



Hexamine as anti-pit agent for mild steel in sulphuric acid medium

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Mild steel corrodes very heavily when exposed to dilute sulphuric acid medium. Pitting corrosion takes place on the surface of mild steel in absence of corrosion inhibitor. Hexamine is a long chain amine having nitrogen atom as heteroatom. Hexamine was tested as corrosion inhibitor for mild steel in 0.1 N H₂SO₄ as corroding medium at 25.0, 30.0 and 35.0°C by weight loss, electrochemical polarization and impedance spectroscopy methods. Hexamine was proved to be a very good anti-pitting agent for mild steel in dilute sulphuric acid medium. Surface study of corroded and un-corroded specimens of mild steel was carried out by Metallurgical Research Microscopy (MRM) and Scanning Electron Microscope (SEM) techniques. Surface study confirms that the adsorption of hexamine takes place through nitrogen atom resulting in the formation of uniform, non-porous, passive film confirmed by decrease in Warburg Impedance (Z_w), decrease in Faradaic current, increase in capacitive current and significant increase in capacitive loop in Nyquist plot with increase in concentration of hexamine which results in significant decrease in corrosion rate of mild steel in dilute sulphuric acid medium especially eliminating pitting corrosion.

Keywords: Mild steel, pitting corrosion, electrochemical polarization, hexamine, weight loss.

Introduction

Mild steel [MS] because of its unique properties like softness, ease in mouldability, cost-effective, easy availability etc. makes it suitable for non-specialist steel products such as cars, tanks, structural body parts and domestic goods. However, mild steel is very reactive and corrodes heavily when exposed to dilute acidic medium as compared to other alloys of steel. Chemical corrosion inhibitors are required essentially for mild steel in order to minimize the rate of corrosion. Different researchers have devoted their research in minimizing the rate of corrosion in acidic medium. Chen *et al.*¹ carried out an investigation to study the effect of sulphur dioxide on the corrosion of a low alloy steel. Polyvinyl pyrrolidone acted as anodic and cathodic corrosion inhibitor for CS in 2.0 M HCl as a corroding medium and an increased PCIE was detected as the concentration of PVP was increased². Zaafarany³ studied the cationic surfactants of Olyel-amido derivatives for MS corrosion in 1.0 M HCl and the PCIE was found to linear with respect to inhibitor. The het-

erocyclic compounds having polar groups are better corrosion inhibitors in acidic solutions^{4,5}. Singh *et al.*⁶ concluded that Ceftazidime is a good corrosion inhibitor for MS in dilute acidic medium. The PCIE of Ceftazidime decreased with temperature leading to an increase in activation energy of corrosion process. Mild steel corrosion studies were carried out with diallylamine, 1-benzylimidazole and 5-aminotetrazole as corrosion inhibitors in aggressive atmospheric conditions and found that these corrosion inhibitors were better for mild steel corrosion⁷. Amiery *et al.*⁸, investigated the PCIE properties of Schiff base derived from 4-aminoantipyrine for MS in 1.0 M H₂SO₄ medium. Some other organic compounds also used for the corrosion inhibitors for steel and their alloys in acidic medium such as fluoroquinolones⁹, 4-methyl-4H-1,2,4-triazole-3-thiol and 2-mercaptopyridine¹⁰, *N,N,N,N*-tetramethylethylenediamine and 1,3-diaminopropane¹¹, triethylamine, ethylamine, *N*-ethylaniline¹², *n*-caprylic acid, *n*-butyric acid, *N,N*-dimethyl propylene urea and 2-amino benzothiazole¹³, dithizone¹⁴, oxo-triazole derivative¹⁵, 2-

amino-5-mercapto-1,3,4-thiadiazole and 2-mercaptothiazoline¹⁶, carboxymethyl cellulose¹⁷. Green corrosion inhibitors such as, *Citrus sinensis*¹⁸, *Azadirachta indica*¹⁹, *Andrographis paniculata* and *Vernonia amygdalina*²⁰, bark and leaves extract of *Neolamarckia cadamba*²¹, *Tagetes erecta*²², *Musa paradisiac*²³, 2-amino-3-((4-((S)-2-amino-2-carboxyethyl)-1H-imidazol-2-yl)thio) propionic acid²⁴, *Plantago ovata*²⁵, *Longan* seed and peel²⁶ were reported as good corrosion inhibitors at different concentration and environmental conditions.

From the current literature survey, it is found that a very little research was carried out corrosion inhibition properties of hexamine in 1.0 N H₂SO₄ at 25.0, 30.0 and 35.0°C temperatures. So we have investigated corrosion inhibition properties of hexamine for mild steel at three different temperatures in 1.0 N H₂SO₄ by weight loss, linear polarization, impedance spectroscopy techniques followed by surface study by MRM and SEM techniques.

Materials and methods

Material used: The corrosion inhibitor used was of Fisher Scientific made (AR grade). Acetone was used for cleaning the metal strips. 0.1 N H₂SO₄ solution was prepared from Himedia Laboratories Pvt. Ltd. (AR grade). All test solutions were prepared with high purity double distilled water. Emery papers of 100, 220, 350 and 600 micron grade were used for polishing the mild steel specimens of size 3.0×1.5 cm² for weight loss method and of size 1.0×1.0 cm² for electrochemical polarization experiments.

