳	----	-----
0.5	485	14۵	۶۸	۵۱	0.36	36
1	482	10۵	۷۸	۵۷	0.53	53
1.5	481	9۲	79	۶۰	0.59	59
2	480	80	75	58	0.64	64

Figure 2. Tafel plots without and with corrosion inhibitor:blank(1), 0.5 (2), 1(3), 1.5(4), 2(5) mg/L.

3.3. AC Impedance Spectroscopy Technique

The Nyquist curves for mild steel in to the 1N H$_2$SO$_4$ solution in the absence and presence of Schiff base ligand (2-PPM) are shown in the Figure 3. The size of the diameter of semicircle enhances with rise in the concentration of 2-PPM. The increase in the size of the depressed plots with rise in the concentration of the inhibitor clearly shows the steel corrosion inhibition property of Schiff base compound against mild steel' corrosion in 1N H$_2$SO$_4$ solution. The figure of the

Nyquist plots in unprotected system and protected systems is same, shows that there is no change in the steel corrosion mechanism in 1N H$_2$SO$_4$ solution without and with inhibitor system mode [36]. The deviation from the perfect half of a circle in the impedance spectra is due to the frequency dispersion and surface heterogeneities which is due to the steel corrosion in acidic environment.
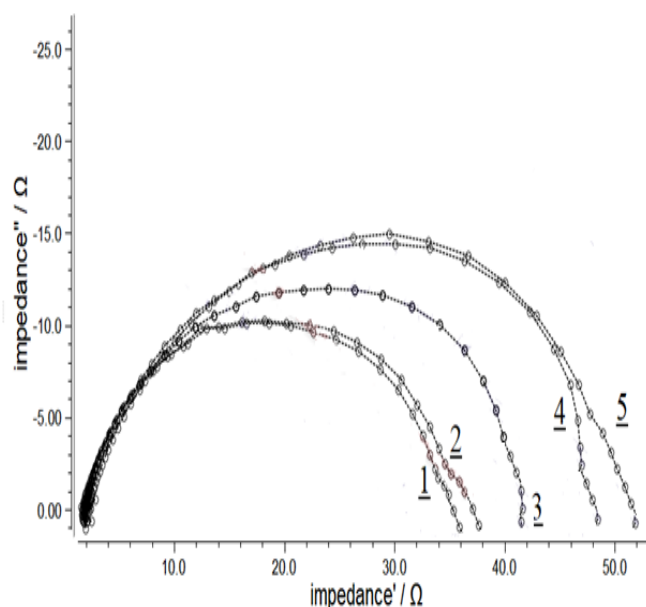

Figure 3. Nyquist plots in the absence and presence of the corrosion inhibitor: blank (1), 0.5 (2), 1(3), 1.5(4), 2(5) mg/L.

Table 3 illustrates the equivalent circuit parameters for the impedance spectra of corrosion of steel in 1N H$_2$SO$_4$ solution. The results demonstrate that the presence of inhibitor enhance the value of R_{ct} obtained in the pure medium while that of Q_{dl} is reduced. The decrease in Q_{dl} values was caused by adsorption of inhibitor indicating that the exposed area decreased. On the other hand, a decrease in Q_{dl}, which can result from a decrease in local dielectric constant and/or an increase in the thickness of the electrical double layer, suggests that inhibitors act by adsorption at the metal–solution interface.

As the Q_{dl}, exponent (*n*) is a measure of the surface heterogeneity, values of n indicates that the steel surface becomes more and more homogeneous as the concentration of inhibitor increases as a result of its adsorption on the steel surface and corrosion inhibition [19]. The increase in values of Rct and the decrease in values of Q_{dl} with increasing the concentration also indicate that inhibitors act as primary interface inhibitors and the charge transfer controls the corrosion of steel under the open circuit conditions.

3.4. Analytical Imaging

SEM images of abraded mild steel metal in 1N H$_2$SO$_4$ solution without and with 2 mg/L of 2-PPM is shown in the Figure 4.a and 4.b. The surface of steel in the nonattendance of the inhibitor is highly corroded which is due to aggressive attack of Sulfuric Acid acid ions.

Table 3. AC Impedance studies

Concentration,	$R_s (\pm 10^{-1})$,	$R_{ct} (\pm 1)$,	$R_f (\pm 10^{-1})$,	$R_p (\pm 1)$,	$Q_{dl}(\pm 10^{-\gamma})$,	$C_{dl}(\pm 10^{-\gamma})$,	$n (\pm 10^{-2})$	$\theta (\pm 10^{-2})$
Mg/L	Ω cm^2	Ω cm^2	Ω cm^2	Ω cm^2	mF cm^2	mF cm^2		
blank	1.5	31	1.5	31	0.5$^{\vee}$	0.21	0.73	----
0.5	1.6	38	1.6	38	0.54	0.21	0.75	18
1	1.6	43	1.6	43	0.5$^{\gamma}$	0.20	0.75	27
1.5	1.7	47	1.7	47	0.41	0.19	0.74	34
2	1.8	54	1.8	54	0.4$^{\delta}$	0.17	0.74	40

Whereas, in the presence of Schiff base compound, the significant enhancement in the steel surface smoothness was observed. This is due to adsorption of the inhibitor on the mild steel in 1N H_2SO_4 solution.
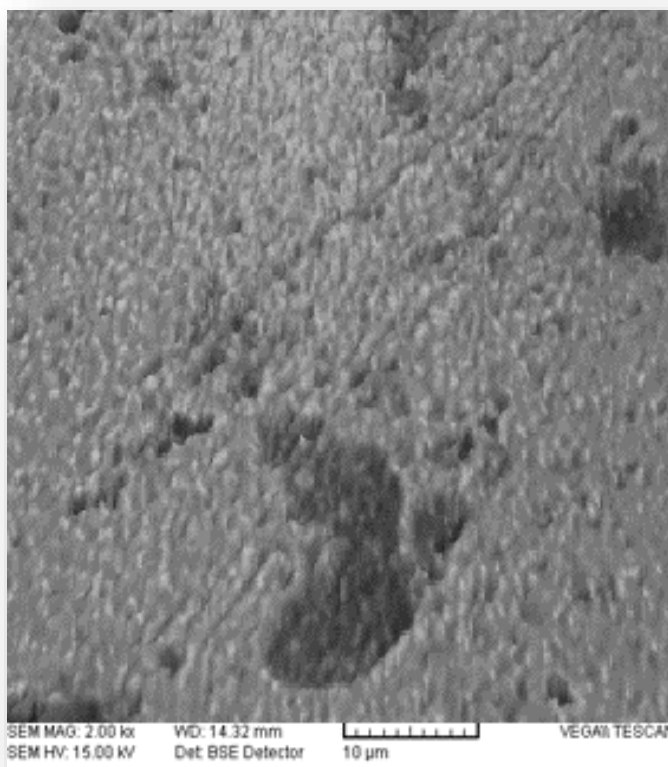
Figure 4.a. SEM images without corrosion inhibitor.

AFM images presented in Fig.5.a and 5.b show the morphologies of the electrode surface in acidic solution with and without inhibitor. The metallography were recorded for steel surface after exposure to H_2SO_4 solution for 6 h. Surface of electrode in the absence of inhibitor was observed in Fig. 5a.
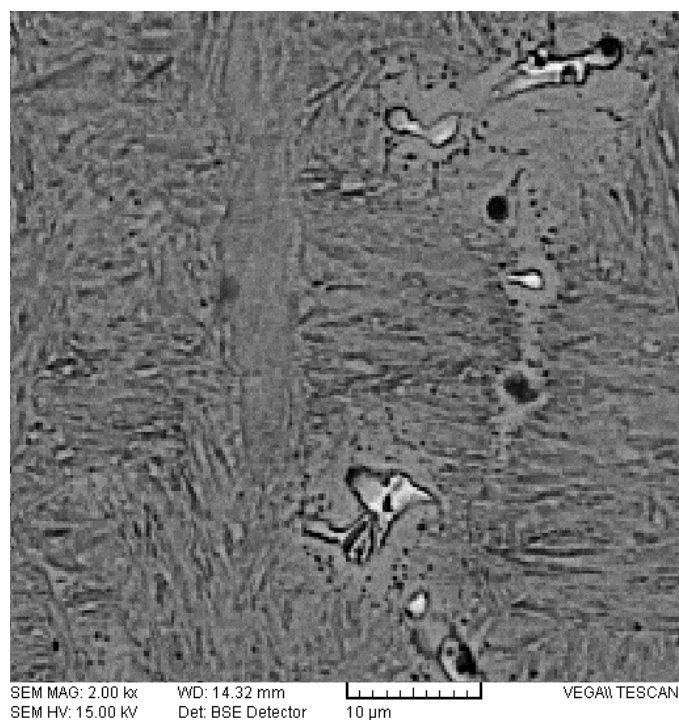
Figure 4.b. SEM images with corrosion inhibitor.