Mild steel: ASTM-283, coupons of dimensions 3.5×1.5×0.025 cm³ having chemical composition:

Element	Composition (w/w)	Element	Composition (w/w)	Element	Composition (w/w)
Carbon	0.17	Nickel	0.01	Copper	0.01
Silicon	0.35	Sulphur	0.05	Chromium	0.01
Phosphorous	0.20	Manganese	0.42	Iron	Rest

Equipments: *Weighing Balance:* Research Analytical Balance of precision 0.01 mg having Model AB135S/FACT, make Mettler Toledo, Japan.

Potentiostat/Galvanostat: make Metrohm Autolab PGSTAT 128N, Netherlands.

Metallurgical microscope: Research grade Metallurgical Microscope make Kyowa Getner, Japan.

SEM: Scanning Electron Microscope, JSM 6510 LV, JEOL.

Methods

Weight loss measurements:

Mild steel specimens were mechanically polished with the help of emery paper of different grades and then washed with double distilled water followed by acetone and dried with the help of hot air dryer in order to remove any adsorbed solid impurities and gases. Corrosion rate and %age Corrosion Inhibition Efficiency (PCIE) were calculated by weighing the specimens with and without hexamine after immersion in 100.0 ml of 0.1 N H₂SO₄ as corroding medium for 24.0 h at 25.0, 30.0 and 35.0°C using formula (1) and (2).

$$CR \text{ (mpy)} = \frac{534 \times W}{DAT} \quad (1)$$

Here W is the weight loss (mg), D is the density of MS (g/cm³), A is the exposed area of sample (sq. inch), T = Exposure time (h).

$$(\text{PCIE}) = \frac{(\text{CR}^{\circ} - \text{CR})}{\text{CR}^{\circ}} \times 100 \quad (2)$$

Here, CR^o and CR are the CR in absence and in presence of inhibitor, respectively.

Electrochemical polarization measurements:

Electrochemical polarization experiments were carried out by using a Potentiostat/Galvanostat Metrohm Autolab, Netherland (PGSTAT 128N)^{27,28}. MS specimens of size 5.0×1.0 cm² was taken as working electrode, Pt electrode was acted as auxillary electrode and Ag/AgCl electrode as non-polarizable electrode for three-electrode system assembly. Working area of 1.0×1.0 cm² was polished with emery papers of grade 100, 220, 350 and 600 microns. Remaining area of the working electrode was covered with lacquer. Working electrode and counter electrode were kept at minimum distance apart in order to minimize the Ohmic drop and this distance was kept constant during all electrochemical experiments. The specimens were immersed in the test solution with and without hexamine (different concentration i.e. 200 to 1000 ppm) until a constant OCP is not observed. All experiments were performed at three different temperatures i.e. 25.0, 30.0 and 35.0°C.

The CR and PCIE were calculated from Stern Gerry and eq. (2), respectively.

EIS measurements:

Impedance measurements were carried out using class one handheld Potentiostat, Netherland. Current density, resistance and capacitance measured for MS in different concentrations of hexamine as a function of time and frequency AC. Frequency of AC 0.1–10 kHz and amplitude 1–10 mV was used for present investigation. Percentage Corrosion Inhibition Efficiency of the inhibitor was calculated using the value of Charge Transfer Resistance, R_{ct} .

Surface study (Metallurgical research microscope):

The specimens of size $3.0 \times 1.5 \text{ cm}^2$ used for weight loss investigations were investigated for the morphological studies. Morphology of the acid treated specimens of the MS were studied under a MRM (Kyowa Getner, Japan) attached to a computer and the micrographs were recorded. Different morphological parameters like pore length, coating thickness, percentage porosity, maximum perimeter (μ) were recorded. The morphological parameters recorded for corroded samples in presence and in absence of corrosion inhibitor (hexamine) gives an information about mechanism and mode of action of corrosion inhibitor.

Scanning Electron Microscopic studies (SEM):

SEM micrographs were recorded for all the corroded specimens undergoing weight loss study using SEM (model JEOL JSM 6510 LV). SEM image tells us about the type, mode of action and mechanism of corrosion. Smooth surface of the specimen without any pit formation proves the effectiveness of hexamine as corrosion inhibitor for MS in $0.1 \text{ N H}_2\text{SO}_4$ medium.

Results and discussion

Weight loss technique:

Corrosion rate and PCIE observed from the weight loss experiments at different concentrations of hexamine in $0.1 \text{ N H}_2\text{SO}_4$ solution at 25.0, 30.0 and 35.0°C and are tabulated in Table 1. As the concentration of hexamine was increased from 200 to 1000 ppm, it was observed that PCIE increases linearly with concentration of hexamine. The results show that maximum PCIE of hexamine was found to be 91.46 % in $0.1 \text{ N H}_2\text{SO}_4$ solution. An appreciable PCIE was observed i.e. 74.0% at 200 ppm concentration of hexamine at 25.0°C. Weight loss results confirms that the amount of adsorption of hexamine on the surface of MS increases with increase in the hexamine concentration. Fig. 1 shows variation of corrosion rate and PCIE with increase in concentration hexamine i.e. 200 to 1000 ppm at 25.0, 30.0 and 35.0°C temperature.