It was observed that the sample in contact solution gave high corrosion attack on the other hand, in presence of inhibitors smooth surface obtained, and the degree of attack decreased (Fig.5b). The roughness Ra of steel in 1 N H_2SO_4 solution without inhibitor was calculated as 2.53 µm by atomic force microscopy (Fig. 5a). The surface roughness of steel after the addition of 2-PPM is 715nm (Fig.5b), as a consequence of low corrosion damage and the protective formation of an inhibitor layer on steel surface. This is in good agreement with the result obtained from the EIS tests as can be seen.
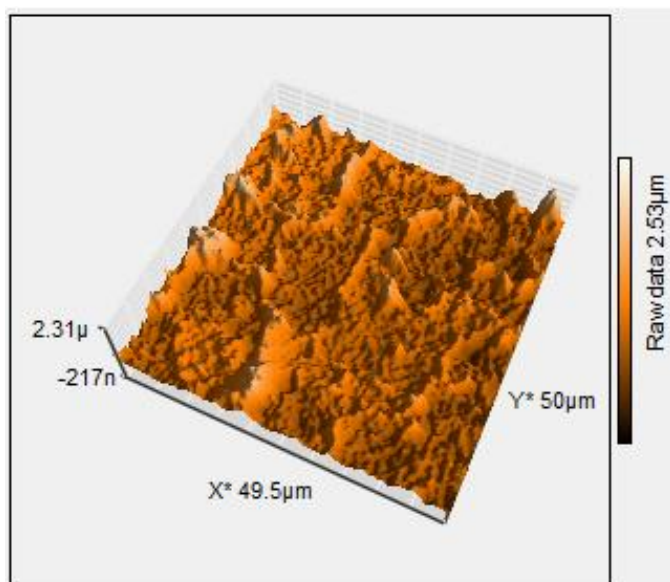
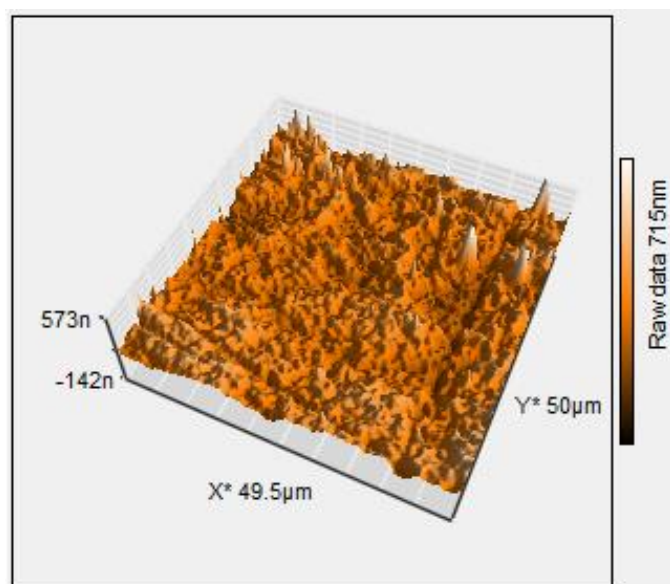**Figure 5.a.** AFM images without corrosion inhibitor.

Figure 5.b. AFM images with corrosion inhibitor.

3.5. AC Impedance Spectroscopy Technique

Schiff base ligand adsorbed on a mild steel surface and caused a retardation of the anodic dissolution of steel and cathodic evolution of hydrogen. Trabanelli [37] established several mechanisms:

1. Formation of a physical barrier.
2. Reduction of metal reactivity.
3. Changes in the electrical double layer.
4. Participation of the inhibitor molecule in partial electrochemical reactions.

According to Figure 2, the cathodic current–potential curves giving rise to parallel lines indicates that the addition of 2-PPM to the 1N H_2SO_4 solution does not modify the reduction mecha¬nism and the reduction at steel sur¬face takes place mainly through the changes in the electrical of double layer system [31-34]. The change

in the mechanism of EDL due to adsorption of 2-PPM molecules is probably one of the great significance mechanisms of corrosion inhibition. Electrostatic adsorption occurs via an interaction of the double layer charge with the charge (formal or dipole-induced) of 2-PPM molecule. The slopes do not display an order with the inhibi¬tor concentration; this feature in¬dicates that inhibition oc¬curred by the formation of a physical barrier mecha¬nism on the available metal surface [32-36]. The physical barrier mechanism included the barrier of reactants touching the mild steel surface and the products of corrosion liberated where adsorbed 2-PPM molecules prevent movement adjacent the steel surface. This mechanism requires the adsorbed layer to have impermeable coverage for effective inhibition.

4. Conclusion

The present study deals with investigation of corrosion inhibition behavior of 2,2'-((1Z,1'Z)-(((propane-1,3-diylbis(oxy))bis(2,1-phenylene))bis(methanylylidene)) bis (azanylylidene)) diethanol (2-PPM) on the mild steel surface in 1N H_2SO_4 solution by application of weight loss, Tafel plot and impedance spectroscopy techniques. Weight loss studies shows that, the corrosion inhibition property of 2-PPM on the external area of mild steel samples in the 1N H_2SO_4 solution is mainly depends on the inhibitor concentration and immersion period. Polarization measurements demonstrate that inhibitor behaved as mixed type corrosion inhibitor by inhibiting both anodic metal dissolution and cathodic hydrogen evolution reactions. Impedance measurements indicate that in presence of inhibitor, the polarization resistance (R_{ct}) increased, while the double-layer capacitance (C_{dl}) decreased. The high resolution micrographs were showed that the corrosion of steel in acidic solution was described by corrosion attack and the addition of inhibitors to the aggressive solutions diminished the corrosion of steel. SEM studies show the adsorption process of the Schiff base ligand on the steel metal surface in acidic environment.

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