Electrochemical polarization technique:

The electrochemical parameters like OCP, β_c , β_a and I_{corr} and PCIE are shown in Table 2. It is clear from the Table 2 that value of I_{corr} decreases with concentration of hexamine. No definite drift was seen in the values of E_{corr} with the increase in the concentrations of hexamine in $0.1 \text{ N H}_2\text{SO}_4$ solution. The values of β_a and β_c supports the adsorption of hexamine on the surface of MS. The value of Faradiac current decreases and value of capacitive current increase with rise in concentration of hexamine proves that thickness of adsorbed film increases with rise in concentration of hexamine. Electrochemical polarization results prove that hexamine acts as a very good corrosion inhibitor for MS. It is also seen that as the concentration of hexamine increases, there is reduction in the anodic dissolution rate supported by decrease in anodic Tafel slope which results in decrease in the rate of hydrogen evolution reaction¹⁸.

Table 1. Corrosion rate (CR) and Percentage Corrosion Inhibition Efficiency (PCIE) shown by hexamine by weight loss method at 25.0, 30.0 and 35.0°C for 24.0 h

Conc. (ppm)	CR 25.0°C (mpy)	PCIE at 25.0°C	CR 30.0°C (mpy)	PCIE at 30.0°C	CR 35.0°C (mpy)	PCIE at 35.0°C
0	119.889	–	144.689	–	147.11	–
200	31.15	74.07	37.385	74.16	44.924	69.46
400	16.91	85.89	33.458	76.87	36.619	75.1
600	13.62	88.63	23.505	83.75	30.593	79.2
800	12.39	89.66	23.215	83.95	25.771	82.48
1000	10.23	91.46	21.992	84.80	24.902	83.07

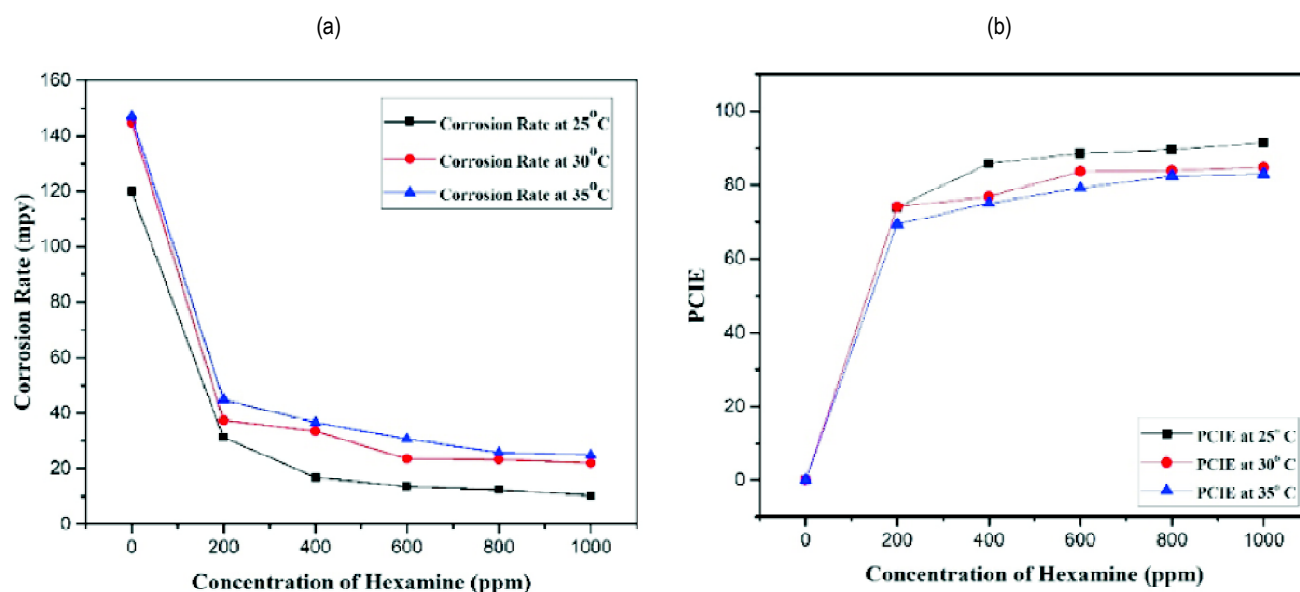


Fig. 1. Variation of corrosion rate (a) and PCIE (b) with concentration of hexamine (200 to 1000 ppm) at 25.0, 30.0 and 35.0°C.

Table 2. OCP, i_{corr} , R_p , β_a , β_c , CR and PCIE of hexamine for mild steel in 0.1 N H_2SO_4 at 25.0, 30.0 and 35.0°C

Corrosion inhibitor	Conc. (ppm)	β_a (mVd ⁻¹)	β_c (mVd ⁻¹)	R_p (Ω cm ²)	OCP (mV vs SCE)	i_{corr} (μA cm ⁻²)	% IE
Blank	0	426	1479	27.92	-0.450	5.14	-
Hexamine at 25.0°C	200	529	1376	71.92	-0.450	2.30	55.25
	400	423	1160	63.50	-0.466	2.11	58.94
	600	397	1407	87.63	-0.485	1.53	70.23
	800	383	1168	102.17	-0.473	1.24	75.87
	1000	395	1410	113.52	-0.493	1.18	77.04
Blank	0	423	1315	14.6	-0.489	9.61	-
Hexamine at 30.0°C	200	947	1334	102.4	-0.530	2.34	75.65
	400	646	1096	77.4	-0.511	2.28	76.27
	600	735	1420	96.2	-0.510	2.18	77.31
	800	691	1080	98.1	-0.512	1.86	80.64
	1000	402	862	68.1	0.510	1.74	81.89
Blank	0	588	1370	11.20	-0.480	15.9	-
Hexamine at 35.0°C	200	936	1219	92.7	-0.530	2.51	84.21
	400	923	1358	98.9	-0.548	2.47	84.46
	600	742	1031	78.1	-0.514	2.39	84.96
	800	446	896	57.4	-0.510	2.25	85.84
	1000	384	880	58.2	-0.506	1.99	87.48

Electrochemical Impedance Spectroscopy:

Impedance parameters for MS in 0.1 N H_2SO_4 solution in presence and absence of hexamine at 25.0, 30.0 and

35.0°C are shown in Table 3. Nyquist plots of MS in 0.1 N H_2SO_4 with different concentration of hexamine at 25.0, 30.0 and 35.0°C temperatures shown in Figs. 2, 3 and 4. PCIE

Table 3. Electrochemical impedance parameters for mild steel samples when exposed to 0.1 N H₂SO₄ solution with and without hexamine as corrosion inhibitor at 25.0, 30.0 and 35.0°C temperature

Corrosion inhibitor	Conc. of hexamine (ppm)	C _{dl} (Fcm ²)	R _{ct} (Ω cm ²)	% IE
Blank	0	3.8×10 ⁻²	41	-
Hexamine at 25.0°C	200	3.1×10 ⁻²	49.95	17.91
	400	2.4×10 ⁻²	65.60	37.50
	600	2.2×10 ⁻²	70	41.42
	800	2.0×10 ⁻²	79	48.10
	1000	1.3×10 ⁻²	117	64.95
Blank	0	3.18×10 ⁻²	50	-
Hexamine at 30.0°C	200	14.7×10 ⁻³	108	53.68
	400	13.2×10 ⁻³	120	58.31
	600	12.8×10 ⁻³	124	59.66
	800	11.7×10 ⁻³	136	63.22
	1000	9.7×10 ⁻³	164	69.50
Blank	0	3.18×10 ⁻²	59	-
Hexamine at 35.0°C	200	11.97×10 ⁻³	133	55.58
	400	11.95×10 ⁻³	133.25	55.66
	600	11.70×10 ⁻³	136	56.56
	800	11.38×10 ⁻³	139.92	57.78
	1000	11.0×10 ⁻³	144	58.97

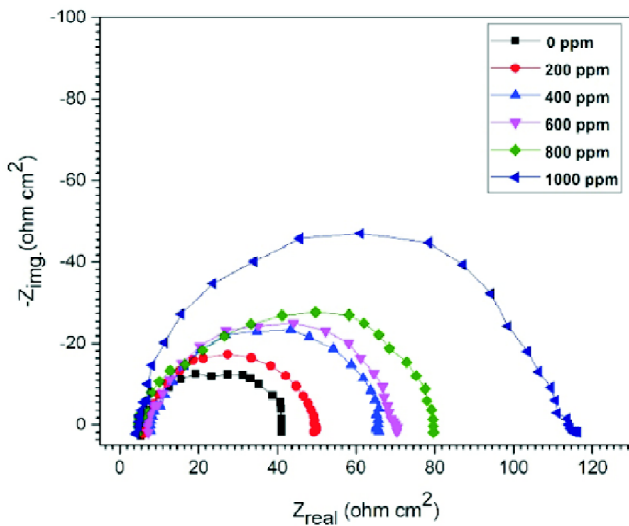


Fig. 2. Nyquist plots (EIS) of mild steel immersed in 0.1 N H₂SO₄ in absence and presence of different concentrations of hexamine at 25.0°C temperature.

was calculated from the R_{ct} values in presence and in absence of corrosion inhibitor. PCIE were found to upsurge with

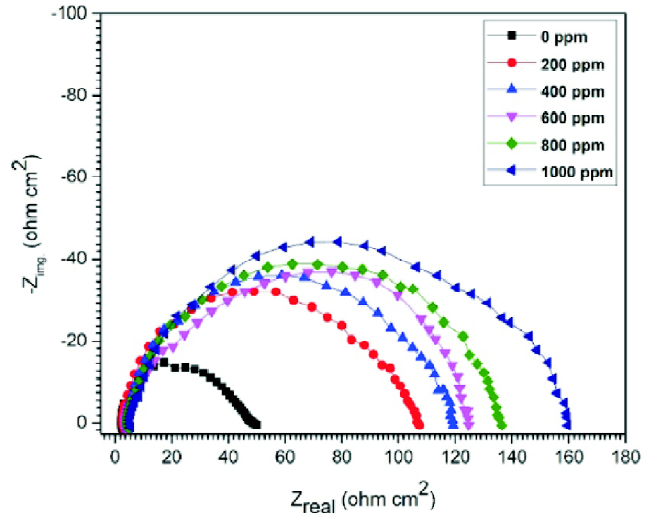


Fig. 3. Nyquist plots (EIS) of mild steel immersed in 0.1 N H₂SO₄ in absence and presence of different concentrations of hexamine at 30.0°C temperature.

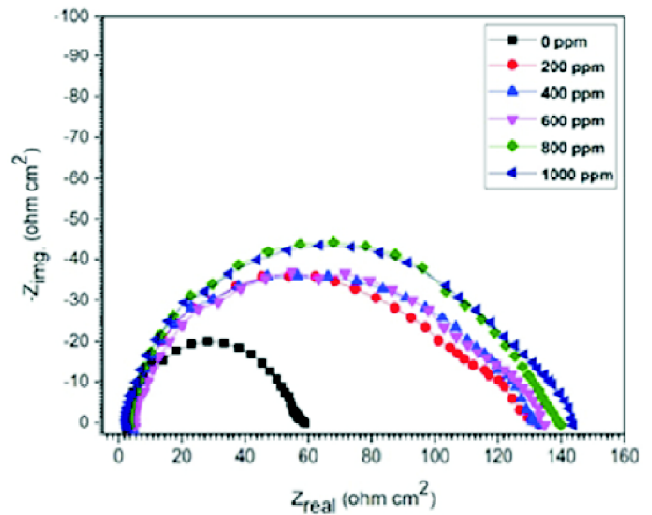


Fig. 4. Nyquist plots (EIS) of mild steel immersed in 0.1 N H₂SO₄ in absence and presence of different concentrations of hexamine at 35.0°C temperature.

surge in the concentration of hexamine. Fig. 5 shows equivalent circuit (Randles circuit) corresponding to 1000 ppm concentration of hexamine as corrosion inhibitor for MS at 25.0°C taken from impedance spectroscopy data when performed in potentiostatic mode at a constant potential (OCP) at AC perturbation amplitude of 10.0 mV/s in a frequency range of 1 to 10 kHz with 10 points per decade. It includes a resistor

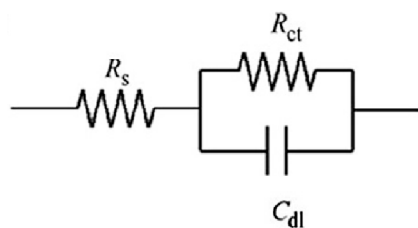


Fig. 5. Equivalent Circuit (Randles circuit) corresponding to 1000 ppm concentration of hexamine as corrosion inhibitor for mild steel at 25.0°C taken from impedance spectroscopy data when performed in potentiostatic mode at a constant potential (OCP) at AC perturbation amplitude of 10.0 mV/s in a frequency range of 1 to 100 kHz with 10 points per decade.

(R_{ct}) which is due to formation of electrical double layer at the interface between MS and corroding electrolyte termed as charge-transfer resistance which is connected in parallel with a double layer capacitor (C_{dl}) and this RC electrical unit is connected in series with another resistor (R_s).

Nyquist plot shows the increase in the value of charge transfer resistance (R_{ct}) with increase in concentration of hexamine which means that thickness of double layer increases with increase in concentration of hexamine resulting in the formation of more uniform, nonporous, barrier film of corrosion inhibitor over the surface of MS. In the low frequency region, inductive loop is almost same at all the five investigated concentration and but there is significant increase in capacitive loop at 1000 ppm of hexamine concentration at all the three investigated temperatures suggesting that there is significant decrease in Warburg Impedance (Z_w). Thickness of the adsorbed film was calculated from the relation (3).

$$d = \frac{\epsilon_0 \epsilon}{C_{dl}} S \quad (3)$$

where, d is thickness of the adsorbed film form by inhibitor

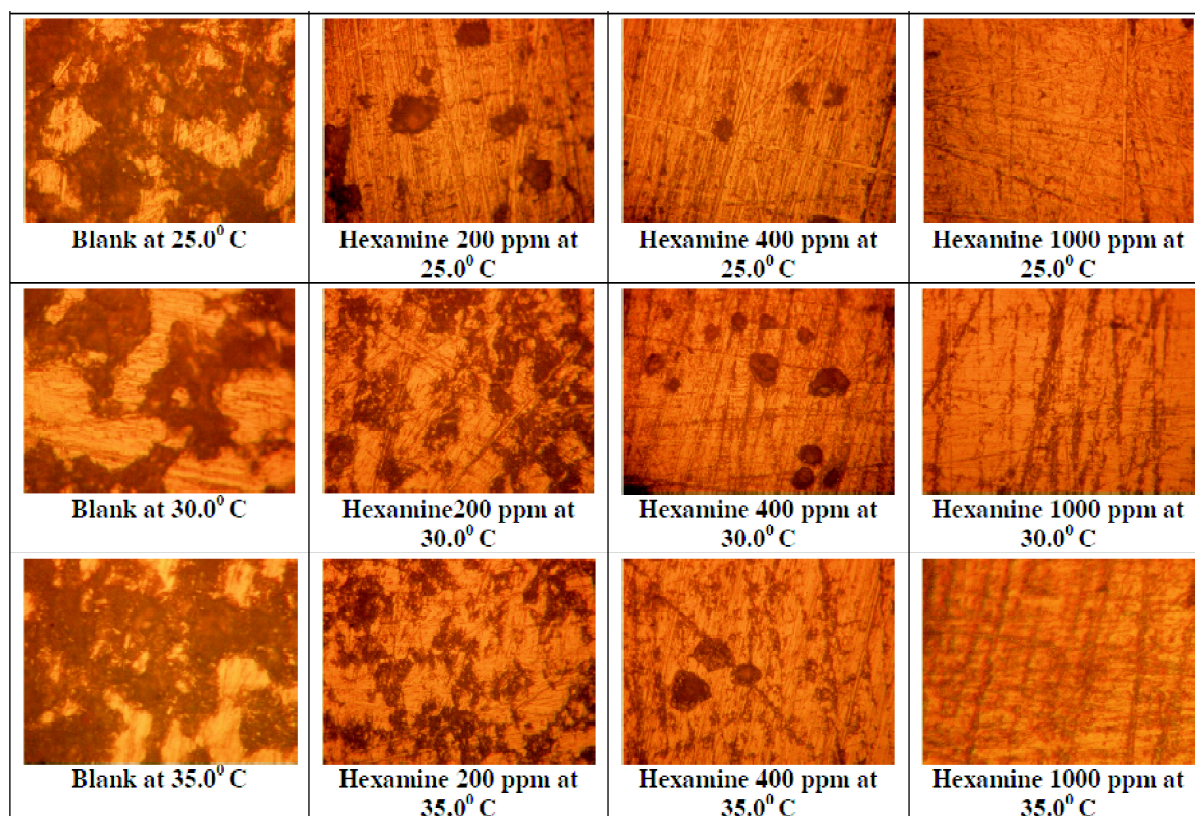


Fig. 6. Trinocular Inverted Metallurgical Research Micrographs of different mild steel samples with and without different concentrations hexamine at 25.0, 30.0 and 35.0°C temperatures.

molecule, S is the surface area of electrode, ϵ is the dielectric constant of the medium, ϵ_0 is the permittivity of the air, C_{dl} is the double layer capacitance. It was observed that thickness of adsorbed film increases with increase in concentration of hexamine from 200 to 1000 ppm. Thus, hexamine proves to be a very good corrosion inhibitor for mild steel in 0.1 N H₂SO₄ solution at three investigated temperatures.

Metallurgical research microscopy technique:

Micrographs of the corroded specimens with and without corrosion inhibitor were taken after immersion in 0.1 N H₂SO₄ for 24.0 h at 25.0, 30.0 and 35.0°C temperature by Metallurgical Microscope (Kyowa Getner, Japan) and are shown in Fig. 6. Various morphological parameters such as porosity, coating thickness, length of pores, maximum and minimum perimeter were measured for mild steel surface. The data pertaining to percentage porosity, coating thickness, length

of pores, maximum and minimum perimeters (μ) were reported in Table 4. Data shows that the surface of mild steel becomes porous after exposing to 0.1 N H₂SO₄ for 24.0 h. It is observed that percentage porosity decreases and coating thickness increases with increase in concentration of hexamine. Length of pores also decreases with increasing concentration of inhibitor.

SEM analysis:

SEM images of mild steel specimens after exposing to 0.1 N H₂SO₄ for 24.0 h in presence and absence of hexamine as corrosion inhibitor were taken at three different temperatures and were shown in Figs. 7–9. In the SEM image (Fig. 7), clear large size pits are visible in absence of Hexamine but as hexamine was introduced into the system pits were disappeared proving that hexamine act as anti-pit agent and retards the corrosion rate of mild steel to a significant effect so that the surface of mild steel get protected from

Table 4. Coating thickness, percentage porosity and pore length of mild steel sample when exposed to 0.1 N H₂SO₄ solution with and without hexamine as corrosion inhibitor at different temperatures

Temp. (°C)	Conc. of inhibitor (ppm)	Max. perimeter (micron)	Max. Area (micron) ²	% porosity	Coating thickness	Length of pores (micron)
25.0	0	7712.58	485803.32	93.36	94.74	185.28
	200	1715.88	29182.82	21.95	357.89	166.87
	400	1447.25	26142.66	20.68	460.53	148.21
	600	1806.28	14473.68	16.28	584.21	122.67
	800	1740.88	20387.81	18.00	644.74	73.68
	1000	983.15	12160.66	11.15	823.68	66.62
30.0	0	6766.84	490304.71	92.70	78.95	265.81
	200	4662.11	46675.90	25.56	213.15	165.87
	400	2990.03	32229.92	21.53	226.32	137.07
	600	2614.90	34515.23	18.83	250.0	108.51
	800	2330.62	24141.27	16.62	400.0	89.51
	1000	1092.23	6031.85	12.51	489.0	66.62
35.0	0	4222.91	511620.49	96.42	268.42	255.38
	200	7205.63	76945.98	36.83	368.42	147.46
	400	2898.21	30512.46	28.06	292.11	118.42
	600	2430.85	24556.79	23.98	278.95	104.27
	800	2270.22	14736.84	19.92	523.68	107.89
	1000	1042.58	13116.34	15.73	626.32	93.11

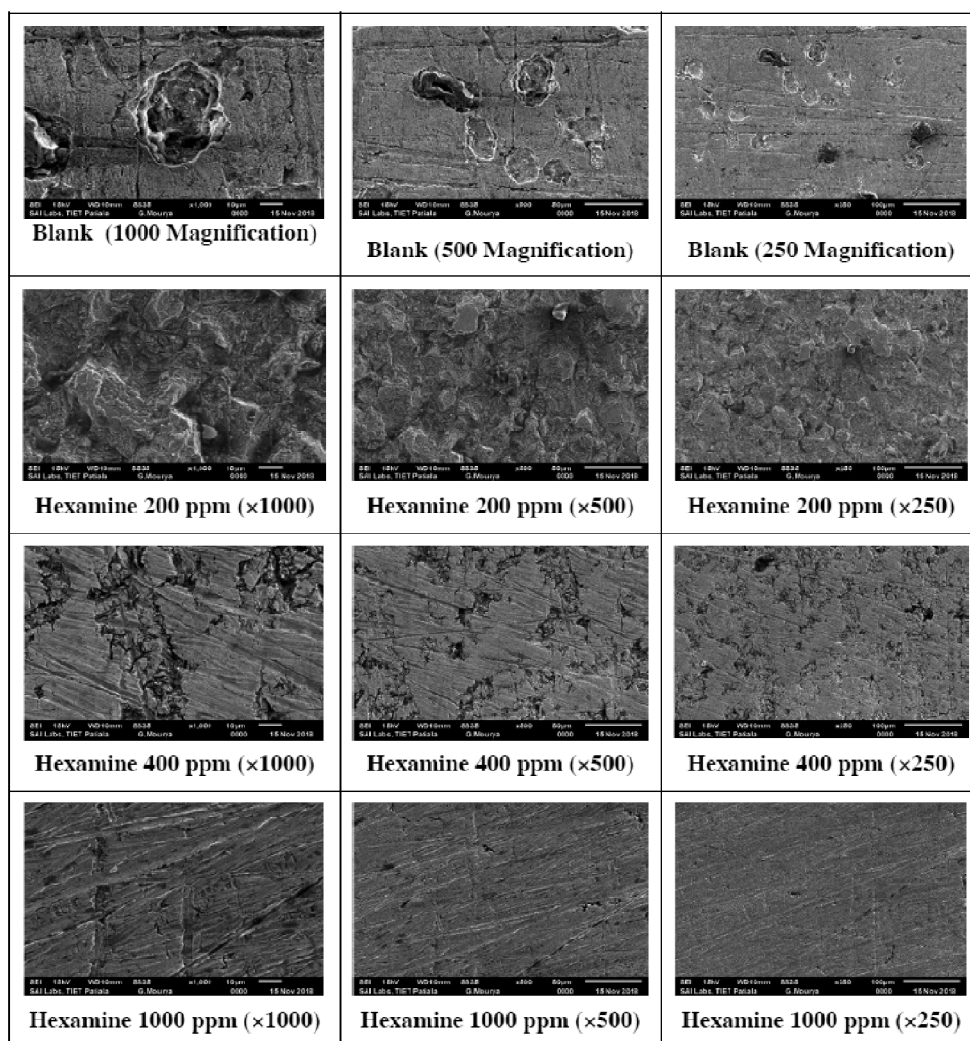


Fig. 7. SEM images of MS samples when exposed to 0.1 N H₂SO₄ medium with and without CTAB at 25.0°C.

corrosion. Surface roughness decreases with the increase in concentration of hexamine. Corrosion products are clearly visible in absence of hexamine but in presence of hexamine corrosion products disappear from the surface of mild steel. With increase in the temperature, there is an increase in cor-

rosion rate and roughness on the surface increases with increase in temperature i.e. 25.0 to 35.0°C as shown in Figs. 8 and 9. SEM images of mild steel specimens shows that Hexamine is a good corrosion inhibitor for mild steel in 0.1 N H₂SO₄ medium at different temperatures.

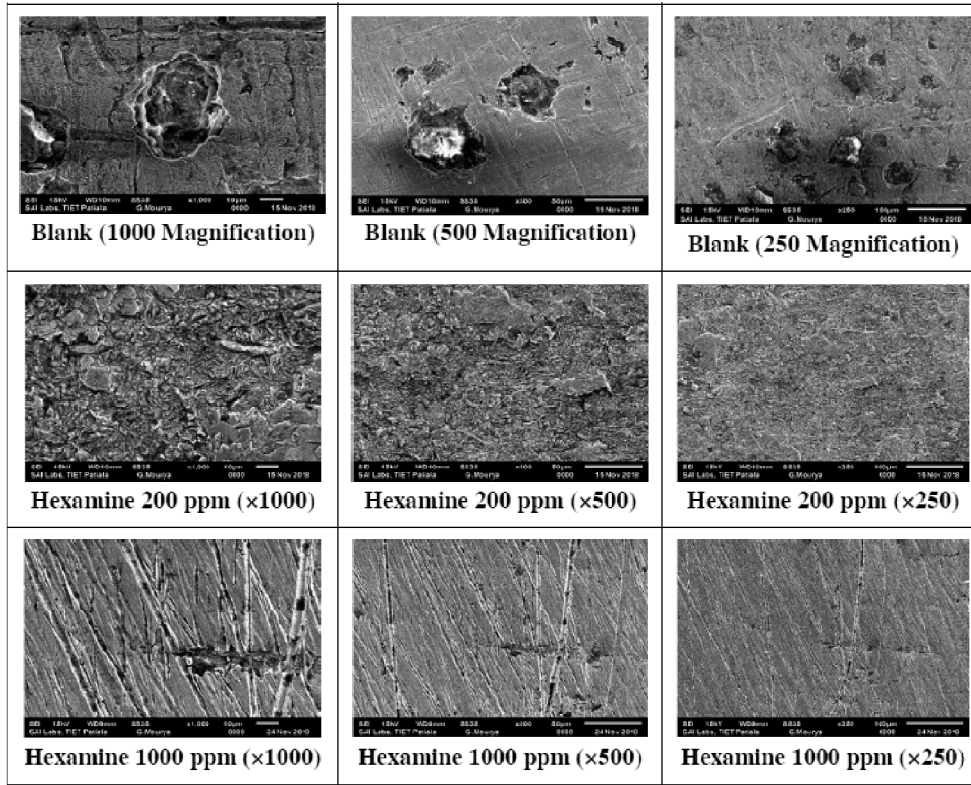


Fig. 8. SEM images of MS samples when exposed to 0.1 N H₂SO₄ medium with and without different concentrations of CTAB at 30.0°C.

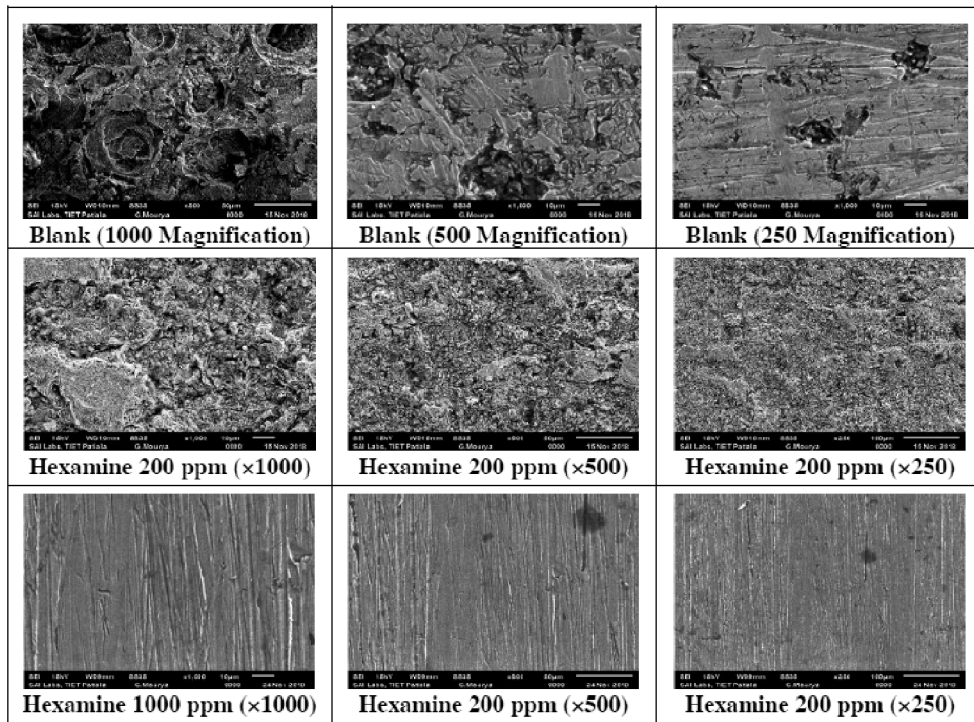


Fig. 9. SEM images of MS samples when exposed to 0.1 N H₂SO₄ medium with and without CTAB at 35.0°C.

Conclusion

Addition of hexamine as a corrosion inhibitor for mild steel in 0.1 N H₂SO₄ solution results in significant increase in corrosion resistant properties. Improvement in corrosion resistant properties are far superior than other organic corrosion inhibitors reported in the literature suggesting that hexamine is a potential alternate to them for inhibiting corrosion of mild steel in dilute sulphuric acid medium. The increase in corrosion resistance properties of hexamine was associated with the formation of uniform, continuous, nonporous, passive film over the surface of mild steel as observed in SEM, Metallurgical Research Microscopy and Impedance Spectroscopy techniques. The thickness of the film increases with increase in concentration of the hexamine and decreases with increase in temperature of the corroding medium. Increase in the thickness of adsorbed film was supported by the decrease in Warburg Impedance (Z_w). Thus, hexamine proves to be a very good anti-pit agent for mild steel in dilute sulphuric acid medium. Increase in temperature of the system has no significant effect on anti-pitting properties of hexamine.

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